APPLICATION BRIEF

Extraction of the SAMSHA-5 Using a Single Sorbent and Reagent Set with the RapidTrace® SPE Workstation

When the RapidTrace SPE Workstation was introduced in 1995, it dramatically changed the way reference laboratories performed solid phase extraction (SPE). Nevertheless, extraction bottlenecks remained. Traditional drugs of abuse methods often used multiple sorbents for different drugs and required as many as fifteen reagents. Given the number of sorbents and reagents, errors can occur easily. Simplifying the system was the obvious solution.

The Challenge: Create robust methods using a single sorbent and a minimal number of reagents.

Can we extract the SAMSHA-5 on one cartridge with fewer reagents?

The most popular extraction methods for drugs of abuse were developed years ago. Since SPE was not widely used, scientists often developed a method for a single drug, not looking at the similarities between the drug classes. Today we have a better overall understanding of sorbent chemistry and its interaction with analytes and reagents. Dramatic improvements have been made in sorbent quality and the processes used to achieve bonded silicas.

While older methods used many types and concentrations of buffers and acids, newer methods allow us to use fewer reagents given our better understanding of the chemistry and the availability of high quality sorbents.

Why use only one sorbent?

Simplicity. One sorbent allows laboratories to stock a single cartridge, eliminating confusion. The sorbent used is a mixed-mode, combining C8 with a strong cation exchanger. Mixed-mode has the ability to selectively elute acidic, basic and neutral fractions. These extractions were performed using ISOLUTE Confirm™ HCX mixed-mode cartridges from Jones Chromatography and International Sorbent Technology (IST).

Am I sacrificing extract cleanliness and recovery using only one sorbent and fewer solvents?

Mixed-mode columns are typically less matrix dependent than pure ion-exchange. They provide ionic interaction with positively charged analytes as well as non-polar interaction with hydrophobic analytes. The ionic retention also permits a stronger organic rinse step, ultimately yielding a cleaner extract.

Using methods provided by Jones Chromatography on ISOLUTE® solid phase extraction cartridges, here are recoveries that can be achieved using RapidTrace in your laboratory.

Analyte	Recovery(%)
Benzoylecgonine	91
Amphetamine	90
Opiates	84 - 95
Phencyclidine (PCP)	88
THC	97

The reagents used for the above extractions include:

- Water
- Methanol
- 0.05M Potassium Phosphate Buffer, pH 6.0
- 0.01M Hydrochloric Acid
- 1.0 M Ammonium Acetate Buffer, pH 8.0
- Acetonitrile/Acetone/0.01M Hydrochloric Acid (15/15/70)
- Hexane/Ethyl Acetate/Acetone (50/40/10)
- Methanol/Ammonia (98/2)

SPE Methodology Trends

Conforming to the industry demands of improved simplicity, these RapidTrace methods are somewhat more generic than traditional methods. Each SAMSHA-5 method uses the same ISOLUTE Confirm™ HCX cartridge and the same reagent set, however, the methods themselves are not identical. Below is an example of one of the five RapidTrace methods:

SPE Sorbent Manufacturer:	Jones Chromatography/IST
Procedure:	THC COOH in Urine
Extraction Column:	ISOLUTE Confirm™ HCX, (preferably 1 mL/130 mg)
Sample Volume:	3 mL
Sample Pretreatment:	Add 100 uL ISTD, Hydrolyze 3 mL urine with 0.1 mL 10M NaOH, Heat 15 minutes at 60°C. Cool, add 0.5 mL glacial acetic acid.
Post Extraction Treatment	Evaporate extract to dryness. Add 50 uL BSTFA, heat for 15 minutes at 60°C.

STEP	REAGENT	OUTPUT	VOLUME (mL)	FLOW RATE (mL/min)
Condition	Methanol	Waste 2	1	10
Condition	Water	Waste 1	1	10
Condition	0.01 M Hydrochloric Acid	Waste 1	1	10
Load	Sample	Waste 1	3.8	2
Rinse	Water	Waste 1	1	6
Purge-Cannula	Water	Cannula	4	30
Rinse	Acetonitrile/Acetone/0.01M HCl (15/15/70)	Waste 1	2	6
Dry	1 minute			
Collect	Hexane/Ethyl Acetate/Acetone (50/40/10)	Fraction 1	1.5	1.5
Purge-Cannula	Methanol	Cannula	5	30
Purge-Cannula	Water	Cannula	5	30

Interested in learning more or getting copies of all of the SAMSHA-5 methods? Simply call CaliperLS at (508)435-9500 and ask to speak with someone about forensic urine drug testing. You can also contact us via email at sales@caliperLS.com.

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