



SPE EXTRACTION OF THC-DELTA-9-CARBOXY METABOLITE FROM URINE

CLEAN
SCREEN XCEL II

January 5, 2010

130mg Clean Screen Xcel II Column
PART #: CSXCE2106 6 ML - 130 MG CARTRIDGE



1. SAMPLE PREPARATION

Hydrolysis of Urine Sample for THC-delta-9-COOH

To 2 mL urine add appropriate internal standards prepared.

Add 50 μ L of 10 N NaOH. Heat for 15 minutes at 60-70 $^{\circ}$ C

Add 50 μ L 1:1 acetic acid: DI water. (pH should be 7.0+1.0)

Add 200 μ L pH 7.0 0.1M Phosphate buffer)

(The sample is ready to be extracted.)

2. APPLYING SAMPLE TO COLUMN

Load sample directly to column without any preconditioning.

Pull sample through at a rate of 1-2 mL/ minute.

Dry column thoroughly under vacuum (10 mm Hg) or positive pressure

(~ 80-100 psi) for 2 minutes.

3. WASH

Wash sample with 1-2 mL of Hexane. (Be sure no sample droplets remain on sides of column.)

Dry column thoroughly under vacuum (10 mm Hg) or positive pressure

(~ 80-100 psi) for a minimum of 10 minutes.



FORENSICS

APPLICATIONS

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NOTE 1: (It is important to dry the column thoroughly before elution to achieve the highest recovery of THC-delta-9-COOH. Any residual moisture will slow down the drying of the elution solvents prior to derivatization for GC/MS analysis. Also, any residual moisture could reduce the reactivity of the derivatization agent resulting in low GC/MS sensitivity.)

4. ELUTION

Elute samples with 1 mL ethyl acetate/ hexane/ acetic acid (49/49/2).

Evaporate fraction to complete dryness under stream of dry air or nitrogen at ~ 35 °C.

GC/MS Analysis

It is recommended to add 50 µL of Ethyl Acetate to 50 µL of derivatization agent and react at ~70 °C for 15 minutes. Inject 1-2 µL of cooled solution in the GC/MS system for analysis.

LC/MS Analysis

Reconstitute in methanol or appropriate mobile phase.

GC/MS Derivatization Ions

<u>Derivatizing Agent</u>	<u>THC-delta-9-COOH (D9 THC-delta-9-COOH)</u>	<u>(Cerilliant Part # T-006) (Cerilliant Part # T-007)</u>
BSTFA	371, 473, 488	(380, 479, 497)
MTBSTFA	413, 515, 572	(422, 524, 581)