UCT

DULOXETINE IN BLOOD AND URINE BY LC-MS/MS*

Part #

ZSDAU020 - CLEAN SCREEN® DAU 200 mg, 10 mL Tube

1. PREPARE SAMPLE:

To 1 mL of 100 mM prosphate buffer (pH 6.0) add internal standard.* Add 1 mL of blood or urine. Add 2 mL of 100 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. Mix/vortex.

Centrifuge as appropriate.

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 out.

3. APPLY SAMPLE:

Load sample at 1-2 mL / minute.

4. WASH COLUMN:

1 x 3 mL D.I. H₂O.

1 x 3 mL 100 mM acetic acid.

1 x 3 mL CH₃OH.

Dry column (5 minutes at > 10 inches Hg).

5. ELUTE DULOXETINE:

1 x 3 mL CH₂Cl₂/ IPA/ NH₄OH (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 (5 µm) C₁₈

Mobile phase:

Time/ min	% Acetonitrile	% 0.1 % Formic Acid
0	5	95
4	90	10
4.1	5	95
5	5	95

Flowrate: 0.5 mL/minite. Column Temperature: ambient. Detector: API

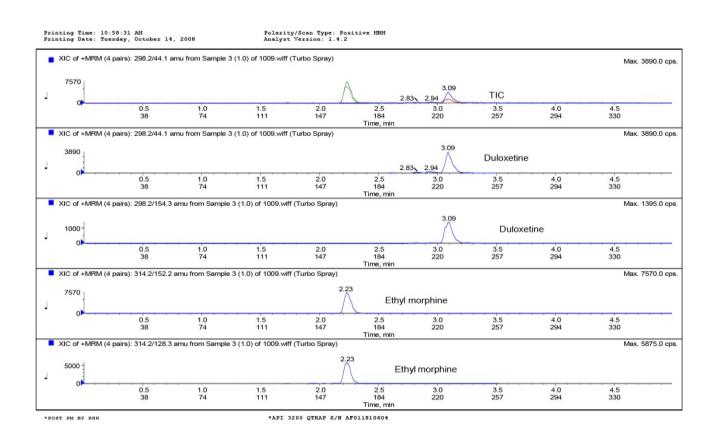
3200 Q-Trap MS/MS.

Compound

MRM Transistion

* Ethyl Morphine (Internal Standard) 314.2/ 152.2 Duloxetine 298.1/44.1

Chromatogram of Ethyl Morphine and Duloxetine



^{*}Presented at SOFT annual meeting 2008 by A.A. Elian