Determination of THC in Marijuana Samples Using Solid Phase Extraction and LC-MC/MS



UCT Part Numbers

CSTHC206 Clean Screen® THC 200 mg, 6 mL

SLDA100ID21-5UM Selectra® DA Column 100 x 2.1 mm, 5 µm **SLDAGDC21-5UM** Selectra® DA Guard Column 10 x 2.1 mm, 5 μm

SPHPHO7001-5 0.1M Phosphate Buffer pH 7

Procedure:

1. Extraction

- a) Add 100 mg of marijuana sample into a clean glass sample tube
- b) Add 5 mL of methanol and cap
- c) Sonicate for approximately 60 minutes at room temperature
- d) Centrifuge for 10 minutes at 3000 rpm
- e) Aliquot 0.5 to 1 mL of methanol extract into a clean glass sample tube
- f) Add internal standard and mix
- g) Add 4 mL of 0.1 M phosphate buffer (pH 7) and mix

2. Condition Clean Screen® THC Extraction Column

- a) 1 x 3 mL CH₃OH
- b) 1 x 3 mL D.I. H₂O
- c) 1 x 1 mL 0.1 M phosphate buffer (pH = 7.0)

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

3. Apply Sample

a) Load sample at 1-2 mL / minute.

4. Wash Column

- a) 1 x 3 mL D.I. H₂O
- b) 1 x 3 mL 0.1 M phosphate buffer (pH 7.0)
- c) Dry column (5 minutes at > 10 inches Hg)

5. Elute THC

- a) 1 x 3 mL hexane/ethyl acetate/acetic acid (49:49:2)
- b) Collect eluate at 1 to 2 mL / minute.

6. Dry Eluate

- a) Evaporate to dryness under nitrogen < 40°C
- b) Reconstitute in 100 µL of mobile phase



Instrument Conditions					
Column	Selectra® DA 100 x 2.1 mm, 5 μm				
Column Temperature	40 °C				
Mobile phase	CH ₃ OH w/ 0.1% formic acid: DI H ₂ O w/ 0.1% formic acid (75:25)				
Flow rate	0.5 mL/ minute				
Detector	API 4000 MS/MS				

MS Parameters					
Polarity	ESI+				
Spray Voltage V	4500 V				
Source Temperature	650 °C				
Curtain Gas	10				
Gas 1	40				
Gas 2	40				
CAD Gas	Medium				
Dwell Time	150 ms				

Compound	Q1	Q3	Time/ms	DP/volts	EP/volts	CXP/volts	CE/volts
THC (1)	315.2	193.2	46	4	18.8	29	4
THC (2)	315.2	123.1	46	4	18.8	45	4
*THC-D3 (1)	318.2	196.2	46	4	18.8	29	4
*THC-D3 (2)	318.2	123.2	46	4	18.8	43	4

Note: Q1=Precursor Ion; Q3=Product Ion; DP= Declustering Potential; EP= Entrance Potential; CEP= Collision Entrance Potential; CE= Collision Energy; CXP= Collision Exit Potential















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