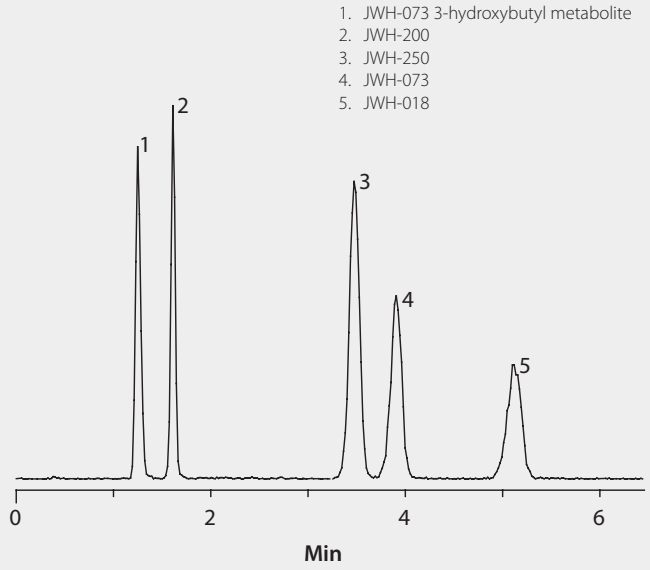
Spice Cannabinoids in Plasma – HybridSPE®-Phospholipid and Ascentis® Express F5

LC-MS/MS Analysis of Synthetic (Spice) Cannabinoids from Plasma on Ascentis Express F5 after SPE using HybridSPE-Phospholipid

Synthetic cannabinoids (Spice) are a relatively new type of designer drug used as a pseudo-legal means to get a cannabis-type high. New synthetic cannabinoids are continually being introduced as suppliers tweak the molecular structures. The ability to rapidly and reliably identify the continually changing population of these compounds in the blood or urine of suspected users is a significant analytical challenge facing forensic chemists. A four prong approach using column selectivity, high purity solvents, effective sample prep, and reference standards, was used to develop a method to rapidly isolate and identify Spice cannabinoids from plasma. The Ascentis® Express F5 column gave the necessary resolution, and the LC-MS CHROMASOLV® solvents and additives gave adduct-free response for maximum sensitivity. Sample prep employing the HybridSPE®-Phospholipid was rapid and effective and the Cerilliant reference standards enabled confident identification.

LC-MS/MS Analysis of Spice Compounds from Plasma on Ascentis Express F5 after SPE using HybridSPE-Phospholipid

sample/matrix:	rabbit plasma, unfiltered K2-EDTA spiked with Spice cannabinoids (5 ng/mL each. Standards from Cerilliant)
SPE:	HybridSPE-Phospholipid, 96-well plate (575656-U)
sample addition:	to each well add 100 µL plasma, followed by a 300 µL of 1% formic acid in acetonitrile, agitate on orbital shaker for 2 minutes
elution:	attach collection plate and apply vacuum at 10" Hg for 4 minutes
column:	Ascentis Express F5, 5 cm x 2.1 mm, 2.7 µm (53567-U)
mobile phase:	(A) 10 mM ammonium formate in water, pH 6.8 (unadjusted); (B) acetonitrile; (50:50; A:B) Solvents LC-MS Ultra CHROMASOLV grade.
flow rate:	0.3 mL/min
temp.:	35 °C
pressure (column):	1296 psi (89.4 bar)
detector:	MS, ESI(+), MRM, m/z 344/155 (JWH-073 metabolite), 385/155 (JWH-200), 336/121 (JWH-250), 328/155 (JWH-073), and 342/155 (JWH-018)
injection:	2 µL
system:	Agilent 1100 HPLC, 3200 QTrap (AB/Sciex)



Spice Cannabinoids in Plasma – HybridSPE®-Phospholipid and Ascentis® Express F5

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 and mixes	
S-038	Spice Cannabinoid Mix (JWH-250; JWH-200; HU-211; (±)-CP 47,497; and (±)-CP 47,497 C8 homologue), 100 µg/mL of each component in acetonitrile, 1 mL/ampoule
S-041	Spice Cannabinoid Mix 2 (JWH-019, JWH-122, JWH-081, AM-2201), 100 µg/mL of each component in acetonitrile, 1 mL/ampoule
S-047	Spice Cannabinoid Mix 3 (RCS-4, RCS-8, JWH-015, JWH-203, JWH-210, AM-2233), 100 µg/mL of each component in acetonitrile, 1 mL/ampoule
S-027	JWH-073, 100 µg/mL in acetonitrile, 1 mL/ampoule
S-025	JWH-018, 100 µg/mL in methanol, 1 mL/ampoule
S-024	HU-210, 100 µg/mL in methanol, 1 mL/ampoule
Metabolites (-OH)	
S-037	JWH-073 3-Hydroxybutyl metabolite, 100 µg/mL in methanol, 1 mL/ampoule
S-035	JWH-018 4-Hydroxypentyl metabolite, 100 µg/mL in methanol, 1 mL/ampoule
S-043	JWH-019 6-Hydroxyhexyl metabolite, 100 µg/mL in methanol, 1 mL/ampoule
S-044	JWH-019 5-Hydroxyhexyl metabolite, 100 µg/mL in methanol, 1 mL/ampoule
S-045	JWH-250 5-Hydroxypentyl metabolite, 100 µg/mL in methanol, 1 mL/ampoule
Metabolites (-COOH)	
S-033	JWH-018 5-Pentanoic acid metabolite, 100 µg/mL in methanol, 1 mL/ampoule
S-036	JWH-073 4-Butanoic acid metabolite, 100 µg/mL in methanol, 1 mL/ampoule
Internal Standards	
S-039	JWH-018 4-Hydroxypentyl metabolite-D_5 (indole-D_5), 100 µg/mL in methanol, 1 mL/ampoule
S-040	JWH-073 3-Hydroxybutyl metabolite-D_5 (indole-D_5), 100 µg/mL 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**

HybridSPE-Phospholipid Plates, Tubes and Accessories

Cat. No.	Description
52813-U	HybridSPE-Phospholipid 96-Well Essentials Kit. Includes: HybridSPE-Phospholipid 96-well plate (575656-U), cap mat (575655-U), and collection plate (575653-U).
575656-U	HybridSPE-Phospholipid 96-well plate (50 mg/2 mL per well), pk of 1 plate
575657-U	HybridSPE-Phospholipid 96-well plate (50 mg/2 mL per well), pk of 20 plates
52794-U	HybridSPE-Phospholipid 96-well plate (15 mg/0.8 mL per well), pk of 1 plate
52798-U	HybridSPE-Phospholipid 96-well plate (15 mg/0.8 mL per well), pk of 20 plates
55269-U	HybridSPE-Phospholipid Ultra Cartridge for on-line phospholipid and protein removal (30 mg/1 mL), pk of 100 cartridges
575654-U	Disposable Reservoir / Waste Tray configured for collecting eluate waste during PlatePrep SPE processing, PVC, pk of 25
575651-U	SPE 96-Deep Square Well Collection Plate, well volume 0.35 mL, polypropylene, pk of 50
575652-U	SPE 96-Deep Square Well Collection Plate, well volume 1 mL, polypropylene, pk of 50
575653-U	SPE 96-Deep Square Well Collection Plate, well volume 2 mL, polypropylene, pk of 50
575655-U	96 Square Well Pierceable Cap Mats configured for sealing Discovery® SPE and square well collection plates, pk of 50
R9259	Texan™ reagent reservoir for multichannel pipettes without lid, non-sterile, pack of 10 × 10
Z654779	IKA® VORTEX 3, vortex mixer (230 V)
Z654760	IKA VORTEX 3, vortex mixer (115 V)

HPLC Columns and Guard Columns

Cat. No.	Description
53569-U	Ascentis Express F5, 10 cm x 2.1 mm I.D., 2.7 µm
53567-U	Ascentis Express F5, 5 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

Enabling Science to Improve the Quality of Life

Order/Customer Service (800) 325-3010 • Fax (800) 325-5052
Technical Service (800) 325-5832 • sigma-aldrich.com/techservice
Development/Custom Manufacturing Inquiries SAFC® (800) 244-1173
Safety-related Information sigma-aldrich.com/safetycenter

World Headquarters
3050 Spruce St.
St. Louis, MO 63103
(314) 771-5765
sigma-aldrich.com

©2012 Sigma-Aldrich Co. LLC. All rights reserved. Cerilliant, SAFC, SIGMA-ALDRICH and SUPELCO are trademarks of Sigma-Aldrich Co. LLC, registered in the US and other countries. Solutions within is a trademark of Sigma-Aldrich Co. LLC. HybridSPE, Discovery and Ascentis are registered trademarks of Sigma-Aldrich Co. LLC. CHROMASOLV is a registered trademark of Sigma-Aldrich Laborchemikalien GmbH. Texan is a trademark of Excel Scientific, Inc. IKA is a registered trademark of IKA-Werke GmbH & Co. KG.

PCL
11621-513940
1102