



## AMPHETAMINES IN URINE, OXIDATION WITH PERIODATE FOR GC OR GC/MS CONFIRMATIONS USING:

### 200 mg CLEAN SCREEN<sup>®</sup> EXTRACTION COLUMN

Part #: ZSDAU020 without Tips or ZCDAU020 with CLEAN-THRU<sup>®</sup> Tips

#### 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<sup>®</sup> EXTRACTION COLUMN

1 x 3 mL CH<sub>3</sub>OH.

1 x 3 mL D.I. H<sub>2</sub>O.

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<sub>2</sub>O.

1 x 1 mL 100 mM acetic acid.

1 x 3 mL CH<sub>3</sub>OH.

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

#### 5. ELUTE AMPHETAMINES

1 x 3 mL CH<sub>2</sub>Cl<sub>2</sub>/IPA/NH<sub>4</sub>OH (78:20:2);

Collect eluate at 1 to 2 mL/minutes.

**NOTE:** Prepare elution solvent daily. Add IPA/NH<sub>4</sub>OH, mix, then add CH<sub>2</sub>Cl<sub>2</sub> (pH 11-12).

\* Suggested internal standards for GC/MS: Amphetamine-D<sub>5</sub> and Methamphetamine-D<sub>5</sub>

\*\* Quantification Ion

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

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

#### 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<sub>2</sub> 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  $\mu$ L onto gas chromatograph.

For MSD monitor the following ions:

<b>ANALYTE (TMS)</b>	<b><u>Primary Ion**</u></b>	<b><u>Secondary</u></b>	<b><u>Tertiary</u></b>	<b><u>Cerilliant #</u></b>
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