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FLORIDA INTERNATIONAL UNIVERSITY

Miami, Florida

DEVELOPMENT OF IMPROVED EXTRACTION/PURIFICATION METHODS AND COMPREHENSIVE SCREENING/CONFIRMATION BY LC-QqQ-MS ANALYSIS FOR NOVEL PSYCHOACTIVE SUBSTANCES

A dissertation submitted in partial fulfillment of

the requirements for the degree of

DOCTOR OF PHILOSOPHY

in

CHEMISTRY

by

Ashley Nicole Kimble

2019

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To: Dean Micheal R. Heithaus
College of Arts, Sciences and Education

This dissertation, written by Ashley Nicole Kimble, and entitled Development of Improved Extraction/Purification Methods and Comprehensive Screening/Confirmation by LC-QqQ-MS Analysis for Novel Psychoactive Substances, having been approved in respect to style and intellectual content, is referred to you for judgment.

We have read this dissertation and recommend that it be approved.

_____
Jose Almirall

_____
Piero Gardinali

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Rudolf Jaffe

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DeEtta Mills

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Anthony P. DeCaprio, Major Professor

Date of Defense: June 10, 2019

The dissertation of Ashley Nicole Kimble is approved.

_____
Dean Micheal R. Heithaus
College of Arts, Science and Education

_____
Andres G. Gil
Vice President for Research and Economic Development
and Dean of the University Graduate School

Florida International University, 2019

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ABSTRACT OF THE DISSERTATION

DEVELOPMENT OF IMPROVED EXTRACTION/PURIFICATION METHODS AND COMPREHENSIVE SCREENING/CONFIRMATION BY LC-QqQ-MS ANALYSIS FOR NOVEL PSYCHOACTIVE SUBSTANCES

by

Ashley Nicole Kimble

Florida International University, 2019

Miami, Florida

Professor Anthony P. DeCaprio, Major Professor

The presence of novel psychoactive substances (NPS) in forensic casework poses major difficulties for detection, since there are many structural variations of NPS circulating in the street market. There are currently no comprehensive screening/confirmatory/quantitation methods available that encompass the majority of NPS encountered in forensic toxicology. A major issue faced with developing such a method is that full validation is extremely time consuming. The use of a liquid chromatography triple quadrupole tandem mass spectrometry (LC-QqQ-MS/MS) method makes the detection of a large number of NPS possible because of high selectivity and sensitivity.

This research included four main tasks: 1) development of a dynamic multiple reaction monitoring (dMRM) LC-QqQ-MS method for 800+ NPS, 2) validation of the dMRM method for screening and confirmation of 800+ NPS using a series of mixtures of non-coeluting standards, 3) comparison and optimization of NPS extraction methods for

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urine and whole blood, and 4) screening of spiked and authentic specimens to determine the real-world potential of the dMRM method.

Validation was completed for the parameters of selectivity, limit of detection (LOD), limit of quantitation (LOQ), carry over, linearity, bias, precision, freeze-thaw stability, and matrix effects. A method that ultimately included a total of 729 compounds was validated with LOD and LOQ in the pg/mL range. The research presented here implements the largest validated method of its kind for NPS with capabilities as a screening method for NPS in urine and whole blood and as a confirmatory method in urine.

Several extraction methods were also compared to determine their efficacy for the extraction of NPS from urine and whole blood. These included dilute- and crash-and-shoot, online and classical solid phase extraction, and QuEChERS. Techniques were compared for elimination of matrix effects, recovery, process efficiency, time, and cost.

Through the analysis of blind spiked and authentic specimens, the applicability of the validated method as a screening and confirmatory method was successfully demonstrated. The method developed in this project will aid in reliable identification of NPS in clinical and forensic toxicological samples. Additionally, this work provided data to improve the reliability of extraction of NPS from biological matrices.

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TABLE OF CONTENTS

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LIST OF TABLES

TABLES PAGE

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LIST OF FIGURES

FIGURES PAGE

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LIST OF ABBREVIATIONS, ACRONYMS, AND SYMBOLS

AF	Ammonium Formate
ANOVA	Analysis of Variance
BE	Bond Elut
CB1	Cannabinoid Type 1 Receptor
CB2	Cannabinoid Type 2 Receptor
CDC	Center for Disease Control and Prevention
CE	Collision Energy
CI	Chemical Ionization
CSA	Controlled Substance Act
CV	Coefficient of Variation
d-SPE	Dispersive SPE
DEA	Drug Enforcement Administration
DFC	Drug Facilitated Crime
dMRM	Dynamic Multiple Reaction Monitoring
DMSO	Dimethyl sulfoxide
DUI	Driving Under the Influence
EC	Endocannabinoids
EC C18	Encapped C18
EDTA	Ethylenediaminetetraacetic acid
EI	Electron Ionization
EIC	Extracted Ion Chromatogram

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ELISA	Enzyme Linked Immunosorbent Assay
EMIT	Enzyme Multiplied Immunoassay Technique
ESI	Electrospray Ionization
FA	Formic Acid
FIA	Flow Injection Analysis
GC-MS	Gas Chromatography Mass Spectrometry
H2O	Water
HCl	Hydrochloric Acid
HPLC	High Performance Liquid Chromatography
HRMS	High Resolution Mass Spectrometry
HSD	Honestly Significant Difference
IS	Internal Standard
LC-MS	Liquid Chromatography Mass Spectrometry
LLE	Liquid-Liquid Extraction
LOD	Limit of detection
LOQ	Limit of Quantitation
m/z	Mass to Charge Ratio
ME	Matrix Effects
MeCN	Acetonitrile
MeOH	Methanol
mg	Milligram
mL	Milliliter
MRM	Multiple Reaction Monitoring

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MS	Mass Spectrometry
MS/MS	Tandem Mass Spectrometry
NaCl	Sodium Chloride
ng	Nanogram
NIST	National Institute of Standards and Technology
NPS	Novel Psychoactive Substances
OSAC	Organization of Scientific Area Committees
OTC	Over the Counter
PCP	Phencyclidine
PE	Process Efficiency
PP	Protein Precipitation
ppb	Parts Per Billion
ppm	Parts Per Million
PSA	Primary Secondary Amine
QC	Quality Control
QqQ	Triple Quadrupole Mass Spectrometry
QTOF	Quadrupole Time-of-Flight Mass Spectrometry
QuEChERS	quick, easy, cheap, effective, rugged, and safe
RE	Recovery
RP	Reversed Phase
RT	Retention Time
SAMHSA	Substance Abuse and Metal Health Services Administration
SIM	Selected Ion Monitoring

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SPE	Solid Phase Extraction
SRM	Selected Reaction Monitoring
SWGTOX	Scientific Working Group for Forensic Toxicology
THC	Δ^9-Tetrahydrocannabinol
TIC	Total Ion Chromatogram
UPLC	Ultra Performance Liquid Chromatography
Δc	Change in Concentration
µg	Microgram
µL	Microliter

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1. INTRODUCTION

1.1 Statement of the Problem

Novel psychoactive substances (NPS) have been a global health hazard for the past several decades. Novel psychoactive substances are structural alterations of controlled substances created to evade drug law. There have been reported fatal overdoses that can be attributed to NPS, especially from synthetic cannabinoids and opioids.[1-4] Novel psychoactive substances are difficult to control and detect in biological fluids, because of their constantly changing structures introduced by illicit manufacturers as current drugs become scheduled and illegal to possess. Drugs of abuse are scheduled according to their unique chemical structure, therefore every small structural alteration results in a compound no longer being regulated by controlled substance laws.[5] Changes to structure can be as small as the addition or removal of a functional group or single atom, such as a halogen. Consequently, there are practically endless structural possibilities for NPS. Such derivatives can have extremely varied pharmacological effects, ranging from minimal effect to severe toxicity. [2,6]

Many screening methods used in clinical and forensic toxicology detect compounds on the basis of their structure or specific functional groups. Since screening methods tend to be structure-specific, NPS can be missed during screening, resulting in false negatives. In a forensic or clinical setting, if a sample is wrongly reported as negative it is possible that the sample will be discarded, making it impossible to retest the sample with advanced methodology. False negatives are especially problematic when the results of these tests are being used for treatment and potentially determining cause of death. One of the biggest issues faced by law enforcement in terms of detecting NPS is that many manufacturers

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have different structures waiting for distribution as soon as an existing NPS structure becomes known, scheduled, and detectable.[7] Consequently, it is difficult for clinical and forensic toxicology laboratories to detect NPS as new ones become available.

A possible solution for the detection of NPS involves the creation of comprehensive libraries and databases containing chromatographic and mass spectral data for individual NPS entities. When combined with libraries and databases, comprehensive targeted screening and confirmatory methods have the potential to detect large numbers of NPS in clinical and forensic toxicology. Currently, there are a number of libraries for gas chromatography (GC) generated using electron ionization (EI) mass spectrometry (MS). Additionally, there exist libraries for liquid chromatography (LC) using electrospray ionization (ESI) MS, but these lack the comprehensiveness and standardization associated with existing GC libraries.[8,9] Even though such MS libraries exist, many of them are theoretical (*i.e.,* determined by calculations of fragmentation rather than using actual reference standards to determine fragmentation) or contain few to no NPS. The majority of forensic toxicological laboratories have in place GC and/or LC-MS methods capable of detecting typical drugs of abuse and other compounds commonly found in their casework. In recent years, many such laboratories have also begun including NPS, but the number of NPS entities included in these types of methods is generally only a small representation of the sheer number of NPS that are potentially available.[10] Clinical and forensic toxicology laboratories struggle to identify NPS in a timely and reliable manner. Misidentification can lead to major health epidemics if, for example, a potent NPS is not detected until after there have been multiple overdoses in a specific area. Research has been done in order to combat

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these issues, but more needs to be done before clinical and forensic toxicology laboratories can properly detect NPS as they are produced and released to the illicit market.

In recent decades, NPS have become a major global public health issue, especially in the United States, Europe, and China.[11,12] Specifically, in the United States synthetic opioids, especially fentanyl derivatives, are contributing to the opioid epidemic. Commonly used opioids and a number of novel derivatives have been the cause of death of thousands in the U.S. over the past few years. Not only are NPS concerning to the forensic toxicology communities, they are also a health hazard. Many NPS are first detected in Europe before being distributed to the U.S. Novel psychoactive substances are relatively easy to obtain since they can often be purchased via the internet. The internet has made the sale and purchasing of NPS easier than it would have been in the past. There is generally little pharmaceutical information available for the majority of NPS, which leads to the risk of increased overdoses and toxicity.[13] Many consumers falsely believe that "legal" means safer, which is not the case for many NPS, further worsening their health hazard.[13] The health concerns revolving around NPS need to be combatted, starting with identification in clinical and forensic toxicology. Being able to detect NPS quickly and reliably will aid in scheduling, further research, and informing the public of their hazards. Consequently, the development of screening and confirmatory methods for the detection of a wide variety of NPS will aid clinical and forensic toxicology laboratories in the timely and reliable detection of such harmful substances, helping to prevent or manage further epidemics.

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1.2 Rationale for Research

Many toxicological laboratories use immunoassays for initial drug screening, which are robust when used for the detection of common drugs of abuse but can be problematic when working with NPS. Immunoassays are capable of detecting drug compounds through an antigen-antibody interaction which relies on the compound's structure. As a result of the structure relation requirement, issues arise when trying to screen for NPS, since they exhibit structural differences as compared to common drugs of abuse. Screening methods using LC and GC MS are more adaptable to the structural changes that NPS undergo than immunoassays. Analytical methods are also routinely used in forensic toxicological laboratories.[9] However, most commonly these methods function based on library matches or a targeted method. Many of these methods and/or libraries only contain a small set of NPS, and some methods contain NPS that are no longer seen in modern case work. In addition, even though screening methods are available for some NPS, very few of those methods have been validated and are capable of quantitative results.

A similar issue to the detection of NPS arises when trying to extract NPS from biological matrices. Extraction methods for common drugs of abuse are well established in clinical and forensic toxicological laboratories. However, it is not always possible to use those same methods for the majority of NPS compounds. Many extraction methods rely on the structure and chemical properties of the compound to successfully extract the analyte of interest from its biological matrix. Since NPS undergo structural alterations, some of these methods may no longer be able to reliably separate the analyte of interest from the matrix. It is important to have extraction methods capable of isolating NPS from biological matrices and ensuring that they are not discarded as waste during the extraction process.

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1.3 Significance of Study

The current research is designed to be applicable to forensic science, clinical and forensic toxicology, and law enforcement. The research described here has revolved around the development and validation of a comprehensive dynamic multiple reaction monitoring (dMRM) method for screening and confirmation of hundreds of NPS. The validated method was then used to evaluate the effectiveness of different extraction methods for NPS and to determine statistically significant differences among the methods. The ultimate goal is to implement optimized extraction approaches and a validated analytical method into forensic toxicology laboratories to aid in the timely and reliable detection of NPS in case samples.

The NPS to be included in the database generated for the project were determined by researching published articles, forensic case work reports, drug user blogs/forums, and overdose reports to ensure that the NPS being included were still in use and relevant for clinical and forensic toxicological samples. In addition to common NPS, the database also includes common adulterants, to ensure that the method is capable of differentiating the NPS from them. The research presented here was divided into four major tasks.

1.3.1 Task 1 – *Creation of a dMRM database and screening/confirmatory method*

This task was the foundation for all the tasks to come after. Up to 10 precursor-product ion transitions were collected for all NPS to be included in the final analytical method. Once transitions were established, all compounds were analyzed to determine retention times in order to create a dMRM method capable of screening for over 800 NPS.

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1.3.2 Task 2 - *Validation of the dMRM screening/confirmatory method using a mixture approach*

After method development was finalized, full method validation was completed using a set of defined mixtures of non-coeluting NPS in order to decrease the time required to complete the validation. Method validation was performed following established toxicological guidelines. The parameters validated for included linearity, limit of detection, limit of quantitation, carry over, bias, precision, matrix effect, and freeze-thaw-stability.

1.3.3 Task 3 - *Evaluation and optimization of NPS extraction/purification methods*

The third portion of the research was completed by comparing crash-/dilute-and-shoot, QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe), online SPE, and classical SPE to determine which protocols have statistically significant benefits over the others for the extraction of NPS from urine and whole blood. Comparison was done using drug recovery, elimination of matrix effects, and improved process efficiency. Even though extraction methods for common drugs of abuse have been thoroughly studied, extraction methods for NPS have not been comprehensively evaluated. Extraction methods were compared and then optimized to ensure that the ideal conditions were used for each method.

1.3.4 Task 4 – *Analysis of blind spikes and authentic specimens*

To apply the current research to actual casework samples, blind spiked urine and whole blood were analyzed quantitatively and qualitatively. Blind spiked samples were used to ensure that the validated method is capable of correctly identifying multiple compounds

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per sample in different matrices. Additionally, blind spikes were used to test the effectiveness of extraction methods used throughout the present research. Blind spikes were treated identically to how case samples would be prepared and tested. Finally, authentic urine specimens were also collected and tested using the validated method to identify the presence of NPS and to ensure that there were no interferences by common drugs of abuse and medications that were likely to be present in authentic samples.

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2. LITERATURE REVIEW

2.1 Novel Psychoactive Substances

Novel psychoactive substances (NPS) have been gaining popularity over the past few decades by consumers, sellers, and manufacturers.[14] Novel psychoactive substances are also known as "legal highs," "designer drugs," and "spice." According to the Controlled Substances Act (CSA) passed in 1970, drugs of abuse are scheduled on the basis of their structure, pharmacological effect, potential for abuse, and accepted medical uses.[15] These compounds can fall under one of five schedules. Schedule V compounds have a very low potential for abuse and many accepted medical uses. Schedule IV and III substances have a low potential for abuse and a moderate potential for abuse, respectively. Schedule I and II compounds have high potential for abuse and no or limited medical use, respectively. Novel psychoactive substances are specifically created to evade drug laws, as a consequence of their structural differences from common, scheduled drugs of abuse. These structural changes can be as simple as the addition of a methyl group, a small change that renders the compound novel and, therefore, unscheduled. Prime examples of this are the NBOMe compounds, which all have the same base structure but with the simple addition of a halogen atom that creates a new compound that is no longer scheduled.[16]

An NPS is a compound that is structurally and pharmacologically "substantially similar" to a Schedule I or II compound. The Drug Enforcement Administration (DEA) has been making an effort to schedule NPS through regulations such as The Federal Analog Act passed in 1986, which stated that new compounds can be scheduled if they are proven to be structurally and pharmacologically similar to a Schedule I or II compound. Although the act was helpful, proving structural and pharmacological similarity can be difficult. Not

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all NPS have the same structural backbone as the common drugs of abuse to which they are designed to have similar pharmacological effects. A prime example of this are the synthetic cannabinoids. The structures of many synthetic cannabinoids vary greatly from that of Δ^9-tetrahydrocannabinol (THC), which is the active component in marijuana. Synthetic cannabinoids can have extremely varied structural characteristics and are divided into separate classes within the general class. Some of these subclasses include cyclohexylphenols, naphthoylindoles, phenylacetylindoles, and indole carboxylates.[17]

There are efforts for temporary scheduling of NPS that are capable of leading to permanent scheduling within two years and adding an additional 12-month extension to continue research efforts. It is generally very difficult to monitor the distribution of NPS, since they are often sold online under the label of "not for human consumption." It is not uncommon for NPS to be developed for research purposes but then diverted for illicit use, with their potential for abuse discovered at a later time.[18] There are published books and online forums that give step-by-step instructions on the synthesis of different NPS, which only makes it easier for illicit drug manufacturers. Unfortunately, as a result of all the resources available to clandestine laboratories, it is difficult for entities such as the DEA to act proactively to schedule NPS.[5]

The speed at which drug manufacturers are able to place structurally different NPS on the market also makes it very difficult for forensic toxicologists to detect all of the different possible compounds that can be found in a specimen. New compounds that are found on the market and not detectible by current methods can then lead to false negatives, which is undesirable from a clinical and forensic viewpoint. It is important to have a method capable of detecting the majority of NPS that are available to consumers. Another issue that

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forensic laboratories face when screening for NPS is that it is impossible to develop and validate methods as quickly as manufacturers can create new structures.

2.2 Classes of Novel Psychoactive Substances

There are a wide variety of NPS that have been recorded in literature and which can be placed into various drug classes depending on structure and pharmacological effects. Novel psychoactive substances can generally be classified according to the following structural categories; benzodiazepines, cathinones, phenethylamines, synthetic cannabinoids, synthetic opioids, and tryptamines.[6] Different classes of NPS need to be treated differently for analysis depending on the structure and the chemical properties of their functional groups. For many NPS, the mechanism of action and pharmacological effects are unknown. The lack of understanding around NPS poses a health risk, since people are ingesting compounds that are not well understood. It is not uncommon for NPS to be more potent than many common drugs of abuse, contributing further to their status as health hazards.[4]

All drug classes of NPS are of public health concern. There are reported overdoses and in some cases fatalities for the majority of them in the past decade in the U.S. and around the world.[19,20] There is typically little to no reliable pharmacological information available for these compounds and many of their mechanisms of actions are not well understood. For example, there have been numerous fatalities due to NBOMes, which are part of the phenethylamine class of NPS.[16,21,22] Synthetic benzodiazepines have also been detected in forensic cases, with the most common compounds being flubromazolam and flubromazepam. Often, illicit benzodiazepines are seen in cases in combination with THC and amphetamine.[23]

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Three of the most prevalent classes of NPS are synthetic cathinones, synthetic cannabinoids, and synthetic opioids. Fatalities involving these drug classes have been reported all over the world, some of which can be considered to be epidemics. There have been cases of mass fatalities for all three of these categories in the U.S. alone. The phenomenon of increased potency of some of these compounds as compared to typical drugs of abuse is not well appreciated by users, further leading to overdoses and related fatalities. The most extensive research on NPS has been conducted on these three classes, resulting in a number of review articles focusing on use, mechanism of action, structures, and pharmacological effects.[24-26]

2.2.1 Synthetic Cathinones

Synthetic cathinones, also referred to as "bath salts," have been abused in the U.S. and around the world for many years and are still being identified in clinical and forensic case samples.[27,28] Synthetic cathinone abuse can be seen in both impaired driving cases and fatal intoxications.[29] Synthetic cathinones can vary considerably in structure, and, because of their varied structures, they can also differ in mechanism of action and pharmacological effects.[30] Synthetic cathinones act upon the monoamine transporters for dopamine, noradrenaline, and serotonin.[31] As a result of variable structures of synthetic cathinones, their affinity for the transporters and ability to inhibit reuptake of these neurotransmitter molecules can vary greatly. These differences can lead to complex combinations of dopaminergic, adrenergic, and/or serotonergic effects in users.[32] These effects contribute to the stimulant and mood-altering feelings associated with synthetic cathinone abuse.

As with many NPS, there is little to no pharmacological information available on synthetic cathinones. The pharmacology of synthetic cathinones is not well understood,

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however, synthetic cathinones can exhibit similar effects to amphetamines. Side effects from synthetic cathinone use in high doses can include hallucinations, delirium, hyperthermia, and tachycardia. Chronic users can exhibit extreme agitation and violent behavior associated with "excited delirium."[31] Additional side effects can include dehydration, muscle damage, and organ failure. Heavy synthetic cathinone abuse in some cases can lead to death.

Detecting synthetic cathinones in human specimens poses numerous difficulties. For example, it is known that cathinones are not stable in plasma.[33] There are issues associated with the quantitative repeatability of synthetic cathinone test results. The stability and changes in concentration that can occur during storage are not well understood for synthetic cathinones, leading to issues with detection and quantification.[34] Another issue with analysis is that synthetic cathinones can undergo *in situ* degradation when analyzed using GC-MS, because of their low boiling point and thermal instability.[35] Synthetic cathinones need to be treated properly to ensure that they are not degraded during sample preparation or analysis.

2.2.2 Synthetic Cannabinoids

There have been findings of synthetic cannabinoids presenting much higher potencies than THC, which can lead to an increased number of overdose cases.[36] Synthetic cannabinoids are considered dangerous and the Center for Disease Control and Prevention (CDC) has posted warnings on their website suggesting that people under no circumstances use anything they purchased after March, 2018 because of recorded cases of extreme bleeding after use of synthetic cannabinoids.[37] Synthetic cannabinoids can be sprayed onto plant material and smoked, vaped from a liquid form, or used in different foods and

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consumed orally. They can be found in convenience stores, but more often are purchased online.[7]

Not all synthetic cannabinoids were developed for illicit distribution; many were developed as part of legitimate scientific research. Some prime examples are the JWH compounds, which were discovered in the laboratory of John W. Huffman at Clemson University. The JWH compounds were synthesized to study their reactions with cannabinoid receptors in the brain. The initial research was published in 1998 however, JWH 018 was not found being used as an alternative to cannabis until 2008.[18,38]

Synthetic cannabinoids can be subcategorized into several different classes determined by their structure. These subclasses include, but are not limited to, cyclohexylphenols, naphthoylindoles, benzoylindoles, phenylacetylindoles, alkoylindoles, indole carboxylates, indole carboxamides, and indazole carboxamides.[17] Cyclohexylphenol cannabinoids are bicyclic derivatives of classical cannabinoids exhibiting the most similar structure to THC.[17] Naphthoylindoles are considered to be the "first generation" of synthetic cannabinoids and were originally identified in herbal substances. The other subclasses are newer synthetic cannabinoids that arose from changing the naphthoyl moiety with varying aromatic and non-aromatic groups. There are practically endless possibilities of structures for synthetic cannabinoids to exhibit.

The cannabinoid system in the human body has naturally occurring neurotransmitters known as endocannabinoids (EC). Many synthetic cannabinoids act on the same receptors as THC, however there are hundreds of possible structures, some that vary greatly from THC. Similar to natural cannabinoids, synthetic cannabinoids compete with endogenous EC at CB receptor sites. The most common receptors in the cannabinoid system are CB_1

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and CB_2.[39] The CB_1 and CB_2 receptors are located in different areas of the body and responsible for different effects once activated. The CB_1 receptor is expressed primarily in the brain and is associated with the psychotropic effects of THC.[31] In contrast, the CB_2 receptor is primarily a peripheral receptor expressed in the immune, gastrointestinal, and other organ systems, although recent work has also reported the presence of CB_2 receptors in the brain. As a result of the complexity of the cannabinoid system in the body it is difficult to determine the targeted receptor and clinical effects of synthetic cannabinoids.

Synthetic cannabinoids exhibit many effects similar to commonly used cannabis products, although there are some differences. Physical effects caused by synthetic cannabinoids can include, but are not limited to, tachycardia, anxiety, hallucinations, acute kidney injury, convulsions, and psychosis.[31,40] Synthetic cannabinoids have been associated with severe toxicity and deaths by consumers leading to a number of mass intoxication reports in the United States between 2013 and 2015.[17] An example of an outbreak occurred in a small radius in New York City involving 33 intoxications due to AMB-FUBINACA.[41]

Synthetic cannabinoids can be metabolized into phase I and phase II metabolites. The metabolism of some synthetic cannabinoids has been studied and metabolites are readily found in clinical and forensic toxicological samples when testing urine.[42] In fact, it is common to only detect metabolites of synthetic cannabinoids in urine rather than detecting the parent compound. Therefore, it is important that methods designed for the detection of synthetic cannabinoids also includes metabolites.

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2.2.3 Synthetic Opioids

Since 2013 there has been an opioid crisis in the United States and synthetic opioids, especially fentanyl derivatives, are part of the epidemic,.[43] Synthetic opioids are often much cheaper than heroin and other opioids, which is a major factor as to why they show up unknown to the consumer.[44] Not only are synthetic opioids inexpensive, they are also easily purchased online anonymously from countries such as China. Illicit drug sellers will purchase synthetic opioids online and, unknown to their customers, they lace heroin with it to make even more of a profit. It is important to realize that fentanyl, carfentanil, and other fentanyl derivatives are much more potent than other opiates and opioids like heroin and morphine.[43,45] The fact that many fentanyl derivatives are so potent has led to an overabundance of synthetic opioid related overdoses and deaths. A large part of the opioid crisis revolves around drug users not knowing that heroin has been laced with more potent synthetic opioids, leading to fatalities.[46] During 2013 in Rhode Island, Pennsylvania, and North Carolina there were many fatal overdoses due to acetylfentanyl. However, acetylfentanyl was not scheduled until 2015 by the DEA.[47,48] The DEA reported a 300% increase in fentanyl cases from 2014 to 2015. Additionally, the CDC reported a 72% increase in synthetic opioid related deaths in that same time frame.[43]

New fentanyl derivatives continue to appear in the U.S. and Europe. Just like all other NPS, they are difficult to detect as new structures frequently appear in clinical and forensic toxicological cases. For example, during the years 2016 and 2017, over ten new fentanyl derivatives appeared in the U.S. contributing to overdoses and fatalities. Some examples include 4-methoxy-burtyryl-fentanyl, o-fluoro-fentanyl, tetrahydrofuranylfentanyl, and cyclopropylfentanyl.[45] The prevalence of synthetic opioids in the U.S. is a public health

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threat. It is important to be able to detect them and control them in order to combat the opioid epidemic in the U.S. and elsewhere.

Fentanyl and many of its derivatives act upon the μ-, δ-, and/or κ-opioid receptors in the human brain.[43] Common effects of synthetic opioid abuse include respiratory depression, miosis, and a changed mental status. There is a lack of research around the pharmacokinetics and pharmacodynamics in humans for illicit synthetic opioids. The information that does exist is derived primarily from animal models.[45] What is known is that many fentanyl derivatives have increased potency because of their lipophilic nature, ability to cross the blood brain barrier, and their high receptor affinity.[43] As an example, U-47700 is 7.5 times more potent in binding to the opioid receptor than morphine.[43] The increased potency exhibited by many synthetic opioids can lead to life-threatening respiratory and central nervous system depression. As a result of increased potency, many synthetic opioids require a higher dose of naloxone to counteract the opioid effect, which is not always known at the time of an overdose.

2.3 Identification of Drugs of Abuse and Novel Psychoactive Substances

Biological matrices for toxicologic analysis can include but are not limited to urine, oral fluid, exhaled breath, serum, plasma, whole blood, breast milk, meconium, and hair. The matrix chosen for analysis depends on the type of test being performed, what the analytes of interest are, what fluids are can reasonably be collected, and the window of detection desired. For example, breast milk and meconium are tested when a new mother is suspected to be abusing illegal drugs and there is a possibility that the baby was exposed *in utero*.[49] Hair can be used to determine long term abuse since it is possible that the analyte of interest stays in the hair as it grows. Segmental analysis can be performed to determine

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abuse during a certain time frame in someone's life.[50] Different matrices have different windows of detection that can range from minutes to potentially years.

Urine and blood are typically used for routine testing for drugs of abuse (*i.e.,* work place testing, rehabilitation, child welfare tests, and drug-facilitated assault).[51] Urine is often preferred over blood for routine testing for a number of reasons. Urine has a longer window of detection than blood; urine's window of detection can last days while blood is generally only a few hours. The collection process for urine is less invasive than blood and often results in a higher sample volume. A higher sample volume can be beneficial since there will be enough sample to retest if needed. Additionally, metabolites found in urine are concentrated, making them easier to detect. Even with many benefits, urine does have disadvantages when it comes to quantification.[52] Since the metabolites are concentrated in the urine it is difficult to calculate the actual amount of parent compound in the system.[53] Consequently, urine is a useful matrix for screening methods, but blood is often preferred when quantitation is important. Blood has some advantages over urine for both screening and quantitation.[54] Since quantitation is easier using blood samples, screening and quantitation can be done with using just one matrix. Additionally, compared to urine, blood levels often correlate with impairment, which is generally not true for urine. Additionally, the body regulates blood volume and only allows it to vary within a small window.[54] Finally, parent drugs of abuse can often be detected in blood prior to metabolism, unlike urine where metabolites are more common.

Urine and blood are the most common sample matrices used for the detection of NPS. When urine is the matrix being analyzed it is important that metabolites are screened for, especially when synthetic cannabinoids are of interest. Typically, only metabolites of

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synthetic cannabinoids are found in urine, therefore looking for parent drugs only may not be sufficient. Analysis of synthetic cannabinoid metabolites in urine can be challenging, due to their multiplicity (often up to two dozen metabolites may be present) and possible instability.[40,42] Blood can be used as an alternative matrix to urine for NPS with longer half-lives where it is more pertinent to screen for the parent compound. Unfortunately, most NPS are not well understood or researched, therefore the metabolism and metabolites of many NPS are not known. Since many metabolites are unknown, blood may be the desired matrix since the parent ion can be screened for. It is important to consider what NPS a method is capable of screening for when choosing the ideal matrix to use.

2.3.1 Traditional Forensic Toxicological Analysis

Toxicological sample analysis for forensic and clinical laboratories typically requires two steps. The first stage is screening for a wide variety of drugs of abuse, while the second stage typically involves confirmation with a more selective and sensitive method of detection.[55] Only samples that show a positive result on the screening method move on to be confirmed via the second analysis approach. This poses a problem for the detection of NPS, since many screening methods are not designed to specifically detect them. Therefore, samples that may be positive for NPS may be overlooked and never confirmed.

Forensic toxicological analysis is well understood and established for common drugs of abuse. The Substance Abuse and Mental Health Services Administration (SAMHSA) is an agency under the Department of Health and Human Services. SAMHSA has developed the term SAMHSA 5, which are five drug group analytes that are typically tested for in clinical and forensic toxicology laboratories. These five groups of drugs include phencyclidine (PCP), cocaine, amphetamines, THC, and opiates. Forensic and clinical

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laboratories have well established protocols for testing the SAMHSA 5, unfortunately those protocols are inadequate for assessing the presence of NPS that may be found in case samples.

2.3.2 Immunoassays

Immunoassays are one of the most common screening tools used in toxicology, followed by the use of GC-MS or LC-MS for confirmation. Immunoassays are an effective, inexpensive, and rapid screening method for common drugs of abuse but have their disadvantages when it comes to screening for NPS.[56] Immunoassays are designed to react with a specific structure or functional group. Since NPS are constantly undergoing structural changes, it is unlikely that they will show cross reactivity with immunoassays commonly used in forensic and clinical toxicology.

Frequently used immunoassays include enzyme linked immune sorbent assay (ELISA) and enzyme multiplied immunoassay technique (EMIT). Unfortunately, since NPS continuously undergo structural changes it is difficult to detect new compounds emerging on the market. There are disadvantages when using immunoassays to screen for NPS. Immunoassays work through an antigen-antibody interaction, which relies on the structure of the analyte in order to show cross reactivity, which is required for a positive result. Typically, each immunoassay only cross reacts with a small number of compounds, since the antigen-antibody interaction must be specific in order to be selective. When new NPS appear on the market they often are missed by immunoassay screenings since the antibody often is not capable of reacting with a structure different than originally intended.

There have been numerous research papers published that tested the cross reactivity of NPS with different immunoassays.[57-59] An example is the work accomplished by

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Swortwood et al. demonstrating the lack of cross reactivity between various NPS and commonly used immunoassays.[60] Beck et al. published research looking into the cross-reactivity of NPS with commercially available immunoassays. Their findings revealed that many of the NPS tested do cross react with commercial immunoassays, leading to an issue of potential false positives. They proposed that these commercial immunoassays could potentially be used to detect for the NPS that showed cross-reactivty.[59] This can pose some issues since compounds are cross-reacting with immunoassays that are not designed to detect them, making the immunoassay less selective and further needing to rely on secondary testing for confirmation.

Recently, there have been immunoassays developed specifically for the detection of NPS, but the creation of new immunoassays can be expensive and a very lengthy process.[61,62] In 2011 Wang et al. published data on an immunoassay designed to detect fentanyl in urine.[63] However, as different fentanyl derivatives became a concern, the immunoassay did not show cross reactivity with the derivatives. Randox Toxicology does have some commercial ELISAs for fentanyl, MT-45, AH-7921, and U-47700, however, this is a small subset of NPS that are available. Ellefsen et al. validated a commercially available immunoassay for synthetic cathinones in urine showing that there is substantial cross-reactivity with a number of synthetic cathinones.[64] Because of the high risk of false negatives and false positives for NPS using commercial immunoassays, a reliable screening method needs to be available that can easily be adapted for the everchanging structures of NPS that can be seen in clinical and forensic cases.

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2.3.3 Instrumental Analysis

Instrumental analysis tends to be more selective and sensitive than other analysis methods. The most common instrumentation used in clinical and forensic toxicology laboratories are GC-MS and LC-MS. Such instrumentation can provide high through-put and more sensitive and selective results than other screening methods. Instrumental analysis is most commonly used for confirmation and is also capable of quantitating drug compounds in biological matrices. Instrumental analysis can also be used for screening purposes.

2.3.3.1 Gas Chromatography Mass Spectrometry

Gas chromatography mass spectrometry (GC-MS) has been the gold standard in toxicological screening for many years. Gas chromatography is well understood and established in many laboratories. It is a rugged, selective, and sensitive technique that can be used for a number of applications including screening for drugs of abuse.

Gas chromatography can be coupled to different mass spectrometers for a variety of detection purposes. There are different types of sources that can be utilized for GC-MS. Ionization sources can either be hard or soft. Hard ionization sources are extremely energetic and result in extreme fragmentation. Soft ionization techniques only produce ions of the molecular species being analyzed.[65] There are three ionization sources that are typically used in GC-MS, including electron ionization, chemical ionization, and field ionization.[65] Electron ionization, chemical ionization, and field ionization are generally considered to me hard, intermediate, and soft ionization techniques, respectively.

Gas chromatography MS is excellent for the detection of volatile, non-polar, and thermally stable compounds. However, many other compounds require derivatization

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before analysis. Gas chromatography MS has increased resolving ability and selectivity as compared to LC-MS. Even though there are a number of benefits associated with GC-MS, it is also has disadvantages that need to be considered when determining the appropriate analysis technique for drug compounds in biological fluids. Depending on the matrix of the sample that is being tested, GC-MS may require extensive sample preparation such as derivatization before analysis.

2.3.3.2 Liquid Chromatography Mass Spectrometry

Recently, many laboratories have moved towards using liquid chromatography mass spectrometry (LC-MS) for clinical and forensic toxicological analysis.[66,67] There are a number of benefits of using LC-MS over GC-MS for biological samples. One benefit is the ease of sample preparation, since there is no need for derivatization. Liquid chromatography MS is a very good technique for the detection of non-volatile, polar, and thermally-labile compounds. This is important because many drugs of abuse and NPS fall into this category of analyte. These types of compounds need to be analyzed using a direct ion source. These sources can either be in liquid phase or solid state. Analytes are in a liquid state for liquid phase ionization and introduced to the source using nebulization.[65] Examples of liquid phase ionization include electrospray ionization, atmospheric pressure chemical ionization, and atmospheric pressure photoionization.

Electrospray ionization (ESI) is a very common source used with LC-MS. Originally, ESI was most commonly used for the analysis of proteins, but later it was adapted for other polymers, biopolymers, and small polar molecules.[65] Electrospray ionization is appealing

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for LC-MS since it allows for high sensitivity and can easily be coupled to LC. Electrospray ionization is formed by applying a strong electric field, under atmospheric pressure, to a liquid as it passes through a capillary tube. A high potential difference is applied between the capillary and the counter electrode to acquire the electric field. The field produces a charge build up at the liquid surface at the end of the capillary, which disperses to form charged droplets creating a Taylor cone.[68] The droplets then pass through either an inert gas or a heated capillary to remove the remaining solvent. Figure 1 is a schematic of the ESI process.

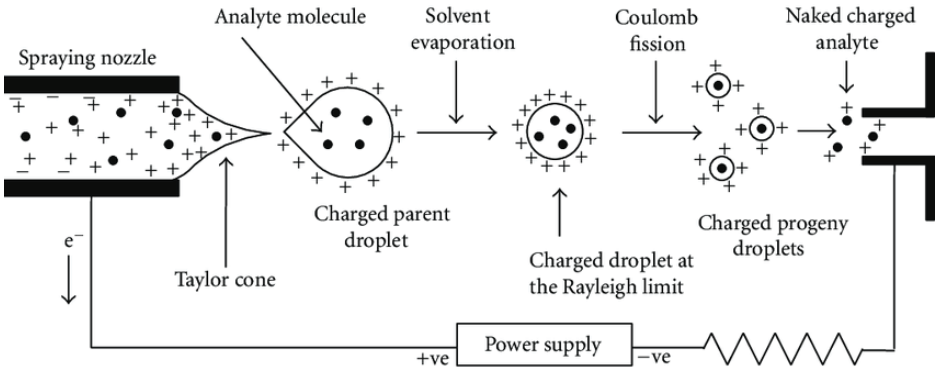

Figure 1. Represents the ESI process [68]

Most commonly, tandem mass spectrometers are used in forensic toxicology laboratories for screening and confirmation of drug compounds in biological matrices. Triple quadrupole mass spectrometers (QqQ-MS) are one example of a tandem mass spectrometer that is commonly used in forensic toxicology laboratories. The first quadrupole sifts out a precursor ion, the second is a collision cell for fragmentation, and the third selects the product ions for detection. Figure 2 is a schematic of a triple quadrupole mass spectrometer.

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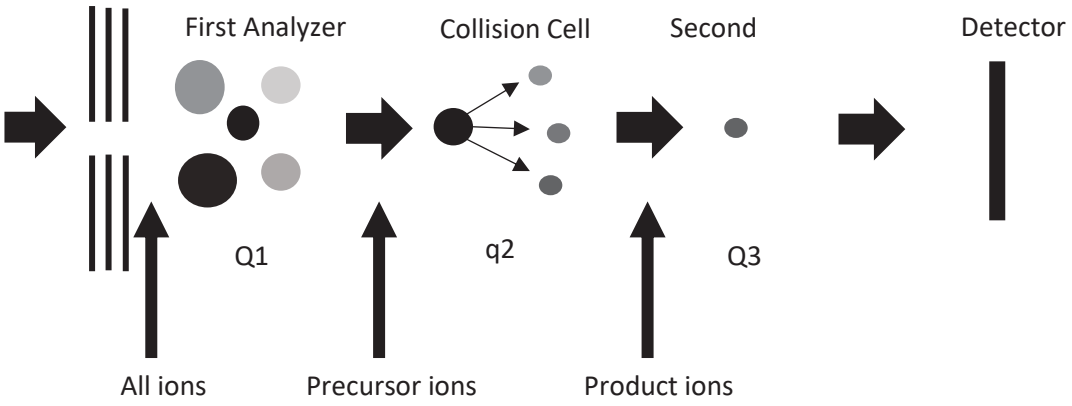

Figure 2. Schematic of a triple quadrupole mass spectrometer showing the function of each quadrupole.

There are four main scanning modes that can be used with tandem mass spectrometry. These scanning modes include product ion scan, precursor ion scan, neutral loss scan, and selected reaction monitoring (SRM). In product ion scan mode, specific precursor ions (*i.e.,* specific *m/z* ratio) are selected for in the first quadrupole, then they go through fragmentation, and finally the third quadrupole scans for all precursor ions resulting from the chosen product ion. In precursor ion scan mode, the precursor ions are scanned for in the first quadrupole and then after fragmentation specific product ions are targeted and looked for in the third quadrupole. In neutral loss scan mode, all precursor ions are scanned for in the first quadrupole, they undergo fragmentation and then the third quadrupole is offset by a selected neutral loss and scanned. Lastly, during SRM, the first and third quadrupole both have *m/z* ratios that have been targeted for. Selected reaction monitoring can either be single reaction monitoring, which targets one precursor ion and one product ion or multiple reaction monitoring (MRM) targeting multiples transitions of each precursor ion.

There are two main acquisition techniques associated with tandem mass spectrometry, targeted and non-targeted. Targeted methods can provide extremely low LOD and are ideal for quantification.[69] Targeted analysis in forensic toxicology labs is often accomplished

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using an MRM method and LC-QqQ-MS.[69] Most MRM methods target two to three transitions per analyte of interest. When using MRM, analytes of interest are detected in samples through the comparison of retention times and ion ratios. Targeted MS/MS approaches typically require database searches or library matches. There are LC-MS libraries available containing a large number of forensically significant compounds. Some examples of libraries and databases are the Wiley Registry MSMS and the NIST 11 MSMS library.[70] Dresen et al. developed an ESI MS/MS library containing 800 forensically relevant compounds in 2006 and added an additional 453 compounds in 2009.[71,72] Electrospray ionization MS/MS libraries are widely used in clinical and forensic toxicology.

There are several libraries available for both low resolution and high-resolution MS instruments. Compounds of interest are typically identified in clinical and forensic toxicological samples on the basis of library matches.[67] Therefore, in order to identify any analyte of interest, it must be in available libraries and/or databases. Non-targeted analysis using HRMS is possible; however, it is not well established in forensic toxicology laboratories at the present time. While non-targeted analysis may become more routine in clinical and forensic toxicology laboratories in the future, current approaches generally utilize available databases and libraries to identify NPS in case samples.

As an alternative to traditionally used low resolution (unit) mass spectrometers, high resolution mass spectrometers (HRMS) are also used in some forensic laboratories for screening purposes. HRMS has increased in popularity over the past decade because of increased selectivity over low resolution MS. There are several reviews and research articles published focusing on HRMS for clinical and forensic toxicology applications.[73-

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[76] Time of flight (TOF) and orbitrap are key examples of tandem HRMS. Disadvantages of HRMS for clinical and forensic toxicological analysis include the cost of the instrumentation, complexity of data analysis software, and the need for a skilled operator.[77] Regardless, HRMS is an excellent technique for screening for drugs of abuse. However, low resolution LC-MS/MS techniques (*i.e.,* LC-QqQ-MS) are still the standard in many forensic laboratories for quantitation.[66]

In 2010 Wohlfarth et al. published a paper describing a LC-MS/MS method for detecting a number of NPS in serum. The classes of NPS included in their work were synthetic amphetamines, trytamines, and piperazines. They were able to screen for a total of 35 NPS.[78] Extensive research on screening and confirmatory methods for use in detecting NPS in different biological matrices has been done since then. Ammann et al. created methods for the detection and quantification of NPS in blood, specifically targeting synthetic cannabinoids and designer cathinones.[79,79] Adamowicz and Tokarczyk developed a method for the rapid screening of 143 NPS by LC-MS/MS. The compounds they focused on varied widely in drug class.[80] A screening and quantitative method was designed by Glicksberg et al. to detect synthetic cathinones in urine and whole blood using LC/QTOF. Their method was designed to detect 22 NPS, which is a small subset of the NPS that have been reported.[81] Swortwood et al. created a method for the LC-QqQ-MS capable of screening for 32 cathinones and tryptamines in serum.[82]

Work has also been done focusing on the detection of synthetic cannabinoids and their metabolites in urine using LC-MS/MS.[42,83] These are examples of class-based methods; with the number of NPS currently available and their varying classes it is important that there exists a more comprehensive method. There are a number of methods that have been

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created that are capable of screening for a much higher number of NPS that are not class focused. For example, recently Patridge et al. created and validated a method for the screening of 320 compounds, including several NPS, using LC-QTOF-MS. However, Patridge's method was only designed to quantitate 39 of the 320 compounds.[84] Vaiano et al published a screening method for 64 NPS in blood using LC-MS/MS. The importance of Vaiano's research was its application to real case samples.[85] Vainano's research is a prime example of the applicability of LC-MS/MS for screening NPS in biological matrices. Work has been published suggesting that LC-MS/MS is a beneficial alternative to immunoassays for screening many drugs of abuse for forensic toxicology.[86]

An increased number of NPS have been detected in clinical and forensic toxicological samples over the past two decades and extensive research has been accomplished in order to combat the detection issues that are associated with NPS. However, there are still several gaps in the research revolving around the detection of NPS. Many of the published methods are only class focused and can only detect a small subset of the possible NPS in that class, especially for synthetic cannabinoids. Even though there are more comprehensive methods available, many of them lack the ability to quantify samples. With the quantity of NPS that are available to consumers and their potential to cause overdose toxicity and death, it is important to have a comprehensive screening and confirmatory method for NPS.

2.4 Extraction Methods

2.4.1 Extraction Methods for Common Drugs of Abuse

Toxicological analysis is completed by analyzing an array of biological fluids, each requiring extraction or purification to detect the analytes of interest. The most effective

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extraction technique highly depends on the matrix the sample is in and the analytes of interest. Some commonly used extraction methods include dilute-and-shoot, crash-and-shoot, liquid-liquid extraction (LLE), and solid phase extraction (SPE).[55]

Matrix effects are of major concern when deciding on a proper extraction and detection methods for different drugs of abuse from biological matrices. The ionization step in LC-MS is susceptible to matrix effects.[67,87,88] Matrix effects are caused by the presence of co-eluting compounds that can increase or decrease the signal of the analyte of interest. An increase of signal is known as ion enhancement and a decrease of signal is referred to as ion suppression. Matrix effects are not always detrimental to an LC-MS method, but can be an issue when detecting analytes of interest in the lower limits of detection and quantitation of the method or when accurate determination of concentration is important (*e.g.*, driving under the influence cases).

Dilute-and-shoot is a common method used in forensic toxicology laboratories for the analysis of urine samples, which involves diluting urine samples with water before analysis. The dilution of the urine samples is necessary to protect instrumentation from the high salt concentration that can be present in urine. The dilution aids in decreasing potential matrix effects in the urine samples although it does not remove them. Crash-and-shoot involves denaturing and precipitating out proteins from whole blood, plasma, or serum samples. This is done by adding cold solvent to the sample and then centrifuging the sample to pellet and remove cellular material and proteins. The supernatant can then be used for analysis. The addition of the solvent will remove most proteins that could damage instrumentation and/or cause matrix effects.

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2.4.2 Solid Phase Extraction

Solid phase extraction (SPE) is a common extraction technique used in forensic toxicology labs. Solid phase extraction is a robust technique that is capable of removing the majority of matrix interferences from various biological matrices, including urine, blood, and hair. Solid phase extraction is composed of four main steps; conditioning, loading, washing, and eluting. Conditioning is done to wet and alter the pH of the extraction cartridge so that the analytes of interest are capable of attaching to the cartridge during the loading process. Cartridges can be made of different adsorbent materials, all of which have different uses. The loading step is completed by slowly running the sample through the cartridge. Washing is done in order to remove any unwanted compounds/substances from the cartridge before elution. Elution occurs when the analytes of interest are removed from the cartridge and recovered in a solution that can then be analyzed and which should be free of most of the contaminants originally present in the complicated matrix.

Solid phase extraction is well understood and researched for common drugs of abuse. Applications of SPE for NPS are less studied and typically are adaptations of protocols for common drugs of abuse of similar classes. Since many NPS have undergone structural alterations changing their chemical interactions, Solid phase extraction protocols may need to be altered in order to properly extract NPS from different matrices. Solid phase extraction relies heavily on the chemical structure of the analytes of interest in order to retain them on the SPE cartridge and to elute them during the proper stage.

2.4.2.1 Online Solid Phase Extraction

Online SPE is an alternative to classical SPE which is designed to decrease the time and overall cost of extracting compounds from various matrices. The extraction method is

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both automated and in line with the instrumentation, which eliminates a number of transfer steps, which can result in increased recovery of the analytes of interest. Often, online SPE is used as an additional cleanup step for environmental water samples.[89] However, work has been done with online SPE for the extraction of drugs of abuse. Heuett and co-workers developed an online SPE method for the extraction of common drugs of abuse from waste water.[90] Moosavi et al. developed an automated SPE method for the extraction of thiopental from plasma.[91] Many of the developed methods are not specifically designed to be implemented into forensic and clinical toxicological laboratories. There is one example of work that has been done focusing on NPS using online SPE, published by Lehman et al.[92] Their work focused on the extraction of 74 NPS from serum using online SPE LC-MS/MS. These are examples of the usefulness of online SPE for the extraction of drugs of abuse from biological matrices.

There a number of benefits to online SPE, but it is not without its challenges. Online SPE can be very complex when developing a new method, has potential for sample loss, and sample can be retained on cartridges, which can also lead to carry over. Online SPE is also limited to the available cartridges for the system, which are not as varied as the options for classical SPE.

2.4.3 QuEChERS

The QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) extraction technique was originally developed in 2003 by Anastassiades et al. to extract pesticides from a wide variety of produce.[93] Since then many environmental laboratories have utilized QuEChERS for different complex matrices, including food, soil samples, and invertebrates.[94-96] In general, QuEChERS is an ideal extraction method for extremely dirty

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and complex samples. The QuEChERS technique was originally designed as a two-step process. The first is a drying and partitioning step, while the second step involves dispersive SPE (d-SPE).[93] During the first step, acetonitrile is added to the sample so that liquid-liquid partitioning can be performed by adding anhydrous magnesium sulfate and sodium chloride. Once the first step is completed, a specific volume of the acetonitrile layer is removed and added to $MgSO_4$ and a sorbent (typically primary secondary amines; PSA) is added to accomplish d-SPE.

Companies such as Agilent Technologies and UCT have developed commercial QuEChERS kits for extraction with various applications, typically advertised for environmental samples, which require large sample volumes. However, some manufacturers do sell QuEChERS kits compatible with small sample sizes.

The QuEChERS approach has also been employed in forensic applications using a variety of biological matrices including whole blood and liver tissue.[97-99] Different research groups have tested a number of different approaches using different sample sizes, combinations of salts and sorbents, and one and two step methods. In 2013, Matsuta et al. designed a one-pot extraction method for 13 compounds of various classes and metabolites in blood. Matsuta's work was one of the earlier examples using QuEChERS for a forensic application.[100] Westland and Dorman also published work in 2013 revolving around using QuEChERS for biological matrices. Their work focused on extracting benzodiazepines from sheep blood and human urine.[99] Soon after, Usui et al. published their QuEChERS method for extracting drugs of abuse from liver samples. Their method was applied to forensic toxicological case samples, showing the potential of QuEChERS for case work.[97] Anzillotti et al. designed a cleanup up method for drugs of abuse and benzodiazepines

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using QuEChERS, showing further applications of QuEChERS in the field of forensic toxicology.[101] Dulaurent et al. designed a QuEChERS approach for a broader set of drug classes including opiates, amphetamines and cocaine in whole blood.[102] Recently Dybowski and Dawidowicz published a QuEChERS method for Δ^9-tetrahydrocannabinol and its metabolites in whole blood.[103] Pouliopoulos et al. designed a QuEChERS approach for the detection of psychotropic drugs in postmortem blood samples.[104]

The publications described above show the potential of QuEChERS in the field of forensic toxicology for the extraction of drugs of abuse from various biological matrices. There are advantages and disadvantages to this approach as described in current literature. Some of these methods require a large volume of sample, which is not ideal for case work. A mini one-pot approach is ideal for forensic applications. Case work samples usually have a limited volume to work with and require high throughput. Using a mini one-pot approach limits the amount of sample needed, cuts down on time and transfer steps, which makes it an ideal alternative extraction technique for forensic toxicology samples.

2.4.4 Evaluation of Extraction Techniques

All of the extraction techniques discussed need to be evaluated and optimized for NPS and compared to determine the benefits and costs associated with each method. There is published literature on the use of SPE for the extraction of NPS from blood and serum.[75,78,82] The majority of methods published for the detection of NPS in serum or blood utilized SPE or a form of protein precipitation.[42,80,82,105] There is very little published data solely focusing on the extraction of NPS from biological fluids, the focus is typically on the detection method. Little research using online SPE and QuEChERS has been reported for the extraction of NPS from biological fluids. Lehmann et al. published a method

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capable of detecting 74 NPS using in-line SPE LC-MS/MS. Lehmann's research is one of the few examples of the use of automated SPE for the extraction of NPS.[92] It is important that the usefulness of these techniques be tested for the extraction of NPS so that they can be implemented into forensic toxicology laboratories. It is not always possible to use a method designed for common drugs of abuse for NPS, often times they need to be optimized specifically for the extraction of NPS. Optimized extraction techniques for NPS resulting in increased recovery of NPS from biological matrices can be beneficial for forensic toxicological laboratories.

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3. DEVELOPMENT OF A DYNAMIC MULTIPLE REACTION MONITORING METHOD

3.1 Introduction

Novel psychoactive substances are a global health hazard. Novel psychoactive substances are structural alterations of drugs of abuse that are manufactured in order to evade drug laws.[2] The detection of NPS poses difficulties for clinical and forensic toxicological laboratories because of the structural alterations. Immunoassays are commonly used for screening biological matrices. However, immunoassays are not capable of detecting the majority of NPS.[82,86] Immunoassays are designed to detect specific drug structures or classes of drug compounds. Therefore, immunoassays are not appropriate for detecting NPS unless specifically designed to do so. There are a few immunoassays capable of screening for a small number of NPS, which still leaves out a large number of NPS that can be found in clinical and forensic toxicological samples.[61,64] As an alternative to immunoassays, MS-based analytical techniques can be used to screen biological matrices for drugs of abuse. Mass spectrometry-based techniques (*e.g.*, GC-MS and LC-MS) typically require a spectral library or compound database in order to screen for compounds. There are a limited number of LC-MS libraries and databases that include NPS. In order to detect NPS they must be included in the spectral libraries and databases for positive matches.

The research presented here reports the collection of MS transitions and development of a comprehensive dMRM method for the detection of 750 chemical compounds, the majority of which can be considered NPS and metabolites. Transitions were collected for all 750 compounds and 76 additional deuterated internal standard compounds. Of the total

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number of compounds, a final method was developed to detect 731 NPS and 22 internal standards, with two transitions per compound.

3.2 Method and Materials

3.2.1 Chemicals and Materials

Reference standards for the NPS compounds, including deuterated standards, were obtained from Cayman Chemical (Ann Arbor, MI) as the neat solid material for the majority of compounds, although some were already in solution. Optima LCMS grade methanol (MeOH), acetonitrile, dichloromethane, dimethyl sulfoxide (DMSO), HPLC water, ammonium formate (99%), and formic acid were purchased from Fisher Scientific (Fair Lawn, NJ).

3.2.2 Standards and Sample Preparation

Neat standards (including deuterated compounds) were dissolved in MeOH or DMSO, depending on the analyte's solubility, to achieve concentrations of 1, 2, 5, or 10 mg/mL. From these initial preparations, 10 µg/mL working solutions were prepared in MeOH for all analytes to be used for transition optimization and method development. In addition, from the 10 µg/mL solutions, 1 µg/mL solutions were prepared in methanol for each of the analytes to collect transitions and LC retention times in order to create the final dMRM method. An arginine reference standard from Cayman Chemical was used as a quality control standard and run daily.

3.2.3 Instrumentation and Software

All samples were analyzed using an Agilent 1290 Infinity Binary Pump LC coupled to an Agilent 6460 triple quadrupole MS/MS with Jet Streaming technology and electrospray ionization (ESI) equipped with Agilent MassHunter software version B7.0.

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Chromatographic separation was performed using an Agilent Zorbax Rapid Resolution HD Eclipse Plus C_{18} column (3.0 x 100 mm, 1.8 μm). Data acquisition was performed in dMRM mode using positive ESI. The dMRM method employed two transitions for each analyte and internal standard, which aids in achieving increased selectivity. Using multiple transitions can help in discerning one compound from another if they have similar retention times or coelute, provided that they have uniquely different transitions.

3.2.4 Methods

All standards were initially analyzed by flow injection analysis (FIA; without LC column) by QqQ-MS using the 1 μg/mL working dilution. Diluted standards were individually injected directly into the Jet Stream ESI ion source. Data were collected in positive ion mode using an isocratic mobile phase of 80:20 0.1% formic acid in methanol:5 mM ammonium formate with 0.1% formic acid in HPLC grade water. If FIA was successful, the standards were then analyzed using Optimizer software, which searches for 4 to 10 product ion transitions that are analyzed via an Optimizer Report. The report includes precursor ion, fragmentor voltage, product ions identified, collision energies, and abundances. All compounds that had four or more transitions with ion abundances above 1000 counts were then separated by LC to obtain standardized retention times.

Collected transitions were used to develop a dMRM method. Chromatographic separation was achieved using gradient elution with a flow rate of 0.3 mL/min using 5 mM ammonium formate/0.1% formic acid in HPLC water as mobile phase A and MeOH with 0.1% formic acid as mobile phase B. The gradient used for analysis was as follows: hold at 5% B for 1 min, followed by 5% B to 98% B from 1 to 9.5 min, then hold at 98% B until

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16 min, followed by a 3-min re-equilibration at 5% B. The analytical column was held at a temperature of 40˚C during separation.

The MS source parameters were as follows: gas temperature, 325˚C; gas flow 6 L/min; nebulizer 40 psi; sheath gas temperature 350˚C; sheath gas flow 11 L/min; capillary voltage 4,000 V; and nozzle voltage 750 V. Agilent MassHunter Optimizer software was used to determine the ideal data acquisition parameters for MRM mode. A dMRM method was chosen to increase selectivity, using analyte retention times, detection windows (Δt_R), and constant scan cycle time to allow for the detection of multiple analytes in a small window. Analyte detection windows ranged from 0.25 min (*i.e.,* ± 0.125 min around t_R) to 0.75 min (*i.e.,* ± 0.375 min around t_R) depending on the analyte.

To collect retention times for method development, individual compounds were injected at concentrations of 1 µg/mL in MeOH at volumes of 3 µL. Separation was conducted over 16 min using an Agilent Zorbax Eclipse Plus C_{18} Rapid Resolution HD column (3.0 x 100 mm; 1.8 µm) and the LC-QqQ-MS method described above. Retention time data were collected using a dMRM method with all retention times set to 8 min and a window of 16 min so that there was a continuous scan. The "Find by MRM" function of MassHunter Qualitative Analysis software was used to isolate the individual compound from each injected solution and the corresponding retention data were recorded.

3.3 Results and Discussion

Agilent MassHunter Optimizer software was used in order to identify the up to 10 fragments, associated collision energy, and optimal fragmentor voltage for each of the analytes included in this method. Appendix 1 shows the in-house identifier, compound name, chemical formula, precursor ion, transitions, collision energies, and abundances for

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all compounds that underwent optimization and were included in the final method. From these data the two most abundant and/or most individualized transitions were chosen for each compound and included in the final dMRM method.

Not all of the compounds analyzed by FIA showed the expected *m/z* ratio in positive mode. Since positive mode was found to be appropriate for the majority of NPS to be included in this method, any compound that required negative mode ionization was excluded. The majority of compounds that required negative mode ionization were synthetic cannabinoids, with a few exceptions. Some of the excluded compounds include delorazepam, THJ 018, multiple CP cannabinoids, and a few RCS cannabinoids.

The information described above was then used to create a dMRM method capable of qualifying and quantifying the analytes of interest that require positive mode ionization. The LC gradient was chosen to separate as many compounds as possible during a 16-min run. The use of a dMRM method allowed for increased selectivity and for compounds with similar retention times but different transitions to be differentiated. An example would be 25I-NBMD and bromazepam, both with $t_R = 9.00$ min, which could be separately identified as a result of their unique transitions.

The final dMRM method included two transitions each for 750 compounds and 22 deuterated internal standards. Table 1 depicts the breakdown of drug entities included in the final method based on drug class. Table 2 depicts the distribution of compounds in the final method based on molecule type. The goal of this dMRM method was to be as comprehensive as possible, based on available standards, for the detection of NPS in clinical and forensic toxicological samples. Common adulterants found with illicit drug

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samples are one of the sub-categories that fall under the "other" category. The method is designed to detect NPS and their common adulterants in case samples.

Table 1. Structural classes for all compounds included in the dMRM database.

Drug Class	Number in Method
Synthetic Cannabinoids	449
Other*	121
Cathinone	112
Phenethylamine	43
Tryptamine	17
Piperazine	8

* includes opioids, amphetamines, benzofuran, and common adulterants

Table 2. Molecule types for all compounds included in the dMRM database.

Molecule Type	Number in Method
Precursor Compounds	470
Metabolites	117
Isomers	128
Analogs	30
Glucuronides	5

The final MRM database included data for 826 individual analytes including 76 deuterated standards (see Appendix). However, the final dMRM method was unable to include transitions for all of the compounds in the database in a single MS run. This limitation is caused by the instrument's ability to collect usable data, which relies greatly on cycle time and dwell time. Dwell time is the amount of time in ms that it takes to collect one transition, while cycle time is the time it takes in ms to collect all transitions associated with a compound.

$$dwell\ time\ (ms) = \frac{peak\ width\ (ms)}{(number\ of\ transitions)(number\ of\ points)} \quad (1)$$

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Dwell time is determined using equation 1. The issue with having too many transitions (*i.e.,* >800) is that there will not be enough points on the peaks of the data collected in order to quantitate the peak area. Ideally there should be about 20 points on the peak; with 1000+ transitions it is impossible to have enough points while still maintaining a reasonable dwell time. In a dMRM method there is a list of transitions that the instrument needs to scan through. Inclusion of all 826 compounds, with two transitions each, would require 1652 transitions. A specific retention time and window is assigned to every compound. Throughout this window the instrument needs to go through the scan 20 times in order to have 20 points on the peak. For certain compounds, retention time windows overlap, therefore they are sharing the total cycle time and that will change the dwell time for each transition. If the dwell time becomes too low the data will not be reproducible or statistically relevant. There are only so many transitions that can share the same retention window. If there are too many sharing the same cycle time, when the instrument tries to cycle through the transitions 20 times it will be trying to collect data from a peak that has already been eluted, because it cannot cycle through fast enough.

Additionally, when attempting to collect such a high number of peaks in a single 16 min run, the resolution between peaks would be very low. Consequently, since there are limitations associated with dwell time and cycle time, it is not possible to measure 800+ compounds in a single dMRM method. Therefore, in order to use the method described, it is necessary to break it into two separate screening runs. This is needed so that the quality of the resulting data are not compromised. For forensic toxicological laboratories to implement this method, each sample would therefore need to be run twice, resulting in a

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32-min rather than 16 min run. This should be acceptable considering the advantages of screening for so many NPS in each specimen.

3.4 Conclusions

The work presented here aimed to create a dMRM method capable of screening for 750 NPS. After undergoing flow injection analysis, it was determined that 729 of the total number of NPS were suitable for positive ion mode. Those 729 NPS were included in the final developed dMRM. The final method is intended to screen for a variety of NPS including metabolites. In order to use this method for forensic purposes it needs to be fully validated.

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4. METHOD VALIDATION USING A MIXTURE APPROACH

4.1 Introduction

In recent years, "designer drugs," also known as novel psychoactive substances (NPS), have become of major concern all over the world, especially in the United States. Novel psychoactive substances are compounds that are considered to be "substantially similar" to Schedule I or II substances determined by chemical structure and pharmacological effects, but that have not been scheduled and therefore are not yet "illegal".[106] Suppliers and consumers use NPS to evade established drug laws. Since every small structural change can result in a new NPS, these compounds are constantly increasing in numbers, making it very difficult for clinical and forensic laboratories to keep up with detection and identification.[107] The popularity of NPS continues to increase, as reflected by Internet content, the media, published scientific research, and the types of forensic and clinical cases reported, including reported fatalities and unexpected side effects.[108,109] It is not uncommon for NPS to have a more potent effect than their scheduled counterparts, leading to more cases of overdose and increased negative side effects. A recent example of potent NPS are the fentanyl derivatives that have been seen during the recent opioid crisis in the United States.[43-45] The continuous rise of NPS makes it clear that new detection methods are needed in order to keep up with the changing structures of compounds being abused.

With the increased importance of reliable detection of NPS in forensic casework, research focusing on creating and validating methods capable of detecting NPS has accelerated.[77,82,110] The majority of published methods that focus on detecting NPS fall into two categories; those that provide quantitation of a relatively small number of NPS and those that can screen for (but not quantitate) a larger number of NPS. As a consequence of

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the limitations of current screening/confirmatory approaches, methods need to be created that encompass both of the goals discussed above.

There are established guidelines by OSAC that need to be followed so that a toxicological method can be considered validated. There are strict instructions on what parameters need to be validated depending on the overall purpose of the method being validated (*i.e.,* qualitative or quantitative).[111] Peters et al. published additional guidelines on method validation as it refers to forensic toxicological analyses. Forensic toxicology methods must be painstakingly validated and periodically tested to ensure the quality of the results.[112] The main parameters for validation are LOD, LOQ, selectivity, linearity, carry over, bias, precision, freeze/thaw stability, and matrix effects. There are specific ranges of acceptable values that all of these results need to fall into in order to be considered validated. Each compound in a method must be validated for all of the parameters listed. For any method consisting of a small set of compounds it is possible to validate the method one compound at a time, however, it is more efficient to validate methods using a mixture approach.

The present work focuses on the development of a validated LC-QqQ-MS method that is designed to confirm and quantitate 800+ NPS. To fully validate a method of this size, a mixture approach was adopted. Specifically, 826 individual NPS and metabolites were incorporated into 16 non-coeluting mixtures for method validation. The mixture approach was utilized to facilitate timely method validation for such a large number of analytes, since validation of one compound at a time was not feasible. Mixture approaches have previously been used for the validation of screening methods.[78,84] However, they are less commonly

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used to validate quantitative methods. Typically, only one mixture is used for methods quantitating 40 or less compounds in total.[80,82,113] The current work employs the mixture approach on a much wider scale, to fully validate a quantitative method capable of detecting a very large number of NPS. The present report focuses on a subset of three mixtures that have been fully validated as a proof-of-concept of this approach.

4.2 Materials and Methods

4.2.1 Chemicals and materials

Reference standards for the NPS compounds, including deuterated standards, were obtained from Cayman Chemical (Ann Arbor, MI) as the neat solid material for the majority of compounds, although some were already in solution. Abbreviations and number association that will be used for a subset of the NPS included in this work can be seen in Table 3, which also separates the compounds into their mixtures. A total of 16 mixtures were designed for method validation, the results of three of those mixtures will be shown here. All other mixtures that have successfully undergone validation can be seen in the Appendix.

Table 3. List of the abbreviations used for the NPS contained in the three mixes used for validation.

#	Mixture 1	#	Mixture 2	#	Mixture 3
1	4-OH MET	26	3,4-DHMA	58	N,N-DMC
2	NMT	27	2-FMC	59	Phenylpiperazine
3	4'-fluoro- α -PPP	28	4-FIC	60	4-hydroxy DiPT
4	4-APDB	29	4-OH MiPT	61	THH
5	4-fluoro PBP	30	Clencyclohexeral	62	MMAI
6	5-MAPB	31	NEB	63	2,3-pentylone isomer
7	3-methyl BP	32	4-MMC	64	(-)-3,4-MDPV
8	4-methyl-α-EAB	33	3-methyl PPP	65	3C-B-fly
9	α-PVP metabolite 1	34	3,4-dimethoxy- α-PVP	66	Para-Fluorofentanyl
10	4-MeO-α-PVP	35	2,3-MDPV	67	PCEEA
11	JWH 200 5-hydroxyindole metabolite	36	4-ethyl-N,N-DMC	68	Benzydamine
12	PCMPA	37	2C-T-2	69	Bromazepam

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13	AM2233 azepane isomer	38	PCPr	70	AB-PINACA N-(5-hydroxypentyl) metabolite
14	4-MeO PV8	39	2C-T-4	71	25E-NBOMe
15	Benocyclidine	40	4'-Methylhexedrone	72	Etaqualone
16	25I-NBMD	41	25I-NBF	73	JWH 198
17	AB-005	42	Loperamide	74	AB-FUBINACA
18	Flubromazepam	43	AB-005 azepine isomer	75	(R)-(-)-JWH 018 N-(4-hydroxypentyl) metabolite
19	AM694 N-(5-hydroxypentyl) metabolite	44	A-796260	76	(+)-WIN 55,212-2
20	5-fluoro SDB-006	45	AB-FUBINACA 3-FB isomer	77	JWH 073 6-MeO indole analog
21	JWH 081 N-(5-hydroxypentyl) metabolite	46	JWH 018 N-(5-hydroxypentyl) metabolite	78	JWH 073 2'-naphthyl-N-(1,1-DME) isomer
22	PB-22 6-hydroxyisoquinoline isomer	47	MAM2201 N-pentanoic acid metabolite	79	BB-22 8-hydroxyisoquinoline isomer
23	NPB-22	48	ADB-PINACA isomer 1	80	JWH 019
24	JWH 203	49	RCS-4 2-MeO isomer		
25	THCA-A	50	PB-22		
		51	XLR11 N-(2-FP) isomer		
		52	UR-144 Degradant		
		53	AKB48 N-(5-FP) analog		
		54	KM 233		
		55	Δ8-THC		
		56	EG-018		
		57	SER-601		

Optima LCMS grade methanol, acetonitrile, HPLC water, ammonium formate (99%) and formic acid were purchased from Fisher Scientific (Fair Lawn, NJ). Certified blank urine was purchased from UTAK Laboratories Inc. (Valencia, CA) and aliquoted into 10 mL portions and stored at -20ºC until needed.

4.2.2 Solution and sample preparation

Neat standards (including deuterated compounds) were dissolved in methanol (MeOH) or dimethyl sulfoxide (DMSO), depending on the analyte's solubility, to achieve concentrations of 1, 2, 5, or 10 mg/mL. From these initial preparations, 10 µg/mL working solutions were prepared in MeOH for all analytes to be used for optimization and method

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validation.

4.2.3 Instrumentation

All samples were analyzed using an Agilent 1290 Infinity Binary Pump LC coupled to an Agilent 6460 triple quadrupole MS/MS with Jet Streaming technology and electrospray ionization (ESI) equipped with Agilent MassHunter software version B7.0. Chromatographic separation was performed using an Agilent Zorbax Rapid Resolution HD Eclipse Plus C_{18} column (3.0 x 100 mm, 1.8 μm). Data acquisition was performed in dMRM mode using positive ESI. The dMRM method employed two transitions for each analyte and internal standard, which aids in achieving increased selectivity. Using multiple transitions can help in discerning one compound from another if they have similar retention times/coelute, as long as they exhibit uniquely different transitions.

4.2.4 Preparation of standard mixtures

The 10 μg/mL solutions were used to prepare NPS mixtures for validation by spiking MeOH with each compound for a final concentration of 200 ng/mL per compound. The spiked MeOH solutions were used as the working solutions to create all samples needed for method validation. Validation mixtures contained anywhere from 22 to 65 compounds of varying NPS structural and pharmacological classes. The 750 compounds included in the final dMRM method were divided into a total of 16 different mixtures for ease of data analysis. Using a series of non-coeluting standard mixtures helped ensured selectivity during method validation. Compounds chosen for each mixture were determined by retention time and primary MRM transitions. Each mix included only non-coeluting compounds to ensure selectivity. In addition, an internal standard (IS) mixture of 22 compounds, each at a concentration of 200 ng/mL, was prepared to be used for

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quantification. The 22 deuterated IS compounds, along with LC retention time and drug class can be found in Table 4. Internal standards were chosen in order to cover all of the drug classes included in the in the final dMRM method. As it was impossible to find a deuterated compound for every NPS used in this research, a set of internal standards were chosen in order to match structures and drug classes as well as possible for each analyte.

Table 4. List of internal standards used for validation, drug class, and retention times.

Compound	RT	Drug Class
JWH 007-d9	11.63	Synthetic Cannabinoid
JWH 018-d9	11.52	Synthetic Cannabinoid
JWH 073 5-Hydroxyindole metabolite-d7	10.69	Synthetic Cannabinoid
JWH 081 N-pentanoic acid metabolite-d5	10.54	Synthetic Cannabinoid
(-)-11-nor-9-carboxy-Δ^9-THC-d3	11.47	Cannabinoid
(±)-CP 47,497-C8-homolog-d7	12.05	Synthetic Cannabinoid
AM 2201 N-(4-hydroxypentyl) metabolite-d5	10.26	Synthetic Cannabinoid
MAM 2201 N-pentanoic acid metabolite-d5	10.67	Synthetic Cannabinoid
PB-22-d9	11.3	Synthetic Cannabinoid
UR-144 N-(4-hydroxypentyl) metabolite-d5	10.85	Synthetic Cannabinoid
XLR 11 N-(4-hydroxypentyl) metabolite-d5	11.59	Synthetic Cannabinoid
RCS-4 N-(5-hydroxypentyl) metabolite-d5	10.08	Synthetic Cannabinoid
25I-NBOMe-d3	9.18	Phenethylamine
Benocyclidine-d10	9.08	Arylcyclohexylamine
3,4-Methylenedioxy pyrovalerone-d8	7.48	Stimulant
AB-PINACA-d9	10.74	Synthetic Cannabinoid
ADB-PINACA-d9	11.03	Synthetic Cannabinoid
AB-FUBINACA-d4	10.25	Synthetic Cannabinoid
Acetyl norfentanyl-d5	5.98	Opioid
Norsufentanil-d3	7.49	Synthetic Opioid
Butylone-d3	6.53	Stimulant
cis-Tramadol-d6	7.15	Opioid

Calibrators and quality control (QC) samples were prepared using pooled certified blank urine. Sample preparation before analysis consisted of using a dilute-and-shoot method with a ratio of 1:5 (urine:HPLC water). Samples were prepared by spiking urine

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with one of the NPS 200 ng/mL mixtures described above in addition to the IS spiking solution. Once spiked, the urine samples were diluted using HPLC water before undergoing LC-MS analysis.

4.2.5 LC Conditions and MS parameters

Chromatographic separation was achieved using gradient elution with a flow rate of 0.3 mL/min using 5 mM ammonium formate/0.1% formic acid in HPLC water as mobile phase A and MeOH with 0.1% formic acid as mobile phase B. The gradient used for analysis was as follows: hold at 5% B for 1 min, followed by 5% B to 98% B from 1 to 9.5 min, then hold at 98% B until 16 min, followed by a 3-min re-equilibration at 5% B. The analytical column was held at a temperature of 40˚C during separation.

The MS source parameters were as follows: gas temperature, 325˚C; gas flow 6 L/min; nebulizer 40 psi; sheath gas temperature 350˚C; sheath gas flow 11 L/min; capillary voltage 4,000 V; nozzle voltage was 750 V. Agilent MassHunter Optimizer software was used to determine the ideal data acquisition parameters for MRM mode. The software uses the mass of each compound to determine optimal fragmentor voltage, resulting product ions, and associated collision energies and can optimize for up to 10 transitions for each precursor ion.

Prior to validation of the method, retention time data for all compounds were collected. Separation was conducted over 16 min using an Agilent Zorbax Eclipse Plus C_{18} Rapid Resolution HD column (3.0 x 100 mm; 1.8 µm) and the LC-QqQ-MS method described above. The “Find by MRM” function of Qualitative Analysis software was used to isolate the individual compound from each injected solution and the corresponding retention data were recorded. These data were used to design the validation mixtures so that no two

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components of a mixture would co-elute, thus minimizing interference with identification and quantitation of the compounds.

4.2.6 Quantification

Agilent MassHunter Quantitative Analysis software version B.07.00 was used for quantification. The software was used to calculate and plot peak area ratios of drug versus internal standard. Using the calibration curves produced, the software then calculated the concentration of each sample.

4.2.7 Assay validation

The LC-MS/MS method was validated in accordance with guidelines for forensic toxicology method validation provided by OSAC and as described by Peters et al.[107-108] The parameters evaluated consisted of selectivity, matrix effects, recovery, linearity, freeze-thaw stability, carry over, accuracy, and precision.

4.2.8 Selectivity

Blank pooled urine samples were prepared using the dilute-and-shoot procedure described above and analyzed using the dMRM method to ensure that there were no peaks present that could interfere with the analytes of interest or internal standards. Blank urine was spiked with the IS mixture at a concentration of 100 ng/mL and analyzed to confirm that the internal standard peaks did not interfere with the detection of the targeted analytes. Lastly, each of the NPS mixtures were spiked into urine and analyzed to ensure that the targeted analytes did not interfere with the internal standard peaks.

4.2.9 Matrix effects and recovery

Traditionally, matrix effects are determined by comparing three different samples sets

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(*i.e.,* analyte of interest in solvent, analyte of interest spiked after extraction, and analyte of interest spiked before extraction); the described approach is not feasible when using a dilute-and-shoot method. As an alternative, matrix effects were determined by comparing the results of the analytes of interest spiked in HPLC water (Set 1) and spiked into pooled urine that was diluted before analysis (Set 2). Matrix effects were determined at three different concentrations, 5 ng/mL (LOW), 20 ng/mL (MED) and 80 ng/mL (HIGH) through the comparison of peak areas. Matrix effects were calculated by using equation 2 shown below. Positive values represent ion enhancement and negative values represent ion suppression; the higher the value, the higher the level of interference. According to OSAC guidelines, a value of ±25% for matrix effect is acceptable for method validation.

$$Matrix\ Effect = \left(\frac{Set\ 1 - Set\ 2}{Set\ 1}\right) * 100 \qquad (2)$$

4.2.10 Linearity of calibration and limits of detection/quantitation

Calibration curves were analyzed by using seven calibration levels ranging from 1 to 100 ng/mL (*i.e.,* 1, 2, 5, 10, 20, 50, and 100 ng/mL). Each of the 22 IS in the IS spiking mixture were present in each calibrator at a concentration of 40 ng/mL. Each calibration level was prepared in pooled blank urine at a volume of 0.4 mL. Replicates (n=4) at each concentration were analyzed using the dMRM method described above over the course of five different days. Regression lines were calculated for each analyte of interest using Agilent MassHunter Quantitative Analysis software with a weighted (1/x) model.

Limit of detection and LOQ were determined using Equations 3 and 4, respectively. Using the calibration curve to derive LOD and LOQ was deemed a viable option since all calibration curves were linear. Alternative methods for determining LOD and LOQ could have been employed, but this approach was appealing since it did not include additional

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analysis, which can shorten the time required for method validation when working with a large number of compounds. The described approach uses the equation of the line, where s_y is the standard deviation of the y-intercept and Avg_m is the average slope of the line:

$$LOD = (3.3s_y)/Avg_m \qquad (3)$$

$$LOQ = (10s_y)/Avg_m \qquad (4)$$

4.2.11 Precision and accuracy

Precision and accuracy were determined through the analysis of QC samples at three different concentrations, 5, 20, and 80 ng/mL. Each QC concentration level was analyzed in replicates (n=3) over five different days. The mean value for each concentration on each day were used to determine interday bias and precision. Each replicate was used to determine intraday bias and precision. Equations 5 and 6 are used to determine interday and intraday percent coefficient of variance (%CV) values, respectively. According to OSAC guidelines, 20% CV and ±20% bias are acceptable for method validation.

$$Interday\ CV\ (\%) = \frac{std\ dev.of\ all\ observations\ for\ each\ concentration}{grand\ mean\ for\ each\ concentration} x\ 100 \qquad (5)$$

$$Intraday\ CV\ (\%) = \frac{std\ dev.of\ a\ single\ run\ of\ samples}{mean\ calculated\ value\ of\ a\ single\ run\ of\ samples} x\ 100 \qquad (6)$$

Recovery was determined using the same QC samples that were used for bias and precision studies. The average of each concentration (LOW, MED, and HIGH) with repeats (n=3) over five different runs were used to determine percent recovery. The average concentration was divided by the expected concentration and multiplied by 100% in order

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to determine percent recovery.

4.2.12 Freeze-thaw stability

Freeze-thaw stability was completed over three freeze thaw cycles at two different concentrations (LOW and HIGH). At the start of the experiment, 5 mL of 5 ng/mL (LOW) and 80 ng/mL (HIGH) of the NPS mix being tested was prepared in matrix and aliquoted into four amber vials. The first vial was used for time zero and the other three vials were placed in the freezer (-20˚C) for 24 h, after which they were all removed and allowed to thaw to room temperature for 2 h. After this time, one vial was analyzed as first thaw and the other two vials were placed back in the freezer for 20 h, before being thawed to room temperature for 2 h. After being thawed, one vial was analyzed as the second thaw cycle and the other was placed back into the freezer for another 20 h and then analyzed once it returned to room temperature as the third thaw. Calibration curves were made fresh daily for quantification. Mean concentrations of first, second, and third thaw samples were compared to the mean concentrations of the analytes of interest at time zero. Compounds were considered stable as long as the mean concentration stayed within ±15% of the mean concentration calculated at time zero.

4.3 Results

The final dMRM method that underwent method validation included two transitions each for 750 compounds and 22 deuterated internal standards. Tables 5, 6, and 7 show the dMRM parameters used for Mixes 1, 2, and 3, respectively and the internal standard match for each compound. Information regarding the dMRM parameters for all other compounds included in the final method can be seen in the Appendix.

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Table 5. Dynamic MRM MS method parameters for NPS in Mix 1 and internal standard matches.

Drug	Precursor Ion	Transitions	CE (V)	Fragmentor (V)	t_R (min)	Internal Standard
4-OH MET	219.1	160 72	16 12	96	5.53	Butylone-d3
NMT	175.1	144 117	8 28	84	6.06	Benocyclidine-d10
4'-fluoro- α -PPP	222.1	123 98	24 28	120	6.31	Butylone-d3
4-APDB	178.1	161 133	8 20	84	6.56	Benocyclidine-d10
4'-fluoro- α -PPP	222.1	123 98	24 28	120	6.31	Butylone-d3
4-APDB	178.1	161 133	8 20	84	6.56	Benocyclidine-d10
4-fluoro PBP	236.1	112 109	24 28	120	6.87	Butylone-d3
5-MAPB	190.1	159 131	8 20	84	7.04	3,4-Methylenedioxy Pyrovalerone-d8
3-methyl BP	192.1	174 144	8 36	96	7.28	Benocyclidine-d10
4-methyl-α-EAB	206.2	188 144	8 32	108	7.48	Butylone-d3
α-PVP metabolite 1	234.2	216 72	16 20	108	7.62	JWH 081 N-pentanoic acid metabolite-d5
4-MeO-α-PVP	262.2	126 121	24 24	120	7.69	Butylone-d3
JWH 200 5-hydroxyindole metabolite	401.2	155 114	20 32	108	8.03	PB-22-d9
PCMPA	248.2	159 91	12 40	84	8.16	Benocyclidine-d10
AM2233 azepane isomer	459.2	58 112	60 24	108	8.55	RCS-4 N-(5-hydroxypentyl) metabolite-d5
4-MeO PV8	290.2	154	28	108	8.79	Butylone-d3

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		121	24			
Benocyclidine	300.2	147 86	32 4	72	8.98	JWH 073 5-hydroxyindole metabolite-d7
25I-NBMD	442.1	135 77	32 60	120	9.00	25I-NBOMe-d3
AB-005	353.3	112 98	24 36	120	9.39	Butylone-d3
Flubromazepam	333.0	226 184	32 32	120	9.77	JWH 018-d9
AM694 N-(5-hydroxypentyl) metabolite	434.1	230.9 202.9	20 56	120	9.99	(-)-11-nor-9-carboxy-Δ9-THC-d3
5-fluoro SDB-006	339.2	232.1 91.1	20 56	120	10.38	3,4-Methylenedioxy Pyrovalerone-d8
PB-22 6-hydroxyisoquinoline isomer	388.2	185.1 157.1	20 48	108	10.57	PB-22-d9
NPB-22	359.2	214.1 144	16 44	108	10.77	(±)-CP 47,497-C8-homolog-d7
THCA-A	359.2	341.2 219.1	12 36	108	13.45	Benocyclidine-d10

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Table 6. Dynamic MRM MS method parameters for NPS in Mix 2 and internal standard matches.

Drug	Precursor Ion	Transitions	CE (V)	Fragmentor (V)	t_R (min)	Internal Standard
Δ8-THC	315.2	193.2 123.1	24 36	108	12.33	(-)-11-nor-9-carboxy-Δ9-THC-d3
2,3-methylenedioxy pyrovalerone	276.2	175.0 126.1	20 32	120	7.41	3,4-Methylenedioxy Pyrovalerone-d8
25I-NBF	416.1	291.0 109.1	20 56	120	8.88	25I-NBOMe-d3
2C-T-2	242.1	225.1 91.1	8 52	72	7.88	25I-NBOMe-d3
2C-T-4	256.1	239.1 197.1	8 16	84	8.36	25I-NBOMe-d3
2-fluoromethcathinone	182.1	164.1 149.0	12 20	96	5.38	Butylone-d3
3,4-DHMA	182.1	123.0 77.1	20 44	84	4.19	Butylone-d3
3,4-dimethoxy-α-Pyrrolidinopentiophenone	292.2	151.1 126.0	28 24	120	7.15	Butylone-d3
3-methyl-α-Pyrrolidinopropiophenone	218.2	119.1 98.1	24 28	120	6.93	Butylone-d3
4'-Methyl-N-methylhexanophenone	220.2	202.2 105.1	8 24	96	8.39	Butylone-d3
4-ethyl-N,N-dimethylcathinone	206.1	105.1 72.1	28 28	120	7.43	Butylone-d3
4-fluoroisocathinone	168.1	123.0 77.1	16 40	72	5.39	Butylone-d3
4-hydroxy MiPT	233.2	160.0 86.1	20 12	96	5.86	Norsufentanil-d3
4-MMC	178.1	160.1 145.1	8 20	84	6.6	Butylone-d3
A-796260	355.2	125.1 114.1	20 32	120	10.2	UR-144 N-(4-hydroxypentyl) metabolite-d5
AB-005 azepane isomer	353.3	112.0	24	120	9.47	UR-144 N-(4-

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		58.1	56			hydroxypentyl) metabolite-d5
AB-FUBINACA 3-fluorobenzyl isomer	369.2	253.0 109.0	24 52	96	10.11	AB-FUBINACA-d4
ADB-PINACA isomer 1	345.2	215.1 145.0	24 48	96	10.72	ADB-PINACA-d9
AKB48 N-(5-fluoropentyl) analog	384.2	135.1 93.0	24 60	120	11.81	AB-FUBINACA-d4
Clencyclohexerol	319.1	203.0 81.1	20 32	96	6.05	UR-144 N-(4-hydroxypentyl) metabolite-d5
EG-018	392.2	155.1 127.1	24 60	120	12.78	JWH 018-d9
JWH 018 N-(5-hydroxypentyl) metabolite	358.2	155.0 127.0	20 52	120	10.37	JWH 081 N-pentanoic acid metabolite-d5
KM 233	363.2	119.1 91.1	20 60	120	12.09	(-)-11-nor-9-carboxy-Δ9-THC-d3
Loperamide	477.2	266.1 210.1	24 60	120	9.22	Acetyl norfentanyl-d5
MAM2201 N-pentanoic acid metabolite	386.2	169.1 141.1	24 48	120	10.55	MAM2201 N-pentanoic acid metabolite-d5
N-Ethylbuphedrone	192.1	130.1 91.1	32 32	96	6.51	Butylone-d3
PB-22	359.2	214.1 116.0	8 60	60	11.2	PB-22-d9
PCPr	218.2	91.1 60.2	28 4	60	8.19	Benocyclindine-d10
RCS-4 2-methoxy isomer	322.2	135.0 77.1	20 60	120	10.96	RCS-4 N-(5-hydroxypentyl) metabolite-d5
SER-601	435.3	284.2 135.1	28 32	120	13.28	JWH 007-d9
UR-144 Degradant	312.2	214.1 55.2	20 60	120	11.64	UR-144 N-(4-hydroxypentyl) metabolite-d5

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Table 7. Dynamic MRM MS method parameters for NPS in Mix 3 and internal standard matches

Drug	Precursor Ion	Transitions	CE (V)	Fragmentor (V)	t_R (min)	Internal Standard
(-)-3,4-Methylenedioxy Pyrovalerone	276.2	135.1 126.1	28 28	120	7.29	3,4-Methylenedioxy Pyrovalerone-d8
(+)-WIN 55,212-2	427.2	155.0 127.0	24 60	120	10.86	UR-144 N-(4-hydroxypentyl) metabolite-d5
(R)-(-)-JWH 018 N-(4-hydroxypentyl) metabolite	358.2	155.1 127.1	20 56	120	10.44	JWH 081 N-pentanoic acid metabolite-d5
2,3-pentylone isomer	236.1	188.1 131.1	12 40	108	7.24	butylone-d3
25E-NBOMe	330.2	121.1 91.1	50 52	120	9.25	25I-NBOMe-d3
3C-B-fly	298.1	281 202.1	12 24	84	7.97	25I-NBOMe-d3
4-hydroxy DiPT	261.2	160.1 114.1	20 12	96	6.48	
AB-FUBINACA	369.2	324.1 109.0	12 48	96	10.15	AB-FUBINACA-d4
AB-PINACA N-(5-hydroxypentyl) metabolite	347.2	302.1 213.0	12 28	96	9.19	AB-PINACA-d9
BB-22 8-hydroxyisoquinoline isomer	385.2	240.2 144.0	20 44	120	11.78	PB-22-d9
Benzydamine	310.2	86.1 58.1	16 56	108	8.65	AB-FUBINACA-d4
Bromazepam	316.0	209.1 182.1	28 36	108	9.00	Cis-tramadol-d6
Etaqualone	265.1	146.0 77.0	28 60	120	9.95	butylone-d3
JWH 019	356.2	228.1 144.0	24 40	120	11.82	JWH 018-d9
	328.2	154.9	28	120	11.4	

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JWH 073 2'-naphthyl-N-(1,1-dimethylethyl) isomer		144.2	24			JWH 073 5-hydroxyindole metabolite-d7
JWH 073 6-methoxyindole analog	358.2	230.1 174.1	24 40	120	11.29	JWH 073 5-hydroxyindole metabolite-d7
JWH 198	415.2	185.1 114.1	24 32	120	10.07	JWH 007-d9
MMAI	178.1	161.0 105.0	8 24	84	7.15	JWH 073 5-hydroxyindole metabolite-d7
N,N-dimethylcathinone	178.1	105.1 72.1	20 24	108	5.48	butylone-d3
p-Fluorofentanyl	355.2	188.2 105.1	24 48	120	8.01	acetyl norfentanyl-d5
PCEEA	248.2	159.1 65.1	8 60	84	8.26	Benocyclidine-d10
Phenylpiperazine	163.1	120.0 77.1	20 44	108	5.77	acetyl norfentanyl-d5
Tetrahydroharmine	217.1	200.0 188.0	8 8	84	6.68	Norsufentanil-d3

4.3.1 Selectivity and carryover

A set of ten diluted purchased pooled certified blank urine samples were analyzed using the developed dMRM method to ensure that there were no interfering peaks. Initially, there was a peak identified as PB-22 6-hydroxyisoquinoline isomer that showed up consistently in every sample. In order to address this issue, the transitions for this analyte were changed and the interfering peak was no longer present when blank urine was re-analyzed. When analyzing IS and NPS drug mixtures spiked in blank urine using dMRM no interfering peaks were observed, confirming the value of using a dMRM method to eliminate interfering peaks that could be present in full scan MS modes. Interferences can be detrimental to a screening and confirmatory

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test and can contribute to false positive results (*i.e.,* the detection of a compound that is not actually in the sample). It is extremely important to avoid false positive results in clinical and forensic toxicology.

Carry over can be the result of compounds not fully eluting from the analytical column and can affect quantitative analysis. In order to address any carry over, five blank matrix samples were injected after analysis of the highest calibrator (100 ng/mL) to determine if there was any carry over. When analyzing the five blank urine samples there was no carryover seen, meaning that the 3-min clean up after the 16 min run was sufficient to eliminate carry over from higher concentrated samples in the next sample or blank. Carry over can also contribute to false positive results, which should be avoided.

4.3.2 Linearity of calibration and limit of detection/quantitation

Agilent MassHunter Quantitative Analysis software was used to find regression lines for each of the analytes. Additionally, the software was used to aid in the determination of precision, accuracy, LOD, and LOQ for all analytes included in this experiment. All regression models were weighted by a factor of 1/x to offset heteroscedasticity. All R^2 values were a minimum of 0.95 for the analytes analyzed. However, the majority of compounds had an R^2 value >0.99. Linear range was 1 to 100 ng/mL for most NPS analyzed. There were a few compounds that did not show linearity, including THCA-A and tetrahydroharmine. Only compounds showing linearity were further analyzed for method validation. At concentrations higher than 100 ng/mL, linearity was lost for the majority of the compounds. Nevertheless, the range used is adequate for the concentration of NPS found in typical case samples, including fatalities.[108] If a sample

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is suspected to be above the 100 ng/mL linearity cutoff, it can be diluted before analysis, which will alleviate the issue.

Limits of detection (LOD) and limits of quantitation (LOQ) were determined using the equation of the regression line for each compound, which was possible because of the linearity of the calibration curves. Limit of detections for all compounds in Mixes 1, 2, and 3 ranged from 0.01 to 0.12 ng/mL and LOQs for all the compounds analyzed ranged from 0.02 to 0.36 ng/mL. Limit of detections and LOQs for all compounds in Mixes 1, 2, and 3 can be found in Tables 8-10, respectively. These LODs and LOQs are similar to those that have been reported previously in literature for selected NPS.[42,80,84,85] It is important to note that LOD and LOQ were determined analyzing diluted samples, therefore the detection and quantification limits represent what is possible in a diluted sample. The ability to detect and quantify NPS in the ppt range will greatly aid in the identification and quantification of some of the more potent NPS, which are often found in low concentrations in case studies.

4.3.3 Precision and accuracy

The QC samples were analyzed at 5, 20, and 80 ng/mL in triplicate on five different days. Accuracy, precision, and percent recovery ware calculated for each analyte at the three different concentrations. Acceptable values were ±20% for bias and 20% for precision (% CV). These values for the compounds included in Mixes 1, 2, and 3 can be found in Tables 8, 9, and 10, respectively. Bias, precision, LOD, and LOQ values for additional mixes can be found in the Appendix. All compounds in Mixes 1, 2, and 3 fell within the acceptable limits for both bias and precision.

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Table 8. LOD, LOQ, R2 values, and precision and bias values for all compounds in Mix 1 at three different concentration levels.

				Low (5 ppb)		Medium (20 ppb)		High (80 ppb)	
Compound Name	**R^2**	**LOD (ng/mL)**	**LOQ (ng/mL)**	**% CV**	**% Bias**	**% CV**	**% Bias**	**% CV**	**% Bias**
4-OH MET	0.9985	0.039	0.117	5.5	-6.3	2.6	-5.1	3.0	-1.5
NMT	0.9982	0.040	0.121	2.8	4.2	2.0	7.2	3.1	-0.8
4-fluoro- α -PPP	0.9953	0.027	0.083	7.2	-4.7	3.9	-8.2	2.3	-1.8
4'-fluoro- α -PPP	0.9987	0.058	0.176	9.2	1.3	5.6	8.4	2.3	0.1
4-APDB	0.9959	0.023	0.071	6.4	-3.4	4.3	-8.2	2.8	-1.6
4-fluoro PBP	0.9936	0.089	0.270	3.1	0.3	2.5	4.4	2.6	-0.2
5-MAPB	0.9843	0.025	0.077	6.3	-1.9	5.0	-3.7	2.3	-2.0
3-methyl BP	0.9994	0.028	0.085	8.1	-3.0	4.2	-8.1	2.7	-1.8
4-methyl-α-EAB	0.9960	0.020	0.060	7.7	-15.7	4.2	-17.8	3.8	-12.1
α-PVP metabolite 1	0.9943	0.056	0.171	8.5	-4.1	6.5	-8.1	2.6	-1.8
4-MeO-α-PVP	0.9983	0.024	0.073	2.7	-3.4	4.0	-2.1	3.3	-0.8
JWH 200 5-hydroxyindole metabolite	0.9941	0.028	0.085	8.7	-3.6	2.0	10.7	1.8	-1.9
PCMPA	0.9986	0.023	0.069	3.5	-3.3	5.2	3.6	2.5	-2.8
AM2233 azepane isomer	0.9992	0.069	0.209	6.3	-7.3	4.7	-6.8	2.8	-1.3
4-MeO PV8	0.9960	0.015	0.046	3.5	-6.5	1.8	-1.0	2.3	-0.5
Benocyclidine	0.9956	0.009	0.028	6.1	-8.0	1.9	1.1	1.6	-0.7
25I-NBMD	0.9991	0.076	0.230	6.5	-3.8	8.6	-15.8	2.0	-5.1
AB-005	0.9995	0.056	0.169	13.0	-0.4	3.4	12.4	4.3	-2.2
Flubromazepam	0.9601	0.017	0.052	11.1	-8.3	2.7	-11.0	3.4	-2.0
AM694 N-(5-hydroxypentyl) metabolite	0.9928	0.036	0.109	6.2	-7.0	4.5	4.6	2.6	-1.1
5-fluoro SDB-006	0.9934	0.015	0.047	4.2	-6.6	2.4	1.9	2.9	-0.4
PB-22 6-hydroxyisoquinoline isomer	0.9976	0.051	0.154	4.9	-12.2	5.5	-13.5	1.7	-2.1
NPB-22	0.9941	0.086	0.260	5.1	-2.3	3.6	-0.5	4.0	2.2

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Table 9. LOD, LOQ. R2 values and precision and bias values for all compounds in Mix 2 at three different concentration levels.

				Low (5 ppb)		Medium (20 ppb)		High (80 ppb)	
Compound Name	**R^2**	**LOD (ng/mL)**	**LOQ (ng/mL)**	**% CV**	**% Bias**	**% CV**	**% Bias**	**% CV**	**% Bias**
3,4-DHMA	0.9821	0.005	0.014	2.5	4.1	2.5	-10.7	0.9	-5.6
2-fluoromethcathinone	0.9854	0.006	0.017	19.3	15.4	4.1	-8.5	0.2	-23.5
4-fluoroisocathinone	0.9515	0.018	0.052	2.8	-1.6	1.4	-6.5	2.2	-9.2
4-hydroxy MiPT	0.9798	0.015	0.045	1.3	-12.9	6.3	-2.6	1.5	-3.1
Clencyclohexerol	0.9989	0.025	0.074	1.9	-22.0	2.4	0.7	1.4	-3.9
N-Ethylbuphedrone	0.9987	0.006	0.017	1.7	0.4	1.6	-0.8	1.6	-0.7
4-MMC	0.9975	0.017	0.050	2.5	-1.5	1.4	2.4	1.8	-5.6
3-methyl-a-Pyrrolidinopropiophenone	0.9973	0.003	0.008	2.6	8.6	1.5	-2.1	2.5	-0.6
3,4-dimethoxy-a-pyrrolidinopentiophenone	0.9968	0.003	0.010	1.5	9.3	1.8	-2.8	1.6	-1.1
2,3-methylenedioxy pyrovalerone	0.9986	0.013	0.040	5.0	-1.4	5.7	-5.2	2.2	-6.1
4-ethyl-N,N-dimethylcathinone	0.9978	0.002	0.006	2.0	9.9	1.5	-0.5	2.2	-2.3
2C-T-2	0.9879	0.019	0.057	2.0	-11.4	1.7	6.3	1.9	-3.8
PCPr	0.9991	0.015	0.046	1.2	-7.1	1.1	2.9	2.6	-0.2
2C-T-4	0.9833	0.022	0.068	3.3	-13.5	1.1	11.6	1.9	-5.9
4'-methyl-N-methylhexanophenone	0.9987	0.005	0.017	2.6	3.8	1.6	0.3	1.8	-3.1
25I-NBF	0.9975	0.001	0.003	2.2	-9.5	1.4	-0.7	2.0	-4.7
Loperamide	0.9961	0.008	0.026	3.2	3.3	0.4	-7.0	1.9	-4.8
AB-005 azepane isomer	0.9980	0.007	0.020	1.5	-15.0	2.1	-4.8	1.2	-7.7
A-796260	0.9974	0.032	0.097	1.5	-7.4	1.7	4.9	1.7	-1.3
AB-FUBINACA 3-fluorobenzyl isomer	0.9985	0.008	0.024	1.0	-9.9	1.3	-5.2	2.2	-4.9
JWH 018 N-(5-hydroxypentyl) metabolite	0.9984	0.011	0.032	2.0	-6.3	2.1	2.0	1.6	-1.0
MAM2201 N-pentanoic acid metabolite	0.9987	0.012	0.035	1.1	-6.9	1.7	4.6	2.3	1.3
ADB-PINACA isomer 1	0.9992	0.009	0.028	0.7	-9.7	4.3	6.4	2.2	0.7
RCS-4 2-methoxy isomer	0.9959	0.011	0.033	2.1	-6.2	1.8	15.8	2.3	-5.5

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PB-22	0.9978	0.004	0.012	1.1	-20.6	2.0	5.2	1.5	0.1
XLR11 N-(2-fluoropentyl) isomer	0.9933	0.002	0.007	7.6	-31.3	1.7	5.0	1.8	-1.0
UR-144 Degradent	0.9944	0.003	0.008	0.6	-30.8	1.8	2.1	1.7	-2.8
AKB48 N-(5-fluoropentyl) analog	0.9960	0.021	0.064	4.0	-22.6	1.2	-4.5	3.3	1.1
KM 233	0.9903	0.017	0.052	2.1	-35.4	0.8	2.4	4.5	1.6
Δ 8- THC	0.9806	0.023	0.069	7.9	-37.6	2.2	-8.4	3.5	2.7
EG-018	0.9598	0.023	0.070	1.1	-37.5	1.4	-12.0	2.4	3.6
SER-601	0.9923	0.011	0.033	1.7	-38.5	0.6	3.6	2.1	2.0

Table 10. LOD, LOQ, R2 values, and precision and bias values for all compounds in Mix 3 at three different concentration levels.

				Low (5 ppb)		Medium (20 ppb)		High (80 ppb)	
Compound Name	**R^2**	**LOD (ng/mL)**	**LOQ (ng/mL)**	**% CV**	**% Bias**	**% CV**	**% Bias**	**% CV**	**% Bias**
N,N-dimethylcathinone	0.9858	0.02	0.06	6.8	-2.8	2.8	-1.7	3.1	4.2
Phenylpiperazine	0.9825	0.02	0.07	6.7	9.3	4.5	4.0	4.5	1.9
tetrahydro_harmine	0.9992	0.02	0.07	13.4	9.2	8.0	0.7	8.7	-2.7
MMAI	0.9987	0.02	0.07	7.9	9.0	7.9	6.6	6.4	5.5
2,3-pentylone isomer	0.9878	0.05	0.15	8.3	-0.3	3.4	-2.2	2.9	3.0
(-)-3,4-Methylenedioxy Pyrovalerone	0.9875	0.04	0.27	5.5	3.0	4.7	4.9	4.9	2.3
3C-B-fly	0.9506	0.06	0.18	7.5	3.6	4.3	5.7	8.5	10.9
para-Fluorofentanyl	0.9832	0.05	0.11	10.5	9.3	3.7	-1.2	3.6	1.0
PCEEA	0.9984	0.07	0.2	3.8	8.5	2.6	2.7	2.7	4.0
Benzydamine	0.9963	0.03	0.09	6.6	12.1	3.7	5.1	3.4	-2.1
Bromazepam	0.9463	0.04	0.13	8.8	5.7	4.3	-1.1	3.9	-0.5
AB-PINACA N-(5-hydroxypentyl) metabolite	0.9880	0.03	0.09	11.0	-0.2	3.7	2.8	2.1	2.8
25E-NBOMe	0.9840	0.01	0.03	9.8	8.6	4.0	3.0	7.3	8.3
Etaqualone	0.9915	0.04	0.12	10.6	-0.3	4.0	-3.5	3.3	6.4
JWH 198	0.9934	0.07	0.21	8.5	-13.9	37.7	-1.8	32.9	2.0
AB-FUBINACA	0.9988	0.02	0.05	4.9	8.7	3.0	2.6	3.0	3.3

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(+)-WIN 55,212-2	0.9990	0.02	0.05	4.4	14.7	3.2	-1.0	4.8	3.0
JWH 073 6-methoxyindole analog	0.9956	0.03	0.09	5.0	27.8	5.5	1.9	5.5	-0.7
JWH 073 2'-naphthyl-N-(1,1-dimethylethyl) isomer	0.9968	0.12	0.36	6.4	16.9	7.4	-6.3	6.1	2.5
BB-22 8-hydroxyisoquinoline isomer	0.9979	0.04	0.13	5.2	25.9	5.1	-6.2	5.3	-4.0

4.3.4 Freeze-thaw stability

Quality control samples at concentrations of 5 and 80 ng/mL were analyzed over three different freeze-thaw cycles to determine stability. Each day a fresh set of calibrators were analyzed with the sets of samples for quantification. Samples after each thaw were compared to the concentration of time zero samples. Concentrations within ±15% of the time zero value were considered to be acceptable. The percent change in concentration for all compounds in Mixes 1, 2, and 3 at two different concentrations, from time zero until the 3rd thaw, can be seen in Tables 11, 12, and 13, respectively. All compounds were within the acceptable range after three freeze-thaw cycles for both concentrations, except for AB-005, etaqualone, benzydamine, 3,4-DHMA, 4-fluoroisocathinone, 4-hydroxy MiPT, 4-MMC, and a few others. The freeze-thaw stability study shows that it will be acceptable to store case work samples at -20˚C for up to three freeze-thaw cycles for the majority of compounds. No information on longer term storage is provided by this work; further testing would be required to assess extended storage.

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Table 11. Freeze-thaw stability for all analytes in Mix 1.

Compound Name	LOW (%Δ_C)	HIGH (%Δ_C)
4-hydroxy-MET	-12.7	-9.7
N-Methyltryptamine	-0.7	-4.2
4'-fluoro-α-pyrrolidinoprophenone	1.7	-2.5
4-APBD	5.2	4.5
4-fluoro-α-pyrrolidinobutiophenone	0.8	-2.4
5-MAPB	-0.1	-0.6
3-Methylbuphedrone	-0.9	-3.6
4-methyl-α-ethylaminobutiophenone	-5.0	-0.9
α-Pyrrolidinopentiophenone	0.01	-0.7
4-methoxy-α-pyrrolidinopentiophenone	1.6	-0.9
PCMPA	-2.8	-1.9
AM2233 azepane isomer	-5.3	-0.5
JWH 200 5-hydroxyindole metabolite	0.7	-0.6
4-methoxy PV8	2.6	-0.4
Benocyclidine	-3.5	0.2
25I-NBMD	5.8	0.2
AB-005	-52.5	15.3
Flubromazepam	14.6	8.2
AM694 N-(5-hydroxypentyl) metabolite	-1.4	2.4
5-fluoro SDB-006	-0.3	-1.1
JWH 081 N-(5-hydroxypentyl) metabolite	-2.6	0.3
JWH 203	-3.5	0.9

Table 12. Freeze-thaw stability for all analytes in Mix 2 (LC-QqQ-MS)

Compound Name	LOW (%Δ_C)	HIGH (%Δ_C)
3,4-DHMA	-62.0	-64.6
2-fluoromethcathinone	-25.1	-20.4
4-fluoroisocathinone	-45.9	-8.2
4-hydroxy MiPT	-34.1	-33.3
Clencyclohexerol	0.7	33.9
N-Ethylbuphedrone	1.4	-8.7
4-MMC	-2.4	-63.0
3-methyl-a-Pyrrolidinopropiophenone	5.0	-3.0
3,4-dimethoxy-a-pyrrolidinopentiophenone	2.6	1.5
2,3-methylenedioxy pyrovalerone	5.8	-51.3
4-ethyl-N,N-dimethylcathinone	1.8	5.4
2C-T-2	11.3	65.8
PCPr	4.7	-32.9
2C-T-4	7.9	66.9
4'-methyl-N-methylhexanophenone	-2.7	-12.4

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25I-NBF	5.7	-9.6
Loperamide	-2.7	-59.2
AB-005 azepane isomer	-2.6	-14.2
A-796260	1.1	-5.8
AB-FUBINACA 3-fluorobenzyl isomer	-2.6	-57.3
JWH 018 N-(5-hydroxypentyl) metabolite	-0.5	38.7
MAM2201 N-pentanoic acid metabolite	-5.3	-18.6
ADB-PINACA isomer 1	-17.3	46.6
RCS-4 2-methoxy isomer	6.1	2.7
PB-22	5.4	-26.2
XLR11 N-(2-fluoropentyl) isomer	-3.4	42.9
UR-144 Degradent	1.9	38.8
AKB48 N-(5-fluoropentyl) analog	3.0	28.9
KM 233	-4.9	8.3
Δ 8- THC	-4.5	-22.9
EG-018	-33.3	-51.2
SER-601	-14.1	1.9

Table 13. Freeze-thaw stability for all analytes in Mix 3 (LC-QqQ-MS)

Compound Name	LOW (%Δ_C)	HIGH (%Δ_C)
N,N-dimethylcathinone	-11.8	9.2
Phenylpiperazine	7.2	14.3
MMAI	19.5	21.1
2,3-pentylone isomer	14.0	16.7
(-)-3,4-Methylenedioxy Pyrovalerone	-4.1	5.0
para-Fluorofentanyl	5.6	-9.3
3C-B-fly	0.2	-1.5
PCEEA	-2.5	10.4
Benzydamine	-28.7	-18.4
Bromazeam	-5.4	-14.9
AB-PINACA N-(5-hydroxypentyl) metabolite	-2.4	-13.9
25E-NBOMe	-10.6	-20.4
JWH 198	7.5	27.0
Etaqualone	1.2	-59.0
AB-FUBINACA	4.9	2.1
(+)-WIN 55,212-2	2.0	1.7
JWH 073 6-methoxyindole analog	6.3	-3.7
JWH 073 2'-naphthyl-N-(1,1-dimethylethyl) isomer	6.7	-8.6
BB-22 8-hydroxyisoquinoline isomer	-6.7	2.7
JWH 019	-1.8	8.8

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4.3.5 Matrix effects and recovery

The ME and percent recovery were determined for each analyte using the procedure previously described for LOW, MED, and HIGH concentrations. A summary of matrix effects and percent recoveries for the compounds in Mixes 1, 2, and 3 can be found in Tables 14, 15, and 16 respectively. When following OSAC guidelines, the ME for each analyte is determined using the highest value noted for the concentrations tested. Acceptable values of ME for method validation must fall within ±25% of the peak area of the analyte in no matrix (*i.e.,* in water). The majority of compounds at MED and HIGH concentrations fell well within these parameters with a few outliers, including 4-APBD, 4-methoxy-α-pyrrolidinopentiophenone, and AB-005. However, at the low concentration of 5 ng/mL, many of the compounds did not fall within the OSAC parameters, a finding that was not completely unexpected when working with such low concentrations. Regardless, the matrix effects experienced with these compounds did not negatively affect detection, as recovery for most compounds was above 85% at all concentration levels. A few compounds, including N-methyltryptamine and 4-APBD, showed recoveries above 100%, which likely reflects ion enhancement.

Figure 3 show how synthetic cannabinoids (n=40) vary in recovery based on their concentration, three concentration levels were analyzed low (5 ng/mL) medium (20 ng/mL) and high (80 ng/mL). As the concentration increases the percent recovery also increases. There is a statistically significant difference between medium and low and high and low concentrations. As can be seen in Figure 3, the recoveries for the lowest level vary more than the other two levels and are significantly lower. This could be due to ion suppression being higher at the lower limits of the calibration curve for the validated

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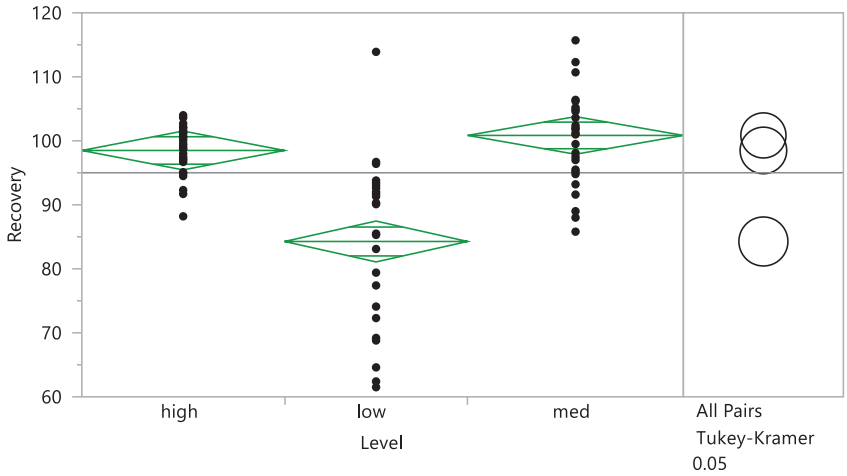
method. These results relate to Figure 4, which shows a one-way ANOVA comparing the matrix effects of the same three levels for synthetic cannabinoids (n=40). It can be seen in Figure 4 that the low-level samples have more ion suppression than the higher level samples. Both ANOVAs were created only using a small subset of the total number of synthetic cannabinoids included in the final dMRM method. There is no clear pattern in terms of recovery or matrix effects for synthetic cannabinoids, which can be attributed to the there being multiple subclasses of synthetic cannabinoids.



Level	- Level	Difference	Std Err Dif	Lower CL	Upper CL	p-Value
med	low	16.57100	2.184544	11.3640	21.77800	<.0001*
high	low	14.22212	2.216655	8.9386	19.50567	<.0001*
med	high	2.34888	2.126592	-2.7200	7.41775	0.5138

Figure 3. The top panel visually represents the results of a Means/ANOVA test used to determine whether results of the recovery of synthetic cannabinoids are statistically different at three different concentration levels (5, 20, and 80 ng/mL). The bottom panel shows the results of Tukey HSD test showing that there is a statistically significant difference in terms of recovery between the medium and low levels and the high and low levels.

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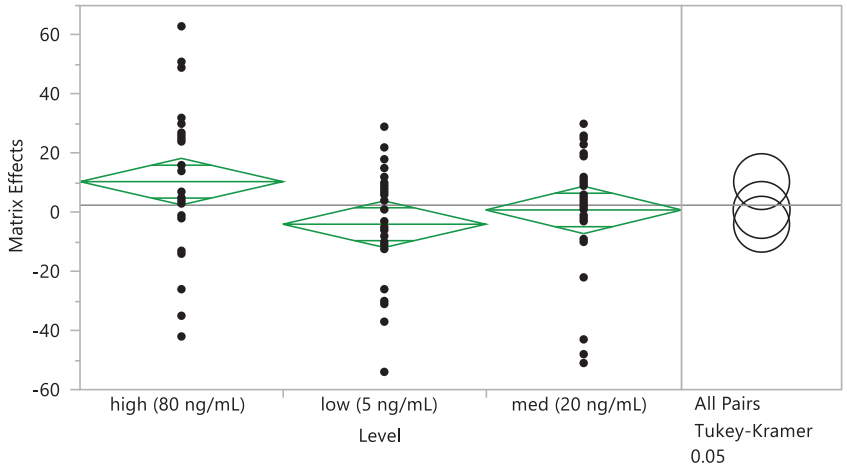

Level	- Level	Difference	Std Err Dif	Lower CL	Upper CL	p-Value
high (80 ng/mL)	low (5 ng/mL)	14.42069	5.601007	1.05394	27.78744	0.0314*
high (80 ng/mL)	med (20 ng/mL)	9.59236	5.650795	-3.89320	23.07793	0.2122
med (20 ng/mL)	low (5 ng/mL)	4.82833	5.650795	-8.65724	18.31389	0.6703

Figure 4. The top panel visually represents the results of a Means/ANOVA test used to determine whether or not matrix effects of synthetic cannabinoids are statistically different at three different concentration levels (5, 20, and 80 ng/mL). The bottom panel shows the results of Tukey HSD test showing that there is a statistically significant difference in terms of matrix effects between the high and low levels.

Table 14. Percent matrix effects and percent recovery for all compounds in Mix 1.

	Low (5 ppb)		Medium (20 ppb)		High (80 ppb)	
Compound Name	**ME (%)**	**%Recovery**	**ME (%)**	**%Recovery**	**ME (%)**	**%Recovery**
4-hydroxy-MET	1.4	93.7	33.9	94.9	13.3	98.5
N-Methyltryptamine	-10.5	104.2	31.7	107.2	23.9	99.2
4'-fluoro-α-pyrrolidinoprophenone	-26.6	95.3	8.9	91.8	7.7	98.2
4-APBD	47.3	101.3	63.3	108.4	55.9	100.1
4-fluoro-α-pyrrolidinobutiophenone	-33.4	96.6	13.6	91.8	4.8	98.4
5-MAPB	-57.7	100.3	5.2	104.4	-7.2	99.8
3-Methylbuphedrone	-60.7	98.1	6.4	96.3	-18.1	98.0
4-methyl-α-ethylaminobutiophenone	-65.8	97.0	-0.8	91.9	-14.2	98.2
α-Pyrrolidinopentiophenone	-50.9	95.6	8.3	93.1	-10.5	99.6
4-methoxy-α-pyrrolidinopentiophenone	-55.2	95.9	0.2	91.9	-15.9	98.2
PCMPA	23.2	96.6	37.0	97.9	27.2	99.2
AM2233 azepane isomer	-54.0	96.4	-1.3	110.7	-13.1	98.1
JWH 200 5-hydroxyindole metabolite	-37.3	96.7	5.4	103.6	3.8	97.2
4-methoxy PV8	-40.6	92.7	7.8	93.2	-5.0	98.7
Benocyclidine	-35.1	93.5	-4.8	99.0	-8.1	99.5
25I-NBMD	-31.6	92.0	-3.5	101.1	-2.2	99.3
AB-005	-122.7	96.2	-48.3	112.3	4.9	94.9
Flubromazepam	-7.6	99.6	15.4	112.4	7.0	97.8
AM694N-(5-hydroxypentyl) metabolite	-10.2	91.7	12.2	89.0	5.2	98.0
5-fluoro SDB-006	-11.1	93.0	5.8	104.6	-2.2	98.9
JWH081N-(5-hydroxypentyl) metabolite	-12.4	93.4	10.3	101.9	2.9	99.6
JWH 203	-30.9	N/A	-43.0	99.5	-0.4	102.2

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Table 15. Percent matrix effects and percent recovery for all compounds in Mix 2.

	Low (5 ppb)		Medium (20 ppb)		High (80 ppb)	
Compound Name	**ME (%)**	**%Recovery**	**ME (%)**	**%Recovery**	**ME (%)**	**%Recovery**
3,4-DHMA	-16.9	104.1	-122.2	89.3	-266.0	94.4
2-fluoromethcathinone	60.7	115.4	-25.3	91.5	-395.7	76.5
4-fluoroisocathinone	60.6	98.4	63.6	93.4	66.0	90.8
4-hydroxy MiPT	18.8	87.1	11.0	97.4	17.2	96.9
Clencyclohexerol	30.2	78.0	23.6	100.7	22.3	96.1
N-Ethylbuphedrone	36.2	100.4	29.4	99.2	-36.4	99.3
4-MMC	26.4	98.5	17.9	102.4	-11.6	94.4
3-methyl-a-Pyrrolidinopropiophenone	5.5	108.6	7.6	97.9	-11.9	99.4
3,4-dimethoxy-a-pyrrolidinopentiophenone	6.3	109.3	5.2	97.2	-14.0	98.9
2,3-methylenedioxy pyrovalerone	7.2	98.6	6.4	94.8	-12.9	93.9
4-ethyl-N,N-dimethylcathinone	14.0	109.9	6.8	99.5	-14.7	97.7
2C-T-2	19.8	88.6	10.6	106.3	-2.7	96.2
PCPr	16.5	92.9	1.9	102.9	-13.5	99.8
2C-T-4	32.1	86.5	26.3	111.6	17.7	94.1
4'-methyl-N-methylhexanophenone	24.4	103.8	14.0	100.3	-2.3	96.9
25I-NBF	11.7	90.5	-9.7	99.3	-18.9	95.3
Loperamide	1.4	103.3	-32.2	93.0	-58.6	95.3
AB-005 azepane isomer	4.0	85.0	-34.3	95.2	-67.4	92.3
A-796260	5.5	92.6	-5.3	104.9	0.3	98.7
AB-FUBINACA 3-fluorobenzyl isomer	-26.1	90.1	-50.7	94.8	-42.4	95.1
JWH 018 N-(5-hydroxypentyl) metabolite	7.7	93.8	0.5	102.0	7.1	99.0
MAM2201 N-pentanoic acid metabolite	14.7	93.1	8.6	104.6	16.3	101.3
ADB-PINACA isomer 1	8.8	90.3	2.4	106.4	5.4	100.7
RCS-4 2-methoxy isomer	6.6	93.8	-2.5	115.7	3.0	94.5
PB-22	7.0	79.4	-10.2	105.2	3.8	100.1
XLR11 N-(2-fluoropentyl) isomer	6.5	68.8	2.6	105.0	14.0	99.0

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UR-144 Degradent	3.6	69.2	-1.3	102.1	14.0	97.2
AKB48 N-(5-fluoropentyl) analog	6.4	77.4	-2.0	95.5	2.9	101.1
KM 233	21.7	64.6	25.3	102.4	49.3	101.6
delta 8- THC	28.5	62.4	29.5	91.6	63.3	102.7
EG-018	17.7	62.5	25.8	88.0	51.5	103.6
SER-601	10.3	61.5	-20.1	103.6	24.9	102.0

Table 16. Percent matrix effects and percent recovery for all compounds in Mix 3.

	Low (5 ppb)		Medium (20 ppb)		High (80 ppb)	
Compound Name	**ME (%)**	**%Recovery**	**ME (%)**	**%Recovery**	**ME (%)**	**%Recovery**
N,N-dimethylcathinone	-0.9	102.8	3.8	101.7	18.1	95.8
Phenylpiperazine	28.7	90.7	19.6	96.0	37.2	98.1
MMAI	18.2	90.8	25.4	99.3	40.2	102.8
2,3-pentylone isomer	19.5	91.0	20.7	93.4	44.8	94.5
(-)-3,4-Methylenedioxy Pyrovalerone	-4.8	100.3	-7.5	102.2	15.9	97.0
p-Fluorofentanyl	-6.9	97.0	-8.8	95.1	9.6	97.7
3C-B-fly	14.3	96.4	15.9	94.3	38.1	89.1
PCEEA	4.7	90.7	7.8	101.2	20.1	99.0
Benzydamine	2.3	91.5	10.3	97.3	24.6	96.0
Bromazepam	-10.7	87.9	-13.1	94.9	-407.0	102.1
AB-PINACA N-(5-hydroxypentyl) metabolite	10.2	94.3	23.4	101.2	25.7	100.5
25E-NBOMe	-23.5	100.2	-11.5	97.3	3.4	97.2
JWH 198	-7.7	91.4	-22.9	97.0	-26.7	91.7
Etaqualone	-7.0	100.3	14.5	103.5	27.4	93.6
AB-FUBINACA	-29.8	113.9	-9.3	101.8	-14.4	98.0
-(-)-JWH 018 N-(4-hydroxypentyl)	-3.4	91.3	11.3	97.5	24.8	96.7

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metabolite						
(+)-WIN 55,212-2	11.7		-182.0	85.8	-35.3	88.2
JWH 073 6-methoxyindole analog	1.4	85.3	18.9	101.0	26.4	97.0
JWH 073 2'-naphthyl-N-(1,1-dimethylethyl) isomer	-5.4	72.3	4.4	98.1	27.0	100.7
BB-22 8-hydroxyisoquinoline isomer	-6.0	83.1	9.5	106.3	29.9	97.5
JWH 019	-3.3	74.1	-2.7	106.2	32.5	104.0

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4.4 Discussion

Recent research has investigated the potential of LC-QqQ-MS for the comprehensive screening, confirmation, and quantitation of NPS in human sample matrices. For example, in 2010 Wohlfarth and co-workers published an LC-MS/MS method capable of detecting over thirty NPS. Their work was one of the first attempts to create a comprehensive screening method for NPS.[78] Since then, other research groups have continued the work in the hopes of creating comprehensive screening and confirmatory methods for these substances. However, many of those methods are focused on a single structural class of NPS (*e.g.*, synthetic cannabinoids or cathinones) and can only quantitate a relatively small number of compounds.[79,81,82,110] A recent review article on different screening methods for detecting NPS in biological matrices indicated that there were only a few methods reportedly capable of detecting >100 NPS in a single run, while the majority of methods could detect fewer than 50 compounds.[114] In addition, some of the methods capable of detecting over 100 individual NPS were not fully validated.[80,84]

Strickland et al. published a method that aimed to be all-inclusive for the detection of designer drugs. Strickland's research is one of the few examples of a method that includes multiple classes of NPS. Their method took 4.5 minutes and targeted 24 compounds.[113] The importance of their method was in assuring that the compounds included were recently found in forensic case samples and are currently being abused. Even with that consideration, it is difficult to cover such a wide array of abused compounds with such a rapid method. Al-Saffar and co-workers also published a method that aimed to be able to detect different classes of NPS in a single run. Their method included 26 compounds, but it was validated for qualitative and quantitative analysis.[115] Even though

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their method is a move in the right direction, it excluded a large portion of abused NPS. These data confirm the need for comprehensive screening methods for NPS in biological matrices that can reliably detect the hundreds of individual chemical entities that can be considered as NPS.

One major challenge in developing comprehensive screening/confirmatory methods for hundreds of NPS involves the approach used to validate such a method. Specifically, validation using a classical "one component at a time" approach is prohibitive in terms of the time required for complete method development. In contrast, validation using non-coeluting analyte mixtures holds promise for substantially reducing the time it takes to fully validate a new method designed to encompass a large number of analytes. Validating screening/confirmatory methods using a mixture approach, while not a new concept, has only been reported on a limited basis for quantitation of NPS.[80-82] For example, Ammann and co-workers used a mixture approach to validate two separate quantitative methods, one for synthetic cathinones and another for synthetic cannabinoid each capable of confirming 25 compounds.[79,110] Two different runs, each optimized for structural class, were required for complete analysis. In contrast, there are currently no available reports using NPS mixtures in order to validate a single comprehensive method capable of detecting multiple NPS drug classes in one run. In the present study, a LC-QqQ-MS dMRM-based assay was fully validated according to OSAC guidelines for 80 NPS using three non-coeluting mixtures of NPS standards as a proof-of-principle of the mixture approach.

Further work is underway to extend validation to additional NPS mixes to ultimately include more than 800 individual compounds. Work is also being done to test different

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extraction methods for NPS from whole blood using the method discussed in the present work, showing its potential to be used to detect NPS in whole blood in addition to urine. The final validated assay could potentially have significant impact on forensic toxicology laboratories, giving them the ability to screen for a higher number of NPS than many are currently capable of. The ability to detect an increased number of NPS will decrease the number of false negatives, allowing for the proper detection of NPS. With time, more and more NPS are being introduced into the illicit market. New compounds can be added to the present assay as soon as commercial standards are produced, allowing for constantly increasing number of analytes that can be screened for.

Although the mixture approach described here has many benefits for the development of comprehensive NPS screening methods, there are also some challenges. For example, the method must be optimized for a large number of analytes in each mixture as a whole. Consequently, some individual compounds in the mixture may not be analyzed under their optimal conditions. In addition, while there is the temptation of adding new analytes to a mixture as standards become available in order to limit analysis time, this must be balanced against the loss of selectivity that can accompany the use of larger mixtures. Nevertheless, the use of analyte mixtures as described in the present work appears to be a promising approach to validate analytical methods for screening large numbers of NPS.

4.5 Conclusions

It is important to have a comprehensive screening technique for NPS in clinical and forensic toxicology laboratories in order to address the large number of NPS currently available and continuously appearing on the illicit drug market. In the present

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study, a comprehensive LC-QqQ-MS method capable of screening and confirmation was developed for the detection of 729 NPS, which is by far the largest number of NPS to be included in a comprehensive analytical method to date. This was accomplished using a mixture approach in order to reduce time required for method validation. The present work demonstrates that it is possible to use a series of standard mixtures for the validation of a method containing a large number of NPS.

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5. COMPARISON OF MULTIPLE EXTRACTION/PURIFICATION METHODS

5.1 Introduction

Novel psychoactive substances are compounds that are manufactured to emulate classically known and used illicit drugs. Novel psychoactive substances are often classified by one of the following drug classes; phenethylamines, amphetamines, synthetic cathinone, synthetic cannabinoids, piperazines, pipradrols/piperidines, benzofurans, and tryptamines. Clandestine laboratories circumvent federal rules and regulations by altering the structure of the parent compounds, creating new synthetic compounds, and ultimately rendering them just outside of federal jurisdiction. Once these "legal highs," "designer drugs," or "bath salts" are released to the public, it is not uncommon that they are followed by a wave of overdoses and potential fatalities. This phenomenon gives rise to an even greater issue for clinical and forensic toxicology laboratories; the extraction, detection, identification, and quantitation of NPS. The work presented here aims to focus on the extraction of NPS from biological matrices, which will then aid in the detection and quantitation of NPS.

The most commonly used extraction/purification techniques in forensic toxicology laboratories are "dilute-and-shoot", protein precipitation ("crash-and-shoot"; PP), solid phase extraction (SPE) and liquid-liquid extraction (LLE). While these methods may not always be ideal for all drugs, due to time, cost, and lack of removal of possible interferences, they are well established for the extraction of common drugs of abuse. However, there is little available research focusing on the development of extraction methods with the sole purpose of extracting NPS from biological matrices. The majority of research done on the extraction of NPS focuses on the screening method rather than the

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extraction technique.[82,116] There are only a few publications that have investigated extraction of a small subset of NPS from biological matrices. [92,117]

The goal of the present research is to compare several extraction methods to determine if any one approach is more reliable than the others for the purpose of extracting NPS from biological matrices. Blood and urine are two of the most important specimens collected in forensic cases, as they can provide accurate detail into endogenous as well as exogenous analytes present in a sample, or lack thereof. Thus, it is imperative to develop near optimal conditions by taking into consideration the amount of solvent used, pH, pK_a of analytes, and possible drug-drug interactions for the ideal extraction of analytes within complex matrices. With the constant emergence of NPS, there is an underlying ambiguity in that NPS that are similar in structure may co-elute. Co-eluting analytes may experience suppression or enhancement and can therefore not be specifically detected. In addition to more traditional extraction methods, the present research investigated the potential benefits of online SPE and "QuEChERS" (Quick, Easy, Cheap, Effective, Rugged and Safe) for the extraction of NPS from urine or whole blood.

Online SPE has the potential to greatly reduce analysis time, transfer steps, and sample handling. However, method development and optimization for online SPE is very complex, time consuming, and relies heavily on having the proper instrumentation. QuEChERS, developed by Anastassiades and co-workers, has become a reliable extraction method for pesticide analysis in agricultural and food produce industries.[93] Initially, QuEChERS served as an approach to extract the wide range of polar and nonpolar pesticide residues left on fruit and vegetables. The process of extraction for a single sample involves a two-step process; partitioning, followed by a dispersive-solid phase extraction (d-SPE)

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cleanup.[93] The method's application has since become expanded to include the extraction of pollutants from complex matrix samples such as soil, sediment, and water. Furthermore, QuEChERS has in a very limited number of cases been modified to include the extraction of NPS found in biological matrices.[117,118] The process of QuEChERS occurring in a "one-pot" process can further improve the process by cutting down on the number of steps, preparation time, cost of SPE cartridges, cleaning of glass, and use of harmful solvents. In this study, a QuEChERS method, modified to involve only a mini one-pot process, was compared to standard SPE methods and protein precipitation through the analysis of chromatographic profiles for mixes of NPS in whole-blood and urine.

5.2 Materials and Methods

5.2.1 Chemicals and Materials

Reference standards for all NPS compounds, including deuterated standards, were obtained from Cayman Chemical (Ann Arbor, MI) as the neat solid material for the majority of compounds, although some were already in solution. Optima LCMS grade MeOH, acetonitrile, DMSO, HPLC water, ammonium formate (99%), formic acid, ammonium hydroxide, HCl, glacial acetic acid, ammonium acetate, magnesium sulfate anhydrous, sodium acetate anhydrous, and sodium chloride were purchased from Fisher Scientific (Fair Lawn, NJ). Bulk sorbents of primary secondary amines (PSA) and end-capped C18 were purchased from United Chemical Technologies (Bristol, PA). Bond Elut Plexa PCX SPE cartridges were purchased from Agilent Technologies (Santa Clara, CA). Certified blank urine was purchased from UTAK Laboratories Inc. (Valencia, CA) and aliquoted into 10 mL portions and stored at -20ºC until needed. Blank human whole blood

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with disodium EDTA as an anticoagulant was purchased from BioIVT (Hicksville, NY) and stored at 4˚C.

5.2.2 Preparation of standard solutions

Neat standards (including deuterated compounds) were dissolved in MeOH or DMSO, depending on the analyte's solubility, to achieve concentrations of 1, 2, 5, or 10 mg/mL. From these initial preparations, 10 µg/mL working solutions were prepared in MeOH for all analytes. The 10 µg/mL working solutions were used to create 200 ng/mL spiking solutions that contained up to 36 compounds. An internal standard for spiking was also created using the 10 µg/mL working solution.

5.2.3 Dilute-and-Shoot/Crash-and-Shoot Methodology

All urine and whole blood samples were spiked with the 200 ng/mL spiking solutions and the IS mix to reach the desired concentration. Dilute-and-shoot was completed by diluting spiked urine at a ratio of 1:5 with HPLC water and then directly injecting the diluted sample into the instrument for analysis. Crash-and-shoot was completed on spiked whole blood samples. This procedure consisted of adding 600 µL of cold acetonitrile (-20˚C) to 200 µL of sample, vortexed for 30 sec and centrifuged for 5 min at 7000 rpm. After centrifuging, 100 µL of acidified MeOH (2% HCl) was added to the organic layer and dried down using a vacufuge (1 h at 45˚C) and then reconstituted in 200 µL of MeOH for analysis.

5.2.4 The QuEChERS Methodology

A mini-one pot QuEChERS kit was developed in-house. For the one-pot method, certified drug free pooled whole blood with EDTA as an anticoagulant was spiked with drug mix and internal standard mix to reach a total sample volume of 0.2 mL. Before the

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addition of sample, 600 µL of cold acetonitrile and 200 µL of HPLC water were added to a pre-weighed mini QuEChERS kit consisting of 200 mg of $MgSO_4$, 50 mg of NaCl, 25 mg of PSA, 25 mg of end-capped C_{18}, and ceramic homogenizer beads. Next, 200 µL of sample was added to the kit then the sample was shaken by hand for 30 s, vortexed for 1 min, and centrifuged for 5 min at 10,000 rpm. Acidified MeOH was added to the supernatant before being dried down using a vacufuge. Once dry, the sample was reconstituted in 200 µL of MeOH before LC-MS/MS analysis.

5.2.5 On-line SPE Methodology

Online SPE was initially performed using an Agilent 1290 Infinity Online SPE Solution in conjunction with an Agilent 1290 FlexCube LC unit, with a Bond Elute (BE) online polymeric sorbent material (PLRP-S) cartridge. An online SPE method was created by altering the dMRM method that had been previously developed for the detection of NPS. The online SPE method included parameters for the Agilent Flex Cube LC unit using the same mobile phases as those developed for the screening method (*i.e.,* initial 95:5 A:B; final 2:98 A:B mobile phases).

After initial experiments using this online SPE method were unsatisfactory, a new approach was taken in order to increase the retention and recovery of all compounds. This revised online SPE method utilized two different cartridges which were loaded and eluted at the same time. For this purpose, the BE cartridge described above was used in tandem with a mixed mode cartridge. This approach was developed in the hope of reducing tailing and peak broadening encountered with the initial method. It was reasoned that a mixed mode cartridge could allow for some the compounds to elute faster rather than being

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retained on the reverse phase column. To properly load and elute using the two cartridges, modified FlexCube parameters were developed (Table 17).

Table 17. Programming timetable for the Flex Cube for Online SPE.

Time (min)	Function	Parameter
0	Pump for volume	Pump 3 mL: Flow at 0.5 mL/min
1.00	Valve change position	Position 2 (Load 2 Elute 1)
2.00	Valve change position	Position 1 (Load 1 Elute 2)
12.5	Pump for volume	Pump 3 mL: Flow at 0.5 mL/min
14.00	Pump for volume	Pump 4 mL: Flow at 0.5 mL/min

5.2.6 SPE Methodology

An SPE method previously created in the lab was altered in order to be used for a wide variety of NPS. Crash-and-shoot, as described above, was completed on 200 µL of sample, then 1 mL of 0.1 M phosphate buffer (pH=6) was added to the organic layer. A mixed mode Plexa PXR cartridge was used for SPE. The cartridge was conditioned with 1 mL of MeOH and 1 mL of phosphate buffer. After conditioning, the sample was loaded onto the cartridge slowly and then washed with 3 mL of buffer and 3 mL of MeOH:H_2O (20:80). After washing, the cartridge was dried for 10 min before elution. The sample was eluted with two 0.5 mL aliquots of MeOH:MeCN (50:50) and one 0.5 mL aliquot of 5% ammonium hydroxide in MeCN. Acidified MeOH (2% HCl) was added to the extract before drying using a vacufuge. Once dried, samples were reconstituted in 200 µL of MeOH before analysis.

5.2.7 Comparison of techniques

Results for all extraction methods except online SPE were compared with regard to matrix effects, recovery, process efficiency, cost, and time. Recovery (RE), matrix effects (ME), and process efficiency (PE) were determined using three sample sets, all with a final NPS concentration of 50 ng/mL. The three sets were designed as follows; neat drug

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dissolved in MeOH (set A), drug spiked into the sample after extraction (set B), and drug spiked into the sample before extraction (set C). Matrix effects, recovery, and process efficiency were determined according to equations 7, 8, and 9, respectively. Cost was determined based on the materials needed to run 50 samples a day for a full year and the time required per batch of samples (20 samples).

$$ME = \frac{Set\ B}{Set\ A} * 100 \qquad (7)$$

$$RE = \frac{Set\ C}{Set\ B} * 100 \qquad (8)$$

$$PE = \frac{Set\ C}{Set\ A} * 100 \qquad (9)$$

Techniques were separated by matrix (*i.e.,* urine or whole blood) and compared accordingly. To determine whether results for any of the techniques were significantly different from one another, a one-way analysis of variance (ANOVA) was completed using JMP software version 14. An independent one-way ANOVA was done for each parameter (ME, RE, and PE) resulting in three ANOVAs for each matrix. Average peak area was used to determine ME, RE, and PE for each compound. Each of the three ANOVAs were created for all compounds in Mix 2 (n=33). An individual ANOVA was not completed per compound, instead ANOVAs were separated based on technique and parameter (ME, RE, PE). An ANOVA is only capable of determining if any of the techniques are significantly different (determined using the F ratio on the basis of sum of squares) not which specific techniques are different from one another. To determine which specific extraction techniques produced significantly different results, a Tukey's honestly significant difference (HSD) test was completed using JMP software. Cost and time were compared

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subjectively and are highly dependent on the needs of the analysis (*i.e.,* whether the elimination of matrix effects is more important than recovery).

5.2.8 LC-QqQ-MS analysis

All samples were analyzed using an Agilent 1290 Infinity Binary Pump LC coupled to an Agilent 6460 triple quadrupole MS/MS with Jet Streaming technology and electrospray ionization (ESI) equipped with Agilent MassHunter software version B7.0. Chromatographic separation was performed using an Agilent Zorbax Rapid Resolution HD Eclipse Plus C_{18} column (3.0 x 100 mm, 1.8 μm). Data acquisition was performed in dMRM mode using positive mode ESI. The dMRM method employed two transitions for each analyte, which aids in achieving increased selectivity.

Chromatographic separation was achieved using gradient elution with a flow rate of 0.3 mL/min using 5 mM ammonium formate/0.1% formic acid in HPLC water as mobile phase A and MeOH with 0.1% formic acid as mobile phase B. The gradient used for analysis was as follows: hold at 5% B for 1 min, followed by 5% B to 98% B from 1 to 9.5 min, then hold at 98% B until 16 min, followed by a 3-min re-equilibration at 5% B. The analytical column was held at a temperature of 40˚C during separation.

MS source parameters were as follows: gas temperature, 325˚C; gas flow 6 L/min; nebulizer 40 psi; sheath gas temperature 350˚C; sheath gas flow 11 L/min; capillary voltage 4,000 V; and nozzle voltage 750 V. Agilent MassHunter Optimizer software was used to determine the ideal data acquisition parameters for MRM mode. The software uses the mass of each compound to determine optimal fragmentor voltage, resulting product ions, and associated collision energies and can optimize for up to 10 transitions for each

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precursor ion. Analyte detection windows ranged from 0.25 min (*i.e.*, ± 0.125 min around t_R) to 0.75 min (*i.e.*, ± 0.375 min around t_R) depending on the analyte.

5.3 Results and Discussion

5.3.1 Dilute-and-shoot/crash-and-shoot methodology

Dilute-and-shoot was determined to be an economical and fast method, however, it exhibited the highest ion suppression/enhancement from matrix effects. This was expected, since no matrix effects were being eliminated by using this technique, just reduced via dilution. Dilute-and-shoot can be used when high throughput is necessary, since it saves both time and money, however it could cause increased instrument down time if proper cleanup precautions are not used.

Crash-and-shoot showed similar results to those of dilute-and-shoot, since it is also a minimal purification technique. The goal of crash-and-shoot is to eliminate proteins by precipitating them out using cold solvent. For many screening purposes in forensic laboratories, crash-and-shoot may be desirable to cut down on time and cost of analysis.

The results for ME, RE, PE for all compounds in Mix 2 for crash-and-shoot can be seen in Table 18. Matrix effects should ideally be 80 - 120%; there were a number of compounds for which ME was not within this range. However, the majority of compounds did exhibit ME within 60 - 85%, indicating some degree of ion suppression. This result may not be surprising, as whole blood is a complicated matrix and eliminating all matrix effects simply by removing proteins present in the sample is not always enough to sufficiently eliminate matrix effects.

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Table 18. Matrix effects, recovery, and process efficiency for Mix 2 compounds in spiked (50 ng/mL) whole blood samples following crash-and-shoot processing.

Compound	ME (%)	RE (%)	PE (%)
3,4-DHMA	134	33	44
2-fluoromethcathinone	97	81	78
4-fluoroisocathinone	74	85	63
4-hydroxy MiPT	0	48	0
Clencyclohexerol	44	73	32
N-Ethylbuphedrone	79	87	69
4-MMC	88	82	72
3-methyl-α-Pyrrolidinopropiophenone	88	83	73
3,4-dimethoxy-α-Pyrrolidinopentiophenone	89	83	74
2,3-methylenedioxy pyrovalerone	89	83	74
4-ethyl-N,N-dimethylcathinone	88	84	74
2C-T-2	61	76	46
PCPr	87	81	71
2C-T-4	61	74	45
4'-Methyl-N-methylhexanophenone	88	83	77
25I-NBF	87	83	72
Loperamide	89	81	72
AB-005 azepane isomer	58	94	55
AB-FUBINACA 3-fluorobenzyl isomer	80	73	58
A-796260	55	68	37
JWH 018 N-(5-hydroxypentyl) metabolite	61	88	54
MAM2201 N-pentanoic acid metabolite	47	112	53
ADB-PINACA isomer 1	73	80	58
RCS-4 2-methoxy isomer	70	81	57
PB-22	75	71	53
XLR11 N-(2-fluoropentyl) isomer	42	73	31
UR-144 Degradant	1	62	1
AKB48 N-(5-fluoropentyl) analog	13	49	6
KM 233	1	200	2
Δ8-THC	2	314	5
EG-018	34	73	25
SER-601	101	35	35

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5.3.2 The QuEChERS Methodology

The QuEChERS method underwent some efforts at optimization before a final in-house method was created and used for the comparison work. At first, a two-step approach using a commercial (Agilent Technologies, Inc.) QuEChERS kit designed for the extraction of 2 mL samples was tested and then compared to the results of an in-house mini QuEChERS kit that was developed. Figure 5 shows the comparison of chromatograms for the one-pot approach and the two-step method when applied to whole blood samples spiked at the 5 ng/mL level. Results of the two approaches were compared based on peak area and recovery (determined using a daily calibration curve). It was determined that at lower concentration levels the in-house mini QuEChERS approach resulted in higher recoveries than the commercial two-step approach. Other concentrations (20 and 80 ng/mL) were also compared, however, there was no significant difference between the two approaches for higher concentrations.

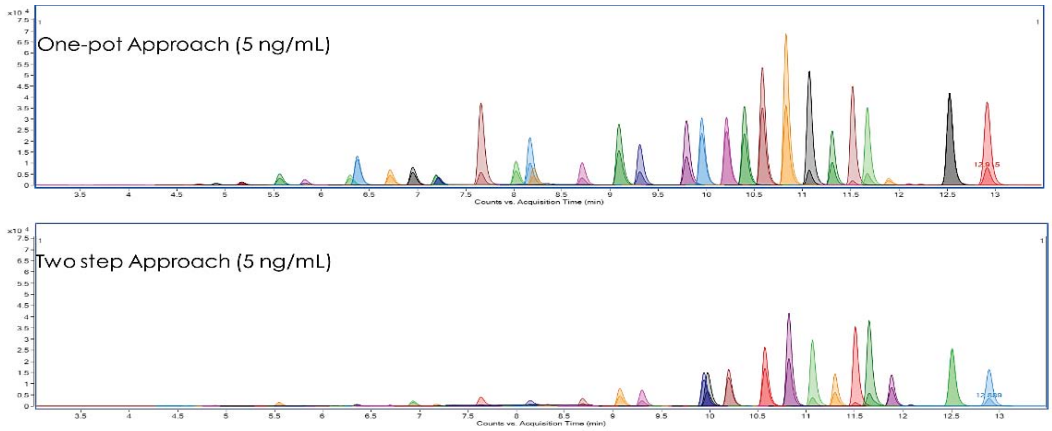

Figure 5. The top panel represents the MRM results of extracting the compounds in Mix 2 from whole blood using an in-house one-pot approach, while the bottom panel is the result of extracting the same compounds from whole blood using a two-step approach.

Figure 6 represents the comparison of the one-pot approach and the two-step approach for QuEChERS in terms of extracting a mix of NPS from whole blood. There was no statistically significant difference between the approaches, however, there was a

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wider spread of results for the two-step approach than the one-pot method. The variation of results for the two-step approach can be attributed to the low recoveries of 5 ng/mL samples in comparison to the results seen with the one-pot approach. Figure 6 shows chromatograms for the one-pot approach and the two-step approach for 5 ng/mL samples. All work moving forward was completed using the one-pot approach.

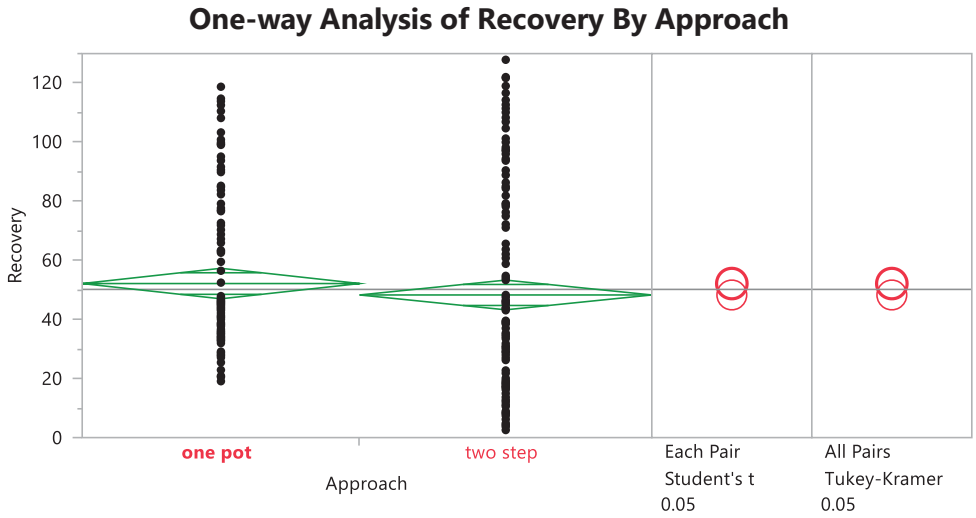

Figure 6. Visually represents the results of a Means/ANOVA test used to determine whether or not there is a statistically significant difference between the commercial two step approach and the in-house one-pot approach in terms of recovery.

Subsequently, attempts were made to further optimize the one-pot mini QuEChERS approach. Ratios and amounts of solids were modified to assess the effect on reduction of matrix effects and drug recovery. However, these efforts did not result in in significant additional improvement in these parameters. Figure 7 represents the elimination of matrix effects by changing salts using QuEChERS. In the figure on the x-axis B=blood U=urine and the number correspond to the amount of $MgSO_4$ in mg in the QuEChERS kit. Since there were no statistically significant differences found through the one-way ANOVA and

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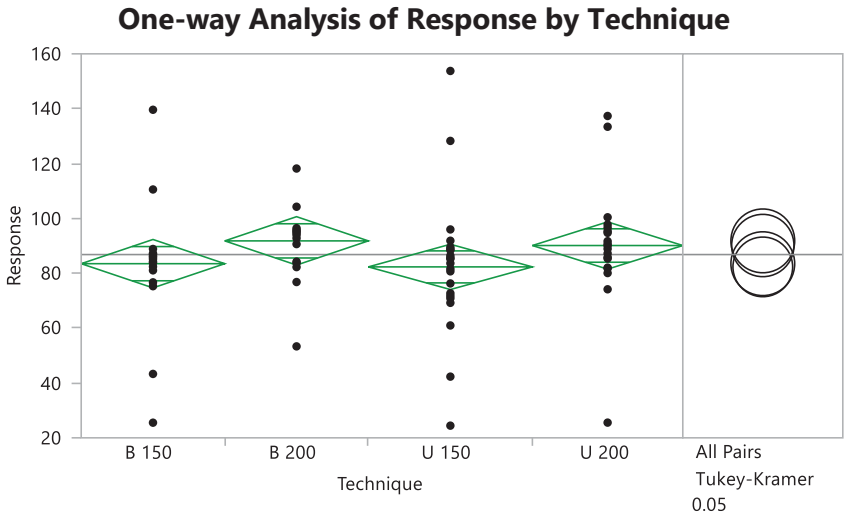
Tukey's HSD test, the original amount of salts (150 mg) were used to create one-pot QuEChERS kits for extraction.



Level	- Level	Difference	Std Err Dif	Lower CL	Upper CL	p-Value
B 200	U 150	9.484481	6.075284	-6.4401	25.40911	0.4063
B 200	B 150	8.312173	6.274526	-8.1347	24.75906	0.5499
U 200	U 150	7.793373	6.001192	-7.9370	23.52379	0.5664
U 200	B 150	6.621065	6.202815	-9.6378	22.87998	0.7102
B 200	U 200	1.691108	6.202815	-14.5678	17.95002	0.9929
B 150	U 150	1.172308	6.075284	-14.7523	17.09694	0.9974

Figure 7. The top panel visually represents the results of a Means/ANOVA test used to determine whether or not the amounts of salts used in the QUEChERS one-pot kit has an effect on elimination of matrix effects. The bottom panel shows the results of Tukey HSD test showing that there are no statistically significant differences.

The results for ME, RE, PE for all compounds spiked into whole blood and extracted by QuEChERS can be seen in Table 19. Matrix effects observed for all compounds in Mix 2 were quite variable; the majority were below 100%, meaning that the compounds were experiencing ion suppression. Most of the compounds fall between 60 and 100% matrix effects, therefore they were experiencing up to 40% ion suppression. While there was a large range of recoveries, the majority of compounds were recovered at levels above 60%. It is not uncommon to see such varied recoveries due to the different structural classes of drugs that are being extracted, all of which have different chemical

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interactions. Process efficiency on average fell between 40 and 60% for the compounds included in Mix 2. Ideally, process efficiency should be higher and the QuEChERS approach could be further optimized specifically to increase PE values.

Table 19. Matrix effects, recovery, and process efficiency for Mix 2 compounds in spiked (50 ng/mL) whole blood samples following QuEChERS processing.

Compound	ME (%)	RE (%)	PE (%)
3,4-DHMA	105	8	9
2-fluoromethcathinone	73	69	50
4-fluoroisocathinone	91	116	106
4-hydroxy MiPT	0.1	51	0.1
Clencyclohexerol	61	69	42
N-Ethylbuphedrone	63	72	45
4-MMC	69	69	47
3-methyl-α-Pyrrolidinopropiophenone	68	64	44
3,4-dimethoxy-α-Pyrrolidinopentiophenone	69	69	48
2,3-methylenedioxy pyrovalerone	68	66	45
4-ethyl-N,N-dimethylcathinone	68	61	42
2C-T-2	17	206	34
PCPr	67	61	41
2C-T-4	21	190	41
4'-Methyl-N-methylhexanophenone	68	64	44
25I-NBF	68	72	49
Loperamide	68	85	57
AB-005 azepane isomer	43	99	42
AB-FUBINACA 3-fluorobenzyl isomer	66	86	56
A-796260	31	139	43
JWH 018 N-(5-hydroxypentyl) metabolite	57	87	51
MAM2201 N-pentanoic acid metabolite	56	92	51
ADB-PINACA isomer 1	61	78	47
RCS-4 2-methoxy isomer	57	84	48
PB-22	56	84	47
XLR11 N-(2-fluoropentyl) isomer	46	87	39
UR-144 Degradant	16	79	13
AKB48 N-(5-fluoropentyl) analog	22	69	15
KM 233	5	112	6
Δ8-THC	3	202	7
EG-018	43	57	24
SER-601	71	78	55

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5.3.3 Online SPE methodology

During the online SPE method development and optimization experiments, initial results were less than ideal, resulting in severe tailing, peak broadening, and carry over. An example of a typical chromatogram collected using the one cartridge approach can be seen in Figure 8. One of the major issues faced when trying to optimize an online SPE approach for the Agilent FlexCube instrumentation was the inability to manipulate pH in the same way as that for a classical SPE approach. In order to get around this limitation, a two-cartridge approach was attempted, but also without success. It was ultimately determined that a different online SPE instrumentation design would be required to successfully extract different classes of NPS from biological matrices in just one run. For

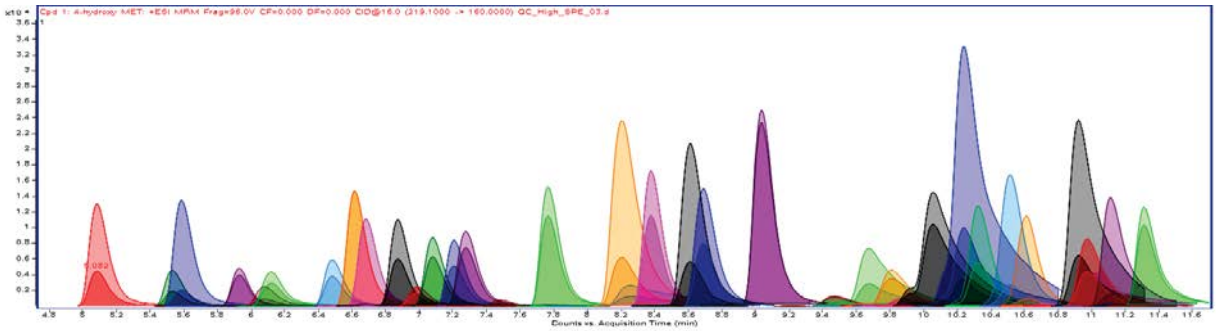

Figure 8. Typical LC-QqQ-MS MRM response for Mix 2 using the reversed phase online SPE approach.

example, Lehman and co-workers developed an in-line SPE method for the extraction of 74 NPS from serum. Their in-line SPE set up was capable of up to 100 µL injection volumes and was capable of higher ranges of pH manipulation. Additionally, an ideal online SPE setup would allow for higher flow rates and the ability to use more than three solvents for the SPE process. Higher flow rates would aid in the washing of impurities from the cartridge and elution of compounds of interest. The more solvent attachments allow for a method with more steps, which may be necessary to extract such varied compounds in one run. Consequently, online SPE was not considered further when

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completing comparison tests for extraction/purification methods. As an alternative, classical SPE was tested and compared to the other extraction/purification methods.

5.3.4 Solid phase extraction methodology

The classical SPE approach was adapted from a previously developed approach in the lab so that it was capable of extracting different classes of NPS. The SPE method was specifically designed to extract drug compounds with varying pKa values through pH manipulation and multiple elution steps. Table 20 shows the ME, RE, and PE for all compounds included in Mix 2. The majority of compounds underwent ion some suppression in terms of matrix effects, however, most of the compounds were found to have ME above 70%, which is desired when working with whole blood. It was shown that SPE is capable of removing a large portion of matrix effects for the majority of compounds. The major exceptions were synthetic cannabinoids, which tend to pose issues when extracting from biological matrices in general. In terms of recovery, the results were generally lower than desired; RE fell within the range of 50-100%, which might be improved with further optimization. Process efficiency varied from compound to compound without a specific trend.

Table 20. Shows the results for mix 2 using SPE in terms of matrix effects, recovery, and process efficiency using 50 ng/mL spiked whole blood samples.

Compound	ME (%)	RE (%)	PE (%)
3,4-DHMA	132	3	4
2-fluoromethcathinone	23	106	24
4-fluoroisocathinone	29	64	18
4-hydroxy MiPT	69	2	1
Clencyclohexerol	85	22	19
N-Ethylbuphedrone	60	72	43
4-MMC	59	62	37
3-methyl-α-Pyrrolidinopropiophenone	77	53	41
3,4-dimethoxy-α-Pyrrolidinopentiophenone	86	55	47
2,3-methylenedioxy pyrovalerone	78	55	43

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4-ethyl-N,N-dimethylcathinone	71	65	46
2C-T-2	84	11	9
PCPr	85	43	37
2C-T-4	87	13	11
4'-Methyl-N-methylhexanophenone	60	65	39
25I-NBF	85	52	44
Loperamide	88	23	20
AB-005 azepane isomer	86	54	46
AB-FUBINACA 3-fluorobenzyl isomer	95	62	59
A-796260	82	61	50
JWH 018 N-(5-hydroxypentyl) metabolite	86	78	67
MAM2201 N-pentanoic acid metabolite	93	79.	73
ADB-PINACA isomer 1	86	64	55
RCS-4 2-methoxy isomer	75	74	56
PB-22	77	61	47
XLR11 N-(2-fluoropentyl) isomer	55	75	42
UR-144 Degradant	31	88	27
AKB48 N-(5-fluoropentyl) analog	15	128	19
KM 233	8	120	10
Δ8-THC	31	79	24
EG-018	33	71	23
SER-601	93	77	72

5.3.5 Comparison of techniques

All extraction methods were compared on the basis of matrix effects, recovery, process efficiency, time, and overall cost. Table 21 shows the breakdown of how much each extraction technique would cost to analyze 20 samples including consumables, solvents, cartridges, and operator time assuming a $22 hourly salary.. Additionally, Table 21 shows the time each method would take to extract 20 samples. It is important to consider overall cost and time when determining which extraction method is ideal for a specific purpose. However, time and cost should not be the only factors to consider in making a final decision. These parameters should be considered in addition to ME, RE, and PE.

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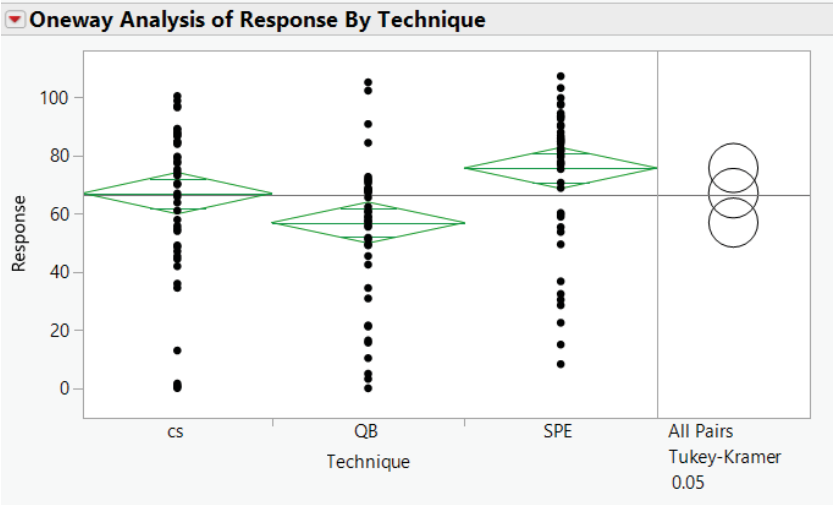
Table 21. Cost of each extraction technique and the time each one takes to prepare a set of 20 samples.

Technique	Cost per set ($)	Time (min)
Dilute-and-shoot (urine)	5	10
Crash-and-shoot (blood)	39	100
QuEChERS (blood)	47	120
QuEChERS (urine)	47	120
Solid phase extraction (blood)	146	210
Solid phase extraction (urine)	132	180

In addition, extraction method performance was statistically compared using a one-way ANOVA based on ME, RE, and PE, using peak area as a measure of response. The ANOVA and results of the Tukey's HSD test can be seen in Figures 9, 10, and 11.



Ordered Differences Report

Level	- Level	Difference	Std Err Dif	Lower CL	Upper CL	p-Value
SPE	QB	18.74810	5.047154	6.80628	30.68993	0.0008*
cs	QB	10.09486	5.070905	-1.90316	22.09288	0.1178
SPE	cs	8.65324	5.070905	-3.34478	20.65127	0.2060

Figure 9 The top panel visually represents the results of a Means/ANOVA test used to determine whether results of the three extraction methods based on matrix effects are significantly different. The bottom panel shows the results of Tukey HSD test showing that there is a statistically significant difference between the results of SPE and QuEChERS with blood in terms of elimination of matrix effects.

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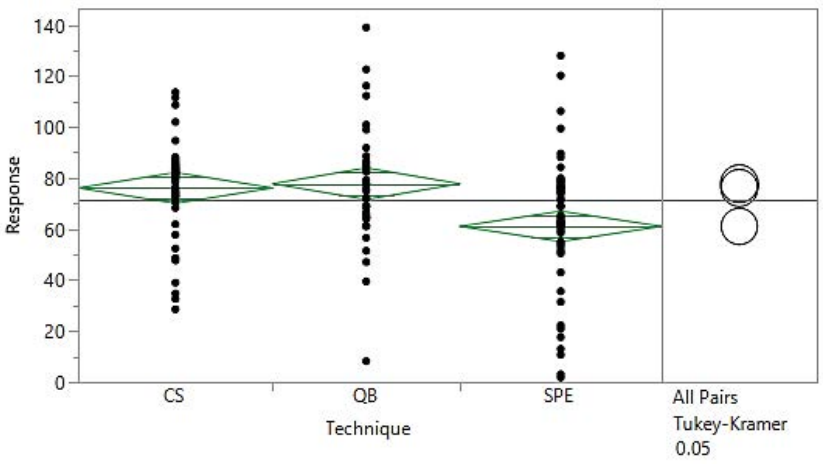
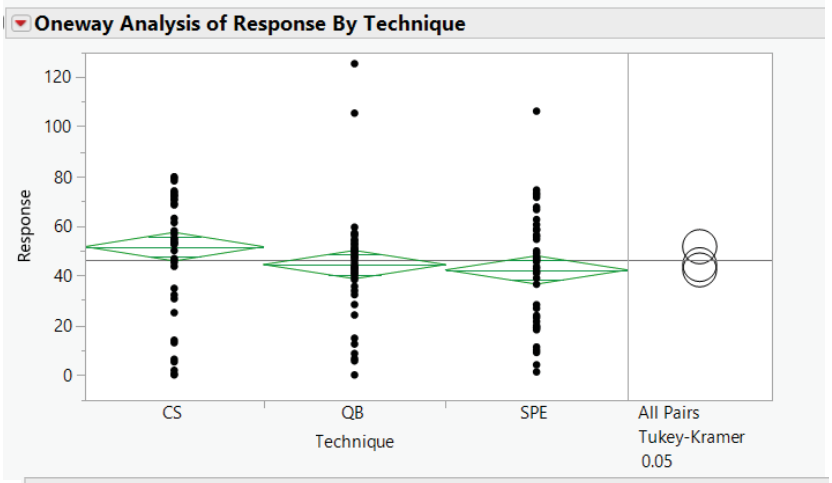


Ordered Differences Report

Level	- Level	Difference	Std Err Dif	Lower CL	Upper CL	p-Value
QB	SPE	27.77594	7.426382	10.2058	45.34609	0.0007*
CS	SPE	21.74123	7.426382	4.1711	39.31138	0.0109*
QB	CS	6.03471	7.426382	-11.5354	23.60487	0.6958

Figure 10. The top panel visually represents the results of a Means/ANOVA test used to determine whether results of the three extraction methods based on recovery are significantly different. The bottom panel shows the results of Tukey HSD test showing that there is a statistically significant difference between the results of SPE and QuEChERS with blood and crash and shoot and SPE in terms of recovery.



Ordered Differences Report

Level	- Level	Difference	Std Err Dif	Lower CL	Upper CL	p-Value
CS	SPE	9.378162	4.105412	-0.33545	19.09178	0.0609
CS	QB	7.224076	4.105412	-2.48954	16.93769	0.1867
QB	SPE	2.154085	4.086183	-7.51403	11.82220	0.8582

Figure 11. The top panel visually represents the results of a Means/ANOVA test used to determine whether results of the three extraction methods based on process efficiency are significantly different. The bottom panel shows the results of Tukey HSD test showing there are no statistically significant differences.

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The one-pot QuEChERS approach when completed for urine samples showed a greater elimination of matrix effects than dilute-and-shoot, but lower elimination of matrix effects than SPE. Even though QuEChERS is capable of removing a higher level of matrix effects from urine as compared to dilute-and-shoot, it has comparable recoveries and process efficiency to dilute-and-shoot. QuEChERS for whole blood showed comparable matrix effects, recovery and process efficiency as compared to crash-and-shoot. While at first glance QuEChERS may appear capable of eliminating more matrix effects based on the ANOVA plot, the p values indicate no statistically significant differences.

The SPE approach, when completed for urine samples, showed much higher elimination of matrix effects than either dilute-and-shoot or QuEChERS. Even though SPE is capable of removing a higher level of matrix effects from urine samples than dilute-and-shoot and QuEChERS, there was no significant difference when considering recovery and process efficiency. Solid phase extraction for whole blood, like the results for urine, showed higher elimination of matrix effects than the other two extraction methods. However, SPE had lower recovery than both QuEChERS and crash-and-shoot. All three extraction methods for whole blood were comparable when looking at process efficiency. SPE is more efficient at eliminating matrix effects than the other options, but is the most time consuming and expensive. The cost and time it takes to complete SPE needs to be weighed against the need for the elimination of matrix effects.

When considering the results of the ANOVAs for all parameters and Tukey HSD tests, there is no clear answer on which technique would be most beneficial for the extraction of NPS. In order to decide which approach is appropriate, it is therefore

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important to consider the overall needs of the analysis. For qualitative purposes, dilute-and-shoot and crash-and-shoot may be appealing since they are quick and cost effective. For qualitative analysis, the elimination of matrix effects is not imperative, therefore it is not necessary to use a more time and cost consuming method. If quantification is needed, then a technique capable of minimizing matrix effects would be more effective. Therefore, SPE may be the best choice for quantitation or when low limits of detection are needed. QuEChERS may be ideal when high throughput and quantification is desired due to the time difference between SPE and QuEChERS.

Finally, the drying down process was analyzed to ensure that it was not contributing to sample loss. Although this step is not necessary for all samples, it was done in this study so that all extracts had the same composition and volume for analysis. Results indicated that the drying down process does account for some analyte loss. For most compounds, there was ~10% loss of analyte in the drying process using the vacuum centrifuge. However, some compounds showed a much higher sample loss, for example 2C-T-4 and KM 233, which had ~90% loss of analyte through the drying process. To avoid potentially high sample losses from drying down, it is also possible to use a gentle stream of nitrogen as an alternative. Ideally, the drying down process should only be used when necessary, such as when the goal is to concentrate samples before analysis.

5.4 Conclusions

Various extraction methods, including dilute-and-shoot, crash-and-shoot, SPE, and QuEChERS, were analyzed and compared based on elimination of matrix effects, and improved recovery and process efficiency. It was determined that none of the methods are clearly statistically better than the others for the extraction of NPS from

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urine and whole blood. Each extraction method was applied to a mixture of 33 NPS that included compounds from various drug classes and metabolites. When analyzing the mixture as a whole it was determined that SPE is capable of eliminating the most matrix effects, however SPE had the lowest recoveries. Ultimately, when deciding which extraction method is best, it is necessary to consider the goals of the final method. Further optimization would need to be completed in order to have one method that is best in all aspects for multiple drug classes. However, QuEChERS has the potential to be that method with additional optimization.

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6. ANALYSIS OF BLIND SPIKES AND AUTHENTIC SPECIMENS

6.1 Introduction

Full method validation and applicability of extraction methods generally need to be tested on blind spikes and authentic specimens in order to prove adaptability to forensic case work samples.[111] A number of methods that have been validated for the detection of NPS have been applied to forensic case work samples.[78,82]Authentic specimens have challenges that are not faced when analyzing spiked samples. Some of these challenges include interferences from licit medications, unknown concentrations of analytes, and increased matrix effects that can be caused by medical issues.

The present study included the analysis of blind spiked urine and whole blood samples and authentic ante-mortem urine specimens using the validated dMRM method for the detection of NPS. Blind spikes were qualitatively and quantitatively analyzed in order to further validate the dMRM method. Additionally, authentic specimens were qualitatively screened for all 826 analytes included in the full dMRM method. Analyzing blind spikes and authentic specimens is important in order to prove the applicability of a developed method to clinical and forensic samples.

6.2 Materials and Methods

6.2.1 Chemicals

Reference standards for all NPS compounds, including deuterated standards, were obtained from Cayman Chemical (Ann Arbor, MI) as the neat solid material for the majority of compounds, although some were already in solution. Optima LCMS grade methanol, acetonitrile, HPLC water, ammonium formate (99%), formic acid, magnesium sulfate anhydrous, sodium acetate anhydrous, and sodium chloride were purchased from

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Fisher Scientific (Fair Lawn, NJ). Bulk sorbents of primary secondary amines (PSA), endcapped C18, and beta-glucuronidase were purchased from United Chemical Technologies (Bristol, PA). Certified blank urine was purchased from UTAK Laboratories Inc. (Valencia, CA) and aliquoted into 10 mL portions and stored at -20ºC until needed. Blank human whole blood with disodium EDTA as an anticoagulant was purchased from BioIVT (Hicksville, NY) and stored at 4˚C.

6.2.2 Collection of Authentic Specimens

Authentic urine specimens that had originally been collected in 2014 were obtained from a local drug testing laboratory, with volumes varying from 2.5 to 6.0 mL. Specimens were obtained from subjects in addiction treatment and pain medication monitoring programs and were supplied deidentified with no subject information provided. Specimens were assigned a sequential ID number for laboratory tracking purposes and were stored in a -20˚C locked freezer until analysis.

6.2.2 Preparation of samples

Blind spiked urine samples were created such that identity and concentration were unknown to the analyst. Samples were created in certified blank urine at a final volume of 200 µL. Analytes were selected from Mixes 1, 2, and/or 3 and samples contained 0 to 9 individual analytes at varying concentrations. An aliquot of internal standard mix was added to each sample, which was then diluted to 1 mL with HPLC water before analysis.

Blind spiked whole blood samples were also created in the same manner as the blind spiked urine samples, *i.e.,* in certified blank human whole blood in a final volume of 200 µL. Analytes were selected from Mixes 1, 2, and/or 3 and samples contained 0 to 2 individual analytes. An aliquot of internal standard mix was added to each sample in the

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first set of blind spiked whole blood samples before undergoing mini one-pot QuEChERS extraction. After extraction, the organic layer was removed and dried down using vacufuge after the addition of acidified MeOH. Once completely dry, samples were reconstituted with 200 µL of MeOH for analysis. A second set of blind spiked whole blood samples were prepared using crash-and-shoot processing. An aliquot of internal standard mix was added to all samples before the addition of 600 µL of cold MeCN. Once the MeCN was added, all samples were vortexed and then centrifuged. After centrifugation, the supernatant was removed and dried down using the same technique as the samples prepared using QuEChERS.

In addition, 50 authentic urine specimens were analyzed using the validated dMRM method. Authentic specimens were prepared by adding internal standard mix to 100 µL of sample and diluting it to 500 µL with HPLC water for analysis. After this initial analysis, the 50 authentic specimens were glucuronidase treated, making it possible to detect metabolites that may be missed otherwise. The glucuronidase solution was prepared using 2 mL of hydrolysis buffer, 18 mL water, and 5 mL of β-glucuronidase. Treatment was done by adding the glucuronidase solution to authentic urine specimens at a ratio of 1:1 and incubating for 2 h at 35˚C before LC analysis. These samples were qualitatively screened for the presence of any of the 826 compounds included in the NPS standard mixes. Qualitative analysis for the blind spiked and authentic specimens was completed using the dMRM method described previously. Any peak with a signal 3 times greater than the noise was considered to be positive for that analyte. Quantitative analysis for blind spiked

samples was completed using the fully validated dMRM method described previously,

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using a daily calibration curve and MassHunter Quantitative software.

6.3 Results and Discussion

A total of 38 blind spiked urine samples were prepared in three different sets for qualitative and quantitative analysis. The first set was designed to test the lower limits of the validated method, the second set was designed in the middle of the calibration curve, and the third was designed to test selectivity. The experimental identity, experimental concentration, true identity, and true concentration for the 15 blind spikes included in the first set are shown in Table 22. The only sample that was not properly identified was sample 1, which should have been identified as THCA-A, but was identified as JWH 200 5-hydroxyindole metabolite. This was likely due to the fact that THCA-A was one of the few compounds that did not show linearity and therefore this method was not capable of properly identifying that compound. In addition, there were three false negative results (samples 2, 4, and 6), *i.e.,* samples that were determined to be blank even though they each contained one compound, and one sample (sample 5) that contained two compounds with only one correctly identified. The false negatives could have been caused by ion suppression, which may have resulted in low ion intensity and levels that were not with in the LOD/LOQ of the dMRM method, since this set of spikes was designed to test the lower limits of the method. Differences between the spiked concentration and the detected concentration may also be attributed to ion suppression or enhancement. Additionally, the use of IS not chemically identical to every analyte can also introduce errors in quantitation, due to differences in relative ionization or other factors.

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Table 22. True identity and concentration and experimental identity and concentration of blind spiked urine samples included in set 1 (low concentration)

#	Compound Spiked	Conc. spiked (ng/mL)	Compound Detected	Conc. Detected (ng/mL)
1	THCA-A	5	JWH 200 5-hydroxyindole metabolite	N/A
2	4-APDB	5	nd	-
3	JWH 200 5-hydroxyindole metabolite	5	JWH 200 5-hydroxyindole metabolite	7
4	N-Methyltryptamine	5	nd	
5	3-Methylbuphedrone	5	3-Methylbuphedrone	7
	5-fluoro SDB-006	5	nd	
6	2-fluoromethcathinone	5	nd	
7	2C-T-4	5	2C-T-4	1
8	PB-22	5	PB-22*	9
9	4-ethyl-N,N-dimethylcathinone	5	4-ethyl-N,N-dimethylcathinone	3
10	JWH 018 N(5-hydroxypentyl) metabolite	5	JWH 018 N(5-hydroxypentyl) metabolite	9
	MAM2201 N-pentanoic acid metabolite	5	MAM2201 N-pentanoic acid metabolite	5
11	25E-NBOMe	5	25E-NBOMe	5
12	AB-FUBINACA	5	AB-FUBINACA*	3
13	3C-B-fly	5	3C-B-fly	4
14	Para-Fluorofentanyl	5	Para-Fluorofentanyl	3
15	N,N-dimethylcathinone	5	N,N-dimethylcathinone	1
	Phenylpiperazine	5	Phenylpiperazine	3

*Isomer of this compound were also detected.
nd- not detected.

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The true identity, true concentration, experimental identity, and experimental concentration can be seen in Table 23 for all compounds in the second set of spike urine samples. This set was spiked with 0 to 2 compounds each. The only sample that was not properly identified was sample 1, which should have been identified as THCA-A, but was not. This was likely due to the fact that THCA-A was one of the few compounds that did not show linearity and therefore this method was not capable of properly identifying that compound. Virtually all of the blind spikes were correctly identified, with quantitative results generally within ±20 of nominal. However, this method is not capable of discerning isomers and, as noted in the table, some isomers were determined in addition to the correct identification.

The true identity, true concentration, experimental identity, and experimental concentration can be seen in Table 24 for all compounds in the third set of spike urine compounds. This set was included 3 to 9 compounds per sample at varying concentrations. All compounds were identified correctly, except for THCA-A and 4'-fluoro-a-pyrrolidinopropiophenone. Again, THCA-A does not show linearity and 4'-fluoro-a-pyrrolidinopropiophenone was eliminated due to low abundance, which may have been caused by ion suppression. This set was designed to test selectivity, however, samples were still quantified and tested the full linear range of the method.

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Table 23. The true identity and concentration and experimental identity and concentration of blind spiked urine samples included in set 3 (intermediate concentration)

#	Compound Spiked	Conc. spiked (ng/mL)	Compound Detected	Conc. Detected (ng/mL)
1	THCA-A	50	Benzydamine	-
2	4-APDB	50	4-APDB	57
3	JWH 200 5-hydroxyindole metabolite	50	JWH 200 5-hydroxyindole metabolite	45
4	blank		None	-
5	N-Methyltryptamine	50	N-Methyltryptamine	43
6	3-Methylbuphedrone	50	3-Methylbuphedrone	56
	5-fluoro SDB-006	50	5-fluoro SDB-006	61
7	2-fluoromethcathinone	50	2-fluoromethcathinone	47
8	2C-T-4	50	2C-T-4	60
9	PB-22	50	PB-22*	70
10	blank		None	-
11	4-ethyl-N,N-dimethylcathinone	50	4-ethyl-N,N-dimethylcathinone	42
12	JWH 018 N(5-hydroxypentyl) metabolite	50	JWH 018 N(5-hydroxypentyl) metabolite*	55
	MAM2201 N-pentanoic acid metabolite	50	MAM2201 N-pentanoic acid metabolite	44
13	25E-NBOMe	50	25E-NBOMe	45
14	AB-FUBINACA	50	AB-FUBINACA*	65
15	3C-B-fly	50	3C-B-fly	57
16	para-Fluorofentanyl	50	para-Fluorofentanyl	46
17	blank		None	-
18	N,N-dimethylcathinone	50	N,N-dimethylcathinone	45
	Phenylpiperazine	50	Phenylpiperazine	53

*Isomers of these compounds were also detected

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Table 24. The true identity and concentration and experimental identity and concentration of blind spiked urine samples included in set 2 (varied concentrations)

#	Compound Spiked	Conc. Spiked (ng/mL)	Compound Detected	Conc. Detected (ng/mL)
1	Benocyclidine	5	Benocyclidine	5
	JWH 073 2'-naphthyl-N-(1,1-dimethyl) isomer	5	JWH 073 2'-naphthyl-N-(1,1-dimethyl) isomer	6
	4-hydroxy MiPT	5	4-hydroxy MiPT	6
	JWH 200 5-hydroxyindole metabolite	35	JWH 200 5-hydroxyindole metabolite	36
	XLR11 N-(2-fluoropentyl) isomer	35	XLR11 N-(2-fluoropentyl) isomer	32
	PB-22	35	PB-22*	70
	MMAI	35	MMAI	32
	4-methoxy PV8	80	4-methoxy PV8	77
	2-fluoromethcathinone	80	2-fluoromethcathinone	50
2	N-Ethylbuphedrone	5	N-Ethylbuphedrone	10
	3-Methylbuphedrone	35	3-Methylbuphedrone	7
	RCS-4 2-methoxy isomer	35	RCS-4 2-methoxy isomer	35
	Etaqualone	35	Etaqualone	31
	PB-22 6-hydroxyisoquinoline isomer	80	PB-22 6-hydroxyisoquinoline isomer*	110
	3-methyl-α-Pyrrolidinopropiophenone	80	3-methyl-α-Pyrrolidinopropiophenone	66
	Δ8-THC	80	Δ8-THC	90
	XLR11 N-(2-fluoropentyl) isomer	80	XLR11 N-(2-fluoropentyl) isomer	91
3	AB-FUBINACA	5	AB-FUBINACA*	7
	4-Methyl-a-ethylaminobutiophenone	5	4-Methyl-α-ethylaminobutiophenone	7
	THCA-A	5	nd	-
	MAM2201 N-pentanoic acid metabolite	5	MAM2201 N-pentanoic acid metabolite	6
	3C-B-fly	35	3C-B-fly	33
	3-methyl-α-Pyrrolidinopropiophenone	35	3-methyl-α-Pyrrolidinopropiophenone	33
	3,4-dimethoxy-α-Pyrrolidinopentiophenone	35	3,4-dimethoxy-α-Pyrrolidinopentiophenone	27

	N,N-dimethylcathinone	35	N,N-dimethylcathinone	42
	AM694 N-(5-hydroxypentyl) metabolite	80	AM694 N-(5-hydroxypentyl) metabolite	72
4	25I-NBF	5	25I-NBF	9
	4'-Methyl-N-methylhexanophenone	5	4'-Methyl-N-methylhexanophenone	8
	2C-T-2	35	2C-T-2	37
	4-hydroxy MET	80	4-hydroxy MET	64
5	4'-fluoro-a-Pyrrolidinopropiophenone	5	nd	-
	A-796260	5	A-796260	7
	N-Methyltryptamine	35	N-Methyltryptamine	40

*Isomer of this compound were also detected.
nd - not detected.

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Finally, two sets of blind spiked whole blood samples were prepared and analyzed qualitatively and quantitatively. The first set was designed to test the lower limits of the validated method, while the second set was designed to be in the middle of the calibration curve. The experimental identity, experimental concentration, true identity, and true concentration for the 15 blind spikes included in the first set are shown in Table 25. All samples in the first set were extracted using an in-house mini one-pot QuEChERS approach. The only sample that was not properly identified was sample 1, which should have been identified as THCA-A, but was not. This was likely due to the fact that THCA-A was one of the few compounds that did not show linearity and therefore this method was not capable of properly identifying that compound. However, there were 3 samples that were determined to be blank even though they all contained one compound. Additionally, sample 5 contained two compounds but only one was identified. Even though the dMRM method used for analysis was validated specifically for urine, the whole blood spiked samples were also quantitated. Many of the compounds were identified as higher concentrations than the true value this could be due to matrix effects that differ from those seen with urine.

The second set was designed to be in the middle of the calibration curve. The experimental identity, experimental concentration, true identity, and true concentration for the 18 blind spikes included in the second set are shown in Table 26. All samples in the second set were extracted prepared using a crash-and-shoot approach. The only sample that was not properly identified was sample 1, which should have been identified as THCA-A, but was not. This was likely due to the fact that THCA-A was one of the few compounds that did not show linearity. Experimental concentrations for the majority

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of compounds fell within ±20 nominal showing that the method is capable of quantifying whole blood extracts with similar percent error to those seen with urine samples.

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Table 25. The true identity and concentration and experimental identity and concentration of blind spiked whole blood samples included in set 1 (low concentration)

#	Compound Spiked	Conc. spiked (ng/mL)	Compound Detected	Conc. Detected (ng/mL)
1	THCA-A	5	JWH 200 5-hydroxyindole metabolite	N/A
2	4-APDB	5	-	-
3	JWH 200 5-hydroxyindole metabolite	5	JWH 200 5-hydroxyindole metabolite	9
4	N-Methyltryptamine	5	-	-
5	3-Methylbuphedrone	5	3-Methylbuphedrone	2
	5-fluoro SDB-006	5	-	-
6	2-fluoromethcathinone	5	-	-
7	2C-T-4	5	2C-T-4	3
8	PB-22	5	PB-22*	8
9	4-ethyl-N,N-dimethylcathinone	5	4-ethyl-N,N-dimethylcathinone	5
10	JWH 018 N(5-hydroxypentyl) metabolite	5	JWH 018 N(5-hydroxypentyl) metabolite	8
	MAM2201 N-pentanoic acid metabolite	5	MAM2201 N-pentanoic acid metabolite	5
11	25E-NBOMe	5	25E-NBOMe	4
12	AB-FUBINACA	5	AB-FUBINACA*	9
13	3C-B-fly	5	3C-B-fly	6
14	Para-Fluorofentanyl	5	Para-Fluorofentanyl	4
15	N,N-dimethylcathinone	5	N,N-dimethylcathinone	2
	Phenylpiperazine	5	Phenylpiperazine	4

*Isomer of this compound were also detected

Table 26. The true identity and concentration and experimental identity and concentration of blind spiked whole blood samples included in set 2 (medium concentration)

#	Compound Spiked into Urine	Conc. spiked (ng/mL)	Compound Detected	Conc. Detected (ng/mL)
1	THCA-A	50	Benzydamine	N/A
2	4-APDB	50	4-APDB	98
3	JWH 200 5-hydroxyindole metabolite	50	JWH 200 5-hydroxyindole metabolite	34
4	blank	0	None	-
5	N-Methyltryptamine	50	N-Methyltryptamine	46
6	3-Methylbuphedrone	50	3-Methylbuphedrone	8
	5-fluoro SDB-006	50	5-fluoro SDB-006	24
7	2-fluoromethcathinone	50	2-fluoromethcathinone	47
8	2C-T-4	50	2C-T-4	55
9	PB-22	50	PB-22*	66
10	blank	0	None	-
11	4-ethyl-N,N-dimethylcathinone	50	4-ethyl-N,N-dimethylcathinone	12
12	JWH 018 N(5-hydroxypentyl) metabolite	50	JWH 018 N(5-hydroxypentyl) metabolite*	36
	MAM2201 N-pentanoic acid metabolite	50	MAM2201 N-pentanoic acid metabolite	26
13	25E-NBOMe	50	25E-NBOMe	40
14	AB-FUBINACA	50	AB-FUBINACA*	43
15	3C-B-fly	50	3C-B-fly	37
16	para-Fluorofentanyl	50	para-Fluorofentanyl	23
17	blank	0	None	-
18	N,N-dimethylcathinone	50	N,N-dimethylcathinone	6
	Phenylpiperazine	50	Phenylpiperazine	41

*Isomer of this compound were also detected

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In a final test of applicability, a total of 50 authentic urine specimens were qualitatively analyzed using a method with transitions for all of the 16 standard mixes, therefore allowing screening for any of the 729 compounds included. Table 27 shows the identity of the compounds found and the number of specimens that were positive for each compound, while Table 28 shows the identity of metabolites found following glucuronidase treatment and the number of specimens positive for each. A general toxicology screen looking for common drugs of abuse and their metabolites was completed in addition to the NPS screen. The results of the general screening are shown in Table 29. The results of the NPS screen and general screening for each of the 50 authentic specimens can be seen in Table 30. Cathine and levamisole were present in the highest number of specimens. Cathine is a metabolite of pseudophedrine and likely represents use of this common over the counter (OTC) drug rather than direct ingestion of cathine.[119] Levamisole is a common adulterant of cocaine, which explains why it is present in the majority of samples that were positive for beonzoylecgonine. Interestingly, several NPS/metabolites were also confirmed present in at least one specimen, illustrating the potential value of this method for identification of compounds not typically screened for in forensic specimens.

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Table 27. The identity of compounds found in authentic specimens and the number of positive specimens for each compound.

Compound Detected	Number of Positives
Levamisole	12
Cathine	11
6-IT	5
2C-I	4
4-methoxy-N,N-dimethylcathinone	4
Hydroxy Bupropion	4
Sildenafil Citrate	4
3-Bromomethcathinone	3
4-FMC	3
3-fluoromethcathinone	2
3-methoxyamphetamine	2
6-APB	2
Mescaline	2
2,3-Dichlorophenylpiperazine	1
4-acetoxy DMT	1
4-hydroxy MET	1
4-methyl-α-ethylaminobutiophenone	1
Benzydamine	1
Deoxypipradol	1
Loperamide	1
Propylhexedrine	1

Table 28. The identity of metabolites found in authentic specimens following glucuronidase treatment and the number of positive specimens for each compound.

Compound Detected	Number of Positives
4-fluoromethcathinone metabolite	10
Buphedrone metabolite	5
Pentedrone metabolite	4
JWH 200 7-hydroxyindole metabolite	3
JWH 073 5-hydroxyindole metabolite	1

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Table 29. The identity of compounds found in authentic specimens after a general screening and the number of positive specimens for each compound.

Compound Detected	Number of Positives
Pregabalin	46
Morphine	19
Buprenorphine	17
Norbuprenorphine	16
Gabapentin	14
Ritalinic Acid	10
Dextroprhan	9
Oxazepam	8
Oxycodone	8
Alprazolam	7
Beonzoylecgonine (cocaine)	6
OH-Alprazolam	6
Oxymorphone	6
Temazepam	6
Doxepin	5
Nordiazepam	5
Ethylmorphine	4
Hydrocodone	4
Amphetamine	3
Hydromorphone	3
M6G	3
Methamphetamine	3
Norephedrine	3
Dextromethorphan	2
EDDP	2
Methadone	2
C6G	1
Codeine	1
Lorazepam	1
MDMA	1
Mitragynine	1
OH-Mitragynine	1
PMMA	1
THC	1
Tramadol	1

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Table 30. The identity of compounds found in each individual authentic specimen during the general screening and the NPS screening

Specimen #	Compounds Detected in NPS Screen	Compounds Detected in Routine Screen
513	cathine, levamisole	beonzoylecgonine, gabapentin, MDMA, norephedrine, pregabalin, ritalinic acid, tramadol
514	cathine, levamisole, 3-fluoromethcathinone, 4-FMC, 4-methoxy-N,N-dimethylcathinone	beonzoylecgonine, buprenorphine, hydrocodone, oxycodone, oxymorphone, pregabalin
515	cathine	alprazolam, amphetamine, methamphetamine, norephedrine, oh-alprazolam, pregabalin, THC
516	3-methoxyamphetamine	hydrocodone, pregabalin
517	3-methoxyamphetamine	alprazolam, doxepin, EDDP, methadone, oxazepam, pregabalin, temazepam
518	levamisole, hydroxy bupropion	doxepin, pregabalin
519	levamisole, 4-acetoxy DMT	beonzoylecgonine, buprenorphine, doxepin, morphine, norbuprenorphine, pregabalin
520	4-hydroxy MET, 4-methoxy-N,N-dimethylcathinone	doxepin, morphine, oxazepam, pregabalin
521	hydroxy bupropion, propylhexedrine	pregabalin
522	2,3-dichlorophenylpiperazine, deoxypipradol, loperamide	morphine, pregabalin
523	sildenafil	morphine, pregabalin
524	nd	buprenorphine, gabapentin, norbuprenorphine, oxymorphone, pregabalin, ritalinic acid
525	sildenafil	pregabalin, ritalinic acid
526	sildenafil	buprenorphine, gabapentin, norbuprenorphine, pregabalin
527	mescaline	alprazolam, EDDP, methadone, oh-alprazolam, pregabalin
528	nd	pregabalin
529	mescaline,	ethylmorphine, gabapentin, pregabalin
530	nd	pregabalin
531	cathine, levamisole	pregabalin
532	nd	pregabalin
533	sildenafil	morphine, pregabalin, ritalinic acid

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534	cathine, 4-FMC	buprenorphine, dextromethorphan, dextroprhan, gabapentin, norbuprenorphine
535	nd	C6G, codeine, dextroprhan, M6G, mitragynine, morphine, oh-mitragynine, pregabalin
536	3-fluoromethcathinone	buprenorphine, dextroprhan, morphine, norbuprenorphine, pregabalin, ritalinic acid
537	nd	buprenorphine, dextroprhan, norbuprenorphine, nordiazepam, oxazepam, pregabalin, temazepam
538	nd	dextroprhan, pregabalin
539	3-bromomethcathinone, cathine	amphetamine, buprenorphine, dextroprhan, lorazepam, morphine, norbuprenorphine, nordiazepam, norephedrine, oxazepam, PMMA, pregabalin, temazepam
540	hydroxy bupropion	ethylmorphine, pregabalin
541	4-methoxy-N,N-dimethylcathinone, 6-IT	amphetamine, pregabalin
542	cathine, 4-FMC	alprazolam, buprenorphine, dextroprhan, hydrocodone, norbuprenorphine, nordiazepam, oh-alprazolam, oxazepam, oxycodone, oxymorphone, pregabalin, temazepam
543	6-IT	buprenorphine, dextroprhan, ethylmorphine, gabapentin, morphine, pregabalin, ritalinic acid
544	levamisole, benzydamine	alprazolam, buprenorphine, ethylmorphine, hydromorphone, M6G, morphine, norbuprenorphine, nordiazepam, oh-alprazolam, oxazepam, oxymorphone, pregabalin, temazepam
545	cathine	alprazolam, buprenorphine, morphine, norbuprenorphine, oh-alprazolam, pregabalin
546	nd	pregabalin
547	levamisole, 6-IT	beonzoylecgonine, buprenorphine, dextromethorphan, dextroprhan, gabapentin, methamphetamine, morphine, norbuprenorphine
548	levamisole, 4-methoxy-N,N-dimethylcathinone	buprenorphine, doxepin, morphine, norbuprenorphine, pregabalin
549	cathine, levamisole	pregabalin, ritalinic acid
550	nd	hydrocodone, morphine, pregabalin
551	4-methyl-α-ethylaminobutiophenone	pregabalin

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552	levamisole	beonzoylecgonine, buprenorphine, morphine, norbuprenorphine, pregabalin
553	3-bromomethcathinone	buprenorphine, gabapentin, morphine, norbuprenorphine, nordiazepam, oxazepam, PMMA, pregabalin, temazepam
554	cathine	alprazolam, methamphetamine, oh-alprazolam, pregabalin
555	cathine	pregabalin
556	hydroxy bupropion	nd
557	nd	beonzoylecgonine, buprenorphine, gabapentin, hydromorphone, morphine, norbuprenorphine, oxycodone, oxymorphone, pregabalin, ritalinic acid
558	nd	gabapentin, hydromorphone, M6G, morphine, oxymorphone, ritalinic acid
559	levamisole	gabapentin, morphine, oxazepam, pregabalin, ritalinic acid
560	levamisole	gabapentin, pregabalin
596	3-bromomethcathinone	gabapentin, pregabalin
597	nd	gabapentin, pregabalin

nd - no detections

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6.3 Conclusions

In order to further test the applicability of the validated dMRM method blind spiked samples in urine and whole blood and authentic urine specimens analyzed. Testing blind spikes and authentic specimens made it possible to determine the method's potential as both a screening and confirmatory method. The results from screening and confirmation of the blind spiked samples it was shown that the dMRM method has potential to be used as a confirmatory method for samples in urine and a screening method for samples in whole blood. Due to the number of NPS included in this method it is a semi-qualitative method because it is impossible to match every compound with a deuterated internal standard. Since some compounds do not have an ideal match when quantitated the compound may undergo different matrix effects and ionization than the paired internal standard. However, the results from the blind spike studies and authentic specimens with the validated method indicate that it shows great potential as a comprehensive screening method for the the largest number of NPS reported to date.

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7. SUMMARY AND PROSPECT

Novel psychoactive substances (NPS) have gained popularity over the past two decades all over the world and it does not seem that there will be an end soon. The increased popularity of NPS makes it imperative that clinical and forensic toxicological laboratories have access to reliable comprehensive screening methods for NPS. Unlike with common drugs of abuse, immunoassays are not capable of selectively detecting NPS due to their multiple structural alterations. Immunoassays are one of the most common screening methods for clinical and forensic human specimens. However, they need to be replaced by methods capable of reliably screening for a large number of NPS within varying drug classes. Alternative screening methods do exist, some of which are capable of detecting NPS. Instrumental screening methods (*i.e.,* GC-MS and LC-MS) can be used to screen clinical and forensic toxicology specimens. These methods typically work based on spectral library or database matches. It is vital that spectral libraries and databases exist that include NPS in order to properly screen for them in clinical and forensic toxicological samples.

The goal of the research presented here was to aid in the screening and confirmation of NPS in clinical and forensic toxicological specimens. An MRM transition ion database and a comprehensive screening and confirmatory dMRM method for the detection of NPS in biological matrices were created and are the largest of their kind. In addition to the creation of a dMRM method, validation using a mixture approach was completed to ensure that method parameters fell within OSAC guidelines. Often, method validation is done

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using small mixtures or a one-at-a-time approach, however, this was not feasible for the quantity of NPS that this method was designed to screen for. Consequently, an approach using a series of mixtures of non-coeluting NPS standards was adapted in order to greatly reduce the time it takes to fully validate a comprehensive screening method. Blind spiked urine samples were screened and quantitated using the described dMRM method. This was done to further validate the selectivity and sensitivity of the method as it would be used in the field of forensics. The majority of NPS were correctly identified and most concentrations were determined to be within ±20% of the spiked concentration showing the selectivity and sensitivity of the overall method. Additionally, the dMRM method was used to screen 50 authentic urine specimens to show real world relevance of the method. From the 50 specimens 21 compounds were detecting including NPS (synthetic cathinones) showing the potential of this method for clinical and forensic toxicological specimens.

It is not uncommon that biological matrices must undergo extraction and/or purification before they can be injected into an instrument and analyzed. Consequently, this project also aimed evaluate extraction techniques for NPS in whole blood and urine. Depending on the complexity of the matrix, developing and optimizing an extraction technique can be very difficult. The research described here was designed to determine if particular extraction methods were more efficient than others for the extraction of NPS from whole blood and urine. Forensic toxicological laboratories in general have standard extraction procedures in place for common drugs of abuse, typically involving protein precipitation and/or SPE. The procedure of protein precipitation does not differ from one compound to the next, however SPE involves complex chemistry and relies on pH and pKa

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of the compounds in the sample. Therefore, SPE and potentially other extraction techniques need to be optimized for NPS. This research delved into the usefulness of dilute-and-shoot, crash-and-shoot, online-SPE, classical SPE, and QuEChERS for the extraction of NPS from biological matrices.

This work investigated the potential of on-line SPE and QuEChERS as alternative extraction technique for NPS, since neither technique has been commonly used in forensic toxicology laboratories. QuEChERS is an appealing technique for complex matrices and is more time and cost efficient than SPE. On-line SPE is much more time efficient but was found to involve extremely complex method development. QuEChERS is an appealing alternative to traditional extraction techniques, since it can easily be implemented into different clinical and forensic toxicological laboratories with simple purchase of reagents but not requiring additional instrumentation. The one disadvantage of QuEChERS as compared to classical SPE is the elimination of matrix effects. This is an important factor to consider when determining the needs of an extraction technique.

Future work will be necessary in order to update and expand upon the database and dMRM method as more NPS are reported in literature, however, this will require the availability of appropriate reference standards. Further optimization of QuEChERS would be needed to increase the elimination of matrix effects with the goal of having comparable results to that of SPE. When considering the time and cost efficiency of QuEChERS, it would be a beneficial extraction technique to be implemented into forensic toxicology laboratories. Since QuEChERS is designed for complex matrices it has even further

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potential to be used to extract NPS from biological matrices other than urine and whole blood.

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APPENDICES

Appendix 1. The compound name, chemical formula, unique in-house identifier, retention time, precursor ion, all product ions and associated collision energies and abundances for all compounds in the final dMRM method separated by mixture number.

Mix #	Compound Name	Chemical Formula	In House ID	RT (min)	Precursor Ion	Product Ion	CE	Abundance
1	**25I-NBMD**	C18H20INO4	**FIU_0379**	9.11	**442.04**	298.1	**16**	26142
						291	**20**	96047
						286.1	**16**	196843
						276	**32**	32969
						174.1	**8**	702207
						164.1	**28**	18476
						145.1	**20**	376605
						135.1	**32**	1196209
						105.1	**60**	73216
						79.1	**60**	232510
						77.1	**60**	326616
						51.1	**60**	24342
	3-Methylbuphedrone	C12H17NO	**FIU_0425**	7.17	**192.13**	161.1	**8**	344234
						161.1	**8**	141099

						159.1	**20**	99200
						146.1	**16**	200785
						144.1	**36**	376141
						105.1	**20**	180964
						91.1	**48**	84682
						77.1	**60**	126436
						65.1	**60**	87906
	4-APDB	C11H15NO	**FIU_0417**	6.44	**178.11**	133.1	**20**	270092
						120.1	**24**	24257
						119.1	**12**	24730
						105.2	**28**	38710
						103.1	**40**	27934
						91.1	**48**	34474
						79.2	**40**	37856
						77.2	**52**	85321
						51.2	**60**	30663
	4-fluoro-a-Pyrrolidinobutiophenone	C14H18FNO	**FIU_0397**	6.74	**236.13**	165.1	**16**	267311
						137.1	**20**	144229
						123	**32**	157117

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						112.1	**24**	262664
						109.1	**28**	458875
						95.1	**56**	207940
						84.1	**32**	113650
						75.1	**60**	89763
						70.1	**20**	99710
						55.1	**48**	44392
	4'-fluoro-a-Pyrrolidinopropiophenone	C13H16FNO	**FIU_0399**	6.19	**222.12**	151.1	**16**	232987
						123.1	**24**	349431
						103.1	**36**	209745
						98.1	**28**	404864
						95.1	**52**	64672
						84.2	**32**	40979
						77.1	**60**	183706
						70.1	**16**	88531
						56.1	**56**	138769
						55.7	**48**	58514
	4-hydroxy MET	C13H18N2O	**FIU_0749**	5.45	**219.14191**	160	**16**	118064

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						142	**32**	6053
						132	**32**	18540
						131.5	**36**	2504
						117	**40**	19684
						115	**48**	75184
						89	**60**	19173
						77.1	**60**	7807
						72.1	**12**	334783
						65.1	**60**	11123
	4-methoxy PV8	C18H27NO2	**FIU_0411**	8.69	**290.20418**	219.2	**16**	485978
						154.2	**28**	619924
						135.1	**32**	263495
						121.1	**24**	896340
						107.1	**44**	72222
						91.1	**60**	64198
						84.2	**48**	175841
						77.2	**60**	312126
						69.2	**60**	72277
						55.2	**60**	65725

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	4-methoxy-a-Pyrrolidinopentiophenone	C16H23NO2	**FIU_0401**	7.57	**262.17288**	191.1	**16**	530903
						188.2	**8**	527600
						135	**32**	232327
						126.1	**24**	450678
						121.1	**24**	684502
						107.1	**44**	56103
						97.1	**48**	60685
						91.7	**60**	88344
						84.1	**40**	125580
						77.1	**60**	305935
						55.2	**56**	55004
	4-Methyl-a-ethylaminobutiophenone	C13H19NO	**FIU_0402**	7.36	**206.14666**	160	**16**	203080
						159.1	**20**	305315
						144.1	**32**	392813
						132.1	**20**	74175
						130.1	**44**	62924
						105.1	**28**	209488
						91.1	**48**	117533

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						77.1	**60**	100580
						65.1	**60**	97836
	5-fluoro SDB-006	C21H23FN2O	**FIU_0440**	10.29	**339.17944**	232.1	**20**	592602
						206.1	**20**	404949
						144	**40**	187950
						132.1	**32**	99024
						118.1	**36**	138347
						116.1	**60**	70792
						91.1	**56**	826346
						69.2	**40**	41150
						65.1	**60**	129623
						55.2	**52**	6729
	5-MAPB	C12H15NO	**FIU_0420**	6.94	**190.11536**	159.1	**8**	861733
						131.1	**20**	1009047
						129.1	**28**	27045
						116.1	**32**	67569
						115.1	**52**	92822
						103.1	**40**	48032
						91.1	**36**	226735

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						77.1	**56**	276351
						65.2	**60**	92666
						51.2	**60**	81525
	AB-005	C23H32N2O	**FIU_0712**	9.38	**353.25146**	256.1	**20**	66931
						229.1	**16**	33336
						158	**40**	11982
						125	**20**	169410
						112	**24**	258186
						98	**36**	291984
						83	**32**	13003
						70	**60**	94389
						58.1	**56**	66682
						55.1	**52**	51963
	AM2233 azepane isomer	C22H23IN2O	**FIU_0633**	8.45	**459.08551**	230.9	**32**	76284
						202.9	**60**	50645
						112.1	**24**	1039252
						98.1	**32**	149448
						84.1	**60**	22846
						81.1	**60**	20338

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						76.1	**60**	19551
						70.1	**60**	68784
						58.1	**60**	238730
						55.2	**60**	23624
	AM694 N-(5-hydroxypentyl) metabolite	C20H20INO2	**FIU_0698**	9.98	**434.05387**	234.1	**40**	12567
						230.9	**20**	850211
						220	**40**	11943
						202.9	**56**	312614
						186.1	**12**	69936
						144	**56**	13714
						130	**44**	6020
						104.7	**60**	11314
						104	**60**	50017
						76	**60**	156720
	a-Pyrrolidinopentiophenone metabolite 1	C15H23NO	**FIU_0407**	7.81	**234.17796**	216.2	**16**	297432
						215.1	**8**	640731
						173.1	**24**	106962

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						145.1	**20**	51346
						117.1	**28**	27466
						103.1	**36**	23254
						91.1	**36**	93734
						79.1	**40**	66749
						77.1	**60**	73524
						72.1	**20**	448673
						57.2	**28**	31397
	Benocyclidine	C19H25NS	**FIU_0391**	8.89	**300.17077**	226.1	**32**	7418
						173	**32**	15404
						147	**32**	620996
						135	**28**	17365
						103.1	**60**	36467
						86.1	**4**	450750
						81.1	**32**	77341
						79.1	**60**	22182
						69.1	**60**	34782
						67.1	**32**	27904
	Flubromazepam	C15H10BrFN2O	**FIU_0678**	9.71	**332.99605**	211	**32**	745

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						206.1	**48**	628
						184	**32**	6882
						179.1	**56**	4337
						105.1	**52**	3359
						104.1	**60**	4786
	JWH 081 N-(5-hydroxypentyl) metabolite	C25H25NO3	**FIU_0516**	10.51	**388.18344**	230.1	**28**	186558
						185.1	**20**	2390607
						157.1	**48**	705961
						144	**40**	224925
						142	**60**	297044
						128.3	**60**	161942
						127	**60**	456377
						116	**60**	68151
						114	**60**	136197
						69.1	**40**	61715
	JWH 200 5-hydroxyindole metabolite	C25H24N2O3	**FIU_0531**	8.29	**401.17869**	160.1	**36**	3014
						155	**20**	564845

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						127	**60**	252431
						114.1	**32**	308841
						100.1	**60**	7421
						86.1	**56**	7861
						84.1	**52**	21667
						70.1	**60**	68606
						68.1	**60**	3790
						56.2	**60**	6072
	JWH 203	C21H22ClNO	**FIU_0534**	11.36	**340.13899**	214.1	**28**	108406
						188.1	**20**	172257
						144	**44**	108807
						132.1	**32**	44098
						130	**52**	55877
						125	**28**	1084924
						118.1	**36**	23717
						116	**60**	50535
						99	**60**	41926
						89.1	**60**	117666
	N-Methyltryptamine	C11H14N2	**FIU_0756**	5.98	**175.1157**	144	**8**	263533

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						132	**8**	54859
						127	**32**	21000
						117	**28**	47145
						115	**44**	37217
						91	**44**	24577
						90	**56**	13644
						89	**60**	21090
						77	**48**	15813
						65.1	**60**	13445
	NPB-22	C22H21N3O2	**FIU_0595**	10.95	**360.16338**	215.1	**16**	1353981
						145	**40**	836951
						129.7	**60**	114
						117	**60**	103623
						90.1	**60**	177918
						71.2	**36**	11694
	PB-22 6-hydroxyisoquinoline isomer	C23H22N2O2	**FIU_0603**	11.16	**359.16813**	214.1	**16**	536563
						158	**40**	6693
						144	**44**	223678

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						130	**48**	4767
						116	**60**	68262
						89	**60**	7707
						71.1	**40**	3705
						55.1	**60**	2183
	PCMPA	C16H25NO	**FIU_0389**	8.04	**248.19361**	159.1	**12**	683975
						117.1	**28**	32506
						91.1	**40**	860073
						81.1	**20**	127867
						79.1	**52**	17255
						73.1	**24**	35540
						67.1	**20**	20122
						65.1	**60**	172066
						58.2	**28**	175286
						55.1	**40**	11968
	THCA-A	C22H30O4	**FIU_0453**	13.22	**359.21441**	341.2	**12**	45770
						234.3	**36**	103
						219.1	**36**	9468
						211.3	**36**	262

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						203.2	**36**	807
						193.2	**24**	21943
						69.2	**52**	1848
						55.2	**60**	2014
2	**?8-THC**	C21H30O2	**FIU_0454**	12.27	**315.22458**	135.1	**20**	16535
						123.1	**36**	18191
						107.1	**36**	6320
						93.1	**24**	10781
						91.1	**56**	7223
						81.2	**48**	4993
						77.2	**60**	7819
						69.2	**36**	6143
						67.2	**56**	6716
	2,3-methylenedioxy pyrovalerone	C16H21NO3	**FIU_0108**	7.29	**276.2**	175	**20**	2047695
						149	**32**	525363
						135	**24**	2924163
						126.1	**32**	1693388
						84.1	**44**	397748
						79.1	**48**	264551

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						77.1	**60**	669203
						70.1	**20**	235520
						65.1	**60**	830244
						55.1	**56**	235166
	25I-NBF	C17H19FINO2	**FIU_0378**	9.04	**416.04445**	291	**20**	681306
						275.9	**32**	257739
						260.9	**48**	121283
						164.1	**28**	149900
						149.1	**40**	126534
						134.1	**40**	111284
						121.1	**52**	78130
						109.1	**56**	252726
						104.1	**52**	78022
						91.1	**60**	135377
	2C-T-2	C12H19NO2S	**FIU_0146**	7.88	**242.1**	225.1	**8**	1011348
						210.1	**16**	132670
						195	**24**	64849
						164.1	**20**	85218
						134.1	**28**	96527

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						121	**36**	48444
						119	**36**	47882
						91.1	**52**	162391
						77.1	**60**	86829
						59	**40**	51093
	2C-T-4	C13H21NO2S	**FIU_0020**	8.57	**256.1**	239.1	**8**	1739095
						197.1	**16**	1081480
						182	**24**	429416
						167	**36**	341836
						164.1	**24**	214683
						164.1	**12**	462601
						134.1	**36**	171171
						121	**44**	106071
						119	**44**	89011
						91.1	**56**	328543
						77.1	**60**	117332
	2-Fluoromethcathinone	C10H12FNO	**FIU_0117**	11.65	**182.1**	149	**20**	323785
						148	**36**	197216
						123	**20**	64393

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						103	**32**	83565
						101	**56**	45896
						77.1	**48**	111780
						75.1	**60**	65655
						58.1	**36**	20636
						51.1	**60**	44179
	3,4-dimethoxy-a-Pyrrolidinopentiophenone	C17H25NO3	**FIU_0393**	7.09	**292.18344**	221.1	**16**	471584
						165	**28**	200975
						151.1	**28**	732198
						126.1	**24**	609709
						107	**60**	106169
						97.1	**52**	55242
						84.1	**40**	113952
						77.1	**60**	124661
						69.1	**60**	67566
						55.2	**60**	60492
	3-methyl-a-Pyrrolidinopropiophenone	C14H19NO	**FIU_0395**	6.88	**218.14666**	202.2	**8**	621004

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						147.1	**16**	310958
						146.1	**16**	407197
						119.1	**24**	518374
						117.1	**36**	91511
						98.1	**28**	332567
						91.1	**48**	253525
						77.1	**60**	97633
						70.1	**20**	100738
						65.1	**60**	90449
						56.2	**56**	122555
						55.1	**48**	55536
	4’-Methyl-N-methylhexanophenone	C14H21NO	**FIU_0396**	8.35	**220.16231**	189.1	**8**	198304
						161.1	**12**	1426357
						158.1	**36**	140871
						145.1	**24**	242869
						144.1	**40**	268172
						133.1	**20**	1419465
						131.1	**28**	85902
						105.1	**24**	275429

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						91.1	**48**	118296
						77.1	**60**	111364
	4-ethyl-N,N-dimethylcathinone	C13H19NO	**FIU_0138**	11.65	**206.2**	151	**4**	145310
						143.1	**16**	190223
						123	**16**	111010
						105.1	**28**	1181578
						103.1	**48**	167663
						103.1	**28**	87754
						91.1	**36**	138899
						79.1	**44**	313607
						77.1	**60**	489298
						72.1	**28**	1381308
						58.1	**32**	93347
	4-fluorousovathinone (hydrochloride)	C9H10FNO	**FIU_0141**	11.66	**168.1**	135.1	**24**	694
						95	**44**	4014
						77.1	**40**	80048
						75.1	**60**	13026
						51.1	**60**	28800

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	4-hydroxy MiPT	C14H20N2O	**FIU_0750**	5.85	**233.15756**	160.1	**8**	3375994
						160	**20**	141533
						132	**32**	19884
						131.5	**36**	2923
						117	**44**	25703
						115	**44**	88696
						105	**44**	6389
						89	**60**	16625
						86.1	**12**	315763
						77	**60**	8381
						65.1	**60**	9635
	4-methylmethcathinone (Mephedrone/4-MMC)	C11H15NO	**FIU_0006**	6.66	**178.1**	145.1	**20**	2302144
						144.1	**36**	1455458
						130	**32**	140954
						119	**20**	309913
						115	**52**	136339
						103.1	**48**	158629
						91.1	**40**	400797
						77.1	**60**	495361

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						65.1	**60**	262038
	A-796260	C22H30N2O2	**FIU_0441**	9.77	**355.23073**	125.1	**20**	1119736
						114.1	**32**	519024
						100.1	**56**	15824
						97.1	**36**	125151
						84.1	**52**	46333
						83.2	**40**	44130
						70.1	**52**	146725
						69.1	**44**	74507
						57.2	**56**	137447
						55.2	**56**	238364
	AB-005 azepane isomer	C23H32N2O	**FIU_0713**	9.5	**353.25146**	352.1	**4**	42452
						324.1	**12**	53475
						253	**24**	44169
						125	**20**	45915
						112	**24**	657114
						98.1	**32**	20312
						84.1	**56**	17451
						81	**52**	11750

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						79	**60**	6790
						70	**60**	47807
						58.1	**56**	204217
						56.1	**60**	7589
						55.1	**60**	42897
	ADB-PINACA isomer 1	C19H28N4O2	**FIU_0734**	10.73	**345.22123**	328.2	**4**	274056
						300.2	**12**	247817
						215.1	**24**	314954
						209	**24**	61
						145	**48**	177721
						117	**60**	19264
						90	**60**	22848
						89.5	**60**	9735
						71.1	**44**	3018
	AKB48 N-(5-fluoropentyl) analog	C23H30FN3O	**FIU_0727**	11.85	**384.23729**	135.1	**24**	470715
						107	**56**	41915
						93	**60**	67050
						91	**60**	11533
						81.1	**60**	19852

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						79	**60**	57951
						77	**60**	11623
						69.1	**60**	5924
						67.1	**60**	24116
						55.1	**60**	9996
	EG-018	C28H25NO	**FIU_0468**	12.73	**392.19361**	350.1	**40**	68
						264.1	**24**	81057
						236.1	**36**	5998
						233.4	**20**	69
						194	**44**	6117
						179.1	**52**	66432
						166.1	**56**	13400
						155.1	**24**	1066831
						127.1	**60**	762090
						77.2	**60**	9722
	JWH 018 N-(5-hydroxypentyl) metabolite	C24H23NO2	**FIU_0484**	10.32	**358.17288**	230.1	**28**	35272
						208.9	**24**	108
						155	**24**	625456

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						155	**20**	1330282
						144	**40**	61181
						127	**52**	867032
						116	**60**	23187
						77.1	**60**	14406
						69.1	**40**	17352
	JWH 018 N-propanoic acid metabolite	C22H17NO3	**FIU_0487**	9.97	**344.12084**	284.2	**24**	8653
						216.1	**24**	122192
						144	**44**	37083
						133.2	**16**	280
						127.9	**60**	6280
						127	**56**	532594
						116.1	**60**	20701
						77.1	**60**	16504
						73.1	**48**	8391
	KM 233	C25H30O2	**FIU_0459**	12.04	**363.22458**	302.1	**16**	63
						194.4	**60**	174
						191.3	**32**	200
						163.1	**32**	4167

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						135.2	**28**	3040
						119.1	**20**	468436
						93.2	**44**	3440
						91.1	**60**	289710
						79.2	**56**	11550
						65.1	**60**	6694
	Loperamide	C29H33ClN2O2	**FIU_0764**	9.25	**477.22306**	266.1	**24**	495262
						238.1	**52**	18421
						223.1	**60**	2768
						222.1	**60**	3490
						210.1	**60**	234375
						193.1	**56**	5052
						178	**60**	7396
						167.1	**60**	9271
						115	**60**	18840
						72.1	**60**	58975
	MAM2201 N-pentanoic acid metabolite	C25H23NO3	**FIU_0643**	10.53	**386.16779**	244.1	**24**	52242
						174.1	**12**	530579
						169.9	**24**	50012

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						169.1	**24**	697090
						146.1	**16**	266630
						144	**40**	52536
						141.8	**52**	24097
						141.1	**48**	395051
						130.1	**32**	326747
						115.1	**60**	134388
						101.1	**36**	12951
						83.1	**40**	15759
						55.1	**56**	41321
	N-Ethylbuphedrone	C12H17NO	**FIU_0431**	6.46	**192.13101**	145.1	**20**	241546
						118.1	**24**	111048
						117.1	**32**	64030
						91.1	**32**	248108
						77.1	**60**	224389
						65.1	**60**	72657
						51.1	**60**	74430
	PB-22	C23H22N2O2	**FIU_0596**	11.18	**359.16813**	214.1	**8**	1938796
						158.1	**36**	22440

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						144	**40**	753166
						130.1	**48**	15143
						116	**60**	246065
						89.1	**60**	31084
						71.1	**40**	11476
						55.2	**60**	7274
	PCPr	C15H23N	**FIU_0390**	8.16	**218.18305**	159.1	**8**	680438
						117	**20**	25073
						115	**48**	14536
						91.1	**28**	738575
						81.1	**16**	102919
						79.1	**40**	12197
						67.1	**16**	15195
						65.1	**60**	235075
						60.2	**4**	488820
						55.1	**40**	9181
	RCS-4 2-methoxy isomer	C21H23NO2	**FIU_0085**	10.97	**322.2**	144	**36**	40072
						135	**20**	6826092
						120	**52**	111190

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						107.1	**40**	53326
						105	**48**	105693
						92	**60**	751008
						79.1	**44**	401234
						77.1	**60**	3790055
						64.1	**60**	78160
						51.1	**60**	226015
	SER-601	C28H38N2O2	**FIU_0463**	13.21	**435.29333**	417.3	**16**	23680
						284.2	**28**	181091
						214.1	**48**	50506
						135.1	**32**	839440
						107.1	**60**	99991
						93.1	**60**	132123
						81.2	**60**	35563
						79.2	**60**	115502
						67.2	**60**	45300
						55.2	**60**	17024
	UR-144 Degradant	C21H29NO	**FIU_0645**	11.63	**312.22491**	214.1	**20**	302746
						206.9	**60**	69

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						158.1	**36**	4585
						144	**40**	129095
						130.1	**52**	3131
						116	**60**	46762
						89	**60**	11851
						83.1	**32**	4550
						71.2	**36**	2381
						55.2	**60**	10718
	XLR11 N-(2-fluoropentyl) isomer	C21H28FNO	**FIU_0661**	11.4	**330.21549**	312.2	**20**	11935
						232.1	**24**	33302
						144.1	**44**	11056
						130	**56**	4618
						125.1	**24**	83427
						97.1	**28**	17325
						83.1	**24**	12727
						69.1	**44**	13842
						57.2	**48**	19229
						55.1	**44**	42930

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3	**(-)-3,4-Methylenedioxy Pyrovalerone**	C16H21NO3	**FIU_0356**	7.73	**276.15214**	315.3	**4**	40769
						247.2	**12**	39282
						205.1	**16**	347586
						175.1	**20**	408998
						175.1	**4**	58816
						149	**32**	253614
						135.1	**28**	383560
						126.1	**28**	506535
						121	**48**	140927
						84.1	**40**	146297
						77.1	**60**	112730
						65.1	**60**	297826
						55.1	**56**	77494
	(+)-WIN 55,212-2 (mesylate)	C27H26N2O3	**FIU_0094**	10.79	**427.2**	328.1	**28**	80845
						299.1	**28**	62119
						212	**36**	52652
						200.1	**44**	72975
						175.1	**4**	108163

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						155	**24**	5725977
						127	**60**	2896559
						100.1	**48**	778004
						72.1	**60**	62162
						71.1	**12**	48803
						70.1	**60**	135203
						56.1	**60**	126552
	(±)-CP 47,497-C8-homolog	C22H36O2	**FIU_0098**	11.67	**333.3**	149.1	**24**	4793
						133	**52**	8726
						121	**28**	18823
						107	**20**	23500
						85.1	**12**	17066
						71.2	**16**	17695
						57.1	**36**	21577
	(R)-(-)-JWH 018 N-(4-hydroxypentyl) metabolite	C24H23NO2	**FIU_0472**	10.33	**358.17288**	340.2	**16**	11643
						284.2	**24**	18984
						230.1	**20**	11666
						186.2	**12**	21818

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						155.1	**20**	938068
						144.1	**40**	32667
						127.1	**56**	607512
						116.1	**60**	11799
						77.1	**60**	10917
						69.2	**40**	25517
	?9-THC	C21H30O2	**FIU_0455**	12.15	**315.22458**	193.1	**20**	46218
						135.2	**20**	20933
						123.1	**36**	29699
						107.1	**36**	11891
						93.1	**24**	18446
						91.1	**60**	11394
						81.2	**20**	11724
						69.2	**40**	13427
						67.2	**56**	12520
						55.2	**56**	10339
	2,3-pentylone isomer	C13H17NO3	**FIU_0026**	7.47	**236.1**	218.1	**8**	727720
						188.1	**12**	1974111
						175.1	**12**	857458

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						159.7	**24**	604545
						159.1	**28**	424298
						135	**20**	667530
						131.1	**40**	907532
						86.1	**20**	209571
						77.1	**60**	454804
						65.1	**60**	250844
	3C-B-fly	C13H16BrNO2	**FIU_0315**	8.49	**298.03644**	281	**12**	193399
						253	**24**	24972
						202.2	**8**	598438
						202.1	**24**	104498
						187.1	**36**	61286
						173.1	**36**	39237
						159.1	**40**	26262
						145.1	**52**	18648
						131.1	**56**	22939
						115.1	**60**	23843
						91.1	**60**	26066

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	4-Methyl-a-ethylaminopentiophenone	C14H21NO	**FIU_0403**	7.92	**220.16231**	324.1	**12**	72894
						175.1	**8**	216047
						160.1	**16**	250979
						159.1	**20**	155713
						144.1	**36**	398270
						132.1	**24**	148883
						105.1	**24**	258233
						91.1	**56**	156085
						77.1	**60**	94195
						65.1	**60**	117031
	AB-FUBINACA	C20H21FN4O2	**FIU_0715**	10.13	**369.16485**	352.1	**4**	57840
						330.1	**4**	70311
						302.1	**12**	72370
						253	**24**	59804
						109	**48**	60703
	BB-22 8-hydroxyisoquinoline isomer	C25H24N2O2	**FIU_0626**	11.64	**385.18378**	240.2	**20**	341445
						144	**44**	135629

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						116	**60**	21440
						97.1	**40**	20753
						69.1	**52**	11923
						55.2	**56**	116465
	Benzydamine	C19H23N3O	**FIU_0772**	8.69	**310.18411**	265.1	**12**	29173
						174	**28**	11795
						150.1	**24**	63
						146	**40**	4248
						91.1	**32**	8077
						86.1	**16**	520989
						85.6	**20**	52030
						71.1	**60**	7750
						58.1	**56**	285414
						56.1	**60**	4928
	Bromazepam	C14H10BrN3O	**FIU_0674**	8.91	**316.00072**	288	**20**	32641
						261	**24**	24219
						260	**36**	14712
						209.1	**28**	70869
						208.1	**40**	43604

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						184	**28**	19204
						182.1	**36**	107649
						105.1	**52**	12246
						80.1	**32**	18638
						78.1	**60**	13747
	Etaqualone	C17H16N2O	**FIU_0760**	9.93	**265.12626**	155	**24**	4476587
						146	**28**	171874
						131	**40**	72970
						130	**56**	56170
						118	**36**	33889
						117	**48**	25580
						106	**36**	14604
						105	**40**	23400
						103	**56**	26953
						79.1	**52**	47869
						77	**60**	89059
	JWH 019	C25H25NO	**FIU_0204**	11.87	**356.2**	228.1	**24**	820152
						158	**36**	14899
						144	**40**	504711

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						130	**52**	14808
						127	**56**	4018455
						116	**60**	188942
						89.1	**60**	26097
						77.1	**60**	98227
						57.1	**44**	27368
	JWH 073 2'-naphthyl-N-(1,1-dimethylethyl) isomer	C23H21NO	**FIU_0049**	11.42	**328.2**	272.2	**8**	782
						154.9	**28**	988
						144.2	**24**	890
						127.1	**44**	986
	JWH 073 6-methoxyindole analog	C24H23NO2	**FIU_0504**	11.21	**358.17288**	230.1	**24**	95875
						174.1	**40**	63493
						159.1	**56**	12528
						155	**24**	2648765
						146.1	**52**	31299
						131	**60**	12150
						127	**56**	1982006
						119.1	**60**	14586

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						77.1	**60**	44841
						57.1	**44**	6139
	JWH 198	C26H26N2O3	**FIU_0529**	9.71	**415.19434**	185.1	**24**	1071980
						170	**52**	7293
						157	**52**	258683
						142	**60**	85256
						127	**60**	123126
						114.1	**32**	430279
						100.1	**52**	9723
						86.1	**52**	11111
						84.1	**60**	33976
						70.1	**60**	103310
	Methylhexanamine	C7H17N	**FIU_0249**	6.99	**116.2**	99.1	**4**	16762
						57.1	**12**	352833
						55.2	**28**	1541
	MMAI	C11H15NO	**FIU_0759**	7.27	**178.11536**	161	**8**	114350
						146	**24**	17813
						131	**32**	14037
						128	**32**	5207

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						115	**52**	8603
						105.1	**20**	2191683
						105	**24**	22136
						103	**48**	16676
						100	**12**	2386
						91	**36**	8894
						77.1	**60**	21797
	N,N-dimethylcathinone	C11H15NO	**FIU_0252**	5.552	**178.1**	188.2	**24**	1405182
						133	**12**	1388177
						103	**36**	235494
						79.1	**36**	410746
						77.1	**48**	1129270
						72.1	**24**	1582470
						70.1	**48**	92944
						58.1	**28**	128139
						57.7	**52**	67377
						51.1	**60**	493223
	para-Fluorofentanyl	C22H27FN2O	**FIU_0670**	7.93	**355.21074**	234.2	**24**	137932
						150.1	**36**	208005

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						146.1	**32**	47935
						134.1	**32**	142300
						105.1	**48**	1323016
						103.1	**60**	149472
						79.2	**60**	201995
						77.2	**60**	127959
						57.2	**40**	43315
	PCEEA	C16H25NO	**FIU_0388**	8.22	**248.19361**	159.1	**8**	724788
						117.1	**32**	29668
						115.1	**56**	17043
						105.1	**28**	4746
						91.1	**36**	837643
						81.1	**20**	125235
						79.1	**56**	14687
						67.1	**20**	18474
						65.1	**60**	191147
						55.1	**40**	11641
	Phenylpiperazine	C10H14N2	**FIU_0259**	5.772	**163.1**	161.1	**16**	96806
						120	**20**	1095572

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						118	**28**	227891
						106.1	**28**	66941
						103	**32**	110897
						93.1	**32**	49409
						91.1	**40**	112231
						77.1	**44**	585727
						65.1	**56**	73134
						51.1	**60**	365911
	tetrahydro-Harmine	C13H16N2O	**FIU_0701**	6.76	**217.12626**	200	**8**	95891
						188	**8**	106388
						185	**20**	11859
						173	**28**	38572
						158	**36**	12319
						156	**40**	9915
						145	**36**	17625
						144	**48**	11702
						132.1	**8**	278224
						130	**48**	24165
						103	**60**	9530

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4	**(±)-Cannabichromene**	C21H30O2	**FIU_0435**	12.34	**315.22458**	259.1	**12**	13764
						233.2	**12**	10679
						193.1	**16**	29978
						123	**40**	10291
						121.1	**20**	1222329
						109.1	**16**	4276
						107.1	**32**	3106
						81.1	**16**	12887
						69.2	**32**	11537
						67.1	**60**	5033
						55.1	**60**	4706
	(±)-epi CP 47,497	C21H34O2	**FIU_0565**	14.45	**319.25588**	167	**52**	1768
						155.1	**28**	957268
						137	**60**	1011
	25T2-NBOMe	C19H25NO3S	**FIU_0384**	8.51	**348.15551**	331.1	**12**	103037
						211.1	**16**	177422
						196	**32**	26276
						181	**44**	15374
						174.1	**8**	2249439

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						134.1	**44**	16848
						93.1	**36**	171318
						91.1	**52**	1091684
						77.1	**60**	36293
						65.1	**60**	159936
	2-methylethcathinone	C12H17NO	**FIU_0119**	6.69	**192.1**	159.1	**20**	345503
						146	**16**	902266
						145.1	**20**	1064793
						144.1	**32**	1086867
						131.1	**28**	419458
						130	**44**	403533
						115	**52**	165517
						91.1	**44**	364123
						77.1	**60**	320344
	3,4-methylenedioxy pyrovalerone (MDPV)	C16H21NO3	**FIU_0024**	7.52	**276.2**	205.1	**16**	1080770
						175.1	**24**	1324664
						149	**32**	824011
						135	**24**	1208863
						126.1	**28**	1595907

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						121	**48**	419197
						84.1	**44**	464988
						77.1	**60**	344910
						65.1	**60**	879407
						55.1	**56**	246234
	3'-4'-methylenedioxy-a-pyrrolidinopropiophenone	C14H17NO3	**FIU_0125**	6.02	**248.1**	189.1	**4**	245662
						177	**16**	674037
						149	**24**	574241
						147	**24**	1585673
						119	**36**	527575
						98.1	**24**	2094454
						91.1	**48**	1099682
						70.1	**40**	87480
						65.1	**60**	725244
						56.1	**60**	537012
						55.6	**56**	222319
	4-acetoxy DiPT	C18H26N2O2	**FIU_0745**	5.79	**303.19943**	202	**16**	132374
						160	**28**	344673

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						142	**48**	16664
						134	**24**	4674
						132	**48**	50606
						117	**60**	42606
						114.1	**16**	312530
						105.1	**60**	13505
						102.1	**16**	33201
						72.1	**32**	70273
	4-methoxy PCP	C18H27NO	**FIU_0387**	7.99	**274.20926**	147.1	**36**	5712
						121.1	**36**	213978
						106	**60**	4664
						91.1	**60**	28386
						86.1	**0**	95305
						81.1	**24**	10763
						79.5	**60**	3980
						78.1	**60**	26378
						77.1	**60**	38397
	5-fluoro AMB	C19H26FN3O3	**FIU_0708**	10.62	**364.19582**	304.1	**12**	342091
						233	**20**	403551

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						213	**32**	132149
						177	**36**	49611
						171	**44**	13501
						145	**44**	219791
						116.9	**60**	38235
						90	**60**	50298
						88.9	**60**	21975
						69.1	**40**	67911
	5-fluoro NPB-22	C22H20FN3O2	**FIU_0577**	10.42	**378.15396**	233.1	**16**	1170458
						213.1	**28**	341088
						202.1	**4**	193221
						185.1	**36**	22004
						177.1	**32**	140963
						171.1	**40**	36609
						145	**44**	592556
						121	**44**	18387
						117	**60**	106271
						90.1	**60**	149594
						69.1	**36**	167724

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	5-methoxy-a-Ethyltryptamine	C13H18N2O	**FIU_0752**	7.12	**219.14191**	162	**8**	10255
						160	**16**	162585
						159.1	**4**	465869
						148	**12**	12454
						145	**36**	57929
						130	**44**	7229
						117	**48**	63976
						90	**60**	27254
						89	**60**	18770
						58.1	**28**	7383
	6-APB	C11H13NO	**FIU_0422**	6.9	**176.09971**	131.1	**16**	429828
						129.1	**24**	11710
						116.1	**32**	34084
						115.1	**52**	45379
						103.1	**40**	20205
						91.1	**32**	106119
						77.2	**52**	111589
						65.2	**60**	43537
						51.2	**60**	46292

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	A-834735	C22H29NO2	**FIU_0689**	11.02	**340.21983**	242.1	**24**	12609
						125.1	**24**	193742
						99.1	**36**	12845
						97.1	**32**	27588
						83.1	**36**	11888
						81.1	**44**	5513
						79.1	**52**	3563
						69.1	**40**	32027
						57.2	**48**	33240
						55.2	**44**	61735
	AB-PINACA N-(5-hydroxypentyl) metabolite	C18H26N4O3	**FIU_0725**	9.18	**347.20049**	231.1	**20**	30297
						213	**28**	62387
						175	**32**	5497
						171	**44**	6001
						145	**48**	23199
						131	**52**	2331
						90	**60**	3271
						69.1	**44**	11777

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	AB-PINACA pentanoic acid metabolite	C18H24N4O4	**FIU_0726**	9.09	**361.17976**	344.1	**4**	55075
						316.1	**12**	56332
						298.1	**20**	19586
						245.1	**20**	19036
						227	**36**	20998
						217	**32**	28767
						199	**48**	4506
						175	**44**	4950
						145	**56**	9793
						55.1	**60**	16211
	Acetyl fentanyl	C21H26N2O	**FIU_0667**	7.32	**323.20451**	324.3	**4**	239588
						202.1	**20**	220466
						188.1	**24**	1463153
						134.1	**28**	175597
						132.1	**36**	261461
						117	**60**	58630
						105.1	**40**	1637881
						103.1	**60**	228792
						79.1	**60**	338140

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						77.1	**60**	303902
	AM2201 N-(3-chloropentyl) isomer	C24H22ClNO	**FIU_0636**	11.44	**376.13899**	373.6	**20**	51
						248.1	**24**	15892
						212.1	**32**	5993
						155.1	**28**	90270
						144.1	**44**	6273
						127	**60**	80271
						116	**60**	1484
						69.1	**48**	5612
	ATM4 4-acetoxy analog	C23H25NO5	**FIU_0672**	10.02	**396.17327**	378.2	**12**	243807
						305.2	**16**	253412
						249.1	**28**	225275
						221.1	**40**	290304
						217.1	**40**	62553
						206.1	**60**	69305
						189.1	**60**	95620
						178.1	**60**	107687

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	BB-22 5-hydroxyisoquinoline isomer	C25H24N2O2	**FIU_0620**	11.64	**385.18378**	384.1	**0**	2371
						240.2	**24**	326734
						188.1	**24**	1781082
						144	**44**	160396
						116	**60**	23928
						97.1	**44**	22789
						69.2	**52**	13316
						55.2	**60**	124549
	Butyryl fentanyl	C23H30N2O	**FIU_0669**	8.29	**351.23581**	352.3	**0**	362383
						230.2	**24**	230241
						146.1	**36**	86916
						134.1	**28**	261634
						132.1	**36**	322205
						105.1	**48**	1643249
						103.1	**60**	194889
						79.1	**60**	252033
						77.1	**60**	192673
	CB-13	C26H24O2	**FIU_0176**	13.07	**369.2**	299.1	**16**	763302

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						281.1	**28**	43985
						252.1	**60**	31559
						241.1	**16**	202408
						171	**28**	1940179
						155	**24**	2253863
						143	**52**	659771
						127	**60**	1738277
						115	**60**	609693
						77.1	**60**	27331
	CP 47,497-C9-homolog	C23H38O2	**FIU_0568**	14.51	**347.28718**	210.8	**28**	104
						121	**24**	2295
						107.1	**20**	2713
						71.1	**16**	2149
						57.1	**32**	2818
	D2PM	C17H19NO	**FIU_0181**	7.72	**254.2**	236.1	**12**	3588641
						178.1	**48**	306780
						167.1	**32**	217205
						165.1	**60**	322611
						158.1	**20**	439424

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						152	**56**	266546
						130	**32**	914499
						117	**40**	244293
						91.1	**40**	241134
						77.1	**60**	275464
	Delorazepam	C15H10Cl2N2O	**FIU_0675**	9.57	**305.01702**	196.1	**60**	66
						193.1	**48**	209
						179.2	**60**	1705
						165.1	**36**	2143
						140	**32**	4973
						99.2	**56**	235
	JWH 200 4-hydroxyindole metabolite	C25H24N2O3	**FIU_0530**	9.82	**401.17869**	155	**24**	673867
						127	**60**	340709
						114.1	**32**	734541
						100.1	**56**	13405
						86.1	**52**	17245
						84.1	**52**	52158
						70.1	**60**	143643
						68.1	**60**	9477

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						58.1	**60**	7900
						56.1	**60**	10934
	JWH 203 N-(5-hydroxypentyl) metabolite	C21H22ClNO2	**FIU_0536**	10.22	**356.13391**	230.1	**28**	9102
						204.1	**16**	109231
						186.1	**16**	184811
						144.1	**48**	26584
						130	**36**	46178
						125	**28**	542463
						118	**28**	10319
						99	**60**	16453
						89.1	**60**	43399
						69.1	**36**	14538
	JWH 387	C24H22BrNO	**FIU_0550**	12.06	**420.08848**	233	**28**	511810
						214.1	**28**	84677
						205	**52**	305239
						144	**52**	53243
						126	**60**	224465
						116.1	**60**	16024

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	Methiopropamine	C8H13NS	**FIU_0308**	5.52	**156.07687**	125	**8**	283684
						97	**24**	374348
						91.1	**24**	18916
						81.1	**28**	20771
						79.1	**36**	17333
						69.1	**48**	11566
						66.1	**48**	11342
						65.1	**56**	15550
						58.2	**8**	359989
						53.1	**48**	98382
	PB-22 N-(4-hydroxypentyl)-3-carboxyindole metabolite	C14H17NO3	**FIU_0609**	8.83	**248.12084**	230.1	**4**	11815
						186	**8**	5420
						174	**20**	3847
						157.1	**28**	188
						130	**32**	3473
						128.1	**44**	641
						93	**12**	92
						77	**60**	1812

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						69.1	**20**	2403
	UR-144	C21H29NO	**FIU_0268**	11.85	**312.2**	214.1	**24**	928611
						144	**36**	589452
						130	**56**	129459
						125.1	**20**	2475727
						116	**60**	241218
						97.1	**28**	552399
						83.1	**24**	326052
						69.1	**40**	375001
						57.1	**48**	639495
						55.1	**40**	1224554
	XLR12	C20H24F3NO	**FIU_0666**	11.23	**352.181**	353.3	**0**	351590
						254.1	**28**	326414
						144	**48**	177183
						125.1	**24**	806231
						116	**60**	138509
						97.1	**32**	164250
						83.1	**24**	153361
						69.1	**44**	113809

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						57.2	**52**	200408
						55.2	**48**	390987
5	**(±)-ORG 28611**	C23H33N3O2	**FIU_0436**	9.37	**384.25728**	338.3	**8**	4540
						270.1	**16**	1394881
						174.1	**40**	730840
						159	**60**	311991
						146.1	**52**	12847
						131	**60**	27362
						118.1	**60**	10297
						97.1	**40**	65038
						69.2	**52**	38785
						55.1	**60**	364862
	25I-NBOMe 4-methoxy isomer	C18H22INO3	**FIU_0381**	8.91	**428.06444**	301.2	**8**	25474
						272.2	**16**	16838
						121.1	**20**	1768680
						106.1	**60**	22922
						93.1	**60**	9792
						91.1	**60**	121035
						78	**60**	76861

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						77.1	**60**	183073
						65.1	**60**	12998
						55.1	**60**	6140
	2C-E	C12H19NO2	**FIU_0014**	8.43	**210.1**	193.1	**8**	1855745
						178.1	**16**	738861
						163.1	**28**	369121
						135.1	**20**	175389
						115	**52**	89190
						105.1	**28**	289192
						103.1	**40**	103167
						91.1	**52**	274033
						79.1	**40**	174784
						77.1	**60**	309902
	2C-I	C10H14INO2	**FIU_0025**	8.18	**308**	291	**8**	1001652
						276	**20**	327561
						260.9	**32**	149200
						164.1	**20**	159682
						149	**28**	141339
						134.1	**32**	154742

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						106	**52**	219173
						105.6	**60**	61119
						91.1	**56**	321837
						78.1	**60**	215637
	2-methoxymethcathinone	C11H15NO2	**FIU_0118**	6.24	**194.1**	176.1	**8**	991788
						161	**20**	569396
						146	**28**	152952
						144	**36**	104320
						132	**36**	122990
						118	**40**	191448
						117	**56**	107569
						91.1	**52**	109691
						77.1	**60**	160028
						58.1	**20**	75381
	3-fluoromethcathinone (hydrochloride)	C10H12FNO	**FIU_0131**	11.65	**182.1**	164.1	**12**	1605492
						149	**20**	1119625
						148	**36**	758213
						123	**20**	187934
						103.1	**32**	259411

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						101	**56**	168349
						95	**52**	76280
						77.1	**48**	352149
						75	**60**	239356
						51.1	**60**	129921
	4-fluoro PV8	C17H24FNO	**FIU_0409**	8.67	**278.18419**	207.2	**20**	95752
						154.2	**28**	382888
						137.1	**20**	98781
						123.1	**32**	232441
						109.1	**24**	651031
						95.1	**60**	264425
						84.2	**44**	164865
						72.2	**32**	56197
						70.2	**20**	109277
						55.2	**56**	46963
	4-fluoro PV9	C18H26FNO	**FIU_0410**	9.16	**292.19984**	177.1	**16**	570049
						168.2	**32**	406329
						137.1	**20**	90019
						125.1	**20**	100235

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						123.1	**36**	225314
						109.1	**28**	841614
						95.1	**60**	268112
						84.2	**48**	171654
						72.2	**36**	75278
						70.2	**20**	114176
						55.2	**56**	79203
	4-methoxy-a-Pyrrolidinobutiophenone	C15H21NO2	**FIU_0400**	6.97	**248.15723**	149.1	**20**	389778
						135	**28**	200754
						121.1	**32**	419348
						112.1	**24**	435277
						91.1	**52**	106767
						84.1	**36**	85157
						77.1	**56**	264095
						70.1	**48**	77796
						55.2	**52**	64271
	4-methyl-a-pyrrolidinobutiophenone (HCl)	C15H21NO	**FIU_0148**	7.4	**232.2**	161.1	**16**	1488009

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						133.1	**16**	531978
						119	**28**	681065
						112.1	**28**	976569
						105.1	**28**	2205142
						91.1	**48**	964711
						84.1	**32**	377228
						77.1	**60**	314627
						70.1	**44**	218660
						65.1	**60**	506222
	5-fluoro ADBICA	C20H28FN3O2	**FIU_0706**	10.26	**362.21656**	345.2	**8**	262237
						273.6	**4**	53
						232.1	**20**	297629
						144	**48**	129991
						116	**60**	45854
						89	**60**	4351
						69.1	**48**	10037
	5-fluoro PB-22 8-hydroxyisoquinoline isomer	C23H21FN2O2	**FIU_0589**	10.71	**377.15871**	232.1	**20**	2515332
						212.1	**40**	24396

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						176.1	**40**	15269
						161.1	**8**	255598
						158	**40**	19570
						144	**48**	1168634
						130	**60**	19029
						116	**60**	430178
						89.1	**60**	35737
						69.1	**44**	93366
						61.1	**60**	15522
	6-APDB	C11H15NO	**FIU_0423**	6.45	**178.11536**	133.1	**20**	216061
						120.1	**28**	11970
						105.1	**28**	29797
						103.1	**40**	18627
						100.2	**4**	6466
						91.1	**48**	19956
						79.2	**40**	27938
						77.1	**52**	65486
						51.2	**60**	20410
	Acetyl norfentanyl	C13H18N2O	**FIU_0668**	5.8	**219.14191**	136.1	**16**	54754

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						94.1	**36**	38367
						84.1	**16**	925546
						82.1	**32**	11214
						77.1	**60**	24360
						67.1	**28**	24080
						57.1	**28**	7642
						56.2	**28**	228000
						55.1	**44**	338411
						53.1	**60**	14730
	ADB-PINACA	C19H28N4O2	**FIU_0733**	10.92	**345.22123**	344.2	**4**	93835
						328.2	**4**	270236
						316.1	**12**	98571
						300.1	**12**	277681
						232.1	**20**	5162
						215	**24**	343729
						145	**48**	203544
						125.8	**36**	50
						117	**60**	21225
						90	**60**	24901

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						89.5	**60**	12483
						71.1	**44**	2929
	ADB-PINACA N-(5-hydroxypentyl) metabolite	C19H28N4O3	**FIU_0738**	9.63	**361.21614**	231	**20**	44215
						213	**32**	82008
						188.1	**8**	465922
						185.1	**40**	3689
						175	**36**	8114
						171	**48**	8589
						145	**52**	32910
						131	**52**	3603
						69.1	**48**	17081
	a-Ethylaminopentiophenone	C13H19NO	**FIU_0404**	7.17	**206.14666**	146.1	**16**	251766
						130.1	**36**	254640
						118.1	**24**	164429
						117.6	**36**	65938
						105.1	**24**	57604
						91.1	**32**	255679

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						77.1	**60**	236677
						65.1	**60**	79354
						51.1	**60**	75160
	AKB48 N-(4-fluorobenzyl) analog	C25H26FN3O	**FIU_0740**	11.94	**404.20599**	135.1	**20**	669326
						107	**60**	55132
						93	**60**	88456
						91	**60**	12840
						81.1	**60**	25167
						79.1	**60**	74219
						77	**60**	13774
						69.1	**60**	8223
						67.1	**60**	27495
						55.1	**60**	12570
	BB-22 6-hydroxyisoquinoline isomer	C25H24N2O2	**FIU_0622**	11.52	**385.18378**	240.2	**20**	501239
						144	**44**	193649
						138.5	**24**	64
						116	**60**	30511

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						97.1	**40**	31800
						69.1	**48**	18234
						55.2	**60**	173871
	JWH 193	C26H26N2O2	**FIU_0528**	9.86	**399.19943**	169.1	**20**	1001650
						141	**56**	424479
						114.1	**32**	459911
						100.1	**60**	10409
						86.1	**52**	11791
						84.1	**56**	38421
						70.1	**60**	111000
						68.1	**60**	6438
						58.1	**60**	7454
						56.2	**60**	8185
	JWH 210	C26H27NO	**FIU_0225**	12.095	**370.2**	214.1	**24**	1230945
						183.1	**28**	4075202
						155.1	**44**	1247308
						153.1	**56**	1045567
						144	**44**	659238
						129	**60**	388347

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						128.2	**60**	219700
						116	**60**	222546
						115	**60**	419288
						77.1	**60**	197334
	JWH 210 5-hydroxyindole metabolite	C26H27NO2	**FIU_0539**	11.32	**386.20418**	230.1	**24**	92449
						183.1	**28**	374825
						178	**20**	146366
						160	**44**	59085
						155	**44**	121471
						153	**60**	93409
						132	**60**	21012
						129.1	**60**	32997
						127.9	**60**	14656
						115	**60**	34942
						77.1	**60**	17021
	Levamisole	C11H12N2S	**FIU_0763**	5.55	**205.07212**	149	**4**	2033
						130	**44**	13591
						128	**36**	20736
						123	**28**	44966

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						117	**28**	28490
						103	**40**	16127
						91	**40**	99914
						77	**56**	44320
						65	**60**	34975
	PB-22 3-carboxyindole metabolite	C14H17NO2	**FIU_0597**	10.51	**232.12593**	188.1	**8**	8886
						186.3	**20**	116
						144	**28**	1355
						132	**16**	10470
						118	**20**	11328
						91	**44**	4748
	PB-22 N-(4-hydroxypentyl) metabolite	C23H22N2O3	**FIU_0608**	10.06	**375.16304**	281.6	**60**	115
						230.1	**12**	1439072
						225.3	**8**	107
						214.1	**52**	87
						144	**36**	686688
						116	**60**	135422
						89.1	**60**	13211

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						87.1	**32**	19830
						69.1	**40**	410011
						67.1	**56**	7095
	Pentedrone metabolite ((±)-Ephedrine stereochemistry)	C12H19NO	**FIU_0341**	7.6	**194.14666**	176.1	**8**	723346
						145.1	**16**	45243
						133.1	**24**	126644
						132.1	**32**	55838
						117.1	**24**	38017
						91.1	**40**	112699
						79.1	**36**	21077
						77.1	**60**	49015
						65.1	**60**	34924
						56.1	**40**	31100
	RCS-8 4-methoxy isomer	C25H29NO2	**FIU_0088**	11.73	**376.2**	254.1	**32**	496131
						149	**24**	1420456
						144	**48**	435283
						135	**36**	1808051
						121	**36**	4720971

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						107	**56**	309871
						91.1	**60**	401812
						77.1	**60**	1203244
						69.1	**52**	364009
						55.1	**60**	261828
6	**(+)-3,4-Methylenedioxy Pyrovalerone**	C16H21NO3	**FIU_0357**	7.73	**276.15214**	205.1	**16**	342544
						175.1	**20**	397365
						149	**32**	238258
						135	**28**	367160
						126.1	**28**	475979
						121	**48**	127849
						84.1	**44**	144399
						77.1	**60**	107016
						65.1	**60**	290472
						55.1	**56**	73340
	(+/-)-WIN 55,212 (mesylate)	C27H26N2O3	**FIU_0101**	10.77	**427.2**	340.1	**28**	20420
						328.1	**28**	25770
						299.1	**28**	19194

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						212.1	**36**	16268
						200.1	**44**	24450
						155	**24**	1749417
						127	**60**	810686
						100.1	**48**	243642
						70.1	**60**	44863
						56.1	**60**	43262
	(±)-JWH 073 N-(3-hydroxybutyl) metabolite	C23H21NO2	**FIU_0510**	10.3	**344.15723**	284.1	**24**	22739
						216.1	**24**	36391
						158.1	**32**	25448
						155	**20**	759571
						155	**16**	1825048
						144	**44**	17895
						130	**48**	23135
						127	**56**	581475
						116	**60**	9723
						77.1	**60**	16256
						55.2	**56**	8530
	1'-naphthoyl indole	C19H13NO	**FIU_0107**	10.09	**272.1**	254.1	**20**	13489

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						179.1	**8**	834875
						144	**20**	1064251
						127	**40**	1571228
						116	**48**	375694
						101	**60**	60558
						89.1	**60**	343881
						77.1	**60**	329137
						63.1	**60**	30423
						51.1	**60**	16941
	2,5-DMMA	C12H19NO2	**FIU_0304**	7.25	**210.14158**	164.1	**20**	328314
						151.1	**16**	621865
						149.1	**32**	187888
						123.1	**24**	58603
						121.1	**28**	310579
						91.1	**40**	225177
						78.1	**60**	139118
						77.1	**56**	192965
						65.1	**60**	117420
	2C-T-7	C13H21NO2S	**FIU_0312**	9.07	**256.1293**	239.1	**8**	513065

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						224.1	**20**	42920
						197.1	**20**	67597
						182	**28**	58606
						167	**36**	76734
						164.1	**24**	48230
						134.1	**32**	45939
						121.1	**44**	22386
						119	**48**	20742
						91.1	**56**	73590
	2C-TFM	C11H14F3NO2	**FIU_0313**	8.53	**250.09766**	233.1	**8**	161749
						218	**20**	105955
						203.1	**8**	641016
						203	**36**	56012
						175.1	**12**	586087
						151	**40**	6082
						133.1	**36**	12825
						127	**56**	23931
						121.1	**24**	550540
						115.1	**40**	7765

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						113	**48**	20627
						91.1	**60**	14144
						77.1	**60**	13527
	2-methoxy Ketamine	C14H19NO2	**FIU_0283**	7.03	**234.14158**	135.1	**24**	38646
						115.1	**60**	35198
						93.1	**32**	51285
						91.1	**48**	459790
						77.1	**60**	89389
						67.2	**28**	108334
						65.2	**60**	167281
	3,4-EDMC	C12H15NO3	**FIU_0285**	6.28	**222.10519**	204.1	**12**	773196
						189.1	**20**	314874
						163.1	**24**	72046
						148.1	**28**	197052
						133	**36**	227621
						105.1	**52**	73840
						91.1	**44**	116514
						77.1	**60**	96040
						65.1	**60**	113162

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						58.2	**12**	103344
	3-Bromoamphetamine	C9H12BrN	**FIU_0286**	8.02	**214.01531**	197	**4**	56464
						168.9	**16**	102523
						120.9	**16**	100
						118.1	**20**	11776
						117.1	**40**	23442
						115.1	**60**	8202
						91.1	**60**	14528
						90.1	**40**	40223
						89.1	**60**	35928
						64.1	**60**	3923
	3-Methoxyamphetamine	C10H15NO	**FIU_0289**	6.83	**166.11536**	149.1	**4**	398894
						121.1	**16**	443724
						106.1	**36**	9707
						91.1	**32**	143466
						78.1	**48**	90145
						77.1	**36**	63633
						65.1	**48**	78124
						63.1	**60**	9719

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						52.1	**60**	27862
						51.1	**60**	24061
	4-quinolone-3-carboxamide CB2 ligand	C26H34N2O3	**FIU_0151**	12.72	**423.3**	272.6	**24**	846128
						202	**44**	368765
						187	**60**	145440
						135.1	**28**	5354440
						107.1	**60**	588428
						93.1	**60**	843037
						81.1	**60**	231173
						79.1	**60**	728317
						77.1	**60**	123049
						67.1	**60**	271053
	5-fluoro JWH 018 adamantyl analog	C24H30FNO	**FIU_0474**	11.62	**368.23114**	135.2	**36**	1063440
						107.1	**56**	140480
						93.1	**60**	218304
						91.1	**60**	38066
						81.2	**60**	59976
						79.2	**60**	207437

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						77.1	**60**	40949
						69.2	**60**	18282
						67.2	**60**	72180
						55.2	**60**	29403
	5-fluoro PB-22 7-hydroxyisoquinoline isomer	C23H21FN2O2	**FIU_0587**	10.55	**377.15871**	232.1	**24**	1679788
						212.1	**44**	20280
						176.1	**44**	12071
						158	**40**	18539
						144	**48**	1042519
						130.1	**56**	16766
						116	**60**	346830
						89.1	**60**	31060
						69.2	**48**	71486
						61.1	**60**	12336
	AM1248 azepane isomer	C26H34N2O	**FIU_0456**	9.86	**391.26711**	135.2	**32**	241646
						112.2	**28**	496340
						107.1	**56**	24952
						98.1	**44**	37564

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						93.2	**60**	43626
						81.1	**60**	17735
						79.1	**60**	34146
						70.2	**60**	32347
						58.2	**60**	141103
						55.2	**60**	18752
	a-Pyrrolidinobutiothiophenone	C12H17NOS	**FIU_0406**	5.82	**224.10308**	153	**12**	190553
						125	**20**	179496
						112.1	**20**	611336
						111	**36**	111764
						97	**36**	144145
						84.1	**36**	56497
						83.1	**60**	32636
						70.1	**44**	91687
						56.4	**60**	25731
						55.2	**52**	74472
	CB-86	C26H43NO3	**FIU_0179**	11.84	**418.3**	361.3	**16**	295441
						292.2	**12**	268271

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						275.1	**16**	293382
						182.1	**16**	213452
						142.1	**16**	240826
						97.1	**24**	87608
						85.1	**32**	196331
						71.1	**32**	387338
						58.1	**28**	2599650
						55.1	**60**	314099
	DOI	C11H16INO2	**FIU_0299**	8.73	**322.02257**	305	**8**	419743
						277	**20**	113922
						178.1	**20**	163710
						163.1	**28**	89350
						135.1	**36**	110861
						105.1	**52**	99762
						103.1	**60**	44843
						91.1	**60**	62462
						79.1	**60**	48173
						77.1	**60**	101358
	JWH 213	C27H29NO	**FIU_0543**	12.06	**384.22491**	228.1	**28**	362663

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						183.1	**28**	1846841
						158	**44**	231505
						155.1	**44**	559364
						153.1	**56**	467202
						130	**60**	68152
						129.1	**60**	181442
						127.8	**60**	74762
						115.1	**60**	175718
						77.1	**60**	73776
	JWH 251 4-methylphenyl isomer	C22H25NO	**FIU_0077**	11.42	**320.2**	214.1	**24**	754154
						188.2	**20**	199026
						144.1	**40**	567236
						130.1	**48**	134294
						119	**24**	419304
						116.1	**60**	249506
						105.1	**24**	1828336
						91.1	**56**	267202
						79.1	**60**	266183
						77.1	**60**	245281

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	Lisdexamfetamine	C15H25N3O	**FIU_0301**	5.63	**264.19976**	280.2	**0**	232757
						247.2	**12**	24768
						136.1	**12**	9050
						129.1	**8**	19886
						119.1	**20**	8899
						91.1	**40**	22812
						85.2	**28**	136
						84.1	**24**	110392
						67.1	**44**	4084
						56.1	**60**	29628
	Mepirapim	C19H27N3O	**FIU_0329**	9.29	**314.21541**	214.1	**12**	1082746
						158.1	**32**	12956
						144.1	**36**	425625
						130.1	**44**	8942
						116.1	**60**	165617
						91.1	**32**	193
						89.1	**60**	39360
						71.2	**36**	7366
						55.2	**52**	4746

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	Mescaline	C11H17NO3	**FIU_0319**	6.1	**212.12084**	195.1	**4**	221645
						180.1	**16**	51089
						165.1	**20**	38973
						135	**28**	9649
						133.1	**28**	17840
						105.1	**40**	10825
						91.1	**48**	22622
						79.1	**40**	11814
						77.1	**56**	43236
						65.1	**60**	15648
	MT-45	C24H32N2	**FIU_0328**	9.61	**349.25655**	181.1	**24**	1160727
						179.1	**40**	88903
						169.1	**20**	393925
						167.1	**20**	91623
						166.1	**40**	398058
						165.1	**60**	300109
						153.1	**40**	46172
						149.1	**8**	938291
						103.1	**56**	371135

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						87.1	**32**	115883
						77.1	**60**	129098
	para-Methoxymethamphetamine	C11H17NO	**FIU_0307**	6.64	**180.13101**	121	**20**	680782
						119	**36**	14331
						93.1	**24**	20217
						91.1	**36**	204015
						78.1	**52**	161368
						77.1	**40**	119965
						65.1	**56**	114966
						52.1	**60**	29479
						51.1	**60**	38474
	PB-22 7-hydroxyisoquinoline isomer	C23H22N2O2	**FIU_0605**	11.22	**359.16813**	214.1	**24**	1693224
						158	**36**	27828
						144	**44**	975805
						143.2	**56**	2784
						130.1	**56**	20429
						116	**60**	290169

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					89.1	**60**	30487
					71.2	**40**	15875
					55.2	**60**	8068
	Pentedrone metabolite ((±)-Pseudoephedrine stereochemistry)	C12H19NO	**FIU_0342**	7.51	**194.14666**	176.1	**8**	802809
					145.1	**16**	49571
					133.1	**24**	151368
					132.1	**32**	68685
					117.1	**20**	43358
					104.1	**36**	29306
					91.1	**44**	126771
					77.1	**60**	35084
					65.1	**60**	39991
					56.1	**40**	41050
	Propylhexedrine	C10H21N	**FIU_0302**	8.31	**156.1674**	125.1	**8**	21793
						83.1	**16**	116302
						69.2	**16**	371871
						67.2	**12**	5018
						57.2	**12**	52422

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						55.2	**28**	193989
						53.2	**52**	6437
	UR-144 N-(2-hydroxypentyl) metabolite	C21H29NO2	**FIU_0648**	11.01	**328.21983**	230.1	**24**	30608
						144	**40**	23786
						130.1	**48**	6696
						125.1	**24**	152328
						116	**60**	10349
						97.1	**32**	25740
						83.1	**24**	16343
						69.1	**40**	22507
						57.2	**48**	33477
						55.1	**44**	67066
	UR-144 N-(5-methylhexyl) analog	C23H33NO	**FIU_0656**	12.29	**340.25621**	322.3	**20**	17752
						242.2	**24**	41749
						144	**40**	23285
						130.1	**60**	6577
						125.1	**24**	140806

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						97.1	**32**	29712
						83.1	**28**	16883
						69.1	**40**	19898
						57.2	**48**	43015
						55.2	**48**	67582
7	**(R)-(-)-MT-45**	C24H32N2	**FIU_0324**	9.6	**349.25655**	181.1	**24**	1251652
						179.1	**44**	98594
						169.2	**16**	464387
						167.2	**20**	103109
						166.1	**40**	464629
						165.1	**60**	355992
						153.1	**40**	54038
						103.1	**56**	428647
						87.1	**36**	135549
						77.1	**60**	140327
	2,3-Dichlorophenylpiperazine	C10H12Cl2N2	**FIU_0326**	8.49	**231.03775**	188	**20**	131580
						152.7	**28**	73679
						152	**36**	66808
						118.1	**40**	28664

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						117.1	**56**	58659
						90.7	**56**	21105
						90.1	**60**	12302
						89.6	**60**	12413
						75.1	**60**	13446
						70.1	**20**	13990
	2C-T	C11H17NO2S	**FIU_0311**	7.66	**228.098**	211.1	**8**	561379
						196	**20**	101653
						181	**24**	64240
						166	**20**	58717
						164.1	**20**	47394
						134.1	**24**	73037
						121	**36**	31062
						119	**36**	33248
						91.1	**48**	88998
						77.1	**60**	51757
	3,4-Dimethylethcathinone	C13H19NO	**FIU_0331**	7.98	**206.14666**	188.2	**12**	762836
						173.1	**20**	295598
						159.8	**16**	169002

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						159.1	**20**	326413
						158.1	**32**	395250
						144.7	**24**	106532
						144.1	**36**	105431
						115.1	**60**	106431
						105.1	**36**	93991
						91.1	**56**	106950
	3-Bromomethcathinone	C10H12BrNO	**FIU_0352**	7.41	**242.01023**	145.1	**16**	364113
						144.1	**36**	238510
						132.1	**20**	22824
						131	**40**	15869
						128	**56**	18496
						104	**36**	11727
						103.1	**60**	27212
						78	**60**	17422
						77.1	**60**	43051
						58.2	**60**	7153
	3C-P	C14H23NO3	**FIU_0316**	8.29	**254.16779**	237.1	**4**	175322
						195.1	**12**	217228

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						167.1	**20**	46877
						163.1	**16**	47645
						149.1	**4**	625482
						135.1	**20**	25931
						107.1	**28**	83462
						103.1	**40**	15716
						91.1	**60**	26379
						79.1	**40**	24030
						77.1	**60**	53173
	4-Methoxyamphetamine	C10H15NO	**FIU_0293**	6.69	**166.11536**	121.1	**16**	403590
						119	**36**	10174
						93.1	**20**	6156
						91.1	**32**	118106
						78.1	**48**	96133
						77.1	**40**	72439
						65.1	**52**	75461
						52.1	**60**	24971
						51.1	**60**	26844
	4-methoxy-N,N-Dimethylcathinone	C12H17NO2	**FIU_0332**	6.47	**208.12593**	163.1	**12**	268429

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						135.1	**20**	374350
						105.1	**32**	148883
						103.1	**40**	75961
						91.1	**60**	93866
						79.1	**40**	105894
						77.1	**56**	177530
						72.2	**20**	649795
						70.1	**52**	34223
						58.2	**28**	22286
	5-fluoro NNEI	C24H23FN2O	**FIU_0437**	10.55	**375.17944**	232.2	**20**	801759
						212.2	**40**	8266
						206.2	**16**	18743
						176.1	**40**	4557
						158.1	**44**	7466
						144.1	**48**	406051
						130.1	**60**	7367
						116.1	**60**	137737
						89.1	**60**	11688
						69.2	**44**	36116

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	9-octadecenamide (oleamide)	C18H35NO	**FIU_0157**	11.72	**282.3**	111.1	**12**	35047
						97.1	**16**	57377
						95.1	**16**	28919
						83.1	**20**	62044
						81.1	**20**	25419
						71.1	**20**	31999
						69.1	**24**	84689
						67.1	**36**	16158
						57.1	**28**	75143
						55.1	**40**	106311
	BB-22 4-hydroxyquinoline isomer	C25H24N2O2	**FIU_0619**	11.87	**385.18378**	240.1	**12**	679498
						158	**40**	2631
						144	**40**	242106
						125.4	**52**	63
						116	**60**	40566
						97.1	**40**	33999
						89.1	**60**	3288
						69.1	**52**	19312

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						55.1	**60**	198022
	Cathine	C9H13NO	**FIU_0323**	5.68	**152.09971**	134.1	**4**	399942
						117.1	**16**	129016
						115.1	**24**	101578
						91.1	**36**	86537
						89.1	**52**	7744
						77.1	**40**	7708
						65.1	**52**	39152
						63.1	**60**	7549
						56.1	**16**	23121
						51.1	**60**	10832
	Diclofensine	C17H17Cl2NO	**FIU_0298**	9.85	**322.06872**	291	**20**	38008
						279	**20**	85701
						256.1	**28**	27313
						252.1	**8**	1527188
						209.1	**36**	21371
						178.1	**60**	39973
						165.1	**60**	24296
						159	**32**	23787

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						121.1	**24**	178112
						91.1	**52**	48760
						77.1	**52**	19798
	FUB-PB-22	C25H17FN2O2	**FIU_0594**	10.76	**397.12741**	301.1	**20**	106
						224.1	**28**	11390
						109	**44**	1409363
						83.1	**60**	34895
	HMA	C10H15NO2	**FIU_0300**	5.21	**182.11028**	165.1	**4**	222342
						137	**16**	60366
						133.1	**16**	48909
						105.1	**24**	56550
						103.1	**32**	15992
						94.1	**44**	14357
						79.1	**32**	30876
						77.1	**48**	47161
						65.1	**52**	11638
						51.1	**60**	20746
	JWH 018 2-hydroxyindole metabolite	C24H23NO2	**FIU_0475**	12.51	**358.17288**	340.2	**16**	56591
						270.1	**24**	99942

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						254.2	**56**	347
						252.6	**40**	838
						252.1	**40**	49856
						251.1	**60**	24784
						230.1	**20**	9346
						155.1	**20**	15972
						127.1	**56**	11992
	JWH 251 3-methylphenyl isomer	C22H25NO	**FIU_0076**	11.41	**320.2**	214.1	**24**	2540962
						188.2	**16**	607795
						144	**40**	1658027
						130.1	**48**	159603
						119	**24**	269801
						116	**60**	710628
						105.1	**24**	2951181
						91.1	**56**	210814
						79.1	**60**	355442
						77.1	**60**	348348
	MBZP	C12H18N2	**FIU_0327**	5.45	**191.147**	160.1	**8**	765293
						100.1	**16**	39905

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						99.1	**12**	247295
						91.1	**24**	1169459
						84.1	**24**	9708
						70.1	**28**	62134
						65.1	**56**	397011
						63.1	**60**	30028
						58.2	**40**	115136
						56.1	**28**	42801
						51.1	**60**	18793
	Mephedrone	C11H15NO	**FIU_0337**	6.9	**178.11536**	224.1	**12**	435090
						145.1	**20**	580164
						144.1	**36**	376708
						130.1	**32**	39373
						119.1	**24**	86636
						115.1	**52**	36706
						103.1	**48**	42353
						91.1	**36**	104420
						77.1	**60**	133572
						65.1	**60**	65730

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	NRG-3	C16H19NO	**FIU_0340**	9.12	**242.14666**	211.1	**12**	159337
						194.1	**36**	80735
						181.7	**16**	325438
						181.1	**24**	343752
						180.1	**40**	202246
						167.1	**32**	114928
						141.1	**24**	209455
						127.1	**56**	84986
						115.1	**60**	111817
	RCS-4 N-(4-hydroxypentyl) metabolite	C21H23NO3	**FIU_0574**	14.51	**338.16779**	111.1	**20**	19492
						97.1	**24**	31254
						95.1	**20**	16121
						83.1	**24**	30687
						81.1	**24**	13559
						71.1	**28**	19382
						69.1	**36**	41678
						67.1	**40**	9018
						57.2	**40**	37105
						55.1	**52**	51393

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	UR-144 N-(2-chloropentyl) analog	C21H28ClNO	**FIU_0647**	11.64	**346.18594**	248.1	**24**	24585
						144	**40**	15950
						130.1	**56**	3887
						125.1	**24**	77720
						116.1	**60**	6232
						97.1	**32**	15258
						83.1	**24**	10781
						69.1	**44**	16166
						57.2	**56**	17681
						55.1	**48**	37963
8	**(-)-(S)-Cathinone**	C9H11NO	**FIU_0344**	5.59	**150.08406**	133.2	**8**	70853
						117.1	**24**	166242
						105.1	**16**	88675
						103.1	**32**	14783
						89.8	**40**	54402
						89.1	**52**	51167
						79.1	**32**	23572
						77.1	**40**	74263
						51.1	**60**	55674

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	(±)-Ethylphenidate	C15H21NO2	**FIU_0279**	8.12	**248.16**	174.1	**24**	12580
						163.1	**8**	450428
						129.1	**40**	4034
						115.1	**60**	7099
						91.1	**60**	23219
						84.1	**20**	1404865
						70.2	**48**	4475
						69.2	**60**	10177
						67.2	**60**	22288
						65.2	**60**	7635
						56.1	**60**	334424
	2,3-MDA	C10H13NO2	**FIU_0358**	7.05	**180.09463**	174.1	**8**	920493
						135	**16**	297130
						133.1	**16**	74445
						105.1	**24**	177076
						103.1	**32**	38437
						91.1	**40**	7465
						79.1	**32**	90323
						77.1	**44**	191386

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						65.1	**44**	12707
						51.1	**60**	116146
	2,4-Dimethylmethcathinone	C12H17NO	**FIU_0349**	7.86	**192.13101**	159.1	**20**	669562
						158.1	**32**	359343
						144.1	**32**	177906
						116.8	**52**	21510
						115.1	**56**	86963
						105.1	**32**	35737
						91.1	**60**	81412
						77.1	**60**	65270
						58.2	**12**	67072
	30C-NBOMe	C20H26ClNO5	**FIU_0385**	8.35	**396.14995**	181.1	**12**	1313212
						151	**56**	22893
						148	**48**	235340
						137.1	**60**	78969
						123.1	**60**	26954
						120.1	**56**	23410
						107.1	**60**	24969
						91.1	**60**	50605

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						90.5	**60**	31829
						77.1	**60**	25079
	4-Fluoromethcathinone metabolite ((±)-Pseudoephedrine stereochemistry)	C10H14FNO	**FIU_0355**	6.14	**184.10594**	166.1	**8**	957374
						151.1	**20**	163911
						135.1	**20**	123112
						133	**36**	56834
						122.1	**36**	11460
						115.1	**28**	107652
						109	**36**	141519
						83.1	**60**	67403
						70.1	**20**	35481
						56.1	**40**	19331
	4-Methylethcathinone metabolite ((±)-Ephedrine stereochemistry)	C12H19NO	**FIU_0333**	7.32	**194.14666**	176.1	**8**	982397
						161.1	**20**	81004
						147.1	**20**	82802
						146.1	**28**	75159

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						131.1	**20**	131292
						116.1	**32**	52983
						115.1	**52**	69475
						105.1	**24**	44898
						91.1	**36**	151734
	5-fluoro PB-22 N-(3-fluoropentyl) isomer	C23H21FN2O2	**FIU_0591**	10.85	**377.15871**	232.1	**12**	335201
						212.1	**36**	23466
						176	**44**	4804
						158	**40**	6443
						148	**60**	2965
						144	**48**	85426
						130	**56**	5878
						116	**60**	33197
						69.1	**52**	32572
						61	**60**	3730
	A-836339	C16H26N2O2S	**FIU_0442**	10.24	**311.1715**	352	**4**	99027
						253	**20**	72417
						187.1	**16**	2131532
						155.1	**36**	144832

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						129.1	**40**	150865
						125.1	**24**	432296
						97.2	**36**	112367
						87.1	**56**	50246
						69.2	**44**	83446
						59.2	**44**	358874
						57.2	**56**	210341
						55.2	**48**	310195
	AB-FUBINACA isomer 5	C20H21FN4O2	**FIU_0720**	9.8	**369.16485**	324.1	**12**	32111
						109	**44**	67569
	AM1220	C26H26N2O	**FIU_0159**	8.79	**383.2**	286.1	**20**	662973
						177.1	**12**	33086
						158	**36**	137799
						155	**32**	1020648
						127	**60**	730738
						112.1	**20**	2311684
						98.1	**40**	2882892
						84.1	**60**	43078
						70.1	**60**	675347

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						58.1	**60**	451422
						55.1	**60**	67784
	bk-2C-B	C10H12BrNO3	**FIU_0314**	7.58	**274.00006**	257	**4**	15844
						178	**12**	30634
						163.1	**32**	14816
						162.1	**28**	30711
						134.1	**40**	7210
						119	**60**	10714
						105	**44**	4845
						91.1	**60**	6924
						77.1	**60**	11872
	JWH 018 2'-naphthyl-N-(1-methylbutyl) isomer	C24H23NO	**FIU_0037**	11.71	**342.2**	272.1	**20**	61542
						214.1	**20**	320866
						155.1	**24**	3961741
						144	**40**	908068
						127.1	**56**	2875178
						116	**60**	408235
						101	**60**	14966
						89.1	**60**	90970

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						77.1	**60**	73585
						71.2	**32**	14648
	JWH 250 N-(5-hydroxypentyl) metabolite	C22H25NO3	**FIU_0546**	10.02	**352.18344**	204.1	**16**	30070
						186.1	**12**	59991
						144.1	**40**	22438
						131.1	**40**	13908
						130.1	**44**	38757
						121.1	**20**	509301
						93.1	**40**	46476
						91.1	**56**	296378
						69.2	**40**	16174
						65.1	**60**	25483
	JWH 309	C30H27NO	**FIU_0231**	12.445	**418.2**	290.2	**20**	35097
						220.1	**40**	16795
						192.1	**52**	7816
						189	**24**	4001654
						165.1	**60**	14172
						155	**20**	4777497

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						127	**60**	3441735
						101	**60**	7583
						77.1	**60**	38110
	JWH 398 6-chloronaphthyl isomer	C24H22ClNO	**FIU_0081**	11.91	**376.2**	214.1	**24**	603340
						161	**52**	3533857
						158	**36**	9891
						149.1	**20**	10567
						144	**40**	347931
						130.1	**48**	9133
						126	**60**	918657
						116	**60**	112090
						89.1	**60**	15544
	NNEI	C24H24N2O	**FIU_0447**	11.05	**357.18886**	214.2	**20**	733015
						188.2	**16**	16676
						158.1	**44**	10094
						144.1	**44**	323174
						132.1	**28**	3926
						130.1	**52**	7543
						116.1	**60**	99833

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						89.1	**60**	10298
						71.2	**40**	6795
						55.2	**60**	4279
	Pravadoline	C23H26N2O3	**FIU_0260**	9.295	**379.2**	135	**20**	4442637
						114.1	**36**	710049
						107	**52**	437431
						100.1	**56**	24517
						92	**60**	240906
						86.1	**48**	25525
						84.1	**52**	76314
						79.1	**60**	78822
						77.1	**60**	964849
						70.1	**60**	249950
	PV9	C18H27NO	**FIU_0414**	8.99	**274.20926**	168.2	**32**	227170
						119.1	**20**	101200
						107.1	**20**	87006
						105.1	**32**	191455
						91.1	**28**	666212
						84.2	**40**	125960

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						77.1	**60**	314657
						72.2	**36**	62514
						70.2	**20**	128781
						55.2	**56**	56754
9	**(R)-(-)-JWH 073 N-(3-hydroxybutyl) metabolite**	C23H21NO2	**FIU_0511**	10.3	**344.15723**	284.1	**24**	45717
						216.1	**24**	69693
						158	**32**	56847
						155	**24**	1526382
						144	**44**	41505
						130.1	**52**	41771
						127	**52**	1184444
						116	**60**	20368
						77.1	**60**	35078
						55.1	**52**	17162
	(S)-2-Diphenylmethylpyrrolidine (HCl)	C17H19N	**FIU_0102**	7.88	**238.2**	178.1	**52**	88416
						167.1	**20**	97389
						165.1	**52**	95726

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						143.1	**12**	612375
						129	**12**	206428
						128	**32**	196308
						117.1	**16**	1235644
						115	**40**	353612
						91.1	**32**	2262178
						65.1	**60**	673049
	2C-P	C13H21NO2	**FIU_0174**	8.83	**224.2**	207.1	**8**	2370806
						192.1	**16**	794252
						188.1	**8**	1326095
						163.1	**28**	393182
						149.1	**28**	102947
						135	**24**	155614
						105.1	**36**	205058
						103	**48**	116322
						91.1	**56**	235297
						79.1	**52**	176193
						77.1	**60**	382344

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2-methyl-a-Pyrrolidinopropiophenone	C14H19NO	**FIU_0392**	6.8	**218.14666**	160.1	**8**	1004880
					147.1	**16**	261616
					119.1	**24**	420128
					117.1	**36**	98367
					98.1	**24**	434430
					91.1	**40**	231079
					77.1	**60**	109283
					70.1	**20**	47186
					65.1	**60**	97081
					56.1	**60**	127391
					55.1	**48**	53439
3-Methyl-a-Pyrrolidinobutiophenone (HCl)	C15H21NO	**FIU_0135**	7.26	**232.2**	174.1	**8**	2647797
					161.1	**16**	973302
					145.1	**20**	1275444
					133.1	**20**	426623
					119	**28**	581551
					112.1	**28**	829297

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						105.1	**24**	2147103
						91.1	**48**	985537
						84.1	**32**	376516
						77.1	**60**	308484
						70.1	**16**	331020
						65.1	**60**	496869
	4-acetoxy DMT	C14H18N2O2	**FIU_0746**	6.1	**247.13683**	202	**12**	38949
						160	**20**	75052
						159.1	**4**	465959
						142	**40**	4005
						134	**20**	2403
						132	**36**	12099
						131.5	**40**	1825
						117	**48**	11952
						115	**56**	30669
						105	**52**	3793
						58.1	**16**	184611
	4-Fluoropentylindole	C13H16FN	**FIU_0684**	10.73	**206.12668**	186.1	**12**	5057
						141.2	**56**	98

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						132.2	**20**	1970
						130	**16**	12745
						130	**16**	4013
						118.1	**24**	2631
						118	**20**	7318
						91.1	**52**	5001
						91	**44**	1834
						81.2	**20**	96
	4-hydroxy DET	C14H20N2O	**FIU_0747**	5.8	**233.15756**	160	**20**	161985
						142	**36**	9052
						132	**32**	21432
						117	**44**	26820
						115	**48**	94958
						105	**40**	6792
						89	**60**	18928
						86.1	**12**	363422
						77	**60**	8781
						58.1	**40**	35609
	4-methoxy PV9	C19H29NO2	**FIU_0412**	9.18	**304.21983**	233.2	**16**	431945

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						168.2	**28**	615177
						135.1	**36**	258873
						121.1	**28**	901678
						107.1	**52**	67556
						84.2	**44**	178074
						77.1	**60**	300103
						72.2	**36**	40816
						69.2	**60**	64425
						55.2	**56**	72009
	7-APB	C11H13NO	**FIU_0424**	7.02	**176.09971**	159.1	**4**	562444
						131.1	**16**	700125
						116.1	**32**	33120
						115.1	**48**	47814
						103.1	**36**	41825
						102.7	**48**	11973
						91.2	**32**	110412
						77.1	**48**	218512
						65.2	**56**	48948
						51.2	**60**	92417

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	ADB-PINACA N-(4-hydroxypentyl) metabolite	C19H28N4O3	**FIU_0737**	9.64	**361.21614**	358.1	**4**	72811
						231.1	**20**	89654
						213	**32**	124422
						185	**40**	5605
						175	**36**	11360
						171	**44**	5892
						145	**52**	54013
						90	**60**	1827
						69.1	**48**	44417
	AM2201	C24H22FNO	**FIU_0163**	11.01	**360.2**	270.1	**24**	31572
						232.1	**24**	907050
						163	**8**	67680
						144	**40**	619525
						127	**56**	4584774
						116	**60**	269380
						105	**40**	38314
						89	**60**	44472
						77.1	**60**	128925

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						69.1	**40**	44081
	AM2201 7-hydroxyindole metabolite	C24H22FNO2	**FIU_0631**	10.95	**376.16346**	248.1	**28**	65361
						160	**40**	41201
						132.1	**56**	9005
						127	**60**	136036
						104	**60**	17978
						77.1	**60**	2802
						69.1	**44**	3169
	AM2201 8-quinolinyl carboxamide	C23H22FN3O	**FIU_0614**	11.37	**376.17469**	232.1	**16**	302230
						212	**40**	2669
						199.6	**44**	53
						180.4	**48**	74
						171	**8**	2743
						158	**40**	2602
						155.1	**24**	4550430
						144	**44**	135298
						116	**60**	45282
						89	**60**	4037

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						69.1	**44**	10497
	AM2233	C22H23IN2O	**FIU_0165**	8.38	**459.1**	362	**20**	500976
						235.1	**28**	33379
						230.9	**36**	380866
						202.9	**60**	223095
						158	**52**	32211
						112.1	**24**	1649095
						98.1	**40**	2891913
						76.1	**60**	71428
						70.1	**60**	394360
						58.1	**60**	298433
	Buphedrone metabolite ((±)-Ephedrine stereochemistry)	C11H17NO	**FIU_0429**	6.31	**180.13101**	162.1	**8**	825436
						133.1	**20**	183472
						132.1	**32**	76956
						131.1	**16**	87394
						104.1	**36**	33627
						91.1	**32**	190854
						79.1	**36**	28177

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						77.1	**60**	54782
						70.1	**20**	31488
						65.1	**60**	59518
	Desoxypipradrol (hydrochloride)	C18H21N	**FIU_0182**	8.17	**252.2**	167.1	**20**	428612
						165	**52**	204036
						152	**48**	156604
						131.1	**20**	505186
						129.1	**28**	306158
						128	**52**	213080
						117.1	**24**	176782
						115	**48**	281925
						91.1	**36**	1770614
						65.1	**60**	459065
	JWH 018 6-hydroxyindole metabolite	C24H23NO2	**FIU_0478**	10.82	**358.17288**	230.1	**24**	67048
						221.7	**0**	103
						160	**40**	45661
						155	**24**	1157306
						132	**60**	21538

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						127	**60**	885622
						105	**60**	6881
						77.1	**60**	20705
	JWH 018 N-(3-methylbutyl) isomer	C24H23NO	**FIU_0047**	11.57	**342.2**	214.1	**20**	18863
						158.1	**28**	2081
						144	**40**	8185
						127.1	**52**	99556
						119.1	**12**	57
						116.1	**60**	3770
						77.1	**60**	6074
	JWH 081 N-(4-hydroxypentyl) metabolite	C25H25NO3	**FIU_0515**	10.52	**388.18344**	230.1	**24**	77729
						185.1	**24**	1908398
						157.1	**48**	552662
						144	**36**	146817
						142	**60**	224647
						129	**56**	21513
						127	**60**	338286

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						116	**60**	34358
						114	**60**	106689
						69.1	**40**	78971
	JWH 146	C28H29NO	**FIU_0524**	12.28	**396.22491**	268.2	**24**	13823
						170	**40**	10554
						155	**24**	2750418
						127	**60**	1839506
						115.1	**60**	3990
						77.1	**60**	19670
						57.1	**52**	3327
	JWH 167	C21H23NO	**FIU_0526**	11.16	**306.17796**	214.1	**24**	559569
						188.1	**16**	243544
						144	**40**	444672
						132.1	**28**	48261
						130	**44**	91819
						116	**60**	202160
						105	**24**	114231
						91.1	**24**	1346056
						77.1	**56**	78358

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						65.1	**60**	130593
	JWH 200 6-hydroxyindole metabolite	C25H24N2O3	**FIU_0532**	8.62	**401.17869**	155	**24**	461839
						127	**60**	235000
						114.1	**32**	134649
						100.1	**60**	3877
						86.2	**56**	3976
						84.1	**56**	13437
						70.1	**56**	37432
	JWH 200 7-hydroxyindole metabolite	C25H24N2O3	**FIU_0533**	9.85	**401.17869**	351.1	**16**	103
						155	**24**	203327
						127	**60**	104923
						114.1	**28**	217002
						102.5	**60**	160
						100.1	**44**	5560
						86.1	**60**	4730
						84.1	**56**	14248
						70.1	**56**	40410
						56.1	**60**	3525

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	JWH 210 3-ethylnaphthyl isomer	C26H27NO	**FIU_0071**	11.99	**370.2**	214.1	**24**	2296544
						183.1	**24**	5064879
						155.1	**40**	2408535
						153.1	**56**	1421332
						144.1	**44**	1312734
						128.7	**60**	488880
						128.2	**60**	289546
						116.1	**60**	410274
						115.1	**60**	480211
						77.1	**60**	306644
	LY2183240	C17H17N5O	**FIU_0461**	10.09	**308.14331**	280.2	**0**	310087
						192.2	**16**	4093
						167.1	**16**	106535
						165.1	**60**	12344
						152.1	**56**	8932
						87.1	**12**	30449
						72.2	**32**	146626
						59.2	**24**	5470
						56.1	**60**	4204

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	MN-18	C23H23N3O	**FIU_0728**	11.78	**358.18411**	215	**16**	293262
						144.9	**40**	165572
						121.1	**48**	102
						116.9	**60**	20923
						90	**60**	34319
						89.5	**60**	11458
	Phenylpiracetam	C12H14N2O2	**FIU_0766**	7.51	**219.10553**	202	**4**	65359
						174	**8**	107141
						145	**20**	33287
						129	**24**	22618
						128	**44**	6897
						117	**36**	20935
						115	**52**	13111
						91	**60**	19074
						77	**60**	8740
						55.1	**36**	5891
10	**(S)-(+)-JWH 018 N-(4-hydroxypentyl) metabolite**	C24H23NO2	**FIU_0473**	10.33	**358.17288**	340.2	**16**	10864
						284.2	**24**	15422

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						230.1	**24**	11572
						186.2	**12**	19319
						155.1	**20**	837610
						144.1	**40**	31233
						127.1	**56**	568140
						116.1	**60**	11876
						77.2	**60**	9962
						69.2	**40**	23042
	(S)-(+)-MT-45	C24H32N2	**FIU_0325**	9.61	**349.25655**	181.1	**24**	1348728
						179.1	**40**	109145
						169.2	**16**	481881
						167.2	**16**	110702
						166.1	**40**	463168
						165.1	**60**	376021
						153.1	**40**	54200
						103.1	**56**	452122
						87.2	**32**	143251
						77.1	**60**	148661
	2,4,6-Trimethoxyamphetamine	C12H19NO3	**FIU_0280**	8.02	**226.14**	209.1	**4**	1206523

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						181.1	**20**	500507
						151.1	**24**	65931
						136	**32**	83243
						121.1	**28**	176428
						93.1	**52**	54798
						91.1	**40**	144129
						78.1	**60**	80617
						77.1	**60**	110784
						65.1	**60**	104737
	2-Amino-1-phenylbutane	C10H15N	**FIU_0320**	7.19	**150.12045**	133.1	**4**	310981
						105	**8**	1547
						91.1	**16**	654486
						65.1	**44**	215451
						63.1	**60**	20547
						51.1	**60**	19569
	2C-G	C12H19NO2	**FIU_0310**	8.57	**210.14158**	193.1	**4**	344603
						178.1	**12**	658131
						163.1	**28**	284498
						133.1	**20**	32164

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						115.1	**52**	35114
						105.1	**40**	50956
						91.1	**48**	113732
						79.1	**44**	47953
						77.1	**60**	62488
						65.1	**60**	44531
	4-bromo-2,5-DMMA	C12H18BrNO2	**FIU_0306**	8.33	**288.05209**	257	**12**	408487
						229	**20**	204439
						199	**32**	65880
						178.1	**20**	215263
						163.1	**32**	97931
						135.1	**40**	117116
						105.1	**48**	114322
						91.1	**60**	72974
						79.1	**60**	51580
						77.1	**60**	104526
	4-fluoromethcathinone (4-FMC)	C10H12FNO	**FIU_0007**	5.89	**182.1**	164.1	**12**	1980509
						149	**20**	1196810
						148	**36**	642220

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						123	**20**	267291
						103	**32**	343195
						101	**56**	154930
						77.1	**48**	408254
						75.1	**60**	217329
						58.1	**40**	65859
						51.1	**60**	139634
	5-methoxy DMT	C13H18N2O	**FIU_0015**	6.12	**219.1**	174.1	**12**	1425105
						159.1	**28**	435091
						143	**32**	189080
						131.1	**40**	347719
						130	**52**	600002
						115	**52**	79862
						103.1	**60**	155994
						78.1	**60**	80299
						77.1	**60**	138656
						58.1	**12**	2605704
	AM251	C22H21Cl2IN4O	**FIU_0166**	11.85	**555**	454.9	**32**	1667176
						328	**56**	268291

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						299	**60**	54521
						265	**60**	112885
						255.9	**56**	73088
						159.9	**60**	52877
						129	**60**	170832
						99.1	**32**	45102
						84.1	**32**	308062
						55.1	**60**	38288
	AMB	C19H27N3O3	**FIU_0744**	11.23	**346.20524**	314.1	**8**	32421
						300.2	**12**	6456
						286.1	**12**	205903
						215.1	**24**	389298
						145	**44**	225686
						125.8	**16**	62
						117	**56**	27954
						90	**60**	48007
						89	**60**	16840
						89	**12**	29793
						71.1	**40**	2899

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						55.1	**20**	41361
	Diethylcathinone	C13H19NO	**FIU_0347**	6.32	**206.14666**	133.1	**16**	186937
						130.1	**44**	26890
						105.1	**24**	430105
						103.1	**40**	47096
						100.1	**24**	230351
						79.1	**40**	81874
						77.1	**56**	231588
						72.1	**16**	84231
						58.1	**32**	75245
						51.1	**60**	62423
	JWH 073 6-hydroxyindole metabolite	C23H21NO2	**FIU_0503**	10.56	**344.15723**	216.1	**24**	98395
						213.3	**32**	107
						160.1	**40**	64696
						136.9	**16**	144
						132	**60**	33652
						127	**56**	1118593
						105	**60**	13126
						77.1	**60**	39530

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						57.1	**40**	7292
	JWH 203 3-chlorophenyl isomer	C21H22ClNO	**FIU_0068**	11.43	**340.2**	339.4	**0**	8250
						203.4	**0**	40
						124.9	**24**	1178
						75	**56**	93
						57.2	**32**	720
	JWH 203 N-pentanoic acid metabolite	C21H20ClNO3	**FIU_0537**	10.12	**370.11317**	218.1	**16**	77909
						200.1	**16**	182016
						172.1	**32**	34683
						156	**40**	21682
						144	**44**	23472
						130	**60**	19993
						125	**36**	429943
						118	**36**	7164
						89.1	**60**	28460
						55.1	**56**	34506
	JWH 210 2-ethylnaphthyl isomer	C26H27NO	**FIU_0538**	11.64	**370.20926**	214.1	**24**	841275

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						183.1	**24**	1267260
						165.1	**44**	87040
						165.1	**40**	173014
						155.1	**44**	914973
						155.1	**44**	436246
						153.1	**60**	288961
						153.1	**56**	598823
						144.1	**44**	1089196
						144	**44**	540680
						141.1	**40**	623325
						141.1	**36**	299439
						128.7	**60**	204759
						128.7	**60**	103625
						116.1	**60**	382218
						115.6	**60**	260556
						115.6	**60**	177042
						115.2	**60**	126721
	JWH 210 N-pentanoic acid metabolite	C26H25NO3	**FIU_0542**	10.76	**400.18344**	244.1	**24**	38733
						183.1	**28**	400033

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						155.1	**44**	107188
						153.9	**60**	36926
						144	**40**	36043
						129	**60**	31922
						127	**60**	14454
						115.1	**60**	28572
						83.1	**40**	9650
						55.1	**56**	25064
	RCS-4 N-(5-hydroxypentyl) metabolite	C21H23NO3	**FIU_0576**	14.53	**338.16779**	321.3	**8**	20744
						111.1	**20**	10109
						97.1	**24**	17556
						95.1	**20**	8011
						83.1	**24**	15315
						81.1	**28**	8002
						71.1	**20**	9805
						69.1	**36**	22008
						57.2	**36**	20356
						55.2	**56**	26679
11	**2,4-Dimethylethcathinone**	C13H19NO	**FIU_0346**	8.1	**206.14666**	188.2	**8**	795999

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						173.1	**20**	242215
						165.1	**8**	1505695
						160.3	**16**	159358
						159.1	**20**	294710
						158.1	**32**	385094
						145.1	**24**	101115
						144.1	**40**	101386
						115.1	**60**	91814
						91.1	**56**	95586
						72.1	**12**	87081
	25I-NBOMe 3-methoxy isomer	C18H22INO3	**FIU_0380**	8.96	**428.06444**	291	**20**	270909
						276	**32**	99966
						272.1	**16**	223920
						174.1	**8**	2194709
						164.1	**32**	58691
						149.1	**40**	45034
						134.1	**44**	39398
						121.1	**32**	794681
						106.1	**60**	58169

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						91.1	**60**	376311
						77.1	**60**	141918
	2C-C	C10H14ClNO2	**FIU_0143**	7.54	**216.1**	199	**8**	987049
						184	**16**	465013
						174.1	**8**	14572
						169	**32**	260080
						164	**20**	96218
						103	**28**	42645
						91	**48**	130393
						78.1	**60**	60265
						77.1	**48**	221729
						65.1	**60**	70213
						51.1	**60**	67653
	2C-H	C10H15NO2	**FIU_0147**	6.55	**182.1**	150	**16**	937820
						135	**32**	451049
						107	**40**	102394
						105.1	**24**	170097
						103	**32**	114571
						91.1	**48**	97059

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						79.1	**36**	206042
						77.1	**52**	479482
						51.1	**60**	155428
	2-Methyl-a-pyrrolidinobutiophenone (HCl)	C15H21NO	**FIU_0121**	7.2	**232.2**	161.1	**16**	712959
						133.1	**20**	303253
						119	**28**	751316
						112.1	**24**	1055298
						105.1	**24**	1401788
						91.1	**48**	1205154
						84.1	**32**	223744
						77.1	**60**	235736
						70.1	**44**	189210
						65.1	**60**	540930
	3,4-Dimethylmethcathinone (HCl)	C12H17NO	**FIU_0123**	7.21	**192.1**	159.1	**20**	2665048
						158.1	**36**	1503972
						144	**36**	705768
						133.1	**20**	219496

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						117.1	**48**	121135
						115	**60**	333069
						105.1	**32**	169940
						91.1	**56**	336569
						77.1	**60**	285566
	4-Chloroamphetamine	C9H12ClN	**FIU_0291**	7.82	**170.06583**	153	**4**	158378
						125	**16**	207100
						99.1	**48**	20840
						90.9	**52**	2380
						90.1	**44**	21373
						89.1	**48**	46112
						75.1	**48**	3769
						73.1	**60**	11804
						65.1	**52**	5733
						63.1	**60**	24032
	4-fluoro-a-Pyrrolidinopentiophenone	C15H20FNO	**FIU_0398**	7.44	**250.15289**	179.1	**16**	228898
						126.1	**28**	287573
						123	**32**	197197
						109.1	**24**	558343

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						97.1	**48**	45289
						95.1	**56**	252583
						84.1	**36**	128980
						75.1	**60**	84285
						72.2	**28**	45165
						70.1	**20**	96416
	4-Fluoromethcathinone metabolite ((±)-Ephedrine stereochemistry)	C10H14FNO	**FIU_0354**	6.19	**184.10594**	166.1	**8**	818656
						151.1	**20**	142427
						135.1	**20**	115570
						133.1	**32**	48524
						122	**36**	16291
						115.1	**28**	95728
						109.1	**36**	116852
						83.1	**60**	48972
						70.1	**20**	29709
						56.2	**36**	16915
	4-methoxy DMT	C13H18N2O	**FIU_0144**	11.71	**219.1**	186.1	**4**	189442
						174.1	**12**	765906

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						159	**28**	289512
						143.1	**36**	99279
						131	**40**	78350
						130	**52**	234200
						117	**36**	121096
						115	**52**	191205
						91.1	**56**	91946
						77.1	**60**	112737
						58.1	**12**	3365262
	4'-Methoxy-a-pyrrolidinopropiophenone (tosylate)	C14H19NO2	**FIU_0145**	6.53	**234.1**	163.1	**16**	1147358
						135.1	**24**	1348034
						105	**36**	503713
						103.1	**48**	248455
						98.1	**24**	1790794
						91.1	**60**	237389
						79.1	**48**	342539
						77.1	**60**	624919
						56.1	**60**	440154

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						55.1	**52**	189835
	4-methyl-a-Ethyltryptamine	C13H18N2	**FIU_0751**	7.98	**203.147**	146	**8**	15733
						144	**16**	161319
						130	**48**	8537
						129.2	**44**	6810
						128	**56**	6728
						115	**56**	20218
						91	**52**	15633
						77.1	**60**	8418
						58.1	**20**	16352
	4'-Methyl-a-pyrrolidinohexanophenone (HCl)	C17H25NO	**FIU_0149**	8.49	**260.2**	324.1	**12**	63450
						253	**20**	67276
						189.1	**16**	1315960
						140.1	**28**	1054542
						119	**28**	892220
						105.1	**24**	2873232
						91.1	**56**	1121485
						84.1	**40**	487725

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						77.1	**60**	344672
						72.1	**20**	243482
						70.1	**20**	210572
						65.1	**60**	421791
	5-fluoro PB-22	C23H21FN2O2	**FIU_0578**	10.62	**377.15871**	232.1	**12**	2654377
						212.1	**40**	21801
						209.1	**4**	185715
						158.1	**40**	22853
						144	**44**	1192067
						130.1	**52**	19785
						116	**60**	443248
						89.1	**60**	42273
						69.1	**44**	91102
						67.2	**60**	8837
						61.2	**56**	15142
	5-fluoro PB-22 N-(2-fluoropentyl) isomer	C23H21FN2O2	**FIU_0590**	10.82	**377.15871**	232.1	**12**	574530
						212	**40**	28610
						176	**44**	6320
						158	**44**	6128

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						144	**48**	155674
						130	**56**	6148
						129.1	**60**	5787
						116	**60**	45782
						69.1	**48**	30593
						67.1	**56**	3916
	5-methoxy DALT	C17H22N2O	**FIU_0154**	7.02	**271.2**	174.1	**16**	2100684
						159	**36**	696857
						143	**40**	289793
						131	**44**	497275
						130	**60**	904120
						115	**60**	115652
						110.1	**12**	3448027
						81.1	**32**	215470
						79.1	**44**	163195
						68.1	**28**	112500
	6-IT	C11H14N2	**FIU_0688**	6.57	**175.1157**	158.1	**4**	196250
						143.1	**28**	12674
						130.1	**20**	80112

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						117.1	**24**	38179
						115.1	**52**	14362
						103.1	**40**	9831
						91.1	**40**	5100
						90.1	**52**	10716
						89.1	**60**	13410
						77.1	**52**	25675
	AB-FUBINACA isomer 1	C20H21FN4O2	**FIU_0718**	10.15	**369.16485**	352.1	**4**	58898
						109	**52**	60489
	AM2201 benzimidazole analog	C23H21FN2O	**FIU_0457**	11.09	**361.16379**	273.1	**24**	140896
						233.1	**20**	226397
						177.1	**28**	382773
						155.1	**32**	692290
						145.1	**32**	201946
						129.1	**48**	116825
						127.1	**60**	852792
						117.1	**56**	41239
						102.1	**60**	40141
						90.1	**60**	36387

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	AM630	C23H25IN2O3	**FIU_0167**	10.37	**505.1**	135	**24**	1615419
						114.1	**40**	225440
						107	**60**	151319
						100.1	**48**	42491
						92	**60**	28435
						86.1	**60**	7886
						84.1	**60**	23231
						79.1	**60**	17900
						77.1	**60**	171294
						70.1	**60**	68974
	AM694 N-pentanoic acid metabolite	C20H18INO3	**FIU_0699**	9.9	**448.03314**	244	**36**	7692
						234.1	**40**	11226
						230.9	**24**	414779
						220	**28**	11728
						202.9	**60**	173851
						200.1	**12**	12623
						144	**56**	10121
						104	**60**	24861
						76	**60**	69513

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						55.1	**60**	13929
	Buphedrone metabolite ((±)-Pseudoephedrine stereochemistry)	C11H17NO	**FIU_0430**	6.27	**180.13101**	162.1	**8**	1088855
						133.1	**20**	213663
						132.1	**32**	98325
						131.1	**16**	100686
						117.1	**52**	29999
						104.1	**36**	45698
						91.1	**36**	210167
						77.1	**60**	46459
						70.1	**24**	34837
						65.1	**60**	72759
	CB-52	C26H43NO3	**FIU_0178**	11.95	**418.3**	361.2	**12**	442484
						224.2	**12**	122835
						125	**24**	161615
						123	**44**	184095
						83.1	**32**	69180
						81.1	**40**	48298
						69.1	**44**	60095

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						67.1	**60**	62669
						58.1	**20**	2474757
						55.1	**60**	166212
	Dimethocaine	C16H26N2O2	**FIU_0432**	6.27	**279.19943**	206.1	**16**	75459
						160.2	**16**	92041
						149	**12**	7976
						142.1	**16**	496900
						120	**24**	841008
						98.1	**44**	25077
						92.1	**52**	384112
						86.2	**32**	466953
						65.1	**60**	384141
						58.2	**48**	102919
	DPT	C16H24N2	**FIU_0755**	7.42	**245.19395**	144	**24**	171262
						128	**48**	6590
						127	**48**	22985
						117	**40**	38226
						114.1	**12**	341500
						102.1	**12**	14512

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						91	**60**	28506
						86.1	**28**	55626
						77	**60**	15700
						72.1	**32**	23049
	Escaline	C12H19NO3	**FIU_0318**	6.99	**226.13649**	181.1	**12**	137130
						166.1	**20**	17023
						121.1	**20**	26900
						103.1	**28**	20788
						93.1	**24**	22839
						91.1	**36**	51623
						78.3	**56**	11686
						77.1	**48**	47579
						65.1	**60**	23350
	Etizolam	C17H15ClN4S	**FIU_0677**	9.55	**343.07059**	314.1	**24**	73954
						289.1	**24**	11811
						279.1	**28**	5588
						259.1	**40**	13381
						224.1	**48**	11313
						223	**52**	9352

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						210	**48**	6615
						206.1	**24**	11288
						191.1	**52**	1735
						138	**40**	12639
	Eutylone (hydrochloride)	C13H17NO3	**FIU_0185**	6.7	**236.1**	218.1	**12**	1383223
						189.1	**20**	842136
						188.1	**16**	1843317
						174	**36**	756834
						160.1	**24**	400784
						145.1	**40**	250103
						116.6	**48**	279272
						116	**56**	167123
						86.1	**16**	323008
						65.1	**60**	374725
	Hydroxy Bupropion	C13H18ClNO2	**FIU_0761**	7.58	**256.10261**	238.1	**8**	220908
						167	**20**	40913
						166.5	**20**	20295
						139	**28**	41290
						131	**28**	31026

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						130	**60**	43493
						115	**52**	11580
						103	**48**	25437
						77	**60**	24691
						55.1	**32**	13209
	JWH 018 benzimidazole analog	C23H22N2O	**FIU_0481**	11.69	**343.17321**	273.1	**20**	368881
						215.1	**20**	750681
						159.1	**28**	243050
						155	**32**	1345243
						155	**24**	1835622
						147	**28**	105809
						145	**28**	515310
						131.1	**36**	212214
						127	**60**	1789735
						117	**52**	107340
						90.1	**60**	96763
	JWH 018 N-(4-oxo-pentyl) metabolite	C24H21NO2	**FIU_0483**	10.31	**356.15723**	272.1	**20**	60913
						228.1	**16**	29547

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						211.6	**8**	90
						184	**16**	9683
						155	**24**	922434
						155	**20**	2588699
						144	**36**	61251
						127	**56**	1541042
						116	**60**	18891
						85.1	**32**	246131
						77.1	**60**	33814
	JWH 019 N-(5-fluorohexyl) isomer	C25H24FNO	**FIU_0492**	11.09	**374.18419**	354.2	**20**	119050
						284.2	**24**	23901
						246.1	**24**	137610
						226.1	**32**	31390
						176	**36**	20211
						155	**28**	1411279
						144	**48**	103486
						127	**60**	1170744
						116	**60**	46287
						55.1	**52**	48828

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	JWH 030 2-naphthoyl isomer	C20H21NO	**FIU_0497**	11.52	**292.16231**	169.1	**28**	1262
						164.1	**16**	198288
						155	**16**	1700750
						136.1	**24**	18390
						127	**48**	1009673
						108	**32**	34589
						101.1	**60**	19416
						94	**36**	97720
						80.1	**32**	29778
						77.1	**60**	101277
						66.1	**60**	43408
	JWH 073 5-hydroxyindole metabolite	C23H21NO2	**FIU_0502**	10.6	**344.15723**	310.7	**4**	45
						216.1	**24**	134220
						160	**40**	89532
						132	**60**	40736
						127	**56**	824013
						120.9	**20**	150
						105.1	**60**	5121

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						77.1	**60**	26304
						57.2	**44**	9523
	JWH 073 7-hydroxyindole metabolite	C23H21NO2	**FIU_0505**	10.85	**344.15723**	216.1	**24**	291460
						174.1	**36**	4426
						160	**40**	258390
						132	**56**	49055
						127	**56**	1651577
						104.1	**60**	102079
						101.1	**60**	9350
						77.1	**60**	62160
						57.1	**40**	19100
	JWH 073 N-butanoic acid metabolite	C23H19NO3	**FIU_0509**	10.1	**358.13649**	230.1	**20**	36441
						212	**24**	12852
						155	**24**	682790
						144	**40**	41386
						127	**52**	478799
						116	**60**	12998
						87.1	**36**	22745

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						77.1	**60**	10267
	JWH 122 2-methylnaphthyl isomer	C25H25NO	**FIU_0061**	11.49	**356.2**	214.2	**20**	535
						141	**44**	996
	JWH 122 7-methylnaphthyl isomer	C25H25NO	**FIU_0065**	11.72	**356.2**	141.1	**44**	1111
						115	**60**	630
	JWH 122 N-(5-hydroxypentyl) metabolite	C25H25NO2	**FIU_0521**	10.61	**372.18853**	230.1	**28**	71118
						227.8	**60**	129
						223.4	**56**	144
						169.1	**24**	1345702
						144	**44**	94599
						141.1	**52**	735704
						140.4	**52**	41798
						115.1	**60**	263250
						69.2	**44**	26437
	JWH 210 5-ethylnaphthyl isomer	C26H27NO	**FIU_0072**	11.96	**370.2**	214.2	**24**	1244992
						183.1	**24**	5912549

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						155.1	**40**	2383227
						153.1	**56**	1658477
						144.1	**44**	786248
						128.7	**60**	611379
						128.2	**60**	363568
						116	**60**	259945
						115.1	**60**	649101
						77.1	**60**	294169
	JWH 210 N-(5-hydroxypentyl) metabolite	C26H27NO2	**FIU_0541**	10.84	**386.20418**	230.1	**28**	84540
						183	**24**	1298232
						155.1	**44**	317176
						153.9	**60**	109044
						153	**56**	243491
						144	**48**	92372
						129	**60**	93591
						127.9	**60**	36917
						115	**60**	96031
						77.1	**60**	38162

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	JWH 250 N-pentanoic acid metabolite	C22H23NO4	**FIU_0547**	9.93	**366.16271**	200.1	**16**	29494
						131.1	**32**	13369
						130.1	**40**	31165
						121.1	**20**	513783
						93.1	**40**	48991
						91.1	**56**	291466
						83.1	**44**	11566
						77.1	**60**	17423
						65.1	**60**	20063
						55.2	**60**	42411
	N-Phenylacetyl-L-prolylglycine ethyl ester	C17H22N2O4	**FIU_0765**	8.52	**319.15796**	216	**4**	280812
						201.1	**12**	6715
						188.1	**12**	265785
						91	**60**	84296
						70.1	**24**	409046
						65.1	**60**	6486
	PB-22 5-hydroxyisoquinoline isomer	C23H22N2O2	**FIU_0601**	11.3	**359.16813**	214.1	**24**	1875489

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						174.1	**40**	117
						158.1	**40**	28091
						144	**44**	940990
						130.1	**56**	19928
						116	**60**	301147
						89.1	**60**	32696
						71.2	**40**	14989
						55.2	**60**	8188
	PB-22 8-hydroxyisoquinoline isomer	C23H22N2O2	**FIU_0607**	11.3	**359.16813**	214.1	**16**	549358
						158	**40**	7238
						144	**44**	230136
						130	**48**	5274
						116	**60**	74609
						89	**60**	8750
						71.1	**36**	4206
						55.1	**60**	2421
	PB-22 N-pentanoic acid metabolite	C23H20N2O4	**FIU_0612**	9.94	**389.14231**	244.1	**12**	1226669

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						172.1	**32**	9977
						156.1	**36**	6109
						144	**40**	425898
						116	**60**	107190
						101.1	**36**	122565
						89.1	**60**	8807
						83.1	**40**	130835
						59.1	**60**	67042
						55.2	**60**	293916
	PV8	C17H25NO	**FIU_0413**	8.47	**260.19361**	189.2	**16**	87330
						154.2	**28**	248551
						119.1	**20**	115271
						105.1	**32**	195068
						91.1	**28**	633622
						84.2	**40**	128743
						77.2	**60**	323753
						72.2	**32**	56424
						70.2	**20**	125628
						55.2	**56**	40688

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	RCS-4 3-methoxy isomer	C21H23NO2	**FIU_0086**	11.3	**322.2**	214.1	**24**	419753
						144	**40**	363539
						135	**24**	5293292
						116	**60**	165554
						107	**36**	2406541
						92	**60**	1221544
						79.1	**48**	222296
						77.1	**60**	3034318
						64.1	**60**	193370
						51.1	**60**	125214
	Sildenafil	C22H30N6O4S	**FIU_0768**	8.69	**475.20492**	291.1	**48**	56
						283	**44**	2094
						100.1	**32**	6705
						99.6	**32**	4373
						58.1	**52**	16295
						56	**52**	1140
	Sildenafil Citrate	C22H30N6O4S	**FIU_0769**	8.69	**475.20492**	283.1	**44**	1906
						100.1	**32**	6317
						99.6	**28**	3824

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						70.1	**56**	999
						58.1	**48**	15123
						56.1	**56**	1181
	URB937	C20H22N2O4	**FIU_0273**	9.56	**355.2**	230	**8**	330117
						213	**24**	363074
						187	**28**	59223
						185	**36**	41119
						169	**40**	40962
						157	**48**	60945
						141	**52**	77129
						139	**56**	21273
						128	**60**	48593
						115	**60**	52598
	XLR11 Degradant	C21H28FNO	**FIU_0660**	11.09	**330.21549**	232.1	**24**	247404
						216.8	**40**	53
						167.1	**56**	179
						144	**40**	117723
						130.1	**56**	2416
						116.1	**60**	50927

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						89.1	**60**	9664
						83.1	**32**	4543
						69.2	**40**	10825
						55.2	**56**	8715
	XLR11 N-(4-hydroxypentyl) metabolite	C21H28FNO2	**FIU_0664**	10.42	**346.21041**	347.3	**0**	201476
						248.7	**20**	66194
						248.1	**20**	1230020
						210	**8**	292584
						144	**36**	829514
						116	**60**	238449
						87.1	**36**	121677
						67.1	**48**	205737
						59.1	**60**	137686
						57.1	**44**	49883
						55.1	**56**	105980
12	**(-)-CP 55,940**	C24H40O3	**FIU_0091**	11.68	**377.3**	301.2	**4**	43476
						233.2	**12**	58611
						216.1	**20**	131
						175.1	**4**	61840

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						71.1	**20**	30646
	(±)-JWH 018 N-(3-hydroxypentyl) metabolite	C24H23NO2	**FIU_0470**	10.58	**358.17288**	284.1	**24**	47167
						230.2	**24**	32699
						174.1	**8**	720367
						158.1	**32**	34602
						155.1	**24**	965041
						144.1	**44**	19174
						141.1	**32**	7420
						130.1	**52**	23549
						127.1	**56**	691464
						77.2	**60**	14360
						59.2	**48**	7184
	1'-Naphthoyl-2-methylindole	C20H15NO	**FIU_0681**	10.3	**286.11536**	158.1	**20**	61936
						130.1	**44**	16449
						128	**56**	2163
						127	**40**	151965
						121.1	**16**	1677157
						103.1	**56**	9937

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						101	**60**	4656
						77.1	**60**	34676
	2,3-Dimethylmethcathinone	C12H17NO	**FIU_0348**	7.58	**192.13101**	159.1	**20**	666939
						158.1	**32**	340794
						144.1	**36**	181175
						116.8	**52**	20047
						115.1	**56**	85202
						105.1	**32**	32381
						91.1	**56**	76106
						77.1	**60**	69453
						58.2	**24**	28737
	25N-NBOMe	C18H22N2O5	**FIU_0383**	7.98	**347.15287**	93.1	**36**	200830
						91.1	**52**	1330738
						77.1	**60**	34996
						65.1	**60**	187609
	2C-N	C10H14N2O4	**FIU_0155**	6.55	**227.1**	151	**16**	65265
						137	**16**	27450
						107	**28**	14687
						105.1	**36**	19878

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						103.1	**40**	26554
						91	**44**	66672
						79.1	**40**	27742
						77.1	**56**	93303
						65.1	**60**	60175
	2-Ethylamino-1-phenylbutane	C12H19N	**FIU_0321**	7.43	**178.15175**	133.1	**8**	169511
						105.1	**12**	3808
						91.1	**16**	925276
						65.1	**56**	309318
						63.1	**60**	24781
						51.1	**60**	16920
	2-Ethylmethcathinone (hydrochloride)	C12H17NO	**FIU_0113**	7.21	**192.1**	146.1	**16**	736520
						145.1	**20**	1376349
						144.1	**36**	1432023
						131	**28**	239164
						130	**44**	140197
						128	**48**	139152
						103.1	**56**	127973

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						77.1	**60**	414584
						58.1	**28**	71419
	2-Fluoroamphetamine (hydrochloride)	C9H12FN	**FIU_0114**	6.21	**154.1**	137	**4**	924895
						109	**16**	1881739
						89.1	**40**	46503
						83.1	**44**	604590
						81.1	**48**	16666
						75.1	**60**	13315
						65.1	**52**	15318
						63.1	**56**	122396
						59.1	**48**	94382
						57.1	**60**	304545
	3,4-Dimethylmethcathinone metabolite ((±)-Ephedrine stereochemistry)	C12H19NO	**FIU_0350**	7.78	**194.14666**	176.1	**8**	669818
						161.1	**20**	99275
						145.1	**20**	56679
						130.1	**28**	47662
						129.1	**36**	33898

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						115.1	**56**	31667
						105.1	**32**	58958
						91.1	**48**	27896
						77.1	**60**	34026
						56.1	**28**	41907
	3,4-Dimethylmethcathinone metabolite ((±)-Pseudoephedrine stereochemistry)	C12H19NO	**FIU_0351**	7.74	**194.14666**	176.1	**8**	910702
						174.1	**8**	715283
						161.1	**24**	135654
						145.7	**28**	55552
						145.1	**20**	75197
						130.1	**28**	59990
						129.1	**40**	45849
						115.1	**52**	44416
						105.1	**32**	67546
						91.1	**52**	41421
						56.2	**32**	57003

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	3-Ethylmethcathinone (HCl)	C13H19NO	**FIU_0127**	7.33	**206.2**	178.1	**8**	491172
						146	**16**	4561
						145.1	**24**	8362
						144.1	**36**	10491
						131.1	**24**	835
						77	**60**	2458
	3-Fluoroamphetamine (HCl)	C13H19NO	**FIU_0128**	6.28	**206.2**	134.8	**20**	115
	3-Iodoamphetamine	C9H12IN	**FIU_0288**	8.39	**262.00144**	245	**8**	116842
						216.9	**16**	164042
						213	**8**	2310
						183.2	**20**	1651
						118.1	**20**	41960
						117.1	**40**	87754
						115.1	**60**	29667
						91	**60**	44327
						90.1	**40**	114642
						89.1	**60**	93152
	3-methoxy PCP	C18H27NO	**FIU_0386**	8.67	**274.20926**	189.1	**12**	476961

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						121.1	**28**	592000
						91.1	**56**	255444
						86.1	**8**	1047493
						81.1	**24**	173567
						79.1	**52**	45930
						78.1	**60**	100685
						77.1	**60**	115538
						69.2	**56**	26960
						65.1	**60**	94306
	4-Bromoamphetamine	C9H12BrN	**FIU_0290**	8.1	**214.01531**	348.1	**4**	27217
						320.1	**12**	30764
						249	**24**	28269
						197	**8**	79688
						169	**16**	86136
						119.5	**20**	98
						118.1	**20**	25826
						117.1	**36**	37896
						115.1	**60**	11525
						91.1	**60**	20024

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						90.1	**44**	31054
						89.1	**60**	27467
						64.2	**60**	2641
	4-Fluorobuphedrone	C11H14FNO	**FIU_0426**	6.54	**196.10594**	150.1	**16**	179807
						149.1	**24**	299614
						148	**40**	165491
						147	**16**	1620514
						135	**32**	41709
						119.1	**24**	2148167
						109.1	**28**	137909
						101.1	**60**	44622
						95.1	**48**	58274
						83.1	**56**	46606
						75.1	**60**	89841
	4'-Methyl-a-pyrrolidinopropiophenone (HCl)	C14H19NO	**FIU_0150**	6.83	**218.2**	117	**36**	365629
						98.1	**28**	1597591
						91	**44**	988360
						77.1	**60**	434401

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						70.1	**20**	255411
						65.1	**60**	368092
						56.1	**56**	518796
						55.1	**48**	215520
	4-Methylbuphedrone	C12H17NO	**FIU_0427**	7.15	**192.13101**	161.1	**8**	186293
						159.1	**16**	93287
						146.1	**16**	183068
						145.1	**20**	356441
						144.1	**36**	365359
						105.1	**24**	158574
						91.1	**44**	76908
						77.1	**60**	120838
						65.1	**60**	73948
	4-methylethcathinone (4-MEC)	C12H17NO	**FIU_0009**	7.03	**192.1**	174.1	**8**	2536175
						146.1	**16**	1030649
						145.1	**20**	1207627
						144.1	**32**	1201326
						131.1	**28**	462798
						130.1	**44**	381702

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						119.1	**24**	386789
						115	**56**	209152
						91.1	**40**	577057
						77.1	**60**	376686
	5-fluoro AB-PINACA N-(4-hydroxypentyl) metabolite	C18H25FN4O3	**FIU_0705**	8.93	**365.19107**	222.3	**40**	71
						174.9	**40**	2447
						155.1	**28**	4845629
						152.5	**32**	68
						144.9	**48**	16562
						67.1	**60**	3763
	5-fluoro PB-22 4-hydroxyisoquinoline isomer	C23H21FN2O2	**FIU_0581**	10.8	**377.15871**	232.1	**20**	289686
						212	**40**	2916
						200	**28**	45
						180.6	**60**	61
						158	**40**	2486
						144	**44**	137719
						116	**60**	47185

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						89	**60**	3930
						69.1	**48**	9809
						61.1	**60**	1736
	5-fluoro PB-22 6-hydroxyisoquinoline isomer	C23H21FN2O2	**FIU_0585**	10.44	**377.15871**	232	**20**	282512
						212.1	**40**	3133
						158	**40**	2264
						144	**48**	132682
						141.8	**60**	124
						116	**60**	50425
						89	**60**	4592
						69.1	**44**	10149
						61.1	**56**	1616
	5-fluoro PB-22 N-(4-fluoropentyl) isomer	C23H21FN2O2	**FIU_0592**	10.64	**377.15871**	352	**4**	64567
						324.1	**12**	61697
						253	**24**	59264
						232.1	**12**	459513
						212.1	**36**	36483

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						176	**40**	4295
						158	**48**	4630
						144	**44**	160238
						130	**56**	2890
						116	**60**	57799
						89	**60**	6760
						69.1	**48**	27476
						61.1	**60**	2220
	5-Fluoropentyl-3-pyridinoylindole	C19H19FN2O	**FIU_0685**	9.89	**311.14814**	235.1	**24**	11835
						232.2	**32**	17185
						223.1	**28**	10462
						205.1	**36**	5103
						194.1	**36**	9538
						144.1	**44**	36103
						116.1	**60**	22055
						106	**28**	12614
						80.1	**44**	14298
						78.1	**48**	9683
	AB-FUBINACA isomer 2	C20H21FN4O2	**FIU_0719**	9.94	**369.16485**	109	**48**	59051

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	Acetildenafil	C25H34N6O3	**FIU_0771**	8.53	**467.26924**	297.1	**48**	24767
						166.1	**60**	25303
						127.1	**36**	46811
						112.2	**36**	28000
						111	**32**	66049
						97.1	**60**	22972
						84.1	**48**	36287
						72.1	**48**	32613
						70.1	**60**	34349
						56.1	**60**	11976
	ADBICA N-(4-hydroxypentyl) metabolite	C20H29N3O3	**FIU_0695**	9.63	**360.22089**	343.2	**4**	584215
						315.2	**12**	5344
						257.1	**8**	8459
						230.1	**20**	602090
						144	**36**	280196
						116	**60**	57897
						104.1	**4**	3889
						89	**60**	6065

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						87.1	**32**	8109
						69.1	**44**	185100
	ADBICA N-pentanoic acid metabolite	C20H27N3O4	**FIU_0697**	9.52	**373.20016**	357.1	**8**	322095
						329.1	**12**	7203
						244	**20**	326262
						172.1	**40**	2222
						144	**40**	116045
						116	**60**	26602
						101	**40**	30463
						83	**44**	31052
						59.1	**60**	19717
						55.1	**60**	94469
	AM1241	C22H22IN3O3	**FIU_0161**	8.69	**504.1**	407	**20**	119512
						275.9	**40**	66098
						229.9	**60**	53848
						112.1	**28**	417591
						98.1	**28**	3132890
						84.1	**60**	18049
						81.1	**56**	18914

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						70.1	**60**	306760
						58.1	**60**	151378
						55.1	**60**	21745
	AM2201 N-(3-fluoropentyl) isomer	C24H22FNO	**FIU_0029**	11.23	**360.2**	232.1	**24**	894009
						212.1	**36**	101562
						163.1	**8**	34633
						155.1	**28**	5352422
						144	**44**	332788
						127.1	**56**	4564750
						116.1	**60**	153770
						105	**44**	20278
						77.1	**60**	115583
						69.1	**48**	160157
	AM2201 N-(4-fluoropentyl) isomer	C24H22FNO	**FIU_0030**	11.02	**360.2**	340.2	**20**	120647
						284.1	**24**	83465
						232.1	**24**	851798
						212.1	**36**	127795
						144	**44**	492945

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						127.1	**60**	4729212
						116.1	**60**	235998
						77.1	**60**	112815
						69.1	**44**	99980
	AM2201 N-(4-hydroxypentyl) metabolite	C24H22FNO2	**FIU_0632**	10.15	**376.16346**	248.1	**24**	10447
						155	**28**	170129
						144	**40**	15849
						127	**60**	123105
						116	**60**	3892
						87.1	**40**	1643
						67.1	**48**	3631
						59.1	**60**	2061
	AM2232	C24H20N2O	**FIU_0164**	10.24	**353.2**	225.1	**24**	539640
						155	**24**	718606
						155	**20**	4192055
						144	**48**	339634
						129	**56**	20337
						127	**56**	3291319

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						116	**60**	230106
						89	**60**	47582
						82.1	**40**	24923
						77.1	**60**	98507
						55.1	**60**	92567
	deschloro-N-ethyl-Ketamine	C14H19NO	**FIU_0297**	6.97	**218.14666**	173.1	**8**	551118
						145.1	**16**	432062
						131.1	**28**	32303
						129.1	**24**	34373
						117.1	**32**	58630
						115.1	**52**	55787
						91.1	**36**	767639
						77.1	**60**	75845
						67.1	**20**	98397
						65.1	**60**	184741
	Diethylcathinone (hydrochloride)	C13H19NO	**FIU_0183**	6.08	**206.2**	155	**24**	2056130
						133	**16**	946456
						130	**40**	143864

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						105.1	**24**	2274253
						103.1	**40**	246885
						100.1	**24**	1396183
						79.1	**40**	410062
						77.1	**56**	1202677
						72.1	**16**	462075
						58.1	**32**	401950
						51.1	**60**	340274
	JWH 018 2'-naphthyl-N-(1,2-dimethylpropyl) isomer	C24H23NO	**FIU_0036**	11.63	**342.2**	272.1	**20**	201855
						214.1	**20**	266516
						155	**24**	3598355
						144	**36**	1076583
						127.1	**56**	2466159
						116	**60**	420088
						101	**60**	9991
						89.1	**60**	82543
						77.1	**60**	55947
						71.1	**32**	50244

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	JWH 018 5-hydroxyindole metabolite	C24H23NO2	**FIU_0477**	10.86	**358.17288**	230.1	**24**	97003
						174.1	**36**	3958
						160	**40**	65046
						135.2	**24**	115
						132	**56**	21901
						127	**56**	609279
						77.1	**60**	16508
	JWH 018 7-hydroxyindole metabolite	C24H23NO2	**FIU_0479**	11.08	**358.17288**	230.6	**24**	1551
						230.1	**24**	289567
						160	**40**	279592
						132	**56**	39869
						127.1	**44**	2016
						127	**56**	1842672
						104.1	**60**	91869
						77.1	**60**	46357
	JWH 018 8-quinolinyl carboxamide	C23H23N3O	**FIU_0480**	12	**358.18411**	340.2	**12**	5172
						214.1	**12**	2643200

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						171	**8**	26241
						158.1	**40**	35645
						144	**40**	1171077
						130.1	**48**	20114
						116	**60**	360281
						89.1	**60**	47453
						71.2	**40**	19174
						55.1	**60**	10564
	JWH 018 N-(1,2-dimethylpropyl) isomer	C24H23NO	**FIU_0042**	11.43	**342.2**	272.1	**20**	383059
						254.1	**36**	13295
						214.1	**20**	291346
						155.1	**24**	4168628
						155	**24**	1402048
						144	**36**	1180414
						127.1	**56**	3084598
						116	**60**	431380
						89.1	**60**	90547
						77.1	**60**	69772
						71.1	**32**	68986

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	JWH 019 N-(2-fluorohexyl) isomer	C25H24FNO	**FIU_0489**	11.31	**374.18419**	246.1	**24**	368044
						226.1	**40**	22010
						169.1	**52**	1322
						155	**28**	1900413
						144	**44**	135109
						127	**60**	1593275
						116	**60**	45671
						77.1	**60**	19828
						61.1	**60**	12212
						55.1	**52**	61164
	JWH 019 N-(6-fluorohexyl) isomer	C25H24FNO	**FIU_0494**	11.11	**374.18419**	246.1	**24**	132803
						155	**28**	893384
						144	**44**	87211
						127	**56**	763832
						116	**60**	33645
						94.8	**40**	102
						61.1	**52**	6728
						55.2	**56**	20777

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	JWH 073 N-(2-hydroxybutyl) metabolite	C23H21NO2	**FIU_0506**	10.37	**344.15723**	284.1	**24**	17758
						216.1	**24**	183504
						144	**40**	130902
						127	**52**	1204520
						116	**60**	50957
						89.1	**60**	10544
						77.1	**60**	36919
						73.1	**36**	17045
						55.1	**48**	36990
	JWH 073 N-(2-methylpropyl) isomer	C23H21NO	**FIU_0054**	11.28	**328.2**	155	**20**	1902
	JWH 073 N-(4-hydroxybutyl) metabolite	C23H21NO2	**FIU_0507**	10.1	**344.15723**	216.1	**24**	21941
						205.2	**8**	105
						204.2	**60**	108
						155	**24**	651464
						144.1	**40**	38558
						127	**56**	471078
						116.1	**60**	16213

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						77.1	**60**	12306
						55.1	**48**	18474
	JWH 122 5-methylnaphthyl isomer	C25H25NO	**FIU_0063**	11.73	**356.2**	169	**24**	1385
						141	**40**	1351
						115.1	**60**	870
	JWH 145 2-phenyl isomer	C26H25NO	**FIU_0523**	11.48	**368.19361**	319.1	**4**	7246
						287	**20**	3852
						257	**28**	3209
						240.2	**20**	45902
						227	**40**	3335
						196.9	**52**	3168
						170	**40**	40575
						155	**20**	2204135
						127	**52**	1544608
						77.1	**60**	27336
	JWH 200 2'-naphthyl isomer	C25H24N2O2	**FIU_0067**	9.52	**385.2**	298.2	**16**	4292
						221	**12**	69
						155.1	**20**	195990

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						127	**60**	90687
						114.1	**28**	72068
						84.1	**56**	5913
						70.1	**60**	18366
	JWH 210 6-ethylnaphthyl isomer	C26H27NO	**FIU_0073**	12.01	**370.2**	214.2	**24**	1953525
						183.1	**28**	5373899
						155.1	**44**	4112413
						144	**44**	1022318
						140.1	**60**	1196513
						129.1	**60**	365496
						128.2	**60**	216570
						116.1	**60**	329063
						115.6	**60**	294456
						77.1	**60**	126829
	JWH 210 7-ethylnaphthyl isomer	C26H27NO	**FIU_0074**	11.92	**370.2**	214.1	**24**	1498231
						183.1	**24**	6486207
						155.1	**44**	4399405
						144.1	**44**	835153

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						140.1	**60**	1438861
						128.7	**60**	420728
						128.2	**60**	239839
						116	**60**	274094
						115.1	**60**	337574
						77.1	**60**	136041
	JWH 398 7-chloronaphthyl isomer	C24H22ClNO	**FIU_0082**	11.83	**376.2**	270	**28**	4304
						214.1	**24**	422784
						189	**24**	5322561
						161	**52**	4131989
						158	**36**	7402
						144	**40**	273263
						130	**56**	8230
						126	**60**	1097324
						116	**60**	92140
						89.1	**60**	13907
	JWH 398 N-pentanoic acid metabolite	C24H20ClNO3	**FIU_0553**	10.78	**406.11317**	275.9	**60**	84
						247.1	**40**	101

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						189	**28**	88924
						161	**56**	56748
						126	**60**	7275
						55.2	**56**	2961
	Mephedrone metabolite ((±)-Ephedrine stereochemistry)	C11H17NO	**FIU_0338**	6.99	**180.13101**	162.1	**8**	768126
						147.1	**20**	144546
						131.1	**20**	100460
						116.1	**28**	40766
						115.1	**48**	49576
						105.1	**32**	59998
						91.1	**28**	115468
						77.1	**60**	38070
						65.1	**60**	31402
						56.1	**28**	28849
	MN-25-2-methyl derivative	C27H39N3O3	**FIU_0446**	10.6	**454.29914**	275.2	**20**	551216
						190.2	**36**	30146
						188.2	**40**	26043
						137.2	**36**	24367

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						114.2	**36**	987061
						95.2	**60**	49812
						84.2	**60**	41499
						81.2	**56**	144164
						70.2	**60**	118484
						67.2	**60**	14254
	N,N-DMA	C11H17N	**FIU_0309**	6.32	**164.1361**	119.1	**8**	239934
						91.1	**24**	845533
						77	**60**	7650
						72.2	**12**	5534
						65.1	**52**	258856
						63.1	**60**	20164
						51.1	**60**	22385
	N-ethyl-N-Methylcathinone	C12H17NO	**FIU_0335**	6.13	**192.13101**	133.1	**16**	225851
						130.1	**44**	12605
						105.1	**24**	428456
						103.1	**40**	49636
						86.1	**24**	253295
						79.1	**40**	82229

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						77.1	**52**	217680
						60.2	**16**	17139
						58.2	**44**	115131
						51.1	**60**	78471
	Pyrazolam	C16H12BrN5	**FIU_0680**	8.33	**354.02761**	285.1	**24**	4575
						281.2	**0**	65
						249.5	**20**	82
						228.3	**60**	69
						206.1	**36**	13632
						205.1	**52**	10745
						174.1	**8**	3356067
						167.1	**36**	14923
						155	**60**	2083
						115.9	**60**	319
						78.1	**60**	3592
	Thiosildenafil	C22H30N6O3S2	**FIU_0770**	9.86	**491.18208**	341.1	**32**	10456
						327.1	**32**	5788
						313.1	**40**	4000
						299.1	**44**	10563

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						155.1	**20**	174405
						100.1	**32**	14555
						99.6	**32**	10758
						85.1	**36**	4206
						70.1	**60**	4116
						58.1	**56**	45026
						56.1	**60**	4768
	XLR11 N-(4-pentenyl) analog	C21H27NO	**FIU_0665**	11.53	**310.20926**	311.3	**0**	209681
						212.1	**20**	411479
						144	**40**	198994
						130	**48**	95769
						125.1	**20**	1601517
						97.1	**28**	320808
						83.1	**20**	206324
						69.1	**36**	232328
						57.2	**48**	345974
						55.1	**44**	676564
13	**(-)-11-nor-9-carboxy-Δ9-THC**	C21H28O4	**FIU_0089**	11.54	**345.2**	327.2	**12**	45436

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						299.2	**20**	26249
						193.1	**28**	11488
						187.1	**32**	5960
						123	**44**	5074
						119	**32**	6641
						105	**52**	3620
						91.1	**60**	6413
						79.1	**48**	4070
						69.1	**44**	5538
	1,4-Dibenzylpiperazine (HCl)	C18H22N2	**FIU_0106**	7.51	**267.2**	188.2	**8**	620951
						176.2	**16**	93571
						175.1	**16**	1221085
						146.1	**20**	69539
						134.1	**24**	738685
						120.1	**20**	139521
						118.1	**60**	46551
						104.1	**32**	69276
						91.1	**36**	4171244
						65.1	**60**	914480

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						56.1	**32**	40208
	2-Bromoamphetamine	C9H12BrN	**FIU_0281**	7.76	**214.01531**	197	**8**	93000
						169	**16**	178864
						118.1	**20**	7878
						117.1	**36**	17700
						115.1	**60**	9729
						91.2	**56**	14736
						90.1	**44**	77102
						89.1	**60**	67356
						64.2	**60**	7315
						63.1	**60**	9157
	2-Chloroamphetamine	C9H12ClN	**FIU_0282**	7.52	**170.01**	153.1	**8**	156899
						125	**16**	309933
						115.1	**44**	4499
						99.1	**48**	31493
						91.3	**44**	6609
						90.1	**48**	33007
						89.1	**44**	71313
						73.1	**60**	20150

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						65.2	**56**	15159
						63.1	**60**	35294
	3-Chloroamphetamine	C9H12ClN	**FIU_0287**	7.76	**170.06583**	174.1	**8**	2550771
						153	**4**	101647
						125	**16**	231341
						105.3	**20**	102
						99	**44**	20837
						90.1	**48**	22077
						89.1	**44**	44883
						75.1	**48**	3633
						73.1	**60**	12654
						65.1	**48**	5414
						63.1	**60**	24892
	3'-fluoro-a-Pyrrolidinopropiophenone	C13H16FNO	**FIU_0394**	6.17	**222.12159**	178.1	**12**	1206753
						151	**16**	129977
						123	**24**	349783
						103.1	**36**	227859
						98.1	**28**	513166

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						95.1	**52**	94417
						84.1	**36**	60404
						77.1	**56**	184576
						70.1	**20**	194135
						56.1	**52**	147388
						55.1	**52**	67358
	3-hydroxy Phenazepam	C15H10BrClN2O2	**FIU_0673**	9.56	**364.96142**	258.1	**28**	3609
						257.1	**36**	3255
						213	**44**	3091
						210	**24**	1553
						208.2	**36**	1214
						206.1	**40**	7718
						179.1	**56**	4059
						178.1	**60**	4398
						176.1	**8**	1413195
						151	**60**	3279
	3-Methylethcathinone (hydrochloride)	C12H17NO	**FIU_0133**	6.77	**192.1**	146.1	**16**	1081702
						144.1	**32**	1271568
						131	**28**	517821

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						130	**44**	457786
						119.1	**24**	360883
						91.1	**40**	541974
						77.1	**60**	387880
						65.1	**60**	300296
	4-CAB	C10H14ClN	**FIU_0322**	8.31	**184.08148**	188.1	**8**	2305526
						167	**4**	106482
						125	**12**	285411
						107.1	**20**	85
						99	**48**	28500
						90	**48**	26570
						89.1	**52**	56607
						75.1	**52**	4645
						73	**60**	15538
						65.1	**56**	5701
						63.1	**60**	30559
	4-Ethylmethcathinone (hydrochloride)	C12H17NO	**FIU_0137**	7.33	**192.1**	159.1	**20**	186543
						146.1	**16**	762999
						145.1	**20**	1530849

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						144.1	**36**	1563277
						131.1	**28**	206958
						130	**44**	120557
						105.1	**28**	245244
						103.1	**52**	145054
						77.1	**60**	487937
	4-hydroxy DiPT	C16H24N2O	**FIU_0748**	6.48	**261.18886**	160.1	**20**	1230835
						142.1	**40**	60745
						135.1	**4**	233520
						132.1	**36**	164998
						117	**48**	167989
						114.1	**12**	555085
						105.1	**52**	48410
						102.1	**12**	87423
						89.1	**60**	51912
						77.1	**60**	36219
						72.2	**32**	210003
	4-methyl-N-Methylbuphedrone	C13H19NO	**FIU_0428**	7.12	**206.14666**	161.1	**12**	505408
						133.1	**16**	158480

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						119	**24**	174087
						105.1	**20**	562910
						91.1	**40**	260995
						86.1	**24**	242605
						77.1	**60**	87857
						71.1	**48**	108300
						65.1	**60**	190940
						56.1	**60**	79852
	5-APB (hydrochloride)	C11H13NO	**FIU_0152**	7.01	**176.1**	132.1	**16**	66475
						131	**16**	1378255
						116	**32**	99438
						115	**48**	135102
						103	**40**	68392
						91.1	**32**	374196
						77.1	**48**	416424
						65.1	**60**	144923
						51.1	**60**	163102
	5-EAPB	C13H17NO	**FIU_0419**	7.18	**204.13101**	159.1	**8**	708956
						131.1	**20**	900722

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						129.1	**28**	23444
						116.1	**36**	49139
						115.1	**56**	79483
						103.1	**44**	45986
						91.1	**40**	198594
						77.2	**56**	259075
						65.2	**60**	76006
						51.2	**60**	54056
	5-fluoro ABICA	C19H26FN3O2	**FIU_0703**	9.88	**348.20091**	331.1	**4**	1129723
						304.1	**12**	222680
						232.1	**20**	1433917
						212.1	**36**	11555
						158	**40**	10870
						144	**44**	645384
						130	**56**	10884
						116	**60**	251772
						89	**60**	33780
						69.1	**44**	52556
						61.1	**60**	9220

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	5-fluoro PB-22 3-hydroxyquinoline isomer	C23H21FN2O2	**FIU_0580**	11.02	**377.15871**	232.1	**20**	1882439
						212.1	**40**	17483
						176.1	**40**	11809
						158.1	**44**	16479
						144	**44**	929363
						130.1	**56**	14975
						116	**60**	319989
						89.1	**60**	30929
						69.1	**44**	69993
						61.1	**60**	10396
	5-fluoro PB-22 4-hydroxyquinoline isomer	C23H21FN2O2	**FIU_0582**	10.87	**377.15871**	232.1	**8**	1897728
						212.1	**40**	17054
						176	**40**	9731
						158	**40**	15619
						144	**44**	888120
						130	**52**	12541
						116	**60**	324045
						89.1	**60**	34703

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						69.1	**44**	74062
						61.1	**52**	9706
	5-fluoro PB-22 5-hydroxyquinoline isomer	C23H21FN2O2	**FIU_0584**	10.76	**377.15871**	232.1	**20**	1809130
						212.1	**44**	16787
						176	**44**	10839
						158	**40**	17693
						144	**48**	976533
						130	**60**	15052
						116	**60**	334482
						89.1	**60**	30601
						69.1	**44**	76819
						61.1	**60**	12076
	5-fluoro SDB-005	C23H21FN2O2	**FIU_0439**	11.23	**377.15871**	233.1	**8**	1251693
						213.1	**28**	292691
						185.1	**32**	22057
						177.1	**32**	110835
						171.1	**36**	28304
						145.1	**44**	448148
						121.1	**48**	16250

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						117	**60**	79467
						90.1	**60**	122458
						69.2	**36**	154775
	5-Fluoropentylindole	C13H16FN	**FIU_0686**	10.72	**206.12668**	233.1	**20**	236953
						142.2	**12**	92
						137.6	**16**	44
						132.1	**16**	3395
						132	**16**	12878
						118	**20**	21692
						118	**20**	5697
						91.1	**48**	2746
						91.1	**44**	11067
						69.1	**20**	3575
						65.1	**60**	3595
	5-IT	C11H14N2	**FIU_0687**	5.89	**175.1157**	158.1	**4**	233062
						143.1	**28**	14453
						130.1	**20**	98165
						117.1	**24**	46424
						115.1	**52**	14682

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						103.1	**40**	12302
						91.1	**44**	5086
						90.1	**52**	12945
						89.1	**60**	16807
						77.1	**56**	31415
	5-MAPDB	C12H17NO	**FIU_0421**	6.33	**192.13101**	366.1	**4**	55817
						338.1	**12**	62083
						161.1	**8**	960213
						133.1	**24**	635520
						117.1	**48**	27725
						115.1	**56**	36630
						105.1	**36**	103195
						103.1	**44**	59521
						91.1	**60**	65140
						79.2	**40**	89541
						77.1	**60**	188927
						51.2	**60**	42113
	AB-PINACA N-(4-hydroxypentyl) metabolite	C18H26N4O3	**FIU_0724**	9.2	**347.20049**	284.1	**20**	4799

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						231	**20**	25948
						213	**28**	45745
						174.9	**32**	3039
						171	**44**	2165
						144.9	**48**	17703
						102.8	**40**	66
						69.1	**44**	14503
	ADB-FUBINACA	C21H23FN4O2	**FIU_0732**	10.44	**383.1805**	253	**24**	53397
						225	**40**	712
						211.2	**28**	70
						154.6	**36**	66
						109	**56**	53668
	ADBICA N-(5-hydroxypentyl) metabolite	C20H29N3O3	**FIU_0696**	9.63	**360.22089**	343.1	**8**	599813
						315.1	**12**	9915
						230.1	**20**	690910
						144	**44**	293888
						130	**52**	3493
						116	**60**	74818

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						89	**60**	7235
						87.1	**36**	4688
						69.1	**44**	96537
						67.1	**56**	4104
	ADB-PINACA pentanoic acid metabolite	C19H26N4O4	**FIU_0739**	9.51	**375.19541**	330.1	**12**	69957
						284.1	**16**	737394
						245	**24**	30480
						227	**36**	23476
						217.1	**32**	39583
						199	**48**	5377
						185	**48**	3804
						175	**52**	6631
						145	**60**	11949
						55.1	**60**	17335
	AH 7921	C16H22Cl2N2O	**FIU_0671**	8.51	**329.11092**	202	**20**	100510
						190	**24**	195101
						173	**32**	641453
						145	**60**	293644
						109	**60**	47317

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						95.2	**32**	369619
						93.1	**56**	31944
						67.2	**60**	206759
						55.2	**60**	109266
	AKB48	C21H31N3O	**FIU_0158**	12.94	**342.3**	(blank)	**(blank)**	(blank)
	AM1220 azepane isomer	C26H26N2O	**FIU_0627**	8.9	**383.20451**	286.1	**20**	15779
						155	**28**	125473
						127	**60**	131479
						112.1	**20**	839474
						98.1	**28**	97887
						84.1	**56**	18887
						81.1	**56**	14939
						70.1	**60**	61167
						58.1	**60**	215786
						55.2	**60**	25027
	AM1248	C26H34N2O	**FIU_0162**	9.73	**391.3**	155	**24**	4618817
						135.1	**32**	3376107
						112.1	**36**	1318693
						107.1	**60**	263159

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						98.1	**52**	1078622
						93.1	**60**	391400
						81.1	**60**	112984
						79.1	**60**	324495
						70.1	**60**	231906
						67.1	**60**	119298
						58.1	**60**	396787
	AM2201 5-hydroxyindole metabolite	C24H22FNO2	**FIU_0629**	10.26	**376.16346**	248.1	**24**	17710
						160	**44**	12361
						155	**28**	115739
						132	**60**	5614
						127	**56**	97682
	AMT	C5H10N2S	**FIU_0757**	3.98	**131.05647**	124.5	**16**	41
						112	**12**	83
						104	**12**	2698
						77	**16**	8437
						72	**20**	9163
						60	**52**	25194
						53.1	**32**	1650

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	a-Pyrrolidinopentiothiophenone	C13H19NOS	**FIU_0408**	6.59	**238.11873**	167.1	**12**	156888
						126.2	**20**	810428
						111.1	**40**	109663
						97.1	**24**	336963
						84.2	**40**	124674
						83.6	**56**	42469
						70.2	**16**	46047
						69.2	**60**	108807
						55.8	**56**	37649
						55.2	**48**	65152
	Benzedrone	C17H19NO	**FIU_0343**	8.71	**254.14666**	236.1	**8**	142434
						162.1	**12**	11638
						146.1	**12**	63950
						144.1	**12**	8925
						131.1	**24**	6432
						119.1	**24**	13124
						91.1	**20**	1209268
						65.1	**60**	345750

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						63.1	**60**	13133
						51.1	**60**	6527
	bk-DMBDB (hydrochloride)	C13H17NO3	**FIU_0170**	6.46	**236.1**	191.1	**12**	1389419
						163	**16**	638643
						161	**16**	1482770
						149	**24**	1017098
						121	**40**	496318
						105.1	**32**	494796
						86.1	**24**	2124511
						77.1	**60**	372129
						71.1	**52**	677383
						65.1	**56**	1016942
	CMP	C10H17N	**FIU_0303**	7.03	**152.1361**	155	**24**	4289659
						121.1	**8**	52041
						93.1	**12**	84915
						91.1	**28**	23268
						79.1	**20**	289089
						77.1	**36**	262848
						65.1	**56**	8519

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						58.2	**8**	489652
						55.1	**24**	13917
						53.1	**56**	13086
						51.1	**60**	132741
	Diclazepam	C16H12Cl2N2O	**FIU_0676**	9.98	**319.03267**	256.1	**24**	1554
						227.1	**32**	13124
						205.1	**44**	3994
						165.1	**60**	5018
						155	**24**	2894192
						154	**32**	14542
						125	**56**	5629
						118.1	**60**	2725
						117.1	**48**	3278
						91.1	**52**	2821
						58.1	**36**	3030
	FDU-PB-22	C26H18FNO2	**FIU_0593**	11.43	**396.13216**	224.1	**28**	6092
						109	**40**	723015
						106.4	**56**	98
						83.1	**60**	20354

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	Harmaline	C13H14N2O	**FIU_0186**	7.26	**215.1**	200.1	**24**	1124468
						174.1	**24**	1154314
						172.1	**32**	1180156
						171.1	**44**	808670
						159	**32**	267578
						157.1	**44**	217445
						143.1	**44**	143862
						131.1	**44**	491377
						130	**56**	501056
						68.1	**24**	277321
	HU-210	C25H38O3	**FIU_0187**	12.14	**387.3**	261.1	**12**	44036
						243.1	**16**	96186
						201.1	**24**	35670
						147	**20**	10741
						133.1	**24**	16865
						123	**28**	10340
						105.1	**56**	10650
						85.1	**24**	72769
						71.1	**24**	158553

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						57.1	**28**	104560
	JW 618	C17H14F6N2O2	**FIU_0443**	10.13	**393.09595**	373.1	**36**	24136
						197.1	**44**	126781
						183.8	**48**	7457
						183.1	**48**	60025
						181.1	**60**	14660
						170.3	**44**	24900
						169.1	**44**	686760
						155.1	**60**	67743
						154.1	**60**	110466
						141.1	**60**	12087
	JWH 018 2'-naphthyl-N-(2-methylbutyl) isomer	C24H23NO	**FIU_0039**	11.73	**342.2**	214.1	**24**	551061
						155	**20**	38635
						144	**40**	555355
						130.1	**56**	6344
						127	**56**	3485598
						116	**60**	211698
						101.1	**60**	18963
						89.1	**60**	41824

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						77.1	**60**	106649
						71.1	**36**	46570
	JWH 018 2'-naphthyl-N-(3-methylbutyl) isomer	C24H23NO	**FIU_0040**	11.74	**342.2**	214.1	**24**	458478
						158.1	**36**	50092
						144	**40**	247990
						130.1	**48**	30177
						127	**56**	2328641
						116	**60**	101936
						89.1	**60**	19311
						77.1	**60**	79788
						71.2	**36**	14399
	JWH 018 N-(1-ethylpropyl) isomer	C24H23NO	**FIU_0482**	11.28	**342.17796**	272.1	**20**	206130
						272.1	**20**	83125
						254.1	**40**	13422
						254.1	**40**	5639
						214.1	**20**	402340
						214.1	**20**	152939
						155.1	**24**	381314

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						155	**24**	2155201
						144	**36**	1298335
						144	**36**	558080
						127	**56**	1658579
						116	**60**	551772
						116	**60**	236169
						89.1	**60**	125729
						89.1	**60**	49949
						77.1	**60**	98264
						77.1	**60**	39847
						71.1	**32**	19865
						71.1	**32**	6198
	JWH 018 N-(2-methylbutyl) isomer	C24H23NO	**FIU_0046**	11.53	**342.2**	214.2	**20**	5474
						155	**24**	1606321
						144.1	**36**	5198
						127	**56**	34781
	JWH 019 N-(6-hydroxyhexyl) metabolite	C25H25NO2	**FIU_0495**	10.53	**372.18853**	244.1	**28**	35101
						238.9	**48**	198

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						207.9	**44**	125
						155	**20**	1245939
						144	**48**	40651
						127	**60**	860916
						116.1	**60**	17289
						77.1	**60**	9426
						55.1	**56**	32369
	JWH 071	C21H17NO	**FIU_0499**	10.67	**300.13101**	172.1	**20**	426098
						157.1	**40**	10195
						144	**44**	134235
						129	**56**	22301
						127.1	**48**	1476597
						116	**60**	115658
						101.1	**60**	29869
						89.1	**60**	48266
						77.1	**60**	154180
	JWH 081 4-hydroxynaphthyl metabolite	C24H23NO2	**FIU_0514**	11.11	**358.17288**	214.1	**24**	269636
						188.1	**20**	40299

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						171	**24**	1642061
						144.1	**40**	162541
						143	**48**	618568
						132.1	**32**	10449
						118	**40**	5594
						115.9	**60**	67210
						115	**60**	743543
						89.1	**60**	22114
	JWH 122 N-(4-hydroxypentyl) metabolite	C25H25NO2	**FIU_0520**	10.65	**372.18853**	354.2	**16**	17483
						298.1	**24**	29841
						230.1	**24**	45751
						229.1	**4**	37350
						212.1	**28**	10644
						173.1	**8**	8802
						169.1	**24**	1823193
						144	**40**	106982
						141.1	**52**	1027290
						115.9	**60**	32254

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						115.1	**60**	340781
						85.2	**16**	25633
						69.1	**40**	66305
	JWH 133	C22H32O	**FIU_0522**	11.11	**313.24532**	205.1	**4**	1250
						181.2	**48**	123
						149	**8**	2097
						91	**48**	4136
	JWH 147	C27H27NO	**FIU_0218**	12.18	**382.2**	333	**8**	3008
						254.1	**20**	34243
						226.9	**40**	1340
						170	**44**	29961
						155	**20**	5029637
						142	**52**	8601
						127	**56**	3626908
						115.1	**60**	12122
						101	**60**	11221
						77.1	**60**	60434
	JWH 149	C26H27NO	**FIU_0525**	11.83	**370.20926**	243.4	**48**	107
						228.1	**24**	468716

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						169.1	**28**	2673026
						158.1	**44**	283943
						141.1	**52**	1804444
						130.1	**60**	92031
						115	**60**	658829
						103.1	**60**	22656
						91	**60**	11309
	JWH 203 N-(4-hydroxypentyl) metabolite	C21H22ClNO2	**FIU_0535**	10.26	**356.13391**	338.2	**16**	25992
						282	**20**	30445
						204.1	**16**	42172
						186.1	**16**	430745
						170.1	**48**	17383
						144.1	**44**	23626
						130.1	**24**	79701
						125	**32**	619379
						89.1	**60**	51486
						69.1	**40**	35670

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	JWH 210 N-(4-hydroxypentyl) metabolite	C26H27NO2	**FIU_0540**	10.87	**386.20418**	230.1	**24**	63232
						183.1	**24**	1892839
						155.1	**44**	486182
						153	**56**	393758
						144	**40**	136724
						129.1	**60**	150146
						128.2	**60**	59583
						115	**60**	150287
						77.1	**60**	56364
						69.1	**44**	77445
	JWH 398 3-chloronaphthyl isomer	C24H22ClNO	**FIU_0079**	11.96	**376.2**	214.1	**24**	262038
						189	**28**	2917443
						161	**52**	2289818
						158	**36**	5018
						144	**44**	196136
						130	**56**	5222
						126	**60**	601245

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						116	**60**	65883
						89	**60**	9554
	JWH 398 5-chloronaphthyl isomer	C24H22ClNO	**FIU_0080**	11.97	**376.2**	270.1	**36**	3702
						214.1	**24**	361956
						189	**24**	4063660
						161	**48**	3199653
						158.1	**36**	7012
						144	**44**	254584
						130	**48**	7288
						126	**60**	756038
						116	**60**	83352
						89.1	**60**	12689
	JWH 398 8-chloronaphthyl isomer	C24H22ClNO	**FIU_0083**	11.31	**376.2**	340.2	**24**	38331
						270.1	**36**	34681
						214.1	**28**	238945
						189	**28**	4586007
						161	**52**	3373594
						146.1	**8**	1429267

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						144	**40**	211135
						131	**20**	816522
						130.1	**56**	7816
						126	**60**	925642
						116	**60**	77163
						89.1	**60**	12016
	MDAI (hydrochloride)	C10H11NO2	**FIU_0243**	11.782	**178.1**	131	**20**	277158
						103	**32**	354760
						77.1	**52**	293751
						51.1	**60**	106209
	Methedrone	C11H15NO2	**FIU_0010**	6.32	**194.1**	176.1	**8**	2593400
						161.1	**20**	1379942
						146	**32**	858946
						145.5	**28**	246777
						135.1	**20**	242611
						118.1	**44**	454903
						91.1	**56**	391925
						79.1	**40**	173533
						77.1	**56**	417317

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						58.1	**12**	471655
	MN-25	C26H37N3O3	**FIU_0445**	10.2	**440.28349**	353.3	**28**	78423
						261.2	**24**	267191
						217.1	**36**	48978
						176.1	**36**	71369
						174.1	**40**	47701
						137.2	**36**	39758
						114.1	**40**	504852
						95.2	**52**	66498
						81.2	**60**	186610
						70.2	**60**	68648
	Naphyrone 1-naphthyl isomer	C19H23NO	**FIU_0084**	8.31	**282.2**	211.1	**16**	634288
						169	**24**	145025
						155	**28**	490281
						141.1	**28**	776045
						126.1	**24**	769882
						115	**60**	200937
						84.1	**48**	180376
						70.1	**20**	65982

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						55.1	**60**	76564
	N-methyl-2-AI	C10H13N	**FIU_0317**	5.98	**148.1048**	117.1	**16**	745779
						115.1	**32**	329754
						91.1	**36**	304108
						89.1	**56**	34377
						77.1	**48**	11083
						75.1	**60**	4363
						65.1	**56**	153680
						63.1	**60**	37007
						51.1	**60**	30733
	Nor-Mephedrone (hydrochloride)	C10H13NO	**FIU_0254**	6.61	**164.1**	147.1	**8**	201264
						130	**36**	687326
						119	**16**	207010
						103	**52**	110042
						91.1	**36**	150947
						77.1	**60**	301147
						65.1	**56**	103942
						51.1	**60**	81411

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	PB-22 6-hydroxyquinoline isomer	C23H22N2O2	**FIU_0604**	11.31	**359.16813**	214.1	**24**	411246
						158	**40**	6410
						144	**44**	199712
						130	**56**	4401
						126.7	**60**	65
						116	**60**	61074
						89	**60**	6384
						71.1	**44**	3062
	Pentedrone (hydrochloride)	C12H17NO	**FIU_0256**	7.057	**192.1**	161.1	**8**	510846
						144	**32**	430191
						132.1	**16**	772372
						130	**40**	459333
						117	**32**	336023
						91.1	**28**	1014627
						77.1	**60**	656771
						65.1	**60**	301127
						51.1	**60**	266623
	UR-144 Degradant N-pentanoic acid metabolite	C21H27NO3	**FIU_0646**	10.48	**342.19909**	244.1	**20**	57216

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						176.5	**16**	68
						144	**36**	23063
						116.1	**60**	8985
						101.1	**36**	6142
						83.1	**36**	7757
						59.1	**56**	4559
						55.1	**56**	23410
	Yangonin	C15H14O4	**FIU_0465**	9.95	**259.08921**	231.2	**12**	78628
						171.1	**24**	49168
						161.1	**20**	137944
						139.1	**60**	36375
						133.1	**36**	66535
						128.1	**52**	41466
						115.1	**60**	24714
						90.1	**60**	25123
						77.1	**60**	33963
						69.1	**48**	37128
14	**(±)-CP 47,497**	C21H34O2	**FIU_0097**	11.67	**319.3**	301.2	**4**	24031
						233.1	**12**	28527

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						226.9	**28**	1494
						197.1	**40**	1428
						133	**52**	5222
						121	**32**	11495
						107	**24**	14007
						85.2	**16**	8095
						77.1	**60**	6563
						71.1	**20**	14290
						57.1	**36**	10199
	(±)-JWH 018 N-(2-hydroxypentyl) metabolite	C24H23NO2	**FIU_0469**	10.63	**358.17288**	284.1	**28**	21416
						254.1	**60**	4621
						230.1	**24**	101702
						144.1	**44**	91478
						127.1	**56**	817309
						116.1	**60**	35208
						89.1	**60**	4593
						77.1	**60**	17714
						69.2	**40**	5432

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	2,3-Dimethylethcathinone	C13H19NO	**FIU_0345**	7.83	**206.14666**	173.1	**20**	232208
						163.1	**8**	558776
						160.2	**16**	148676
						158.8	**20**	268980
						158.1	**32**	367489
						145.1	**24**	98906
						144.1	**36**	101749
						115.1	**60**	89230
						91.1	**56**	85830
						77.1	**60**	71684
	2,3-methylenedioxymethcathinone	C11H13NO3	**FIU_0109**	11.65	**208.1**	190.1	**8**	430283
						160	**12**	2882810
						147	**16**	279464
						132.1	**28**	1466222
						117	**40**	633380
						91.1	**40**	420629
						77.1	**56**	222660
						65.1	**60**	497039

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						58.1	**36**	115650
	2-Fluoroethcathinone (hydrochloride)	C11H14FNO	**FIU_0115**	11.66	**196.1**	178.1	**12**	1092910
						151.1	**8**	199124
						149.7	**16**	546160
						149.6	**20**	457069
						148	**36**	353858
						135	**32**	279012
						123.1	**16**	183850
						123	**24**	209992
						115	**32**	89959
						103	**32**	228837
						77.1	**52**	320695
						75.1	**60**	135381
	2-methylmethcathinone	C11H15NO	**FIU_0120**	11.66	**178.1**	151.1	**8**	211745
						145.1	**20**	722045
						144.1	**32**	511387
						130.1	**32**	55545
						119	**20**	74660
						103	**48**	55344

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						91.1	**40**	107801
						77.1	**60**	166297
						65.1	**56**	73074
						58.1	**32**	31495
	3,4-DHMA	C10H15NO2	**FIU_0305**	4.27	**182.11028**	133.1	**20**	18576
						123	**20**	151527
						105.1	**24**	42300
						103	**36**	11144
						79.1	**32**	21121
						77.1	**44**	76833
						65.1	**44**	7688
						58.2	**8**	5874
						51.1	**60**	58179
	3-Fluoroethcathinone (HCl)	C11H14FNO	**FIU_0129**	11.65	**196.1**	150	**16**	892141
						149.6	**20**	467320
						135	**32**	410172
						123	**24**	203519
						108	**52**	95938
						103	**32**	252893

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						95	**48**	95259
						77.1	**52**	320637
						75	**60**	149438
	3-Fluoromethamphetamine (hydrochloride)	C10H14FN	**FIU_0130**	11.65	**168.1**	137	**8**	805811
						109	**20**	2680962
						89	**48**	76510
						83.1	**48**	969544
						81	**60**	16842
						75.1	**60**	16683
						63.1	**60**	197604
						59.1	**52**	143123
						58.1	**12**	97914
						57.1	**60**	398762
	4-MTA	C10H15NS	**FIU_0294**	7.76	**182.09252**	165.1	**4**	462929
						159.1	**4**	1653592
						137	**20**	126330
						137	**20**	126152
						122	**40**	45522

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						122	**36**	43631
						121	**56**	38656
						121	**52**	32766
						118.2	**20**	38467
						118.1	**20**	39402
						117.1	**16**	154489
						117.1	**16**	152897
						115.1	**48**	55193
						115.1	**44**	55333
						91.1	**56**	84746
						91.1	**52**	89665
						78.1	**56**	23063
						78.1	**52**	25419
						65.1	**60**	32902
						65.1	**60**	31975
	5-APDB	C11H15NO	**FIU_0418**	6.35	**178.11536**	161.1	**8**	533807
						133.1	**20**	324650
						115.1	**48**	18506
						105.1	**32**	52434

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						103.1	**40**	28649
						91.1	**52**	33821
						79.2	**40**	46977
						77.1	**52**	93973
						65.2	**60**	17851
						51.2	**60**	31531
	5-fluoro ADB-PINACA	C19H27FN4O2	**FIU_0707**	10.31	**363.2118**	233.1	**24**	129299
						213	**36**	36452
						177	**40**	16380
						171	**48**	3884
						145	**52**	68238
						117	**60**	7985
						90	**60**	8331
						69.1	**44**	19585
	5-fluoro MN-18	C23H22FN3O	**FIU_0709**	11.19	**376.17469**	233	**16**	292485
						213	**28**	81304
						185	**32**	5540
						177	**32**	28956
						171	**40**	7161

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						144.9	**44**	121557
						117	**60**	18824
						100	**12**	440
						90	**60**	27952
						69.1	**36**	37153
	5-fluoro PB-22 6-hydroxyquinoline isomer	C23H21FN2O2	**FIU_0586**	10.7	**377.15871**	232.1	**24**	1724282
						212.1	**44**	17773
						176	**44**	12447
						158.1	**40**	18859
						144	**48**	1028711
						130.1	**56**	15546
						116	**60**	348729
						89.1	**60**	32038
						69.1	**44**	76768
						61.1	**60**	11638
	5-fluoro-AKB48 N-(4-hydroxypentyl) metabolite	C23H30FN3O2	**FIU_0710**	11.14	**400.23221**	135.1	**20**	406809
						107	**56**	37201

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						93.1	**60**	50742
						91	**60**	7116
						81.1	**60**	14845
						79.1	**60**	45566
						77.1	**60**	8398
						69.1	**60**	4909
						67.1	**60**	18195
						55.1	**60**	6684
	AB-CHMINACA	C20H28N4O2	**FIU_0714**	10.98	**357.22123**	352.1	**4**	83982
						340.1	**4**	104201
						324.1	**12**	94885
						312.1	**12**	98202
						253	**28**	82248
						241	**28**	115482
						144.9	**48**	69296
						116.9	**60**	4689
						97	**44**	7288
						90	**60**	1171
						69.1	**56**	3621

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						55.1	**60**	35886
	AB-PINACA N-(4-fluoropentyl) isomer	C18H25FN4O2	**FIU_0723**	9.99	**349.19615**	332.1	**4**	58625
						330.1	**4**	46751
						302.1	**12**	54078
						284.1	**16**	5199
						233	**24**	64428
						213	**36**	16674
						177	**40**	5870
						145	**48**	27110
						117	**60**	4245
						90	**60**	6314
						69.1	**44**	10836
	ADB-PINACA isomer 2	C19H28N4O2	**FIU_0735**	10.88	**345.22123**	328.1	**4**	207429
						300.1	**12**	247452
						232.1	**20**	15493
						215.1	**24**	286784
						145	**48**	167539
						117	**60**	17974
						90	**60**	21948

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						71.1	**48**	2332
	AKB48 N-(4-hydroxypentyl) metabolite	C23H31N3O2	**FIU_0741**	11.31	**382.24163**	135	**24**	310963
						107	**56**	27265
						93	**60**	43090
						91	**60**	7644
						81.1	**60**	12013
						79	**60**	35986
						77	**60**	7698
						69.1	**60**	4118
						67.1	**60**	15021
						55.1	**60**	6471
	AM2201 2'-naphthyl isomer	C24H22FNO	**FIU_0027**	11.17	**360.2**	232.1	**24**	463477
						163	**8**	9926
						144	**44**	330566
						127	**56**	2216269
						116	**60**	146060
						105.1	**40**	5199
						89	**60**	24115

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						77.1	**60**	53409
						69.1	**44**	27086
	AM2201 6-hydroxyindole metabolite	C24H22FNO2	**FIU_0630**	10.27	**376.16346**	248.1	**24**	14665
						160	**44**	7817
						155.1	**24**	173975
						155	**28**	148864
						132	**60**	4293
						127	**56**	144520
						114.6	**28**	61
						77.1	**60**	2585
	AM694	C20H19FINO	**FIU_0169**	10.66	**436.1**	309.1	**20**	515345
						292.1	**32**	119869
						234.1	**32**	327306
						232.1	**36**	206578
						230.9	**28**	3116193
						202.9	**56**	1565046
						144	**56**	150562
						104.9	**60**	89457
						104	**60**	216463

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						76.1	**60**	805764
	AM694 3-iodo isomer	C20H19FINO	**FIU_0031**	11.24	**436.1**	232.2	**32**	48782
						230.9	**28**	2194949
						202.9	**52**	1261167
						144	**48**	53916
						130	**60**	10259
						116	**60**	17989
						105.2	**56**	11374
						104	**60**	67662
						76.1	**60**	680805
						69.1	**48**	4334
	BB-22	C25H24N2O2	**FIU_0615**	11.48	**385.18378**	386.4	**0**	7263
						241.1	**8**	5833
						240.1	**12**	2845950
						158.1	**40**	10635
						144	**44**	1073268
						116	**60**	180879
						97.1	**40**	147856
						89.1	**60**	12550

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						69.1	**52**	82062
						55.1	**56**	780505
	BB-22 3-hydroxyquinoline isomer	C25H24N2O2	**FIU_0617**	11.95	**385.18378**	317.9	**0**	49
						240.1	**20**	503971
						158	**40**	2615
						144	**44**	228525
						116	**60**	33823
						97.1	**40**	29854
						89	**60**	2531
						69.1	**52**	16693
						55.1	**60**	159037
	BB-22 4-hydroxyisoquinoline isomer	C25H24N2O2	**FIU_0618**	11.74	**385.18378**	240.1	**20**	590644
						158	**40**	2578
						144	**44**	249444
						116	**60**	37491
						97.1	**40**	32649
						89	**60**	2815

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						69.1	**52**	18503
						55.1	**60**	178525
	BB-22 7-hydroxyisoquinoline isomer	C25H24N2O2	**FIU_0624**	11.56	**385.18378**	253.4	**4**	63
						240.2	**24**	280690
						156.4	**60**	65
						144	**44**	141992
						116	**60**	22255
						97.1	**40**	19553
						69.1	**52**	11222
						55.1	**60**	114145
	bk-MDDMA (hydrochloride)	C12H15NO3	**FIU_0171**	11.75	**222.1**	177	**12**	497035
						149	**20**	540536
						147	**20**	1146862
						119	**28**	445349
						91.1	**40**	956948
						72.1	**20**	2079145
						70.1	**52**	111698
						65.1	**60**	793312

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						58.1	**32**	79344
						57.1	**56**	82914
	Cannabigerol	C21H32O2	**FIU_0467**	11.27	**317.24023**	193.2	**16**	105095
						137.1	**40**	4064
						123.1	**36**	29015
						95.1	**48**	5148
						81.1	**52**	4998
						79.1	**60**	3657
						77.1	**60**	3774
						69.2	**28**	3690
						67.2	**60**	9192
						55.2	**60**	7308
	CB-25	C25H41NO3	**FIU_0177**	11.71	**404.3**	387.3	**12**	346061
						347.3	**16**	630695
						287.2	**4**	5282
						221.1	**12**	224307
						181.1	**24**	670223
						175.1	**4**	6168
						111	**40**	196515

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						83.1	**28**	92248
						71.1	**36**	113438
						69.1	**40**	94246
						58.1	**24**	2605479
						55.1	**56**	215424
	HU-211	C25H38O3	**FIU_0188**	12.12	**387.3**	261.1	**12**	49403
						243.1	**16**	129988
						201.1	**24**	42624
						147	**20**	11084
						133.1	**24**	17500
						105.1	**52**	12872
						95.1	**20**	13170
						85.1	**24**	91828
						71.1	**24**	175373
						57.1	**36**	117221
	HU-311	C21H28O3	**FIU_0190**	11.73	**329.2**	314.2	**12**	11011
	JW 642	C21H20F6N2O3	**FIU_0444**	10.87	**463.13781**	183.1	**28**	1162802
						168.1	**60**	138467
						165.1	**56**	137652

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						155.1	**48**	129893
						155	**24**	4095481
						153.9	**60**	64958
						129.1	**60**	59785
						127.7	**60**	24063
						127.1	**60**	32187
						115.1	**60**	49317
						77.1	**60**	34529
	JWH 016	C24H23NO	**FIU_0196**	11.439	**342.2**	214.1	**24**	531989
						158	**40**	317344
						155	**24**	1909915
						130.1	**56**	113829
						127	**56**	3720079
						103.1	**60**	42839
						101	**60**	26994
						77.1	**60**	154113
						57.1	**44**	31665
						51	**60**	6351

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	JWH 018 2'-naphthyl-N-(1,1-dimethylpropyl) isomer	C24H23NO	**FIU_0035**	11.59	**342.2**	144	**36**	1885405
						120.7	**16**	101
						116	**60**	691057
						101.1	**60**	5201
						89.1	**60**	136667
						77.1	**60**	27431
						71.1	**32**	60124
	JWH 018 2'-naphthyl-N-(2,2-dimethylpropyl) isomer	C24H23NO	**FIU_0038**	11.65	**342.2**	272.1	**24**	20828
						214.1	**24**	512814
						155.1	**28**	581873
						144	**36**	652737
						127	**56**	3266340
						116	**60**	231221
						101	**60**	16327
						89.1	**60**	41230
						77.1	**60**	89445
						71.1	**36**	59576

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	JWH 018 N-(1-ethylpropyl) isomer	C24H23NO	**FIU_0043**	11.39	**342.2**	155	**24**	4929262
						127	**56**	3847552
	JWH 018 N-(2,2-dimethylpropyl) isomer	C24H23NO	**FIU_0045**	11.43	**342.2**	214.2	**24**	4490
						155	**24**	108778
						144	**32**	6790
						127.1	**56**	36813
						116.1	**60**	2330
						77	**60**	1577
	JWH 073 4-hydroxyindole metabolite	C23H21NO2	**FIU_0501**	11.25	**344.15723**	216.1	**24**	170177
						160	**40**	110347
						132.1	**48**	20566
						127	**60**	399774
						104.1	**60**	53011
						77.1	**60**	14907
						57.2	**44**	11146
	JWH 073 4-methylnaphthyl analog	C24H23NO	**FIU_0212**	11.61	**342.2**	200.1	**24**	694343
						169	**24**	2523920

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						158	**36**	12256
						155	**24**	1365619
						144	**40**	411867
						141	**48**	1687110
						116.1	**60**	154476
						115	**60**	923228
						91.1	**60**	22355
						89.1	**60**	32394
						57.1	**44**	48839
	JWH 081 3-methoxynaphthyl	C25H25NO2	**FIU_0056**	11.66	**372.2**	214.1	**24**	1640497
						185	**24**	3247995
						157.6	**36**	29087
						157	**36**	153891
						144	**40**	989738
						130.2	**52**	25575
						129	**44**	1328825
						128	**60**	1353255
						127.6	**60**	1083946
						116	**60**	331907

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	JWH 081 5-methoxynaphthyl	C25H25NO2	**FIU_0057**	11.69	**372.2**	214.1	**28**	635460
						185.1	**24**	5748473
						185	**24**	2122887
						170	**44**	76497
						157	**40**	1304054
						144	**44**	490156
						142	**60**	69563
						129	**48**	885170
						128.5	**60**	1190426
						127	**60**	2758699
						116	**60**	181210
	JWH 081 8-methoxynaphthyl	C25H25NO2	**FIU_0060**	11.36	**372.2**	185	**16**	5080778
						170	**48**	2856931
						155	**48**	60649
						142	**60**	241953
						141	**40**	112625
						129	**44**	226009
						127.7	**60**	206652

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						127	**60**	592046
						115.1	**60**	54893
						114	**60**	437505
	JWH 116	C26H27NO	**FIU_0519**	11.81	**370.20926**	242.2	**24**	222632
						172.1	**40**	128704
						157	**56**	14423
						155	**28**	2809704
						144.1	**56**	31195
						129.1	**60**	20172
						127.1	**60**	2328859
						124.6	**56**	150
						117.1	**60**	10398
						77.1	**60**	34622
	JWH 210 2-ethylnaphthyl isomer	C26H27NO	**FIU_0070**	11.67	**370.2**	214.1	**24**	1724013
						183.1	**24**	2635884
	JWH 210 8-ethylnaphthyl isomer	C26H27NO	**FIU_0075**	11.68	**370.2**	214.2	**24**	590695
						183.1	**24**	2452270
						165.1	**36**	407856

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						155.1	**44**	1493220
						153.1	**60**	385078
						144.1	**44**	365853
						141.1	**44**	110114
						140.1	**60**	448160
						128.7	**60**	135692
						127.1	**60**	145653
	JWH 307 5'-isomer	C26H24FNO	**FIU_0548**	11.33	**386.18419**	258.1	**24**	62268
						258.1	**24**	33273
						188.1	**40**	33942
						188	**40**	54009
						160	**56**	13896
						155.1	**20**	1703780
						140.9	**44**	111
						133	**60**	29733
						133	**60**	10846
						127.1	**60**	1223076
						101	**60**	18979
						77.1	**60**	104656

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						77.1	**60**	17375
						75.1	**60**	3554
						51.1	**60**	5986
	JWH 398 N-(4-hydroxypentyl) metabolite	C24H22ClNO2	**FIU_0551**	10.9	**392.13391**	374.1	**16**	10095
						318.1	**28**	14989
						254.2	**56**	4877
						243.2	**60**	109
						189	**24**	660972
						186.1	**12**	20003
						161	**56**	407534
						144.1	**44**	12916
						126	**60**	61783
						69.1	**40**	14061
	LY2183240 2'-isomer	C17H17N5O	**FIU_0462**	10.38	**308.14331**	192.1	**16**	5876
						167.1	**16**	141147
						165.1	**60**	16540
						152.1	**56**	12037
						87.1	**12**	42396

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						72.2	**36**	183659
						59.2	**20**	7404
						56.1	**60**	5844
	MAM2201 N-(4-hydroxypentyl) metabolite	C25H24FNO2	**FIU_0640**	10.45	**390.17911**	248.1	**24**	19106
						169.1	**24**	171349
						144	**40**	23459
						141.1	**52**	97499
						117.6	**44**	67
						115	**60**	27474
						87.1	**40**	2348
						67.1	**44**	4512
						59.1	**60**	2865
	MAM2201 N-(5-chloropentyl) analog-d5	C25H19D5ClNO	**FIU_0642**	11.38	**395.18603**	253.1	**28**	25431
						252	**28**	13307
						170	**32**	35394
						169.1	**28**	122216
						149.1	**48**	12245

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						148.1	**48**	6692
						142.2	**56**	19661
						141.1	**56**	88035
						115.1	**60**	22900
						69.2	**44**	8188
	MDA 77	C21H23N3O3	**FIU_0242**	11.78	**366.2**	355	**8**	41
						337.1	**12**	85104
						207.3	**48**	110
						175	**20**	26462
						161	**8**	456167
						119	**16**	21653
						105	**20**	5186463
						77.1	**60**	3741867
						51.1	**60**	161475
	MDMA methylene homolog (hydrochloride)	C12H17NO2	**FIU_0244**	7.19	**208.1**	180	**16**	1054437
						177	**12**	241215
						147.1	**16**	237407
						135	**20**	1816798
						119.1	**20**	142294

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						105	**40**	108255
						91.1	**36**	184690
						79.1	**36**	185885
						77.1	**48**	798463
						55.1	**28**	79055
						51.1	**60**	625342
	Methylphenidate	C14H19NO2	**FIU_0758**	7.15	**234.14158**	174.1	**24**	4008
						134.6	**12**	56
						129	**40**	3527
						128	**56**	3188
						115	**56**	2897
						91	**60**	7695
						84.1	**16**	370067
						67.1	**56**	5769
						65.1	**60**	2959
						56.1	**56**	78057
	NM2201	C24H22FNO2	**FIU_0635**	11.35	**376.16346**	232.1	**8**	165033
						179.7	**20**	66
						144	**44**	63532

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						116	**60**	23804
						89.1	**60**	2949
						69.1	**44**	6403
	NNEI 2'-naphthyl isomer	C24H24N2O	**FIU_0448**	11.35	**357.18886**	214.2	**20**	549904
						188.2	**12**	12488
						158.1	**40**	8189
						144.1	**44**	250574
						132.1	**32**	2227
						130.1	**48**	5579
						116.1	**60**	81782
						89.2	**60**	8571
						71.2	**44**	4211
						55.1	**60**	2750
	Norsufentanil	C16H24N2O2	**FIU_0255**	7.841	**277.2**	245.1	**8**	319177
						128.1	**8**	1196540
						96.1	**20**	1657435
						94.1	**40**	150409
						81.1	**48**	172662
						80.1	**60**	118942

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						77.1	**60**	98955
						69.2	**40**	84199
						68.1	**48**	108879
						67.1	**44**	260507
	PB-22 4-hydroxyisoquinoline isomer	C23H22N2O2	**FIU_0599**	11.39	**359.16813**	214.1	**16**	2225963
						158	**36**	29863
						144	**40**	964732
						130.1	**48**	16363
						116	**60**	319712
						89.1	**60**	35401
						71.2	**40**	16900
						55.1	**60**	9593
15	**(-)-CP 47,497**	C21H34O2	**FIU_0090**	11.67	**319.3**	133	**52**	5390
						121	**32**	7826
						107	**24**	13752
						85.1	**12**	6450
						77.1	**60**	3998
						71.1	**12**	13494

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						57.1	**20**	9928
	(+)-CP 55,940	C24H40O3	**FIU_0093**	11.68	**377.3**	211.3	**12**	104
						201.8	**48**	111
						149.1	**12**	2256
	(±)3-epi CP 47,497-C8-homolog	C22H36O2	**FIU_0559**	12.38	**333.27153**	257	**20**	1476
						227	**28**	1550
						199.3	**44**	106
						196.9	**40**	1519
						167	**60**	1339
						124.3	**20**	93
						71.2	**12**	5630
						57.2	**20**	5896
	(±)5-epi CP 55,940	C24H40O3	**FIU_0096**	11.67	**377.3**	359.3	**0**	14939
						233.1	**4**	6903
						219.3	**8**	119
						215.1	**12**	6483
						158.2	**60**	147
						121.1	**20**	4504
						93.1	**36**	2085

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						71.1	**20**	3807
						57.2	**28**	2824
	(±)-JWH 018 N-(4-hydroxypentyl) metabolite	C24H23NO2	**FIU_0471**	10.34	**358.17288**	340.2	**16**	13207
						284.2	**24**	19432
						230.1	**24**	14344
						186.2	**12**	24746
						155.1	**20**	1042394
						144.1	**40**	36994
						127.1	**56**	705941
						116.1	**60**	13978
						77.1	**60**	11690
						69.2	**36**	28266
	1-(4-Fluorobenzyl) piperazine (HCl)	C11H15FN2	**FIU_0103**	11.65	**195.1**	175.1	**12**	136158
						109	**20**	1787937
						89	**52**	53117
						85.1	**12**	182619
						83.1	**56**	684570

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						81.1	**60**	7712
						63.1	**60**	113071
						59.1	**60**	95812
						57.1	**60**	187981
						56.1	**28**	70798
	2-AI (HCl)	C9H11N	**FIU_0111**	11.66	**134.1**	117	**12**	691352
						115.1	**24**	263781
						91.1	**32**	250680
						89.1	**48**	26626
						77.1	**44**	11406
						75.1	**60**	3814
						74	**60**	3350
						65.1	**52**	129437
						63.1	**60**	36873
						51.1	**60**	33380
	3,4-Dimethoxymethamphetamine (HCl)	C12H19NO2	**FIU_0122**	11.66	**210.1**	179.1	**8**	3108697
						164.1	**20**	239844
						151	**20**	1215109

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						138.1	**20**	181366
						136	**24**	122021
						121	**32**	150430
						107	**40**	274328
						91.1	**40**	295370
						77.1	**60**	591332
						65.1	**60**	167026
	3',4'-Methylenedioxy-a-pyrrolidinobutiophenone (HCl)	C15H19NO3	**FIU_0124**	6.65	**262.2**	191	**16**	1194804
						188.1	**8**	2267557
						163.1	**20**	689317
						161	**20**	1498141
						149	**32**	785534
						133	**28**	416705
						121	**48**	394286
						112.1	**28**	1697208
						105.1	**40**	485438
						84.1	**36**	317090
						65.1	**60**	847141

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	3-Ethylethcathinone (HCl)	C13H19NO	**FIU_0126**	11.65	**206.2**	160.1	**16**	940937
						159.1	**20**	1472649
						144.1	**32**	1749084
						131.7	**20**	382214
						130	**44**	353936
						117	**36**	179114
						105.1	**32**	347628
						91.1	**56**	208802
						77.1	**60**	519681
	3-Methoxymethcathinone (hydrochloride)	C11H15NO2	**FIU_0132**	11.65	**194.1**	161.1	**20**	959356
						146	**28**	456167
						145.6	**24**	156949
						133.1	**28**	127138
						132.1	**36**	248485
						118	**40**	212382
						91.1	**56**	226803
						79.1	**36**	73823
						77.1	**56**	218110
	4-APB	C11H13NO	**FIU_0416**	7.13	**176.09971**	131.1	**16**	408457

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						129.1	**24**	14969
						116.1	**32**	22590
						115.1	**48**	38812
						103.1	**36**	18883
						91.1	**32**	96922
						77.2	**48**	109322
						65.2	**60**	40052
						51.2	**60**	43175
	4-Bromomethcathinone	C10H12BrNO	**FIU_0353**	7.52	**242.01023**	145.1	**16**	467160
						144.1	**40**	289388
						132.1	**20**	29385
						131.1	**48**	19805
						128.1	**60**	21744
						104.1	**40**	19868
						103.1	**56**	31340
						78.2	**60**	25106
						77.1	**60**	46914
						58.2	**60**	8656
	4-FA (4-fluoroamphetamine)	C9H12FN	**FIU_0139**	6.26	**154.1**	137	**4**	762528

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						109	**16**	986151
						101.1	**48**	4093
						89.1	**40**	25518
						83	**44**	314664
						81	**52**	5921
						75.1	**60**	13231
						63.1	**60**	67353
						59.1	**48**	51592
						57.1	**60**	154673
	4-Fluoromethamphetamine (hydrochloride)	C10H14FN	**FIU_0142**	11.69	**168.1**	137	**8**	1278139
						109	**20**	2314861
						101	**56**	6381
						89.1	**48**	63120
						83	**52**	735154
						81	**60**	11987
						75.1	**60**	20924
						63.1	**60**	151872
						59.1	**56**	113028

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						57.1	**60**	313974
	4-Hydroxyamphetamine	C9H13NO	**FIU_0292**	7.82	**152.09971**	110.3	**32**	99
						107.1	**16**	189502
						91.1	**44**	7007
						79.1	**32**	18793
						77.1	**40**	76831
						65.1	**60**	8147
						55.1	**48**	8408
						53.2	**52**	4617
						51.1	**56**	28604
	5-fluoro PB-22 7-hydroxyquinoline isomer	C23H21FN2O2	**FIU_0588**	10.66	**377.15871**	232.1	**20**	399667
						223.1	**20**	55
						212	**40**	3831
						158	**40**	3726
						144	**44**	187525
						130	**56**	3272
						116	**60**	63500
						89	**60**	6519
						69.1	**48**	13519

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						61.1	**60**	2532
	5-fluoro-THJ	C22H21FN4O	**FIU_0711**	11.56	**377.16994**	359.1	**20**	99056
						213.1	**28**	89616
						177	**36**	38771
						171	**40**	11608
						145	**44**	160772
						117	**60**	27113
						90	**60**	34529
						89.2	**60**	14289
						69.1	**36**	44434
	5-IAI (hydrochloride)	C9H10IN	**FIU_0153**	11.72	**260**	242.9	**12**	124229
						159.5	**48**	91
						154.8	**16**	105
						116	**28**	327950
						115	**56**	262746
						89	**60**	10210
	A-834735 degradant	C22H29NO2	**FIU_0690**	10.9	**340.21983**	242.1	**20**	1383090
						144	**44**	121338
						125	**24**	38316

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						99.1	**32**	508813
						83.1	**32**	31822
						81.1	**44**	156458
						79.1	**60**	52332
						69.1	**48**	301286
						57.1	**44**	115133
						55.1	**56**	117964
	AB-FUBINACA 2-fluorobenzyl isomer	C20H21FN4O2	**FIU_0716**	10.26	**369.16485**	109	**60**	74428
	ADBICA	C20H29N3O2	**FIU_0694**	10.89	**344.22598**	327.1	**4**	294416
						299.2	**12**	8506
						214.1	**20**	366724
						158	**40**	4458
						144	**44**	136230
						130	**52**	3416
						121.1	**28**	74
						116	**60**	44077
						89	**60**	5325
	AM1235	C24H21FN2O3	**FIU_0160**	11.16	**405.2**	277.1	**24**	436955
						231.1	**40**	42921

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						189	**36**	189810
						172	**56**	12444
						155	**28**	694010
						144.1	**56**	8474
						143	**56**	208191
						127	**60**	562877
						115	**60**	15184
						69.1	**40**	39604
	AM2201 N-(2-fluoropentyl) isomer	C24H22FNO	**FIU_0028**	11.19	**360.2**	232.1	**24**	996175
						212.1	**40**	65149
						163.1	**8**	55708
						155	**28**	1542918
						144	**44**	343227
						127	**56**	4007228
						116	**60**	134909
						105	**40**	29289
						77.1	**60**	115888
						69.1	**44**	74493
	AM694 4-iodo isomer	C20H19FINO	**FIU_0032**	11.23	**436.1**	232.2	**28**	35373

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						230.9	**28**	1773251
						202.9	**56**	832575
						144	**56**	37593
						130.1	**60**	4162
						116	**60**	12023
						104	**60**	177754
						76.1	**60**	457749
						75	**60**	1955
						50.1	**60**	11667
	BB-22 3-carboxyindole metabolite	C16H19NO2	**FIU_0616**	10.9	**258.14158**	214.1	**12**	4904
						176	**16**	4751
						169.4	**12**	81
						158.1	**12**	56
						132	**16**	6106
						118	**24**	9732
						97.1	**20**	3226
						91	**52**	3165
						69.1	**28**	1686
						55.1	**36**	15064

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	BB-22 7-hydroxyquinoline isomer	C25H24N2O2	**FIU_0625**	11.61	**385.18378**	240.2	**20**	391274
						144	**44**	140304
						138.8	**28**	54
						116	**60**	22713
						97.1	**40**	23426
						69.2	**52**	12572
						55.1	**60**	127830
	Cannabipiperidiethanone	C24H28N2O2	**FIU_0247**	8.336	**377.2**	280.1	**16**	232019
						229.1	**16**	571388
						144	**40**	86872
						121	**24**	1420122
						112.1	**24**	2979710
						98.1	**40**	1866851
						93.1	**48**	141629
						91.1	**60**	1054701
						70.1	**60**	576499
						58.1	**60**	697418
	CP 47,497-C6-homolog	C20H32O2	**FIU_0566**	14.52	**305.24023**	329.3	**4**	5428
						219.1	**16**	4943

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						175.1	**8**	5884
						107	**20**	2547
						71.2	**12**	3268
						57.1	**20**	2973
	D-Amphetamine	C9H13N	**FIU_0296**	6.37	**136.1048**	119.1	**4**	316370
						91.1	**16**	490294
						77.1	**48**	6194
						65.1	**44**	148115
						63.1	**56**	13406
						51.1	**60**	24279
	HU-308	C27H42O3	**FIU_0189**	11.74	**415.3**	271.1	**12**	11983
						229.1	**16**	20287
						215.1	**12**	23499
						151	**16**	22820
						133.1	**16**	7398
						91.1	**56**	15306
						85.1	**24**	10761
						79.1	**52**	3868
						71.2	**28**	24245

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						57.1	**36**	16073
	JP104	C25H30N2O3	**FIU_0192**	10.878	**407.2**	390.2	**4**	38459
						212.1	**8**	33233
						197	**24**	278416
						171.1	**32**	119934
						169.1	**48**	35209
						153	**52**	151802
						141.1	**60**	61728
						95.1	**16**	13345
	JWH 007	C25H25NO	**FIU_0193**	11.73	**356.2**	228.1	**24**	449889
						158	**40**	303826
						144	**44**	3043
						130	**60**	98680
						127	**56**	3494643
						103.1	**60**	27864
						101	**60**	13402
						77.1	**60**	83629
						70.5	**48**	268
	JWH 015	C23H21NO	**FIU_0195**	11.172	**328.2**	200.1	**20**	777955

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						158	**40**	420150
						155	**24**	2581326
						130	**56**	189386
						128.2	**60**	21653
						127	**48**	4510605
						103	**60**	83019
						101	**60**	52107
						77.1	**60**	318626
						51.1	**60**	13237
	JWH 018 2'-naphthyl isomer	C24H23NO	**FIU_0034**	11.75	**342.2**	272.1	**16**	2685647
						214.1	**24**	328459
						158	**36**	7347
						155	**28**	2429736
						144	**40**	206154
						130.1	**52**	5848
						127	**60**	1943403
						127	**60**	1515840
						116	**60**	88552
						101	**60**	8898

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						89.1	**60**	16793
						77.1	**60**	50271
	JWH 018 N-(1-methylbutyl) isomer	C24H23NO	**FIU_0044**	11.49	**342.2**	272.1	**20**	9613
						217	**28**	187
						214.1	**20**	30114
						155.1	**24**	39037
						144.1	**36**	76873
						127	**48**	277031
						116	**60**	32010
						101	**60**	2203
						89.1	**60**	9212
						77.1	**60**	11114
	JWH 019 N-(3-fluorohexyl) isomer	C25H24FNO	**FIU_0490**	11.34	**374.18419**	246.1	**24**	101003
						241.2	**48**	311
						235.3	**32**	115
						234	**44**	96
						155	**28**	639768
						144	**44**	38027

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						128.9	**60**	600
						127	**60**	556165
						116.1	**60**	15061
						55.1	**56**	35903
	JWH 073 2-methylnaphthyl analog	C24H23NO	**FIU_0211**	11.37	**342.2**	200.1	**24**	2120190
						158	**36**	48654
						144	**40**	1439879
						141	**44**	2964161
						130.1	**48**	36665
						116.1	**60**	560384
						115	**60**	1398875
						89.1	**60**	117601
						57.1	**44**	171175
	JWH 073 2'-naphthyl-N-(1-methylpropyl) isomer	C23H21NO	**FIU_0050**	11.44	**328.2**	155	**28**	450081
						127	**52**	1987
	JWH 073 N-(1-methylpropyl) isomer	C23H21NO	**FIU_0053**	11.24	**328.2**	144.1	**32**	755
						127	**48**	2336

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	JWH 080	C24H23NO2	**FIU_0513**	11.35	**358.17288**	200.1	**24**	509551
						185.1	**24**	1681297
						157.1	**44**	656393
						144	**44**	305867
						142	**56**	269199
						128.2	**60**	165048
						127.1	**60**	468608
						116	**60**	121793
						114	**60**	217204
						57.2	**48**	32943
	JWH 081	C25H25NO2	**FIU_0213**	11.76	**372.2**	214.1	**24**	1254254
						185	**28**	4107559
						157	**48**	1494001
						144	**44**	762692
						142	**60**	674803
						129.2	**56**	59356
						128.1	**60**	348682
						127	**60**	970491
						116	**60**	222687

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						114	**60**	391649
	JWH 081 2-methoxynaphthyl	C25H25NO2	**FIU_0055**	11.32	**372.2**	214.1	**24**	151975
						185.1	**20**	5629250
						170	**48**	687030
						155	**48**	29741
						144	**44**	126069
						142	**56**	1906954
						129	**44**	326360
						127	**60**	773441
						116	**60**	58571
						114	**60**	790815
	JWH 081 6-methoxynaphthyl	C25H25NO2	**FIU_0058**	11.6	**372.2**	214.1	**24**	1625473
						157	**44**	1782476
						144	**44**	808030
						142	**60**	1017151
						129.2	**56**	27994
						128	**60**	179017
						127	**60**	154957

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						116	**60**	237373
						114	**60**	157876
	JWH 081 N-pentanoic acid metabolite	C25H23NO4	**FIU_0517**	10.42	**402.16271**	244.1	**24**	46305
						185.1	**28**	424044
						157.1	**48**	135914
						144	**40**	46203
						142	**60**	55310
						128.1	**60**	27248
						127	**60**	80740
						114.1	**60**	18875
						83.1	**44**	11446
						55.1	**56**	28615
	JWH 122 3-methylnaphthyl isomer	C25H25NO	**FIU_0062**	11.77	**356.2**	169.1	**20**	2175
						141.1	**44**	1524
	JWH 176	C25H24	**FIU_0527**	11.12	**325.1878**	268.1	**20**	8116
						255.2	**12**	50942
						254	**16**	11872
						253.1	**40**	18508

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						240.2	**40**	8304
						239.1	**56**	10220
						218.7	**8**	100
						141	**36**	12363
						117	**24**	3150
						115	**60**	6072
	JWH 203 4-chlorophenyl isomer	C21H22ClNO	**FIU_0069**	11.47	**340.2**	124.9	**28**	2654
	JWH 250 5-hydroxyindole metabolite	C22H25NO3	**FIU_0544**	10.63	**352.18344**	216.1	**24**	29318
						204.1	**16**	44666
						160	**40**	42613
						146	**44**	29781
						131	**40**	23001
						121	**20**	573349
						93.1	**36**	52252
						91.1	**60**	326959
						77.1	**60**	20797
						65.1	**60**	25626
	JWH 309 5'-isomer	C30H27NO	**FIU_0549**	11.81	**418.20926**	290.1	**24**	83642

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						220.1	**44**	40195
						192.1	**56**	5214
						165.1	**60**	15594
						155	**24**	1635180
						127	**60**	1138691
						77.1	**60**	7667
	JWH 398 2-chloronaphthyl isomer	C24H22ClNO	**FIU_0078**	11.39	**376.2**	214.1	**24**	231019
						189	**28**	4982582
						161	**56**	3251333
						158	**36**	6216
						144	**40**	217358
						130	**56**	9879
						126	**60**	1124469
						116	**60**	83838
						106.1	**24**	9821
						89.1	**60**	13033
	JWH 398 N-(5-hydroxypentyl) metabolite	C24H22ClNO2	**FIU_0552**	10.87	**392.13391**	236.9	**16**	104

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						230.2	**28**	7004
						189	**20**	465236
						161	**52**	267650
						144.1	**48**	11857
						126.1	**60**	41737
						69.1	**44**	4461
	Ketazolam	C20H17ClN2O3	**FIU_0239**	10.29	**369.1**	285.1	**12**	280998
						257	**32**	16837
						241	**52**	8974
						228	**36**	14853
						222.1	**40**	22339
						193.1	**48**	43350
						180	**40**	7040
						154	**40**	40360
						105	**32**	6998
						91.1	**56**	13109
	MAM2201 N-(2-fluoropentyl) isomer	C25H24FNO	**FIU_0637**	11.33	**374.18419**	232.1	**24**	71184
						212.1	**44**	5095
						169.1	**24**	233041

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						144	**48**	22486
						141.1	**48**	161427
						115.9	**60**	8297
						115.1	**60**	60736
						69.1	**44**	5184
	MAM2201 N-(5-chloropentyl) analog	C25H24ClNO	**FIU_0641**	11.39	**390.15464**	248.1	**24**	43010
						212.1	**40**	2622
						169.1	**28**	174982
						144	**48**	25179
						141.1	**56**	115194
						116.1	**60**	7616
						115.1	**60**	33935
						69.1	**40**	8541
	MDA 19	C21H23N3O2	**FIU_0241**	11.96	**350.2**	322.2	**8**	15488
						321.2	**12**	17301
						105	**16**	4002871
						77.1	**60**	2858803
						51.1	**60**	146489
	Methoxetamine	C15H21NO2	**FIU_0246**	7.104	**248.2**	175.1	**16**	1256161

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						159	**24**	205586
						121	**28**	1834341
						115	**60**	143861
						91.1	**56**	785339
						78.1	**60**	322780
						77.1	**60**	399226
						67.1	**24**	526201
						65.1	**60**	335234
	Mitragynine	C23H30N2O4	**FIU_0251**	8.04	**399.2**	367.2	**20**	467367
						238.1	**24**	1026914
						226.1	**24**	1137626
						174.1	**36**	2745239
						159	**56**	1035371
						143.6	**56**	178496
						129	**36**	373236
						117	**60**	301729
						110.1	**36**	663958
						75.1	**56**	403343
	PB-22 3-hydroxyquinoline isomer	C23H22N2O2	**FIU_0598**	11.57	**359.16813**	214.1	**20**	3192364

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						158.1	**36**	40789
						144	**44**	1346827
						131.1	**40**	142
						130.1	**56**	29738
						116	**60**	427352
						89.1	**60**	51329
						71.2	**40**	22734
						55.1	**60**	11832
	PB-22 N-(5-hydroxypentyl) metabolite	C23H22N2O3	**FIU_0610**	10.05	**375.16304**	230.1	**8**	857722
						197.8	**56**	89
						144	**44**	393476
						130.1	**40**	4500
						116	**60**	100226
						89.1	**60**	8081
						87.1	**36**	6747
						69.1	**44**	117910
						67.1	**52**	5102
						57.1	**52**	3751
	Pentylone (hydrochloride)	C13H17NO3	**FIU_0257**	7.182	**236.1**	218.1	**8**	1222013

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						188.1	**16**	1722198
						175	**20**	737605
						160.1	**24**	353181
						159	**32**	238298
						135	**24**	346013
						131.1	**40**	605366
						86.1	**16**	491089
						77.1	**60**	258434
						65.1	**60**	373987
16	**(-)-CP 47,497**	C21H34O2	**FIU_0555**	14.55	**319.25588**	301.3	**4**	209
						256.9	**16**	1614
						226.9	**28**	1377
						197	**36**	1552
						194.8	**56**	99
						167	**52**	1286
	(+)-CP 47,497	C21H34O2	**FIU_0092**	11.67	**319.3**	233.2	**16**	5304
						175.1	**4**	6556
						133.1	**52**	11635
						121.1	**32**	21203

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						107.1	**28**	3166
						107	**28**	29911
						85.1	**16**	15021
						77.1	**60**	11302
						57.1	**24**	21782
	(±)3-epi CP 47,497-C8-homolog	C22H36O2	**FIU_0095**	11.68	**333.3**	193.1	**0**	17115
						175.1	**4**	12820
						141.1	**4**	30894
						107.1	**24**	45118
						107.1	**20**	6758
						85.1	**12**	42672
						81.1	**36**	7221
						79.1	**60**	9103
						77.1	**60**	8900
						57.1	**20**	48600
	(±)-CP 55,940	C24H40O3	**FIU_0099**	11.67	**377.3**	359.3	**0**	11115
						233.1	**8**	4667
						219.1	**24**	112
						215.3	**12**	4306

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						175.1	**4**	181028
						149	**20**	1142
						121	**16**	5476
						106	**56**	264
						79.1	**48**	1847
						71.2	**24**	3674
						57.1	**44**	2241
	(±)-epi CP 47,497	C21H34O2	**FIU_0100**	11.67	**319.3**	287	**8**	2296
						257.1	**16**	2093
						226.9	**28**	1874
						197	**40**	1993
						193.1	**0**	30928
						175.1	**4**	9216
						127.1	**4**	60202
						107	**28**	3488
						107	**20**	71808
						85.1	**12**	57086
						81.1	**28**	10222
						79.1	**60**	14131

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						77.1	**60**	14460
						71.2	**16**	5251
						71.1	**12**	104540
						57.2	**24**	3800
						57.1	**20**	68024
	(±)-UR-144 N-(4-hydroxypentyl) metabolite	C21H29NO2	**FIU_0644**	10.79	**328.21983**	310.3	**16**	6376
						230.2	**20**	6965
						144.1	**36**	10728
						130.1	**52**	2681
						125.1	**20**	202233
						97.1	**32**	24631
						83.1	**32**	10543
						69.1	**40**	26954
						57.2	**48**	28668
						55.1	**48**	56750
	(S)-(+)-JWH 073 N-(3-hydroxybutyl) metabolite	C23H21NO2	**FIU_0512**	10.31	**344.15723**	284.1	**24**	47787
						216.1	**24**	74270

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						158	**32**	55357
						155.1	**24**	1562802
						144	**44**	36078
						141	**24**	10495
						130.1	**48**	47487
						127	**56**	1188832
						77.1	**60**	35611
						55.2	**52**	17134
	1-(p-Fluorophenyl) piperazine (HCl)	C10H13FN2	**FIU_0105**	11.65	**181.1**	179.1	**16**	82769
						138	**20**	832868
						136	**28**	175034
						110.1	**32**	62902
						109	**40**	106281
						96	**44**	102483
						95	**48**	142506
						91.1	**36**	106754
						83.1	**52**	112263
						75.1	**60**	235458
	2,3-MDMA	C11H15NO2	**FIU_0359**	6.82	**194.11028**	135	**16**	490752

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						133.1	**16**	127405
						105.1	**24**	276094
						103.1	**40**	62415
						79.1	**36**	149327
						77.1	**48**	322564
						65.1	**52**	21005
						58.2	**16**	21083
						51.1	**60**	175635
	2C-D	C11H17NO2	**FIU_0104**	N/A	**196.1**	179.1	**8**	2288847
						164.1	**16**	913337
						149	**28**	465147
						119	**20**	169923
						117	**24**	160816
						115	**32**	124114
						103.1	**44**	59413
						91.1	**40**	462609
						77.1	**56**	359642
						65.1	**60**	181139
	2-Ethylethcathinone (hydrochloride)	C13H19NO	**FIU_0112**	11.66	**206.2**	160.1	**12**	542020

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						159.1	**16**	863271
						144.1	**32**	1027195
						132.1	**20**	219652
						131	**24**	167422
						130.1	**44**	210817
						128	**44**	117071
						91.1	**60**	129534
						77.1	**60**	269153
	2-Fluoroisocathinone	C9H10FNO	**FIU_0330**	5.09	**168.07464**	135	**24**	4109
						113	**40**	65
						103.1	**24**	156217
						97.1	**36**	3077
						95	**48**	4765
						77.1	**40**	158993
						75.1	**60**	15704
						51.1	**60**	70381
	2-Fluoromethamphetamine (hydrochloride)	C10H14FN	**FIU_0116**	11.66	**168.1**	137.1	**8**	750768
						115	**40**	9398

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						109	**16**	2471668
						89.1	**48**	74142
						83.1	**48**	842629
						81.1	**60**	24256
						65.1	**60**	21162
						63.1	**60**	181237
						59.1	**52**	136619
						57.1	**60**	384207
	2-Methoxyamphetamine	C10H15NO	**FIU_0284**	7.19	**166.11536**	149.1	**4**	544004
						121.1	**16**	593311
						115.1	**40**	19178
						93.1	**20**	45978
						91.1	**28**	368652
						78.2	**48**	52770
						77.1	**36**	47385
						65.2	**48**	180614
						63.2	**60**	19293
						51.2	**60**	26662
	3-Methylmethcathinone (hydrochloride)	C11H15NO2	**FIU_0134**	11.65	**194.1**	177	**0**	2631

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						59.1	**12**	2363
	4-Ethylethcathinone (hydrochloride)	C13H19NO	**FIU_0136**	11.65	**206.2**	160.1	**16**	838944
						159.1	**20**	1315599
						144.1	**32**	1419213
						130	**44**	280770
						117	**32**	179624
						115	**60**	224469
						105.1	**32**	354534
						91.1	**56**	209305
						77.1	**60**	448597
	4-Fluoroethcathinone (hydrochloride)	C11H14FNO	**FIU_0140**	11.63	**196.1**	178.1	**12**	2304538
						149.7	**16**	1223699
						149.6	**20**	966491
						148	**36**	780187
						135	**28**	526489
						123	**24**	369768
						115	**32**	171261
						103	**36**	442324

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						77.1	**52**	563787
						75	**60**	224247
	4-MTA	C10H15NS	**FIU_0295**	7.76	**182.09252**	165.1	**4**	473458
	5-chloro AB-PINACA	C18H25ClN4O2	**FIU_0702**	10.34	**365.1666**	348.1	**4**	843149
						320.1	**12**	1045490
						249	**24**	783163
						213.1	**36**	355339
						193	**40**	33221
						171	**48**	43639
						145	**48**	393527
						117	**60**	52354
						90	**60**	58269
						69.1	**48**	138929
	5-fluoro AB-PINACA	C18H25FN4O2	**FIU_0704**	9.93	**349.19615**	346.2	**4**	136707
						332.1	**4**	180571
						318.2	**12**	137353
						233	**24**	186686
						213	**36**	49839
						177	**40**	18735

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						144.9	**48**	92353
						127	**60**	103
						117	**60**	13273
						90	**60**	9513
						69.1	**40**	26330
	5-fluoro NNEI 2'-naphthyl isomer	C24H23FN2O	**FIU_0438**	10.9	**375.17944**	232.2	**20**	510890
						212.2	**44**	4725
						206.2	**16**	11018
						158.1	**44**	4467
						144.1	**44**	257722
						130	**60**	4555
						116.1	**60**	86151
						89.1	**60**	7960
						69.2	**48**	19567
						61.2	**60**	3191
	5-methoxy MiPT	C15H22N2O	**FIU_0156**	6.49	**247.2**	174.1	**16**	1800810
						159	**32**	658412
						143	**36**	285289
						131.1	**44**	506709

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						130	**60**	914787
						117.1	**56**	51664
						115	**60**	119428
						103	**60**	125276
						86.1	**12**	3771516
						77.1	**60**	87730
	AB-PINACA	C18H26N4O2	**FIU_0721**	10.63	**331.20558**	314.1	**4**	96165
						286.1	**12**	105232
						215	**28**	128210
						144.9	**48**	67364
						117	**60**	8023
						90	**60**	11346
						89	**60**	5188
	AB-PINACA N-(2-fluoropentyl) isomer	C18H25FN4O2	**FIU_0722**	10.26	**349.19615**	332.1	**4**	45912
						304.1	**12**	68529
						304.1	**12**	55983
						233	**24**	64244
						221.4	**20**	62
						145	**48**	24467

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	ADB-PINACA isomer 3	C19H28N4O2	**FIU_0736**	10.9	**345.22123**	344.2	**4**	153923
						328.2	**4**	239780
						316.1	**12**	150944
						300.2	**12**	223351
						215.1	**24**	330429
						145	**48**	178614
						117	**60**	19663
						90	**60**	24380
						71.1	**44**	2710
	AKB48 N-(5-hydroxypentyl) metabolite	C23H31N3O2	**FIU_0742**	11.3	**382.24163**	135.1	**24**	355301
						107	**56**	30633
						93	**60**	47552
						91	**60**	7279
						81.1	**60**	14406
						79	**60**	41974
						77	**60**	7750
						69.1	**60**	4276
						67.1	**60**	16265

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						55.1	**60**	6995
	AM679	C20H20INO	**FIU_0168**	11.24	**418.1**	291.1	**20**	426471
						274.1	**28**	115007
						234.1	**32**	260697
						230.9	**28**	2973420
						214.1	**32**	173774
						202.9	**52**	1447955
						144	**56**	140632
						105.1	**60**	85309
						104	**60**	218400
						76.1	**60**	987934
	BB-22 5-hydroxyquinoline isomer	C25H24N2O2	**FIU_0621**	11.69	**385.18378**	240.2	**20**	210609
						144	**44**	84635
						116.1	**60**	11958
						97.1	**40**	12978
						69.1	**48**	7467
						55.2	**60**	72538
	BB-22 6-hydroxyquinoline isomer	C25H24N2O2	**FIU_0623**	11.65	**385.18378**	384.1	**8**	1882

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						240.2	**24**	235990
						144	**48**	110900
						116	**60**	16835
						97.1	**44**	15144
						69.1	**52**	9363
						55.1	**60**	92413
	Buphedrone (hydrochloride)	C11H15NO	**FIU_0173**	11.73	**178.1**	132.1	**16**	429829
						131	**24**	660390
						130.1	**36**	455022
						117	**28**	97297
						103	**52**	81371
						91	**24**	369543
						77.1	**56**	372343
						65.1	**56**	112158
						51.1	**60**	178280
	Cannabidiolic Acid	C22H30O4	**FIU_0466**	11.39	**359.21441**	341.2	**8**	138494
						261.2	**24**	11869
						219.1	**32**	39872
						149	**48**	4109

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						135.1	**52**	3856
						109.1	**32**	3523
						81.1	**56**	3798
						69.2	**44**	3921
						67.1	**56**	4007
						55.2	**56**	3881
	Cannabidol	C21H30O2	**FIU_0175**	11.41	**315.2**	193.1	**20**	125293
						135.1	**16**	49031
						123	**36**	74797
						107.1	**28**	29702
						93.1	**24**	50704
						91.1	**56**	27451
						81.1	**40**	29657
						77.1	**60**	38893
						69.1	**36**	29620
						67.1	**60**	26974
	DiPT	C16H24N2	**FIU_0753**	7.12	**245.19395**	144	**20**	309287
						127.8	**48**	9431
						127	**44**	36728

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						117	**40**	59920
						114.1	**12**	284873
						102.1	**12**	27627
						91	**60**	39868
						77.1	**60**	21887
						72.1	**28**	50462
						65	**60**	5835
	EAM2201	C26H26FNO	**FIU_0634**	11.32	**388.19984**	232.1	**28**	44179
						183.1	**28**	144992
						160.1	**8**	1887233
						155.1	**44**	43132
						153.9	**60**	18501
						144	**44**	23726
						132.1	**16**	1119025
						131.1	**20**	907831
						129.1	**60**	13014
						128	**60**	5573
						115.1	**60**	8554
						108.4	**40**	61

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						77.1	**60**	5325
	Ethcathinone (hydrochloride)	C11H15NO	**FIU_0184**	11.74	**178.1**	252.1	**8**	839218
						130	**32**	796041
						117	**32**	465294
						105.1	**24**	465129
						103	**48**	142508
						79.1	**40**	179079
						77.1	**56**	689864
						51.1	**60**	282425
	FUB-144	C23H24FNO	**FIU_0658**	11.33	**350.18419**	332.2	**20**	12241
						252.1	**20**	18632
						208.1	**40**	4827
						125.1	**24**	89402
						109.1	**48**	110712
						97.1	**32**	14949
						83.1	**60**	14134
						69.1	**36**	10200
						57.2	**48**	16244
						55.1	**44**	33068

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	IMMA	C23H23ClN2O4	**FIU_0191**	10.632	**427.1**	397.6	**8**	56
						340	**12**	12250
						312.1	**16**	555767
						277.1	**24**	3331
						139	**28**	2351337
						111	**60**	1084533
						109.8	**60**	4498
						88.1	**20**	240661
						75.1	**60**	60717
						70.1	**56**	9986
	Isopentedrone	C12H17NO	**FIU_0336**	7.44	**192.13101**	214.1	**12**	289064
						174.1	**8**	373755
						161.1	**8**	371548
						132.1	**16**	88082
						119.1	**16**	72124
						117.1	**32**	13888
						105.1	**16**	34044
						91.1	**24**	845878
						77.1	**52**	29178

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						65.1	**60**	257167
	JWH 011	C27H29NO	**FIU_0194**	12.019	**384.2**	286.1	**20**	249426
						256.1	**20**	178975
						158	**36**	870935
						155	**28**	4555479
						155	**20**	5487416
						130	**60**	257321
						128.2	**60**	17946
						127	**60**	3210086
						103.1	**60**	62054
						77.1	**60**	49903
						57.1	**48**	218834
	JWH 018 2'-naphthyl-N-(1-ethylpropyl) isomer	C24H23NO	**FIU_0033**	11.61	**342.2**	272.1	**20**	117170
						214.1	**20**	332603
						155.1	**24**	4424907
						155	**24**	4123852
						144.1	**36**	1201002
						127	**56**	3278115
						116	**60**	537671

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						101.1	**60**	14286
						89.1	**60**	125795
						77.1	**60**	86488
						71.2	**36**	13697
	JWH 018 4-hydroxyindole metabolite	C24H23NO2	**FIU_0476**	11.47	**358.17288**	230.1	**24**	224326
						160.1	**44**	141597
						136.1	**20**	98
						132.1	**56**	23863
						127.1	**60**	534774
						119.1	**44**	163
						104.1	**60**	60611
						77.2	**60**	13686
	JWH 018 6-methoxyindole analog	C25H25NO2	**FIU_0198**	11.57	**372.2**	244.1	**24**	263233
						174	**40**	132347
						159	**56**	29070
						155	**24**	5443132
						146	**56**	55362
						131	**60**	18367

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						127	**56**	3835770
						119	**60**	34856
						77.1	**60**	79865
						73.1	**24**	2137
	JWH 018 N-(1,1-dimethylpropyl) isomer	C24H23NO	**FIU_0041**	11.39	**342.2**	254.1	**28**	21268
						244.1	**32**	11555
						155.1	**28**	2726479
						144	**32**	1585954
						127	**60**	2405313
						116	**60**	574236
						89.1	**60**	121416
						77.1	**60**	40370
						71.1	**32**	76660
	JWH 019 5-hydroxyindole metabolite	C25H25NO2	**FIU_0488**	11.12	**372.18853**	244.2	**28**	72139
						160.1	**44**	45091
						155	**28**	519271
						153.7	**36**	370
						132	**60**	16848

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						127	**56**	437691
						77.1	**60**	6784
	JWH 019 N-(4-fluorohexyl) isomer	C25H24FNO	**FIU_0491**	11.19	**374.18419**	354.2	**20**	101853
						284.1	**24**	41656
						246.1	**24**	231534
						226.1	**32**	41555
						155	**28**	1937388
						144	**44**	149535
						127	**56**	1647994
						116	**60**	62989
						61.1	**56**	13933
						55.1	**52**	71557
	JWH 031 2'-isomer	C21H23NO	**FIU_0498**	11.79	**306.17796**	178.1	**20**	176562
						155	**20**	2752284
						155	**16**	1951093
						150.1	**24**	44705
						127	**48**	1152641
						108.1	**32**	42143
						94.1	**36**	80856

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						80.1	**36**	47529
						77.1	**60**	80591
						66.1	**60**	45869
						55.1	**36**	21314
	JWH 073 N-(1,1-dimethylethyl) isomer	C23H21NO	**FIU_0052**	11.23	**328.2**	272.1	**16**	1669
						155.1	**20**	2421
						155	**24**	2364
						144	**32**	1288
						126.9	**56**	1728
	JWH 081 7-methoxynaphthyl	C25H25NO2	**FIU_0059**	11.34	**372.2**	185	**20**	4316320
						170	**48**	2352312
						155	**48**	43037
						142	**60**	196452
						141	**44**	91334
						129	**44**	200102
						128	**60**	176903
						127	**60**	512663
						115.1	**60**	45727

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						114	**60**	366408
	JWH 122 6-methylnaphthyl isomer	C25H25NO	**FIU_0064**	11.75	**356.2**	169.1	**24**	1376
						141.1	**40**	987
	MAM2201	C25H24FNO	**FIU_0240**	11.262	**374.2**	232.1	**24**	1392578
						169	**28**	5181833
						144	**44**	955675
						141	**52**	3555141
						130	**48**	17112
						116.1	**60**	342788
						115	**60**	1394190
						91.1	**60**	27397
						89.1	**60**	51675
						69.1	**40**	67392
	MAM2201 N-(3-fluoropentyl) isomer	C25H24FNO	**FIU_0638**	11.37	**374.18419**	240.5	**12**	54
						232.1	**24**	56398
						212.1	**36**	6760
						169.1	**28**	247286
						144	**48**	20148

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						141.1	**52**	149963
						116	**60**	8178
						115.1	**60**	56336
						69.2	**48**	9073
	Meconin	C10H10O4	**FIU_0245**	7.326	**195.1**	162	**20**	505497
						151	**24**	175878
						133.6	**28**	188824
						105	**40**	229156
						79.1	**24**	265361
						78.1	**40**	175613
						77.1	**44**	684716
						65.1	**56**	164058
						51.1	**60**	300277
	Mephedrone metabolite ((±)-Pseudoephedrine stereochemistry)	C11H17NO	**FIU_0339**	6.97	**180.13101**	162.1	**8**	926545
						147.1	**20**	179253
						131.1	**20**	108754
						117.1	**44**	38440
						115.7	**32**	50051

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						115.1	**48**	60206
						105.1	**36**	76106
						91.1	**32**	130880
						77.1	**60**	53499
						56.2	**28**	37194
	methyl-1-(5-fluoropentyl)-1H-indole-3-Carboxylate	C15H18FNO2	**FIU_0691**	10.44	**264.13216**	232.1	**16**	182691
						212	**12**	14638
						144	**28**	80617
						132	**20**	35542
						130	**40**	49536
						117	**44**	35650
						116	**48**	33363
						89	**60**	26384
						77.1	**60**	15587
						69.1	**24**	10189
	methyl-1-(cyclohexylmethyl)-1H-indole-3-Carboxylate	C17H21NO2	**FIU_0692**	11.54	**272.15723**	240.1	**16**	279836
						190	**16**	174692

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						176	**16**	159668
						144	**24**	287596
						132	**20**	75326
						130	**36**	70111
						117	**44**	74110
						116	**48**	83945
						97.1	**20**	61919
						55.1	**36**	398673
	methyl-1-pentyl-1H-indole-3-Carboxylate	C15H19NO2	**FIU_0693**	11.16	**246.14158**	214.1	**12**	627235
						190	**12**	92756
						146.1	**16**	56859
						144	**24**	276000
						132	**20**	92899
						130	**36**	163052
						117	**36**	98496
						116	**44**	97392
						89	**60**	78764
						77.1	**60**	66247

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	Naphyrone (hydrochloride)	C19H23NO	**FIU_0253**	8.69	**282.2**	211.1	**16**	1840801
						155	**28**	927427
						141	**24**	3502131
						127	**56**	692483
						127	**52**	817886
						115	**60**	709417
						97.1	**56**	183118
						84.1	**40**	553901
						72.1	**32**	159245
						70.1	**20**	327611
						55.1	**56**	206401
	PB-22 5-hydroxyquinoline isomer	C23H22N2O2	**FIU_0602**	11.35	**359.16813**	214.1	**20**	2104504
						191.2	**28**	111
						158	**36**	27308
						144	**44**	881828
						130.1	**48**	18855
						116	**60**	282413
						89.1	**60**	31523

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						71.2	**40**	14579
						55.2	**60**	8309
	PB-22 7-hydroxyquinoline isomer	C23H22N2O2	**FIU_0606**	11.26	**359.16813**	214.1	**16**	501342
						158	**40**	6355
						144	**44**	210202
						130	**48**	3891
						126.1	**56**	55
						116	**60**	69366
						89	**60**	7991
						71.1	**36**	3665
	Phenazepam	C15H10BrClN2O	**FIU_0258**	9.936	**349**	242	**28**	45458
						208.9	**32**	32583
						207.2	**40**	21438
						206.1	**40**	91010
						184	**36**	83160
						179.1	**56**	84484
						130	**52**	22823
						125	**48**	16533
						105	**52**	55953

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						104.1	**60**	56184
	Pyrovalerone	C16H23NO	**FIU_0261**	7.962	**246.2**	175.1	**16**	1636949
						126.1	**28**	1157605
						119	**28**	957692
						105.1	**24**	3033329
						91.1	**48**	1247783
						84.1	**36**	496208
						77.1	**60**	387889
						72.1	**20**	228273
						70.1	**16**	230108
						65.1	**60**	561876
	RCS-8 3-methoxy isomer	C25H29NO2	**FIU_0087**	11.77	**376.2**	254.1	**28**	2784967
						228.1	**16**	1197165
						158	**40**	479159
						144	**48**	1545250
						132.1	**28**	686096
						121.1	**24**	5055167
						91.1	**60**	1083878
						69.1	**52**	1397720

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	55.1	**60**	796933	

Appendix 2. LOD, LOQ, and precision and bias values for all compounds in Mix 4 at three different concentration levels

			Low (5 ppb)		Medium (20 ppb)		High (80 ppb)	
Compound Name	**LOD (ng/mL)**	**LOQ (ng/mL)**	**% CV**	**% Bias**	**% CV**	**% Bias**	**% CV**	**% Bias**
Methiopropamine	0.006	0.017	5.0	-0.6	7.0	-0.9	8.2	0.7
3,4'-methylenedioxy-alpha-pyrrolidinopropiophenone	0.003	0.010	4.4	-1.5	6.0	-3.2	7.2	1.3
3,4-MDMA	0.009	0.027	4.4	0.5	7.4	-2.7	7.0	1.2
2-methylethcathinone	0.010	0.029	5.7	-3.9	8.8	-3.7	10.7	-2.9
6-APB	0.005	0.016	6.2	1.4	6.1	1.7	6.5	1.4
5-methoxy-a-ethyltryptamine	0.000	0.001	5.5	7.9	6.9	2.9	7.6	-0.7
acetyl fentanyl	0.002	0.005	3.9	-3.3	6.0	-3.9	7.3	-1.0
4-methoxy PCP	0.005	0.015	7.2	6.1	4.3	2.3	7.1	-0.8
butyryl fentanyl	0.007	0.022	5.9	-3.1	8.1	-0.7	9.4	0.6
25T2 NBOMe	0.001	0.004	4.0	-1.7	6.0	-0.7	6.9	-0.2
PB-22-N-(4	0.023	0.069	3.3	-4.2	5.4	-1.1	7.0	0.9
AB-PINACA-pentanoic acid metabolite	0.020	0.060	8.7	-7.7	7.4	-5.9	7.9	2.5
25G-NBOMe	0.002	0.005	3.1	-7.3	5.7	-8.3	10.5	4.5
Delorazepam	0.000	0.001	3.0	-5.5	5.4	3.6	6.4	0.8
JWH 203 N - (5-hydroxypentyl) metabolite	0.003	0.009	5.0	-0.5	5.5	-0.1	8.2	2.0
JWH 018 6-hydroxyindole metabolite	0.035	0.106	34.7	-38.5	49.1	-47.8	33.3	-28.1
A-834735	0.006	0.017	5.3	-2.4	6.1	0.3	8.0	-1.0
XLR 12	0.001	0.002	7.3	-22.8	10.6	18.2	15.5	10.1
AM2201 N-(3-chloropentyl) isomer	0.001	0.002	4.4	-9.3	4.1	4.2	5.2	-0.2
BB-22 5-hydroxyisoqui	0.004	0.011	15.6	-29.6	8.8	3.2	6.1	-1.3
UR-144	0.004	0.011	8.6	-40.1	9.9	-4.3	9.6	-2.1

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JWH 387	0.001	0.002	7.9	-44.4	7.5	-2.4	8.0	0.6
(+)-cann	0.003	0.010	12.1	-40.8	6.5	9.3	6.7	0.0
Boldenone Cypionate	0.003	0.010	7.7	-39.1	5.8	-9.0	6.6	9.9
CB-13	0.013	0.038	6.1	-49.7	19.5	-31.3	15.2	16.0

Appendix 3. LOD, LOQ. R^2 values and precision and bias values for all compounds in Mix 5 at three different concentration levels

			Low (5 ppb)		Medium (20 ppb)		High (80 ppb)	
Compound Name	**LOD (ng/mL)**	**LOQ (ng/mL)**	**% CV**	**% Bias**	**% CV**	**% Bias**	**% CV**	**% Bias**
Levamisole	0.014	0.042	11.2	1.1	7.3	-1.8	10.3	1.3
3-fluoromethcathinone	0.005	0.014	10.1	-24.0	4.7	-8.4	4.2	-13.3
Acetyl norfentanyl	0.006	0.018	12.8	-0.2	4.2	-4.9	2.5	0.3
2-methoxymethcathinone	0.010	0.030	10.3	-1.7	6.1	-4.9	13.3	-6.1
Pentedrone metabolite ((±)-Ephedrine stereochemistry)	0.005	0.014	10.7	-1.7	6.5	-6.3	12.5	-5.9
6-APDB	0.029	0.087	12.3	6.8	13.7	-10.0	1.8	2.3
4-methoxy-a-Pyrrolidinobutiophenone	0.015	0.044	8.2	-1.0	6.5	-4.9	11.5	-0.8
a-Ethylaminopentiophenone	0.019	0.057	8.4	-1.7	6.0	-7.4	6.7	-1.8
4-methyl-a-pyrrolidinobutiophenone	0.017	0.050	12.8	2.0	10.6	2.3	6.9	4.3
2C-I	0.011	0.033	11.1	6.8	7.2	-1.6	12.5	2.3
4-methyl-a-ethylaminopentiophenone	0.018	0.053	12.4	2.4	10.2	0.0	5.8	3.8
2C-E	0.019	0.058	11.3	10.8	6.6	4.7	8.2	1.8
4-fluoro PV8	0.025	0.075	7.6	-4.3	9.0	-10.6	5.7	-1.2
25I-NBOMe 4-methoxy isomer	0.007	0.022	10.9	2.2	5.1	-4.2	2.4	-0.4
4-fluoro PV9	0.019	0.058	7.3	-4.7	9.1	-10.7	28.6	-7.1
(±)-ORG 28611	0.032	0.097	10.8	2.5	4.7	-2.0	3.7	-0.3
ADB-PINACA N-(5-hydroxypentyl) metabolite	0.008	0.025	13.1	0.5	4.9	-4.4	3.5	0.4

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JWH 193	0.006	0.018	10.7	1.5	7.4	0.4	4.2	-1.9
PB-22 N-(4-hydroxypentyl) metabolite	0.007	0.022	8.6	4.7	6.0	-1.8	1.2	0.0
5-fluoro ADBICA	0.012	0.036	12.7	2.5	9.2	-2.2	2.9	-0.6
PB-22 3-carboxyindole metabolite	0.007	0.022	9.9	1.1	7.7	-4.4	9.7	-1.0
5-fluoro PB-22 8-hydroxyisoquinoline isomer	0.006	0.017	9.7	4.1	6.4	-1.0	1.7	1.6
ADB-PINACA	0.007	0.020	14.1	3.0	10.0	-1.6	2.9	-0.8
JWH 210 5-hydroxyindole metabolite	0.020	0.060	12.0	8.7	5.5	-6.6	3.3	-0.5
BB-22 6-hydroxyisoquinoline isomer	0.008	0.024	11.2	10.1	5.0	-4.5	4.3	1.6
RCS-8 4-methoxy isomer	0.018	0.053	17.5	14.9	5.3	3.9	7.6	3.0
AKB48 N-(4-fluorobenzyl) analog	0.017	0.051	16.6	21.3	9.2	13.2	5.7	0.3
JWH 210	0.011	0.033	10.5	14.3	6.8	-2.0	5.5	3.4

Appendix 4. LOD, LOQ, and precision and bias values for all compounds in Mix 6 at three different concentration levels (LC-QqQ-MS)

			Low (5 ppb)		Medium (20 ppb)		High (80 ppb)	
Compound Name	**LOD (ng/mL)**	**LOQ (ng/mL)**	**% CV**	**% Bias**	**% CV**	**% Bias**	**% CV**	**% Bias**
Mescaline	0.146	0.441	32.0	7.8	14.7	6.0	17.0	11.1
a-Pyrrolidinobutiothiophenone	0.053	0.159	14.3	-6.0	11.5	-12.6	14.3	1.8
3,4-EDMC	0.058	0.175	14.6	-6.4	10.8	-9.5	18.6	2.6
para-Methoxymethamphetamine	0.054	0.163	12.0	-8.4	18.8	-21.5	26.3	-13.2
2-methoxt Ketamine	0.040	0.122	11.7	-7.9	8.9	0.0	15.1	-5.4
3-Methoxyamphetamine	0.100	0.303	11.9	-20.8	5.1	-13.8	22.3	-18.5
2,5-DMMA	0.043	0.129	13.5	-3.4	8.9	-8.5	8.8	1.6
Pentedrone Metabolite ((+/-)-Psuedoephedrine stereochemistry)	0.038	0.114	11.3	-1.9	6.5	-5.6	14.0	-1.6
(+)-3,4-Methylenedioxy Pyrovalerone	0.035	0.105	7.3	-5.1	5.1	-5.4	8.7	2.2
3-Bromoamphetamine	0.041	0.124	11.8	-5.0	12.4	-6.4	20.8	-11.7
Propylhexdrine	0.040	0.121	11.6	-5.5	18.9	-14.0	22.4	-11.8

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2C-TFM	0.037	0.114	8.8	-3.9	12.8	-9.9	14.8	-3.3
DOI	0.068	0.206	11.4	-5.8	9.5	-6.2	12.0	-3.0
2C-T-7	0.041	0.125	10.0	-2.7	9.2	-5.3	11.1	-0.6
Mepirapim	0.022	0.068	10.4	-2.5	9.9	-1.4	12.5	-6.3
MT-45	0.072	0.218	14.7	-0.4	13.2	14.6	19.3	7.3
AM1248 azepane isomer	0.014	0.044	8.5	-3.6	6.5	-6.0	8.3	0.5
1'-naphthoyl indole	0.051	0.153	11.4	-2.7	8.0	-5.8	11.1	-4.4
(+/-) JWH 073 N-(3-hydroxybutyl) metabolite	0.039	0.118	10.3	-1.1	8.0	-0.4	8.6	0.9
5-fluoro PB-22 7-hydroxyisoquinoline isomer	0.038	0.115	8.6	0.1	5.8	-2.8	9.8	1.8
(+/-) WIN 55,212	0.031	0.093	11.7	1.4	10.4	4.5	8.0	7.8
UR-144 N-(2-hydroxypentyl) metabolite	0.032	0.095	14.0	-0.4	6.2	-0.9	8.1	3.2
PB-22 7-hydroxyisoquinoline isomer	0.042	0.127	13.4	-2.5	4.4	-2.1	5.8	0.4
JWH 251 4-methylphenyl isomer	0.045	0.137	20.1	0.7	12.5	4.8	9.2	1.2

Appendix 5. LOD, LOQ, and precision and bias values for all compounds in Mix 7 at three different concentration levels (LC-QqQ-MS)

			Low (5 ppb)		**Medium (20 ppb)**		**High (80 ppb)**	
Compound Name	**LOD (ng/mL)**	**LOQ (ng/mL)**	**% CV**	**% Bias**	**% CV**	**% Bias**	**% CV**	**% Bias**
Mescaline	0.146	0.441	32.0	7.8	14.7	6.0	17.0	11.1
a-Pyrrolidinobutiothiophenone	0.053	0.159	14.3	-6.0	11.5	-12.6	14.3	1.8
3,4-EDMC	0.058	0.175	14.6	-6.4	10.8	-9.5	18.6	2.6
para-Methoxymethamphetamine	0.054	0.163	12.0	-8.4	18.8	-21.5	26.3	-13.2
2-methoxt Ketamine	0.040	0.122	11.7	-7.9	8.9	0.0	15.1	-5.4
3-Methoxyamphetamine	0.100	0.303	11.9	-20.8	5.1	-13.8	22.3	-18.5
2,5-DMMA	0.043	0.129	13.5	-3.4	8.9	-8.5	8.8	1.6
Pentedrone Metabolite ((+/-)-Psuedoephedrine stereochemistry)	0.038	0.114	11.3	-1.9	6.5	-5.6	14.0	-1.6

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(+)-3,4-Methylenedioxy Pyrovalerone	0.035	0.105	7.3	-5.1	5.1	-5.4	8.7	2.2
3-Bromoamphetamine	0.041	0.124	11.8	-5.0	12.4	-6.4	20.8	-11.7
Propylhexdrine	0.040	0.121	11.6	-5.5	18.9	-14.0	22.4	-11.8
2C-TFM	0.037	0.114	8.8	-3.9	12.8	-9.9	14.8	-3.3
DOI	0.068	0.206	11.4	-5.8	9.5	-6.2	12.0	-3.0
2C-T-7	0.041	0.125	10.0	-2.7	9.2	-5.3	11.1	-0.6
Mepirapim	0.022	0.068	10.4	-2.5	9.9	-1.4	12.5	-6.3
MT-45	0.072	0.218	14.7	-0.4	13.2	14.6	19.3	7.3
AM1248 azepane isomer	0.014	0.044	8.5	-3.6	6.5	-6.0	8.3	0.5
1'-naphthoyl indole	0.051	0.153	11.4	-2.7	8.0	-5.8	11.1	-4.4
(+/-) JWH 073 N-(3-hydroxybutyl) metabolite	0.039	0.118	10.3	-1.1	8.0	-0.4	8.6	0.9
5-fluoro PB-22 7-hydroxyisoquinoline isomer	0.038	0.115	8.6	0.1	5.8	-2.8	9.8	1.8
(+/-) WIN 55,212	0.031	0.093	11.7	1.4	10.4	4.5	8.0	7.8
UR-144 N-(2-hydroxypentyl) metabolite	0.032	0.095	14.0	-0.4	6.2	-0.9	8.1	3.2
PB-22 7-hydroxyisoquinoline isomer	0.042	0.127	13.4	-2.5	4.4	-2.1	5.8	0.4
JWH 251 4-methylphenyl isomer	0.045	0.137	20.1	0.7	12.5	4.8	9.2	1.2

Appendix 6. LOD, LOQ, and precision and bias values for all compounds in Mix 7 at three different concentration levels (LC-QqQ-MS)

			Low (5 ppb)		Medium (20 ppb)		High (80 ppb)	
Compound Name	**LOD (ng/mL)**	**LOQ (ng/mL)**	**% CV**	**% Bias**	**% CV**	**% Bias**	**% CV**	**% Bias**
HMA	0.027	0.080	7.3	4.5	6.2	5.9	1.6	-1.3
MBZP	0.023	0.069	5.7	-2.9	6.0	-2.0	1.6	-1.2
Cathine	0.060	0.181	18.7	7.4	5.2	7.7	10.6	-5.6
N-methyl-2-AI	0.246	0.744	23.9	27.1	14.8	10.4	17.8	0.7
Methylenedioxy Provalerone Metabolite 2	0.109	0.332	3.2	-0.5	6.9	-0.1	2.7	-0.1
Mephedrone	0.065	0.197	7.8	-1.9	6.2	2.4	1.8	-2.7

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4-Methoxyamphetamine	1.372	4.157	17.3	-7.7	8.1	-0.8	2.5	-1.9
2C-T	0.026	0.078	6.5	2.6	5.7	5.7	2.3	-1.2
3,4-Dimethylethcathinone	0.033	0.099	2.8	4.1	6.7	3.4	2.5	-1.4
3C-P	0.046	0.139	7.0	4.1	4.6	6.3	2.2	-0.8
2,3-Dichlorophenylpiperazine	0.297	0.900	4.6	-2.8	5.3	-0.9	1.4	-0.9
25H-NBOMe	0.016	0.049	5.9	1.9	5.1	4.8	1.4	-0.9
NRG-3	0.211	0.639	4.0	-1.5	6.3	3.4	4.5	-4.5
Diclofensine	0.056	0.170	54.7	31.8	19.0	9.1	5.6	-2.4
5-fluoro NNEI	0.107	0.324	4.9	-13.7	6.1	4.2	3.9	0.7
FUB-PB-22	0.071	0.215	4.2	-5.6	4.8	10.9	2.5	-0.3
AB-CHMINACA	0.110	0.334	9.0	-14.4	6.1	13.2	3.8	1.3
AKB48 N-pentanoic acid metabolite	0.047	0.143	6.0	-8.6	4.9	7.8	4.2	0.0
JWH 251 3-methylphenyl isomer	0.100	0.303	4.2	-1.7	6.4	3.2	3.2	-0.2
UR-144 N-(2-chloropentyl) analog	0.020	0.062	8.5	-30.2	8.1	12.9	6.0	-0.9
BB-22 4-hydroxyquinoline isomer	0.023	0.069	9.3	-46.7	14.6	-0.6	16.7	0.9
Delta 9 THC	0.425	1.289	10.9	-46.6	9.6	-9.4	5.2	4.5
JWH 018 2-hydroxyindole metabolite	0.083	0.251	20.5	-29.0	8.4	31.4	6.7	-0.7

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PUBLICATIONS AND PRESENTATIONS

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Kimble A., Arroyo, L, DeCaprio, A.P. (2015) *Triggered MRM Database for the Comprehensive Detection of Novel Psychoactive Substances by LC-QqQ-MS.* Society of Forensic Toxicologists, October 2015¸ Atlanta, GA.

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