

# Identification of Bath Salts by SPE and GC-MS

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## Key Words

- Bath salts
- TraceGOLD TG-5MS
- Servo+ Total-B
- Mephedrone
- Methylenedioxypropylvalerone
- MDPV

## Abstract

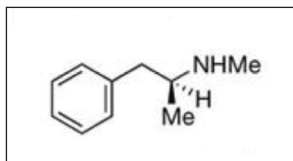
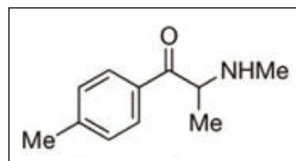
This application note demonstrates a GC-MS method for identifying two compounds commonly found in “bath salts”. An extraction procedure for these compounds was optimized on a Thermo Scientific Servo+ Total B SPE cartridge. A readily available bath salt was analyzed by GC-MS using a Thermo Scientific TraceGOLD TG-5MS column and the active compound along with concentration was determined.

## Introduction

Based on recent news articles and news media attention the U.S. is experiencing a new drug epidemic related to “bath salts.” -All over the U.S., bath salts are being sold with names like “ivory wave,” “white lightning” and “hurricane charlie.” But these particular bath salts are not scented for in-home bath use to soak or relax in – these so-called bath salts are intended to be inhaled, smoked or injected – and users are getting high off of them.

The Drug Enforcement Administration currently do not regulate these substances, but they are under federal scrutiny, as the effects of these salts are comparable to methamphetamine abuse, according to poison control centers and other law enforcement agencies. The DEA is starting a campaign to add bath salts as a Schedule I drug to further regulate its use.

The powders often contain mephedrone and methylenedioxypropylvalerone, also known as MDPV, and can cause hallucinations, paranoia, rapid heart rates and suicidal thoughts, authorities say. The chemicals are in products sold legally at convenience stores and on the Internet as bath salts and even plant foods. However, they aren't necessarily being used for the purposes on the label. As seen below mephedrone is chemically very similar to methamphetamine. Mephedrone (4-methylmethcathinone) is one of a number of synthetic chemical compounds similar to the naturally occurring substance cathinone. Cathinone is the active component of the plant khat, which is chewed in some countries — notably Somalia and Yemen — for its stimulant properties.



Methylenedioxypropylvalerone (MDPV) is a psychoactive drug used as stimulants or weight-loss aids.

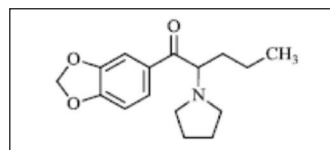


It is commonly known as magic, super coke, cloud 9, ivory wave, peevee, ivory snow, ocean magic, vanilla sky, white dove, white knight, white lightning and many others. Others call it as “fake cocaine” or “complete crank.”

The core chemical structure of mephedrone identifies it as a phenethylamine, and is related in chemical structure to methcathinone differing only by a methyl group (CH<sub>3</sub>) on the ring.

MDPV was reported to have been sold as a drug alternative in the U.S. in 2010 although it has not been clearly said as legal or illegal. In some European countries like the Germany and France, MDPV has long been available over the counter but declined when danger of misuse and dependency were known.

MDPV is a yellowish-white, fine-grained, odorless powder said to have the same effects with cocaine and amphetamine, lasting to 3-8 hours depending on the ingested dosage. It can be taken orally or by injection, smoking, or snorting.



MDPV (1-(1,3-benzodioxol-5-yl)-2-(1-pyrrolidinyl)-1-pentanone; Chemical Abstract Service Number 687603-66-3) is related in chemical structure to schedule I hallucinogenic substances (MDMA, MDEA) and to schedule I stimulants (cathinone, methcathinone). Its molecular formula is C<sub>16</sub>H<sub>21</sub>NO<sub>3</sub> and its molecular weight is 275 g/mol.

## Experimental details

Sample Handling Equipment	Part Number
Liquid handling hardware: eVol with 5 $\mu$ L, 50 $\mu$ L and 500 $\mu$ L	66002-024
SPE hardware: Universal Vacuum Manifold	60104-230
SPE cartridges / consumables:	
Servo+ Total-B 30mg/1 mL	60110-301C
Servo+ Total-B 60mg/3 mL	60110-303C
Vials and closures: 2 mL clear screw top vials	60180-599

### Sample Preparation

Compounds:	Mephedrone, MDPV
Part Number:	60110-301C
Phase:	Servo+ Total B
Volume:	1 mL
Bed Weight:	30 mg
Conditioning:	1 mL of methanol followed by 1 mL of DI water
Application:	Load at 1 to 2 mL/min
Washing:	1 mL of MeOH:H <sub>2</sub> O:NH <sub>4</sub> OH (50:48:2)
Elution:	1 mL of MeOH:NH <sub>4</sub> OH (95:5)

### Separation Conditions

Instrumentation:	Thermo Scientific Trace GC Ultra
GC Column:	TG-5MS, 30 m x 0.25 mm x 0.25 $\mu$ m (26098-1420)
Oven Temp:	180 °C (5 min hold) to 240 °C (1 min hold) at 10 °C/min
Carrier:	Helium (1.5 mL/min)
Injector:	250 °C split (60 mL/min, 40:1 split ratio)
Detector:	FID 300 °C (for SPE elution profiles)
Sample injected:	0.5 $\mu$ L

## Results

Both a sample extraction method and GC-MS method were developed for the identification of mephedrone and MDPV, two common components in readily available “bath salts”. The extraction procedure developed in this application note was the result of SPE optimization. This optimization was done in order to maximize the recovery of each analyte.

A Servo+ Total B SPE cartridge (30 mg/1 mL) was conditioned with 1 mL of methanol followed by 1 mL of water. A 1 mL aliquot of water was spiked with 20  $\mu$ g of both mephedrone and MDPV and loaded on to the Servo+ Total B SPE cartridge. The eluant from the loading was collected. Washes with increasing strengths of methanol in water/ammonium hydroxide (95:5) were applied, starting from 10% methanol/90 % water/NH<sub>4</sub>OH (95:5) and increasing in methanol content by 10% each. Finally four 1 mL aliquots of 95 % methanol / 5 % NH<sub>4</sub>OH were used to wash the SPE cartridge. Each eluant from the washings was collected and analyzed by GC/FID. By plotting the area/detector response for each of these eluants generates an elution profile. This data is shown in Figure 1. These results show an optimum wash profile of 50 % methanol/50 % water/NH<sub>4</sub>OH (95:5) before mephedrone starts to elute.

To demonstrate the robustness of the method and the Servo+ Total B SPE cartridges, percent recoveries of the mephedrone and MDPV were calculated. Using 60 mg/3 mL tubes, 2 mL of dilute urine (1:1 dilution) spiked with 20  $\mu$ g of mephedrone, MDPV, and an internal standard (naphthalene) was loaded onto the SPE devices. Nine SPE

### MS Conditions (if applicable)

Instrumentation:	Thermo Scientific Trace GC Ultra and DSQII
Transfer line temperature:	270 °C
Ionization conditions:	EI
Emission current:	100.00
Electron energy:	-70.00
Filament delay:	4 minutes
Ion Range: Scan	48 - 600
Ions: SIM:	48 m/z, 58 m/z, 126 m/z
Scan time:	Scan 3 sec, SIM 0.36 sec

### Solutions

Mephedrone (5 mg) purchased from Sigma Aldrich. MDPV (5 mg) purchased from Cayman Laboratories. Standards were prepared in house.

### Data Processing

Software:	Xcalibur
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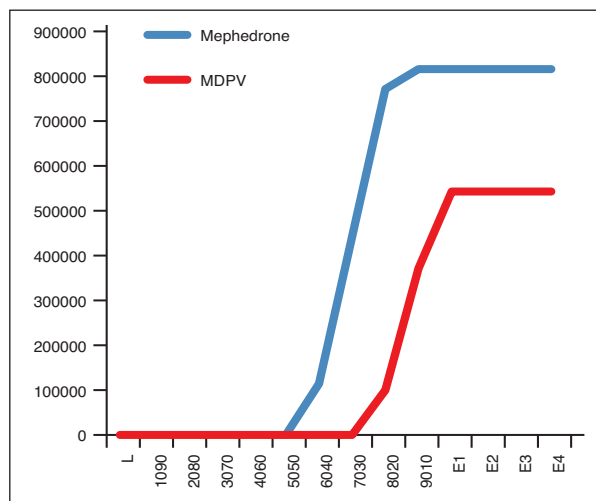


Figure 1: Elution Profiles of Mephedrone and MDPV

tubes were loaded in this manner. All of the tubes were then washed with 2 mL of 50 % methanol/ 50 % water/NH<sub>4</sub>OH (95:5) and then eluted with 1 mL of 95 % methanol/5 % NH<sub>4</sub>OH. These samples, along with a standard prepared with 20  $\mu$ g of mephedrone, MDPV, and naphthalene were analyzed by GC/FID. Using the results from these analysis percent recoveries were calculated resulting, on average with a percent recovery of 87 % for mephedrone and a 99 % for MDPV.

Using GC-MS, an instrument calibration was performed for both mephedrone and MDPV. Amphetamine-d<sub>6</sub> was used as an internal standard. The mass spec was used in SIM mode, monitoring for the parent ions of

amphetamine-d6 (48), mephedrone (58) and MDPV (126). Using 5 different calibration levels and triple injections, results are shown in Table 1. Figures 2 and 3 show the linear correlation for the calibration curve.

Compound	Level	%RSD
Mephedrone	5 µg/mL	1.2 %
	10 µg/mL	0.8 %
	20 µg/mL	2.5 %
	30 µg/mL	0.3 %
	40 µg/mL	2.9 %
MDPV	5 µg/mL	2.4 %
	10 µg/mL	2.6 %
	20 µg/mL	5.7 %
	30 µg/mL	7.4 %
	40 µg/mL	4.1 %

Table 1: Calibration Results showing %RSD for analytes of interest

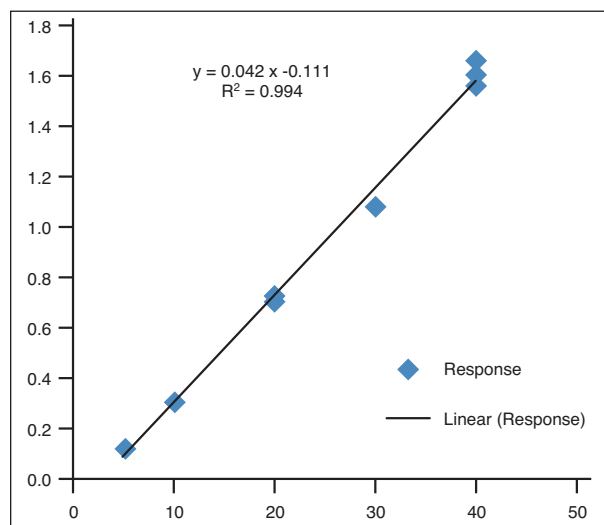


Figure 2: Mephedrone Calibration Curve

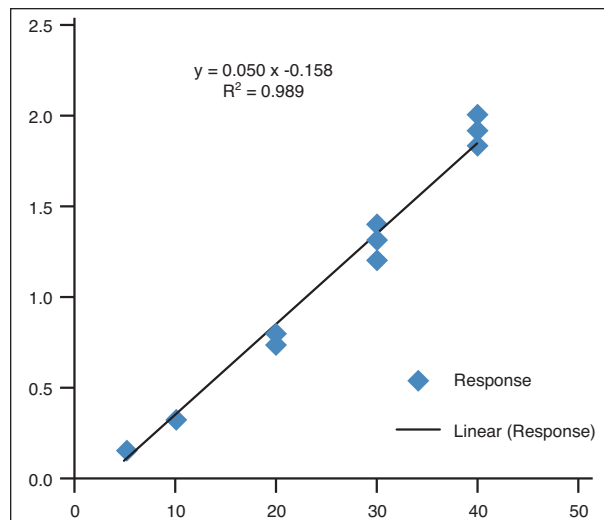


Figure 3: Methylendioxypropylvalerone Calibration Curve

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A bath salt available from a local retail location was purchased for identification of its components. The bath salt was labelled as “white rush”. A 10mg sample was dissolved in 10 mL of methanol using a Class A volumetric flask. The sample was sonicated for 10 minutes and a 25 µl aliquot was transferred to a glass vial, spiked with 20 µg of the internal standard (amphetamine-d6) and diluted 40:1 with methanol. This sample was analyzed via GC-MS in SIM mode. The resulting chromatogram is shown in Figure 4. The only active component found in the White Rush Bath Salt was MDPV at a concentration of 446 mg/mL, or 45 % purity.

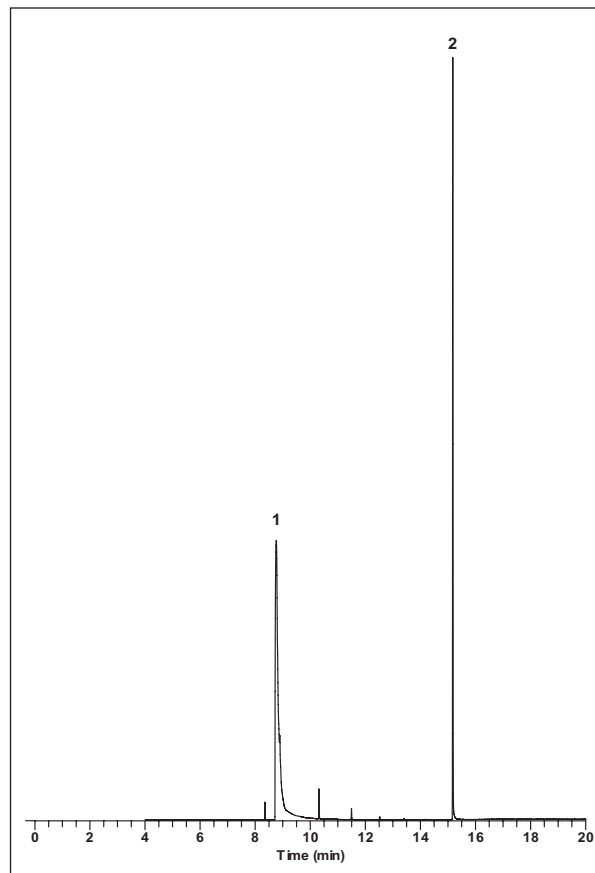


Figure 4: White Rush Bath Salt. 1. I.S. (amphetamine-d6), 2. MDPV

## Conclusions

In this application note an SPE method for the extraction of mephedrone and MDPV from urine was developed. The optimized extraction for these compounds was achieved on a Servo+ Total B SPE column. A GC-MS method was also developed for the identification of active compounds in bath salts. The active compounds of bath salts were identified by GC-MS and quantified by a standard calibration. The TraceGOLD TG-5MS capillary columns provided the inertness and low bleed characteristics necessary for quantifying these compounds.

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