



SPEware uSPE (ultraSPE) Faster / Cleaner Sample Preparation

Cocaine and Benzoylecgonine from Saliva

For GC or GC-MS confirmations using:
Trace-B 35mg.

1. PREPARE SAMPLE

To 0.3ml of saliva sample add internal standard(s) and 1 ml of 100 mM phosphate buffer (pH = 6.0) Mix/Vortex for 30 seconds. Wash Trace-B column with 2ml methanol followed by 1ml H₂O. Transfer the sample into the Trace column and flow through the column at a flow rate of 1.0-2.0ml/minute

2. APPLY SAMPLE

Transfer the supernatant into the Trace-B column and flow through the column at 2-5 ml/min

3 WASH COLUMN (All wash steps flow at 2-5 ml/minute)

1 ml DI H₂O;
1 ml 100 mM HCl
Dry column (3 min at 20 psi N₂)
2 ml CH₃OH;
2ml Ethyl Acetate
Dry column (3 min at 20 psi N₂)

4 ELUTE COCAINE AND BENZOYLECGONINE

1.0ml (80/18/2//Dichloromethane/IPA/NH₄OH); collect eluate at 1mlmin⁻¹
NOTE: Prepare elution solvent daily.

5. DRY ELUATE

Evaporate to dryness at 40 °C.

6. DERIVATIZE

Add 100 ml hexane, 50ml HFIP, 50 ml PFFA
React 10 minutes at 60°C. Remove from heat source to cool.
Evaporate to dryness and reconstitute with 25ml ETOAC

7. QUANTITATE

Inject 1 to 3 ul sample onto chromatograph.
Monitor the following ions (GC/MS):

Cocaine: 182, 198, 303
TMS-BE: 240, 256, 361

D3-Cocaine: 185, 201, 306
TMS-D3-BE: 243, 259, 364