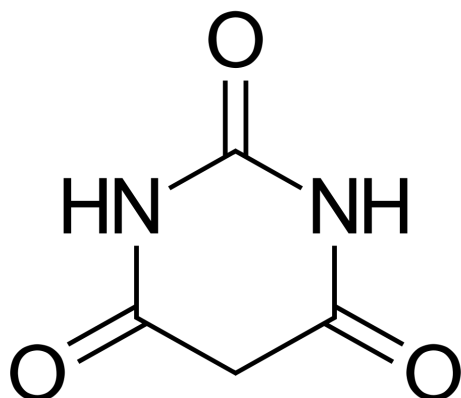


Barbiturates In Equine Urine For GC/MS Confirmations



UCT Part Numbers

XRDAH206

200 mg XtrackT® DAU
Extraction Column in
6 mL cartridge, with
CLEAN-THRU® Tips

or

XCDAH206

200 mg XtrackT® DAU
Extraction Column in 6 mL
cartridge without Tips

SBSTFA-0-1

SELECTRA-SIL® BSTFA,
10 x 1 gm vial pack
optional:

STMPAH-0-1

SELECTRA-SIL® TPAH,
10 x 1 gm vial pack

CLTTP050

CLEAN-THRU® Tips

Procedure:

1. Prepare Sample

- 1) To 2 mL of urine add internal standard(s) and 1 mL of 100 mM phosphate buffer (pH= 5.0) Mix/vortex.
- 2) Sample pH should be 5.0 ± 0.5 .
- 3) Adjust pH accordingly with 100 mM monobasic or dibasic sodium phosphate.

2. Condition XTRACTT® DAU Extraction Column:

- 1) 1 x 3 mL CH₃OH
- 2) 1 x 3 mL D.I. H₂O
- 3) 1 x 1 mL 100 mM phosphate buffer (pH= 5.0)

Note: Aspirate at full vacuum or pressure

3. Apply Sample:

- 1) Load at 1 to 2 mL/ minute

4. Wash Column:

- 1) 1 x 3 mL D.I. H₂O
- 2) 1 x 1 mL 100 mM acetic acid
- 3) Dry column (5 minutes at full vacuum or pressure)
- 4) 1 x 2 mL hexane



5. Elute Barbiturates:

- 1) 1 x 3 mL hexane/ethyl acetate (50:50);
- 2) Collect eluate at 1 to 2 mL / minute

6. Dry Eluate:

- 1) Evaporate to dryness at $< 40^{\circ}\text{C}$
- 2) Reconstitute with 100 μL ethyl acetate

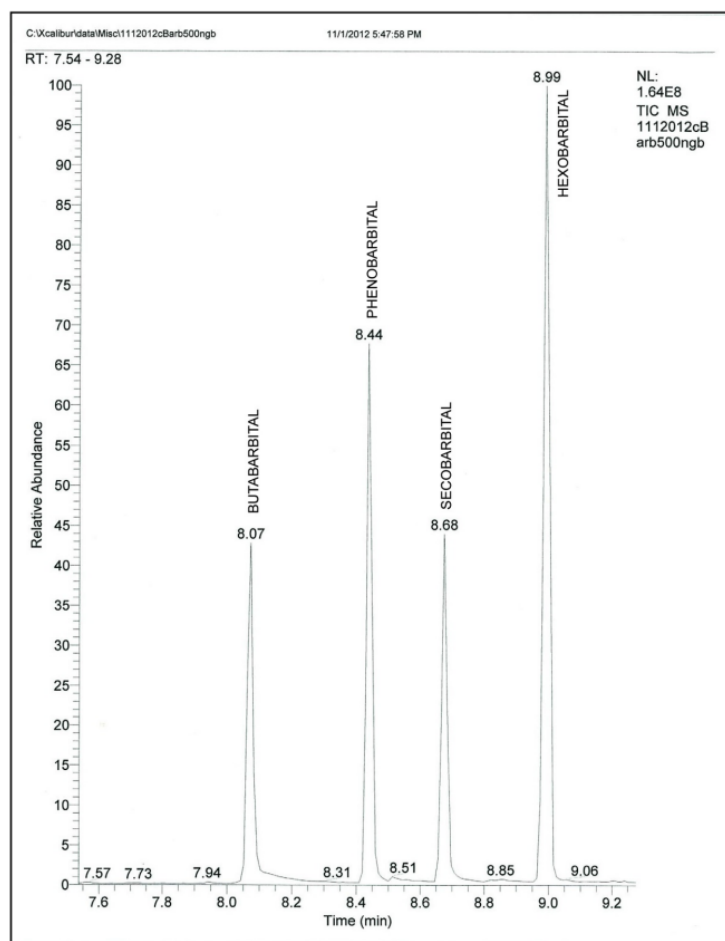
Note: Aspirate at full vacuum or pressure

7. Quantitative:

- 1) Add 50 μL of both Ethyl Acetate and BSTFA
- 2) (OPTIONAL DERIVATIZATION: Add 25-50 μL of 0.2 M TMPAH Reaction occurs in injection port)
- 3) Inject 1 to 2 μL onto gas chromatograph

Chromatogram:

Underivatized Barbiturates



Mass Spec Table

Compound	Primary Ion*	Secondary Ion	Tertiary Ion
Butabarbital	156	141	157
Phenobarbital	204	232	117
Secobarbital	168	167	195
Hexobarbital†	221	157	236

*Quantitation Ion

†Suggested internal standard for GC/MS

Other Barbiturates that can be extracted using this protocol

Compound	Primary Ion*	Secondary Ion	Tertiary Ion
Amobarbital	156	141	157
Butalbital	168	167	181
Pentobarbital	156	141	195
Thiopental	172	157	173

*Quantitation Ion

Mass Spec Ion Table for derivatized barbiturates

Compound	Primary Ion*	Secondary Ion	Tertiary Ion
Butalbital	196	195	209
Butalbital-D5 [†]	201	214	
Amobarbital	169	184	185
Pentobarbital	169	184	112
Secobarbital	196	195	181
¹³ C ₄ Secobarbital _†	200	185	
Phenobarbital	232	146	175
Phenobarbital-D5 _†	237	151	

*Quantitation Ion

†Suggested internal standard for GC/MS



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