

Amphetamines In Urine, Oxidation With Periodate For GC Or GC/MS Confirmations Using: 200 mg Clean Screen® DAU 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

Procedure:

1. Prepare Sample

- To 1 mL of 100 mM phosphate buffer (pH 6.0) add internal standard(s)*. Add 2 mL of urine add, and 1 mL 0.35 M sodium periodate.
- Mix/vortex. Incubate at room temp. for 20 min.
- Sample pH should be 6.0 ± 0.5 .
- Adjust pH accordingly with 100 mM monobasic or dibasic sodium phosphate.

2. Condition Clean Screen® DAU Extraction Column

- 1 x 3 mL CH_3OH .
 - 1 x 3 mL D.I. H_2O .
 - 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 to 2 mL/minute.

4. Wash Column

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

5. Elute Amphetamines

- 1 x 3 mL $\text{CH}_2\text{Cl}_2/\text{IPA}/\text{NH}_4\text{OH}$ (78:20:2);
 - Collect eluate at 1 to 2 mL/minutes.
- Note:** Prepare elution solvent daily. Add IPA/ NH_4OH , mix, then add CH_2Cl_2 (pH 11-12).

6. Concentrate Eluate

- Add 30 μL silylation grade DMF to eluate.
- Evaporate to 30 μL at < 40 °C.
- Alternative: Add 100 μL of 1% HCl in methanol.
- Evaporate to dryness at < 40 °C.

7. Fluoroacylate With PFPA (PFAA)***

- Add 50 μL PFPA (PFAA). Overlay with N_2 and cap.
- Improved derivatization by addition of 50 μL PFPOH****
- React 20 minutes at 70 °C. Evaporate to dryness at < 40 °C.
- Reconstitute with 100 μL ethyl acetate.

8. Quantitate

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

* Suggested internal standards for GC/MS: Amphetamine-D5 and Methamphetamine-D5

** Quantification Ion

*** Part # SPFAA-0-1,10,25,100

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



Analyte (TMS)	Primary Ion**	Secondary	Tertiary	Cerilliant #
Amphetamine-D5*	194	92	123	A-002/A-005
Amphetamine	190	91	118	A-007
Methamphetamine-D5*	208	92	163	A-004
Methamphetamine	204	91	160	A-009

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