

Free (Unbound) Opiates In Blood, Plasma/Serum, Tissue For GC Or GC/MS Confirmations Using: 200 mg Clean Screen® Extraction Column



UCT Part Numbers

ZSDAU020 Clean Screen® DAU 10 mL, 200 mg sorbent Without Tips	Or	ZCDAU020 Clean Screen® DAU 10 mL, 200 mg sorbent With CLEAN-THRU® Tips
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Procedure:

1. Prepare Sample

- To 1 mL of 100 mM phosphate buffer (pH=6) add internal standards*. Add 1 mL of blood, plasma/ serum, or 1 g (1:4) tissue homogenate.
- Mix/vortex and let stand 5 minutes.
- Add 2 mL of 100 mM phosphate buffer (pH 6.0). Mix/vortex.
- Sample pH should be 6.0 ± 0.5 .
- Adjust pH accordingly with 100 mM monobasic or dibasic sodium phosphate.
- Centrifuge for 10 minutes at 2000 rpm and discard pellet

2. Condition Clean Screen® Extraction Column

- 1 x 3 mL CH₃OH.
- 1 x 3 mL D.I. H₂O.
- 1 x 1 mL 100 mM phosphate buffer (pH 6.0).

Note: Aspirate at < 3 inches Hg to prevent sorbent drying.

3. Apply Sample

- Load at 1 mL/minute.

4. Wash Column

- 1 x 2 mL D.I. H₂O.
- 1 x 2 mL 100 mM acetate buffer (pH 4.5).
- 1 x 3 mL CH₃OH.
- Dry column (5 minutes at > 10 inches Hg).

5. Elute Opiates

- 1 x 3 mL CH₂Cl₂/IPA/NH₄OH (78:20:2)
 - Collect eluate at 1 to 2 mL/minute.
- Note:** Prepare elution solvent daily.
- Add IPA/NH₄OH, mix then add CH₂Cl₂ (pH 11-12).

6. Dry Eluate

- Evaporate to dryness at < 40 °C.

7. Derivatize

- Add 50 µL ethyl acetate and 50 µL BSTFA (with 1% TMCS)***.
- Overlay with N₂ and cap. Mix/vortex.
- React 30 minutes at 70 °C.

Remove from heat source to cool.
Note: Do not evaporate BSTFA solution.

8. Quantitate

- Inject 1 to 2 µL onto gas chromatograph.
- For MSD monitor the following ions:

Analyte	Primary Ion****	Secondary	Tertiary	Cerilliant #
Codeine-D ₃ -TMS*	374	237	346	C-039
Codeine-TMS	371	234	343	C-015
Morphine-D ₃ -TMS*	432	290	327	M-003
Morphine-TMS	429	287	324	M-005

* Suggested internal standard for GC/MS: Codeine-D₃, Morphine-D₃

*** Part # SBSTFA-1-1,10, 25, 100

**** Quantitation ion



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