

Extraction of THC-COOH from Urine on RapidTrace® using ISOLUTE® C8 Columns

Application Note

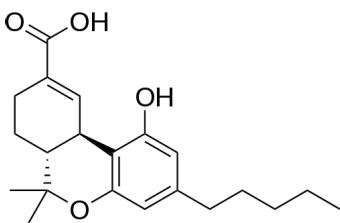


Figure 1. Structure THC-COOH

Introduction

This method demonstrates the automated extraction of THC-COOH from urine using ISOLUTE® C8 columns and the RapidTrace® SPE workstation.

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

Extraction Procedure

ISOLUTE® SPE Column: ISOLUTE C8 100 mg, 1 mL (Part number 290-0010-A) or 100 mg, 3 mL (Part number 290-0010-B)

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 0.25 mL of NaOH, 10 M. Heat for 15 minutes at 60 °C. Cool. Add 1 mL of glacial acetic acid.

Condition: Condition column with 2 mL of methanol at 6 mL/min.

Equilibration: Rinse column with 2 mL of water at 6 mL/min

Sample loading: Load sample volume of 4.4 mL at 1.5 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 2 mL of acetonitrile/acetone/0.01 M HCl, (v/v, 15:15:70), at 6 mL/min
Dry under 20 psig nitrogen for 0.2 min.

Analyte elution: Collect sample with 1 mL methanol/ethyl acetate 50:50 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:

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 (v/v, 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. Methanol/ethyl acetate 50:50. Add 50 mL of ethyl acetate to 50 mL of methanol.

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