



Grace Davison Discovery Sciences

## Solid Phase Extraction of Amphetamine and Methamphetamine from Urine

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**Amphetamine and methamphetamine are both US DEA schedule II controlled substances that have a high potential for abuse and addiction. Solid phase extraction (SPE) can be used to isolate and concentrate methamphetamine and amphetamine from biological fluids such as urine for subsequent analysis by gas or liquid chromatography.**

**M**ethamphetamine, also known as “meth,” “crank,” “speed,” “crystal,” and “ice,” is one of the most abused illegal drugs in the world today. A highly addictive drug, methamphetamine is also easily manufactured in clandestine laboratories with easily obtained materials. There are legal medicinal uses for methamphetamine, including the treatment of narcolepsy, attention deficit disorder (ADD), and attention deficit hyperactivity disorder (ADHD). Amphetamine is also prescribed legally for the treatment of ADD, narcolepsy, and weight control. However, the illegal use and abuse of these drugs is widespread and chromatographic techniques such as GC and HPLC are frequently used in drug screening tests. Chromatographic testing of biological fluids such as urine for amphetamine and methamphetamine requires a cleanup step such as SPE to prevent contamination of the GC or HPLC system. SPE also concentrates the solutes of interest, which allows for detection of lower levels of the drugs.

GracePure™ C18-AQ SPE tubes do not require preconditioning with an organic solvent, eliminating one step from typical SPE procedures. This saves both time and solvent compared to a typical reversed-phase SPE tube. The hydrophilic endcapping used with GracePure™ C18-AQ SPE tubes also permits drying of the tube between steps while maintaining consistently high recoveries. The following procedure shows recovery data for both preconditioned and unconditioned GracePure™ C18-AQ SPE tubes, demonstrating a clean extract with high recovery.

### Experimental Conditions

SPE Tube: GracePure™ C18-AQ, 500 mg/3 mL (Alltech Part No. 5138775)

Sample: Urine containing 1 µg/mL of amphetamine and methamphetamine. Urine was diluted with an equal volume of 2% ammonium hydroxide in deionized water.

- Two column volumes of deionized water
  - 1 mL of 25:75 isopropyl alcohol:deionized water. Dry tube for two minutes under 20" Hg vacuum.
  - 1 mL hexane. Dry tube for two minutes under 20" Hg vacuum.
  - 1 mL isopropyl alcohol
- 4.) Elute amphetamine and methamphetamine with 3 × 1 mL isopropyl alcohol containing 2% ammonium hydroxide
- 5.) Extract was dried under nitrogen and reconstituted with 1 mL isopropyl alcohol

SPE Procedure-Unconditioned GracePure™ C18-AQ SPE Tube:

Same as above extraction procedure except step 1 deleted.

GC Column: Heliflex® AT™-5ms, 30 m × 0.25 mm × 0.25 µm (Alltech Part No. 15807)

Operating Conditions: Helium carrier gas at 1.1 mL/minute

Injection: 1 µL sample, splitless injection (purge time at 1 minute)

Injector: 250 °C

Detector: FID at 300 °C

Oven temperature program: 45 °C (1 minute hold)–140 °C at 20 °C/minute (10 minute hold)–250 °C at 30 °C/minute

### Results

Comparing unconditioned tube recoveries to conditioned tube recoveries shows that GracePure™ C18-AQ gives similar results regardless of conditioning status. This extraction procedure was optimized for GracePure™ C18-AQ to give clean extracts while obtaining absolute recoveries > 80%.

### Conclusion

GracePure™ C18-AQ SPE tubes can be used without preconditioning to extract amphetamine and methamphetamine from urine, giving a clean extract with recoveries greater than 80%.

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Table I: General chromatographic analysis parameters

GracePure™ C18-AQ				
	Amphetamine % Recovery	Methamphetamine % Recovery	Amphetamine Recovery Unconditioned/Conditioned	Methamphetamine Recovery Unconditioned/Conditioned
Unconditioned tube (n=6)	83.1	89.2	0.98	0.98
Conditioned tube (n=4)	84.7	90.7		

SPE Procedure-Conditioned GracePure™ C18-AQ SPE Tube:

- 1.) Condition tube with two column volumes of methanol and two column volumes of deionized water.
- 2.) Apply 10 mL of diluted urine to tube.
- 3.) Wash tube with:

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