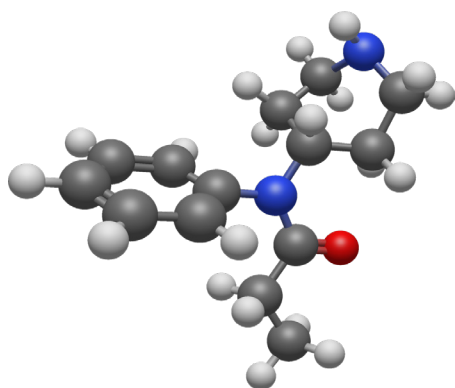


Determination of Norfentanyl / Fentanyl in Urine Samples Using Solid Phase Extraction and GC-MS



UCT Part Numbers

XRDAH206
XtrackT® DAU
200mg x 6mL

SPPHO6001-5
Select pH Buffer Pouch, 100 mM
Phosphate pH 6.0

Procedure:

1. Extraction

- To 1.0 mL of urine add appropriate internal standard* and mix.
- Add 3 mL of 100mM phosphate buffer and mix.
- Centrifuge at 3000 rpm for 10 minutes.
- Discard pellet – retain the liquid.

2. Condition XtrackT® 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 sample at 1 to 2 mL/minute

4. Wash Column

- 1 x 3 mL D.I. H₂O.
- 1 x 3 mL 1.0 M Acetic Acid
- 1 x 3 mL Methanol
- Dry column (5 minutes at > 10 inches Hg).

5. Elute Norfentanyl / Fentanyl

- 1 x 3 mL Dichloromethane: Isopropanol: Ammonium Hydroxide (78:20:2)
Note: Prepare fresh daily by adding 2 mL of ammonium hydroxide to 20 mL of isopropanol, mix and add 78 mL of dichloromethane
- Collect eluate at 1 to 2 mL / minute.

6. Dry Eluate

- Evaporate to dryness under nitrogen < 40°C.
- Dissolve residue in 100 µL of CH₃OH.

7. Inject 1-2 µL onto GC-MS



Instrument Conditions

Instrument	Trace 1300 Gas Chromatograph (Thermo Scientific)
Column	TG-1MS Thermo Scientific (30 m x 0.25 mm (0.25 µm))
Carrier Gas	Helium (1.2 mL/ minute)
Injector Temperature	250°C
Transfer Line Temperature	280 °C
Split Ratio	1:10
Mass Spectrometer	Thermo Scientific ISQ
Mode	Positive EI
Ion Source Temperature	350°C

Oven Program

Time (min)	Temperature °C	Hold time	Ramp Rate °C
0	100	0.5	30
7.8	320	5.0	

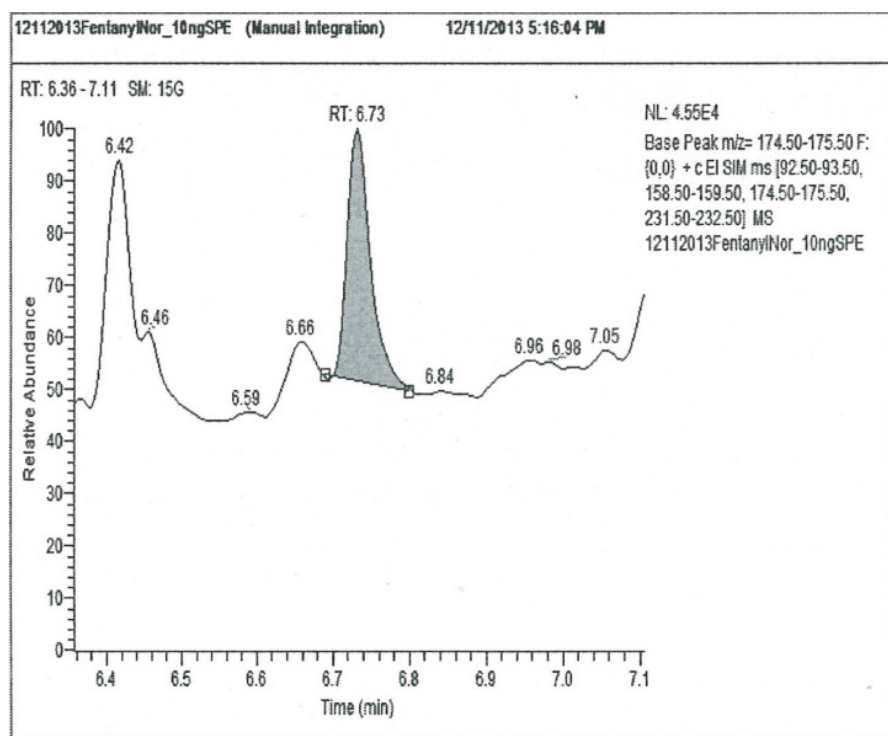
Results

Compound	RT	1° Ion	2° Ion	3° Ion
*Norfentanyl-D5	6.72	98	164	180
Norfentanyl	6.73	93	159	175
*Fentanyl-D5	9.17	250	194	151
Fentanyl	9.18	245	189	146

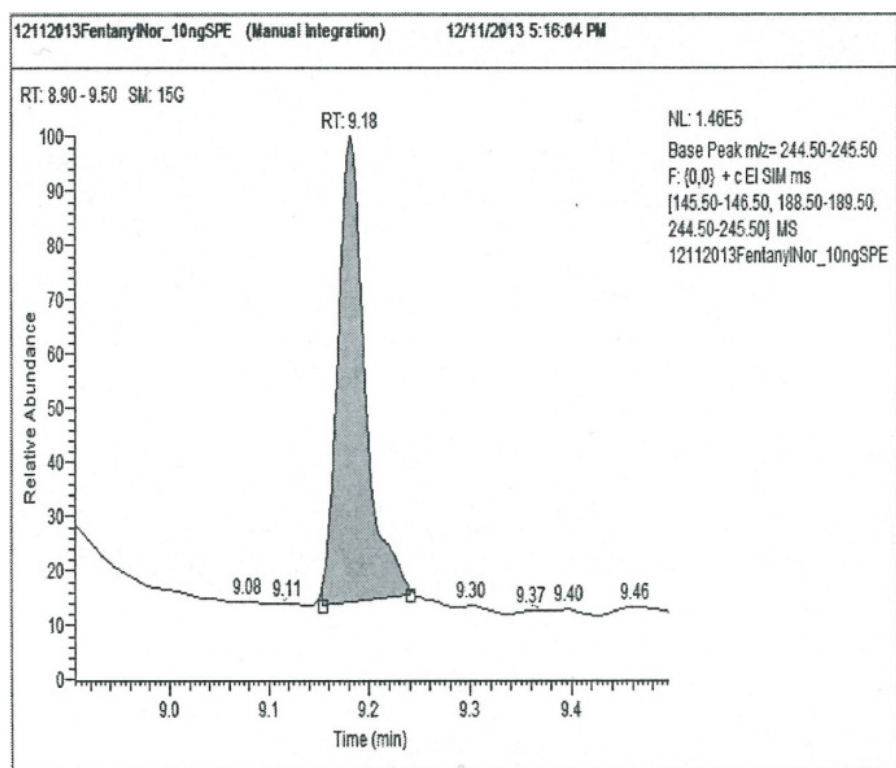
*Internal Standard



Chromatogram of Norfentanyl (10 ng/mL) extracted from 1 mL of Urine



Chromatogram of Fentanyl (10 ng/mL) extracted from 1 mL of Urine



DCN-217121-138

UCT, LLC • 2731 Bartram Road • Bristol, PA 19007 800.385.3153 • 215.781.9255

www.unitedchem.com Email: methods@unitedchem.com

©UCT, LLC 2013 • All rights reserved

