

Extraction of THC-COOH from Urine using ISOLUTE® THC Columns and the RapidTrace® SPE Workstation

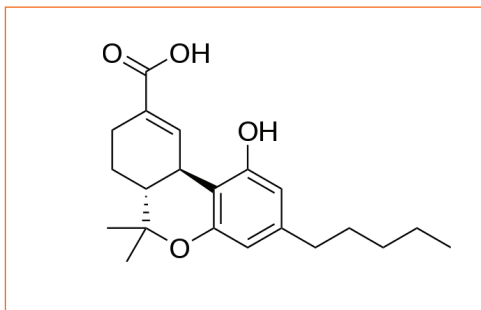


Figure 1. THC-COOH

Introduction

This method has been developed specifically for the RapidTrace® SPE workstation in addition to ISOLUTE THC columns on the 1 mL system setting.

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

Extraction Procedure

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

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

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.

Condition: 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 Loading: Load sample volume of 3.8 mL 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 30 seconds.

Analytical 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 µL BSTFA at 60 °C for 15 min. Inject into GC.

Analytical method: GC-MS

Reagents:

- 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.
- 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.
- 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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