Cocaine and Benzoylecgonine from Urine

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

1. PREPARE SAMPLE

To 2 ml of urine sample add internal standards(s) and 0.5 ml of 100 mM phosphate buffer (pH = 6.0).
Mix/Vortex. (Centrifuge sample for 2 minutes if cloudy.)

2. APPLY SAMPLE

Transfer the supernatant into the column and flow through the column at a flow a rate of
10–20 ml/min.

3. WASH COLUMN (All wash steps flow at 1.0 ml/min)

1.0 ml DI H_2O;
1.0 ml 100 mM HCl
Dry column at 25 psi for 2 minutes
1 ml CH_3OH;
1ml Ethyl Acetate
Dry column at 25 psi for 2 minutes

4. ELUTE COCAINE AND BENZOYLECGONINE

1 x 2.0ml CH_2Cl_2/IPA/NH_4OH (80:18:2); collect eluate at 1 ml/minute or allow to drip by gravity flow.
NOTE: Prepare elution solvent daily.

5. DRY ELUATE

Evaporate to dryness at 40 ºC.

6. DERIVATIZE

Add 50 ul ethyl acetate vortex and transfer to a/s vial
Add 50 ul MSTFA. (Important—do not add MSTFA to dry down vial)
React 20 minutes at 70 ºC. Remove from heat source to cool.
NOTE: Do not evaporate MSTFA solution.

7. QUANTITATE

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

Cocaine: 182, 198, 303	D3-Cocaine: 185, 201, 306
TMS-BE: 240, 256, 361	TMS-D3-BE: 243, 259, 364

HPLC: Reconstitute sample with 50ul of HPLC mobile phase and inject