



Synthetic Cannabinoids “Spice” Drugs

Confirmations LC-MS/MS and GC-MS using CLEAN SCREEN[®] THC SPE Column CSTHC206

April 7, 2011

1. PREPARE SAMPLE

To 1 mL of 100 mM phosphate buffer (pH= 6) add internal standard.
Add 1-2 mL of urine.

Add 3 mL of 100 phosphate buffer (pH= 6). Mix/vortex.

Sample pH should be 6.0 ± 0.5 .

Adjust pH accordingly with 100 mM monobasic or dibasic sodium phosphate.

Mix/vortex.

Centrifuge as appropriate.

2. CONDITION CLEAN SCREEN[®] EXTRACTION COLUMN

1 x 3 mL CH₃OH

1 x 3 mL DI H₂O.

1 x 1 mL 100 mM phosphate buffer (pH= 6).

Note: aspirate at < 3 inches Hg to prevent sorbent drying out.

3. APPLY SAMPLE:

Load sample at 1-2 mL / minute.

4. WASH COLUMN:

1 x 3 mL DI H₂O

1 x 3 mL of 100 mM phosphate buffer containing 20% acetonitrile

Dry column (5 minutes at > 10 inches Hg).

5. ELUTE SPICE

2 x 3 mL ethyl acetate contain 10 % CH₃OH.

Collect eluate at 1-2 mL /minute.

6. EVAPORATION:

Evaporate eluate under a gentle stream of nitrogen < 40°C.

7. RECONSTITUTE sample in 50 µL of mobile phase for LC-MS/MS

Inject 10µL.

Dissolve residue in 50 µL of ethyl acetate and MSTFA for GC-MS

Heat at 70°C for 2 hours

Cool and inject 1 µL onto GC-MS

INSTRUMENT CONDITIONS (LC-MS/MS):

Column: 50 x 2.1 mm (5,µm) Biphenyl (Restek)

Mobile phase:

<u>Time/ min</u>	<u>% Acetonitrile (containing 0.1% formic acid/2 mM ammonium formate)</u>
0	30
8	40
15	40
16	90
20	30

Flowrate: 0.6 mL/minute

Column Temperature: 40°C.

Detector: API 4000 Qtrap MS/MS.

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