



SPE Application Note for THC-COOH from Urine using THC Column on RapidTrace

This method has been developed specifically for application to the Caliper LifeSciences RapidTrace.

EXTRACTION PROCEDURE

ISOLUTE® SPE Column: THC 100 mg / 1mL, Part number 292-0010-A

Pre-treatment: Add 100 μ L 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: Condition column with 1 mL of methanol at 10 mL/min.

Equilibration: Rinse column with 1 mL of water at 10 mL/min
Rinse column with 1 mL of 0.01 M HCl at 10 mL/min

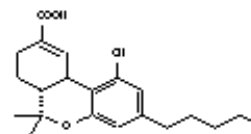
Sample application: Load sample volume of 3.8 at 2 mL/min

Interference elution: Rinse column with 1 mL of water at 6 mL/min.
Purge cannula with 4 mL of water at 30 mL/min.
Rinse column with 1 mL of acetonitrile/acetone/0.01 M HCl, 15:15:70, at 6 mL/min
Dry under 20 psig nitrogen for 1/2 minute.

Analyte elution: Collect sample with 1 mL hexane/ethyl acetate/acetone 50:40:10 at 1.5 mL/min.
Purge cannula with 5 mL of methanol at 30 mL/min.
Purge cannula with 5 mL of deionized water at 30 mL/min.

Evaporate eluate to dryness.
Derivatize with 50 μ L BSTFA at 60 C for 15 min.
Inject into GC.

Structure



Structural considerations Due to the presence of a carboxylic acid group, this compound will require derivatization 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. Add 15 mL of 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.
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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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