



SPE Application Note for THC-COOH from Urine (THC Column)

This method was developed for the extraction of the THC-COOH metabolite for drugs of abuse applications. The analyte is extracted by a non-polar retention mechanism.

EXTRACTION PROCEDURE

ISOLUTE® SPE Column: THC Column: 100 mg/3 mL (Part number 292-0010-B)

Pre-treatment: Add 100 µL of internal standard standard to 3 mL of urine. Hydrolyze with 0.1 mL of NaOH, 10 M. Heat for 15 minutes at 60 C. Cool. Add 0.5 mL of glacial acetic acid.

Solvation: Rinse column with 1 mL of methanol at 2 to 4 mL per/minute.

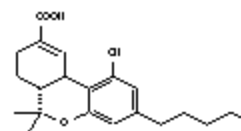
Equilibration: Rinse column with 1 mL of water at 2 to 4 mL/min, followed by 1 mL of 0.01 M HCl at 2 to 4 mL/min.

Sample application: Apply sample at 1.5 mL/min.

Interference elution: Rinse column with 2 mL of acetonitrile/acetone/0.01 M HCl, 15:15:70, v/v at 2 to 4 mL/min. Dry for 30 seconds under -20 psig vacuum or under 20 psig nitrogen.

Analyte elution: Elute sample with 2 mL hexane/ethyl acetate/acetone 50:40:10 v/v. At 1 mL/min. Evaporate to dryness. Derivatize with 0.05 mL BSTFA at 60 C for 15 min. Inject into GC.

Structure As shown.



Structural considerations Due to the presence of a carboxylic acid group, this compound will require hydrolysis prior to the analytical determination.

Matrix considerations The analyte is being extracted from an aqueous matrix of high ionic strength.

Analytical method GC-MS

Reagents 1. 0.01 M HCl. Add 2 mL of concentrated HCl to 200 mL of deionized water in a 250 mL volumetric flask. Dilute to mark with deionized water.

2. Acetonitrile/acetone/0.01 M HCl 15:15:70, v/v. Add 15 mL of

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Last Revised: 29-Nov-05

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ethyl acetate and 15 mL of acetone to a 100 mL volumetric flask. Dilute to mark with 0.01 M HCl.

3. Hexane/ethyl acetate/acetone 50:40:10. Add 40 mL of ethyl acetate and 10 mL of acetone to a 100 mL volumetric flask. Dilute to mark with hexane.

General comments

ISOLUTE column part numbers represent the product configuration of choice for use with a vacuum sample processing station. For 96-well and alternative column configurations compatible with any SPE automation system, please contact Biotage.

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United States and Canada

T: + 1 434 9792319
Toll-Free: +1 800 446 4752
ordermailbox@biotage.com

Sweden

Biotage
T: + 46 18 56 59 00
order@eu.biotage.com

United Kingdom, EIRE

Biotage
T: + 44 1443 811811
eurossales@eu.biotage.com

Japan

Biotage
T: + 81 422 281233
order@biotage.co.jp

