



Bath Salts in Urine – Supel™-Select SCX SPE and Ascentis® Express HILIC

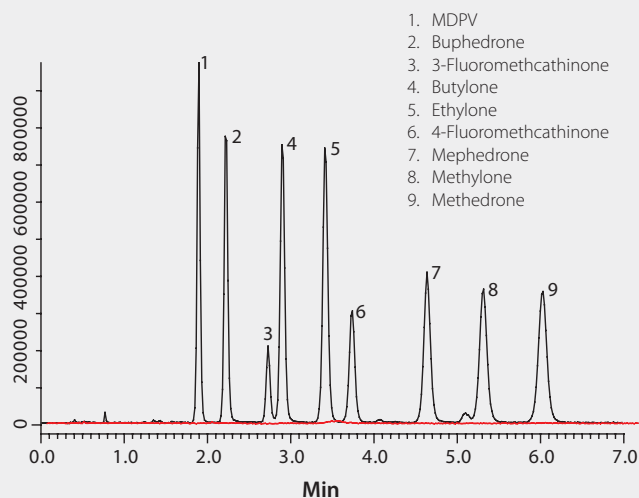
LC-MS Analysis of Illicit Bath Salts in Urine on Ascentis Express HILIC after Solid Phase Extraction (SPE) on Supel-Select SCX

This application demonstrates the analysis of bath salts extracted from human urine samples using polymeric solid phase extraction (SPE) sample preparation followed by hydrophilic interaction liquid chromatography (HILIC) analysis with TOF-MS detection. HILIC conditions on the Ascentis Express HILIC (Si) phase are used for fast, high-resolution separation of nine synthetic bath salts. Recoveries greater than 65% were observed for all analytes except MDPV (43.7%).

The Figure illustrates the detection of bath salts in the spiked urine sample after SPE sample cleanup. Notice there are no interfering peaks in the chromatogram, demonstrating the effectiveness of the SPE sample cleanup. As a reference, the Figure also depicts the monitored bath salt ions for the blank urine sample. Again, there are no interfering peaks that could cause irregularities in analyte detection.

Monitored ions of spiked urine (black) and blank urine (red) sample after SPE cleanup.

sample/matrix: 1 mL urine spiked to 100 ng/mL of bath salt mixture (standards from Cerilliant)
SPE tube: Supel-Select SCX, 30 mg/1 mL (54240-U)
conditioning: 1 mL 1% formic acid acetonitrile, then 1 mL water
sample addition: 1 mL spiked urine
washing: 1 mL water, 1 mL 1% formic acid in acetonitrile, 1 mL water
elution: 2 mL 10% ammonium hydroxide in acetonitrile
eluate post-treatment: thoroughly mix via vortex agitation, evaporate 1 mL aliquot to dryness, reconstitute in 100 µL water:methanol
column: Ascentis Express HILIC (Si), 10 cm X 2.1 mm, 2.7 µm (53939-U)
mobile phase: (A) 5 mM ammonium formate acetonitrile; (B) 5 mM ammonium formate water; (98:2, A:B) (Solvents LC-MS Ultra CHROMASOLV® grade)
flow rate: 0.6 mL/min
pressure: 127 bar
column temp: 35 °C
detector: MS, ESI+, 100-1000 m/z
injection: 1 µL





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Following is a selection of products that can be used in this application. It is meant for research and forensic applications and not for medical or diagnostic purposes.

Cerilliant Certified Reference Materials*

| Cat. No. | Description |
|---------------------|--|
| Parent drugs | |
| B-047 | Buphedrone HCl, 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| B-045 | Butylone HCl, 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| E-071 | Ethylone HCl, 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| F-016 | 3-Fluoromethcathinone HCl, 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| F-015 | 4-Fluoromethcathinone HCl (Flephedrone HCl), 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| M-144 | MDAI HCl, 1.0 mg/mL (as free base) in acetonitrile:water (90:10) with 5% 1 M HCl, 1 mL/ampoule |
| M-154 | MDPBP HCl (3,4'-Methylenedioxy- α -pyrrolidinobutiophenone HCl), 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| M-138 | Mephedrone HCl, 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| M-147 | Methedrone HCl (4-Methoxymethcathinone HCl), 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| M-151 | 3-Methoxymethcathinone HCl, 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| M-146 | 3,4-Methylenedioxypropylvalerone HCl (MDPV), 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| M-155 | 4-Methylethcathinone HCl, 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| M-140 | Methylone HCl, 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| N-067 | Naphyrone HCl, 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| P-087 | Pentredone HCl, 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| P-086 | Pentylone HCl, 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| P-081 | Pyrovalerone HCl, 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| Metabolites | |
| M-158 | (\pm)-4-Methylephedrine HCl (Mephedrone Metabolite), 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |
| B-050 | Buphedrone Ephedrine Metabolite HCl, 1.0 mg/mL (as free base) in methanol, 1 mL/ampoule |

| Cat. No. | Description |
|---------------------------|--|
| Internal Standards | |
| B-046 | Butylone-D3 HCl, 100 μ g/mL (as free base) in methanol, 1 mL/ampoule |
| E-072 | Ethylone-D5 HCl, 100 μ g/mL (as free base) in methanol, 1 mL/ampoule |
| M-139 | Mephedrone-D3 HCl, 100 μ g/mL (as free base) in methanol, 1 mL/ampoule |
| M-150 | 3,4-Methylenedioxypropylvalerone-D8 HCl (MDPV-D8 HCl), 100 μ g/mL (as free base) in methanol, 1 mL/ampoule |
| M-141 | Methylone-D3 HCl, 100 μ g/mL (as free base) in methanol, 1 mL/ampoule |

*New products are continually being introduced. Please consult the Cerilliant website (cerilliant.com) for the up to date listing of reference standards.

For a complete listing of Cerilliant standards and to order, visit cerilliant.com

SPE Tubes and Accessories

| Cat. No. | Description |
|----------|--|
| 54240-U | Supel™-Select SCX, 30 mg/1 mL |
| 57080-U | Visi-1 Single SPE Tube Processor (provides precise flow control through a single 1, 3 or 6 mL SPE tubes) |
| 57044 | Visiprep™-DL 12-Port Vacuum Manifold |
| 57265 | Visiprep-DL 24-Port Vacuum Manifold |

HPLC Columns and Guard Columns

| Cat. No. | Description |
|----------|---|
| 53569-U | Ascentis® Express F5, 10 cm x 2.1 mm I.D., 2.7 μ m |
| 53594-U | Ascentis Express F5, 2.1 mm I.D., 2.7 μ m (pack of 3) |
| 53500-U | Universal Guard Holder |

LC-MS Ultra CHROMASOLV® Solvents and Additives

| Cat. No. | Description |
|----------|---|
| 14261 | Acetonitrile, LC-MS Ultra CHROMASOLV, 1 L, 2 L |
| 14262 | Methanol, LC-MS Ultra CHROMASOLV, 1 L, 2 L |
| 14263 | Water, LC-MS Ultra CHROMASOLV, 1 L, 2 L |
| 14264 | Trifluoroacetic acid, LC-MS Ultra eluent additive, 1 mL, 2 mL |
| 14265 | Formic acid, LC-MS Ultra eluent additive, 1 mL, 2 mL |
| 14266 | Ammonium formate, LC-MS Ultra eluent additive, 25 g |
| 14267 | Ammonium acetate, LC-MS Ultra eluent additive, 25 g |

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