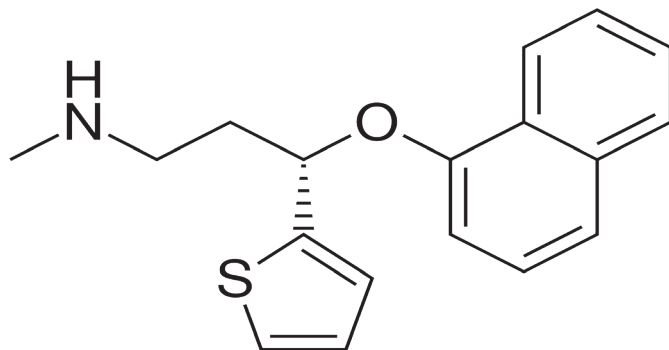


Duloxetine In Blood, Plasma/Serum and Urine. LC-MS/MS Confirmations Using: 200 mg CSDAU Extraction Column



UCT Part Numbers

ZSDAU020
Clean Screen® DAU
10 mL, 200 mg sorbent

Procedure:

1. Prepare Sample

- To 1 mL of 100 mM phosphate buffer (pH= 6) add internal standard.* Add 1 mL of blood or urine. Add 2 mL of 100 phosphate buffer (pH= 6). Mix/vortex.
- Sample pH should be 6.0 ± 0.5 .
- Adjust pH accordingly with 100 mM monobasic or dibasic sodium phosphate.
- Mix/vortex.
- Centrifuge as appropriate.

2. Condition Clean Screen® Extraction Column

- 1 x 3 mL CH_3OH .
 - 1 x 3 mL D.I. H_2O .
 - 1 x 3 mL 100 mM phosphate buffer (pH=6).
- Note:** Aspirate at < 3 inches Hg to prevent sorbent drying out.

3. Apply Sample

- Load sample at 1-2 mL / minute.

4. Wash Column

- 1 x 3 mL D.I. H_2O .
- 1 x 3 mL 100 mM acetic acid.
- 1 x 3 mL CH_3OH .
- Dry column (5 minutes at > 10 inches Hg).

5. Elute Duloxetine

- 1 x 3 mL dichloromethane/ isopropanol/ ammonia (78: 20: 2 v/v).
- Collect eluate at 1-2 mL / minute.

6. Evaporation

- Evaporate eluate under a gentle stream of nitrogen < 40°C.

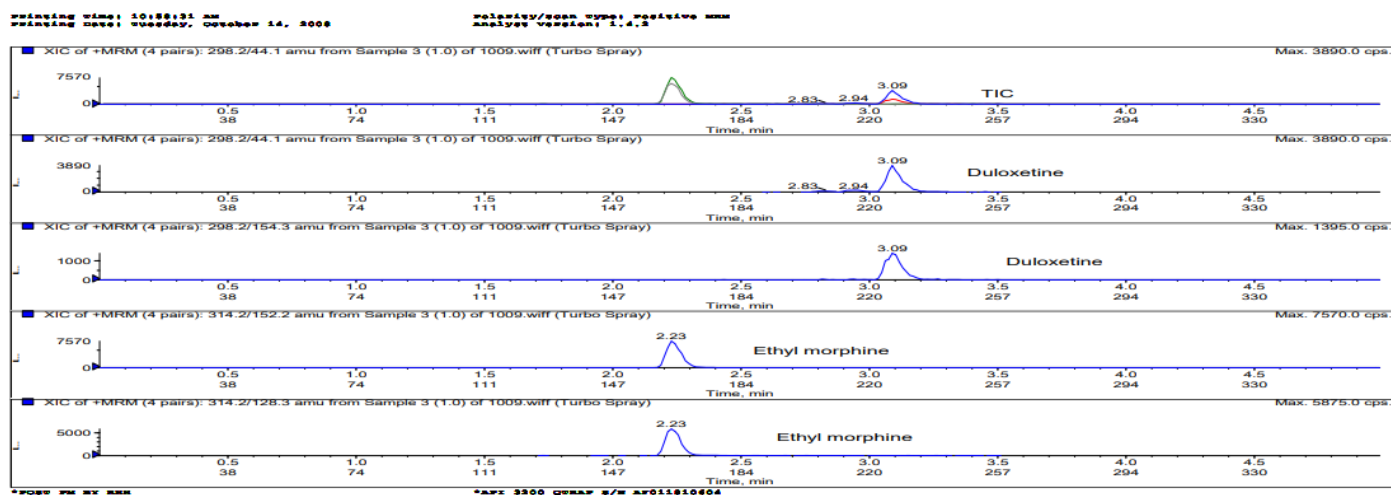
7. Reconstitute

- Sample in 200 μL of 0.1% Formic acid
- Inject 5 μL .



Instrument Conditions		
Column	50 x 2.1 mm (5µm) C ₁₈	
Flow Rate	0.5 mL/minute	
Column Temperature	ambient	
Detector	API 3200 Q-Trap MS/MS	
Mobile Phase		
Time (min)	Acetonitrile %	0.1% Formic Acid
0	5	95
4	90	10
4.1	5	95
5	5	95
Compound	MRM Transition	Cerilliant #
* Ethyl Morphine	314.2/ 152.2	E-052
Duloxetine	298.1/44.1	D-004

Chromatogram of Ethyl Morphine and Duloxetine



*Presented at SOFT annual meeting 2008 by A.A. Elian

Recovery: > 90%

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