

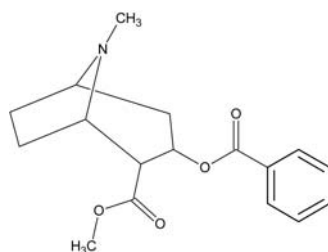


## SPE Application Note for Cocaine and Benzoylecgonine from Urine

The following method has been optimized for the extraction of cocaine and benzoylecgonine from urine for drugs of abuse applications. Typical recoveries of these drugs are > 85%. The procedure may be applied to plasma, serum or whole blood with suitable modifications.

Structure:

Cocaine as shown.



Structural considerations:

The analytes are relatively non-polar, with a basic group which can be utilized in the extraction.

Matrix considerations:

This analyte is present in an aqueous matrix of high ionic strength. A mixed-mode extraction mechanism enables a rigorous interference elution step to minimize interferences.

Analytical Method:

GC-MS

Column: DB-5 capillary 15 m x 0.25 mm i.d. x 25 µL film

Initial Temp: 140 °C for 1 min.

Temp ramp: 15 °C / min.

Final Temp: 260 °C for 3 min.

Detection: MS

Reagents:

a) 0.05 M phosphate buffer, pH 6.0. Add 6.8 g of potassiumhydrogen orthophosphate to a 1 L volumetric flask containing 900 mL of deionized water. Dissolve. Adjust the pH to 6.0 (+/-0.1) with 1.0 M potassium hydroxide, and make up to the mark with deionized water.

b) 0.01M hydrochloric acid. Add 2 mL of concentrated HCl to a 200 mL of deionized water in a 250 mL volumetric flask. Mix thoroughly. Dilute to mark.

c) Methanol/ammonia 98:2. Add 2 mL of concentrated ammonia to 98 mL of methanol.

THIS REAGENT SHOULD BE MADE UP FRESH DAILY.

d) BSTFA containing 1% TMCS.

### Extraction Procedure

ISOLUTE® SPE Column:

Confirm HCX 130 mg/3 mL (Part Number 902-0013-B)

Pre-treatment:

Add 1 mL of 0.05 M phosphate buffer, pH 6.0 to 2 mL urine.

Solvation:

Rinse column with 1 mL of methanol at 2 to 4 mL/min.

Equilibration:

Rinse column with 1 mL of deionized water at 2 to 4 mL/min, followed by 1 mL of 0.05M phosphate buffer (pH 6) at 2 to 4 mL/min.

**Sample Application:** Apply sample at 1-2 mL/min.

**Interference Elution:** Elute interferences with 2 mL of deionized water at 2 to 4 mL/min, followed by 1 mL of 0.01M HCl at 2 to 4 mL/min, and then 2 mL of methanol at 2 to 4 mL/min. Dry the column for 0.5 min at -20psig.

**Analyte Elution:** Elute the analytes with 1.2 mL of methanol/ammonia, 98: 2 (v/v) at 1 to 2 mL/min Evaporate to dryness. Add 0.5 mL of BSTFA and heat for 15 min at 60 °C. Inject into GC. Prepare fresh elution solvent daily.



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