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

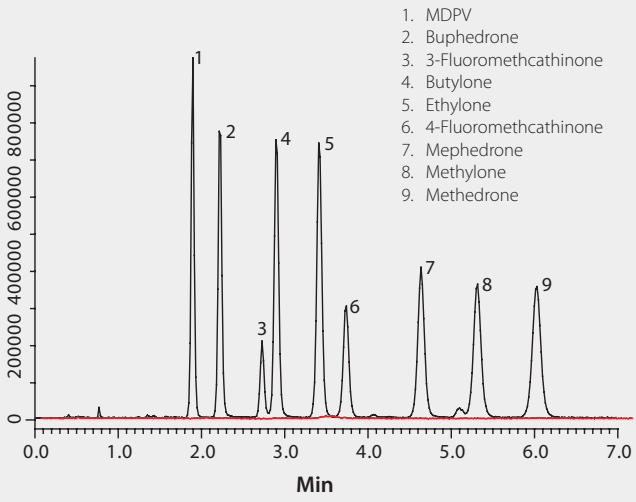
LC-MS Analysis of Illicit Bath Salts in Urine on Ascentis Express HILIC after Solid Phase Extraction (SPE) on Supel-Select SCX

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



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

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-Methylenedioxypyrovalerone 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	Pentedrone 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	(±)-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 µg/mL (as free base) in methanol, 1 mL/ampoule
E-072	Ethylone-D5 HCl, 100 µg/mL (as free base) in methanol, 1 mL/ampoule
M-139	Mephedrone-D3 HCl, 100 µg/mL (as free base) in methanol, 1 mL/ampoule
M-150	3,4-Methylenedioxypyrovalerone-D8 HCl (MDPV-D8 HCl), 100 µg/mL (as free base) in methanol, 1 mL/ampoule
M-141	Methylone-D3 HCl, 100 µg/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 µm
53594-U	Ascentis Express F5, 2.1 mm I.D., 2.7 µm (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	Trifluroacetic 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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