

Paul Liang, Brittany Laramee, Frederick Li, Brian Musselman
IonSense, Inc., Saugus, MA 01906

Overview

- Solid phase microextraction fibers (SPE-its) improves DART detection sensitivity in matrix
- SPME methods are slow and requires frequent operator attention
- SPME with DART can be automated to reduce operator time from 35 minutes to under 1 minute

Introduction

- Drugs of abuse can be detected from urine specimen, but lack of chromatographic separation reduces detection sensitivity with DART-MS
- DART-MS can utilize SPE-its to extract and concentrate drug compounds from urine
- Current workflow is slow and requires frequent operator attention
- The addition of a flat surface module allows automated systems to transfer entire boxes of SPE-its, enabling high throughput SPE-it analysis

Method

- Current workflow involves operator to fill wells with conditioning, wash, and extraction solutions and manually filling racks with 12-SPE-its and manually transferring racks between wells for each step: conditioning, washing, extraction, washing, and analyzing
- New workflow adds an elution step to the current workflow but can reduce operator time to DART-MS analysis only
 - Solvents and extraction solutions can be automated with liquid handlers
 - SPE-it boxes can be moved with automated arm
 - Eluent can be deposited with a liquid handler robot onto QuickStrip (QS) mesh for analysis with DART-MS

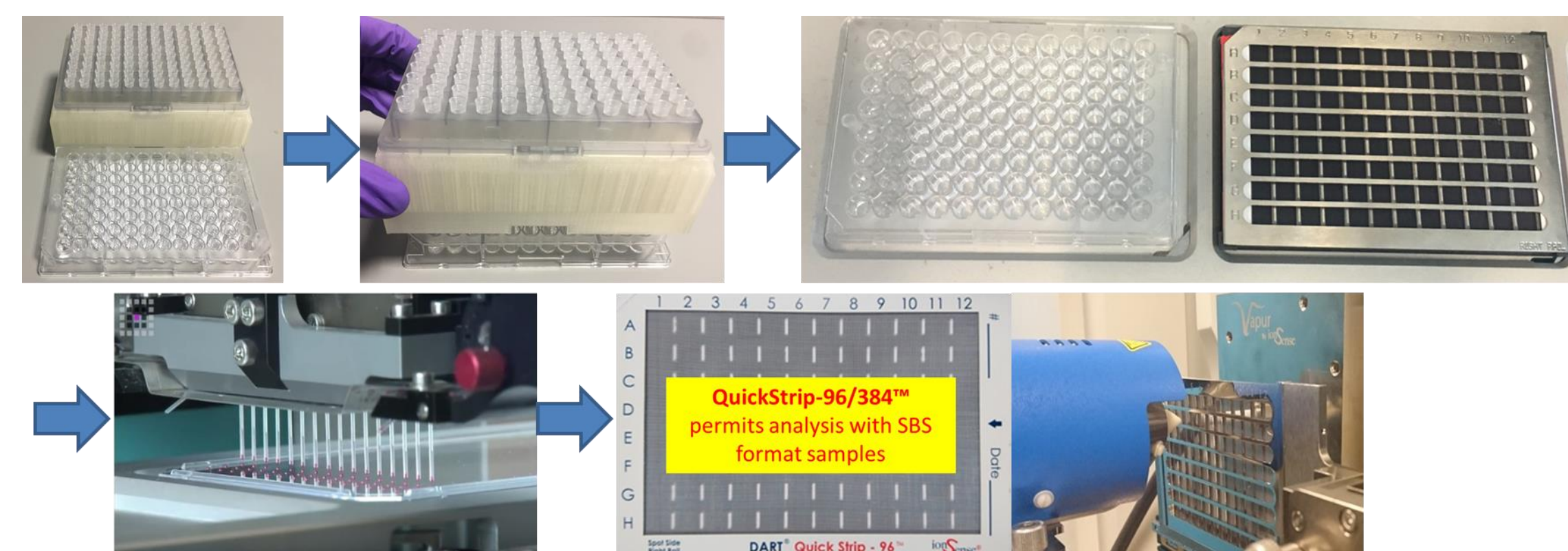


Figure 1 – Automated Workflow

Results

Decreased Sensitivity

- Detection of certain drugs of abuse such as opioids like codeine and heroin or cannabinoids like $\Delta 9$ -THC can be reduced in matrices such as urine
- Direct analysis of spiked urine on QS cards with 3 μ L deposits resulted in limits of detections of **1 ppm** for codeine and $\Delta 9$ - THC and **greater than 1 ppm** for heroin

Elution Solvent

- With the additional elution step for automated workflow, various elution solvents were examined for three drugs at 1 ppm: codeine, $\Delta 9$ -THC, and heroin
- 10 – minute elution time with acetonitrile resulted in the highest signal response

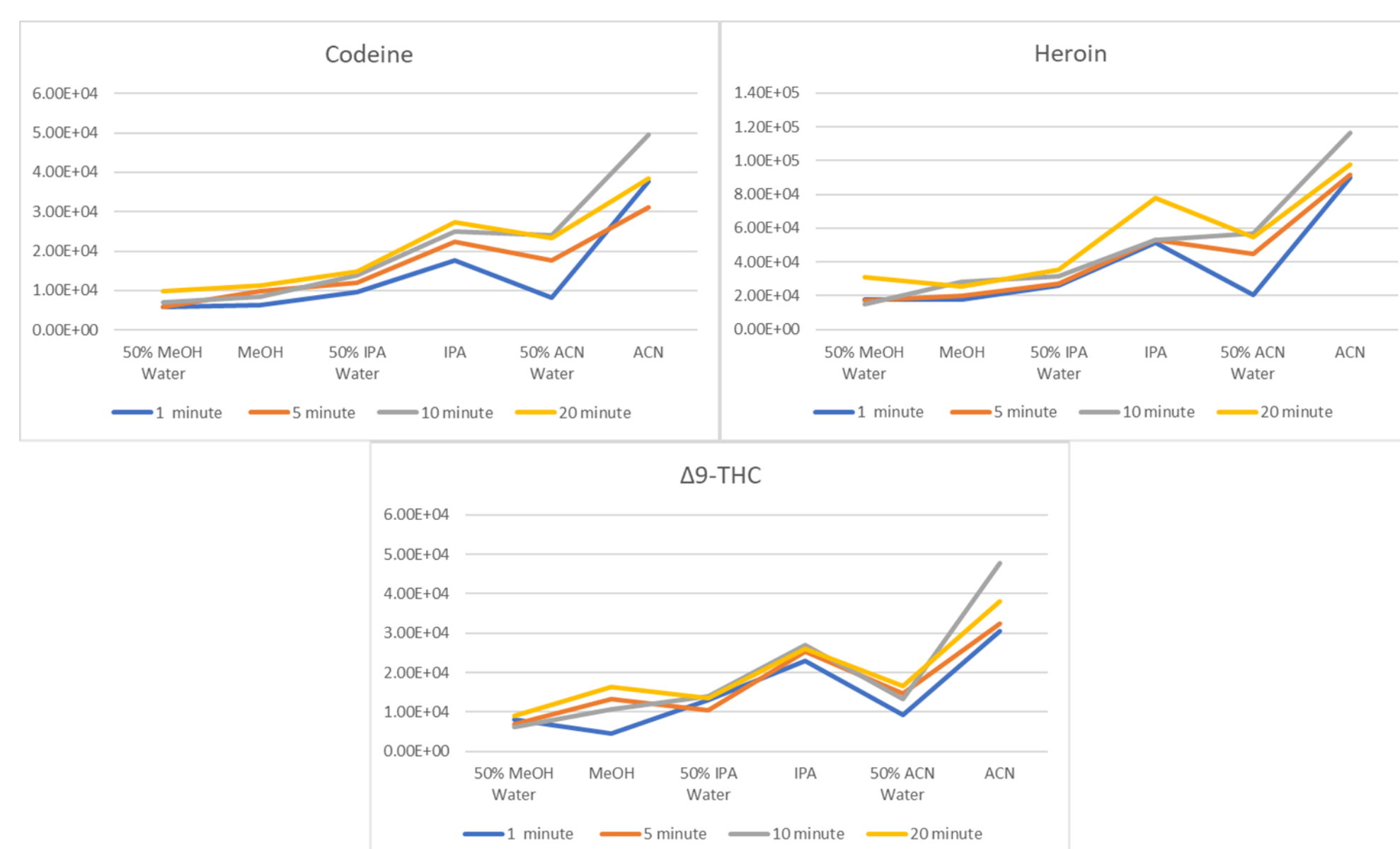


Figure 2 – Elution Solvent and Elution Time Optimization

Limit of Detection

- With the optimized elution solvent and elution times, LOD of the three drugs were examined
- LOD was improved from direct analysis: codeine and THC were detected at **125 ppb** compared to **1 ppm** and heroin was detected at **31 ppb** compared to **>1 ppm**

Table 1 – Drug LOD Comparison

Limit of Detection			
Method	Codeine	$\Delta 9$ -THC	Heroin
Urine Direct Analysis	1 ppm	1 ppm	>1 ppm
SPE-it Extraction	125 ppb	125 ppb	31 ppb

Lowest Concentration with S/N>3

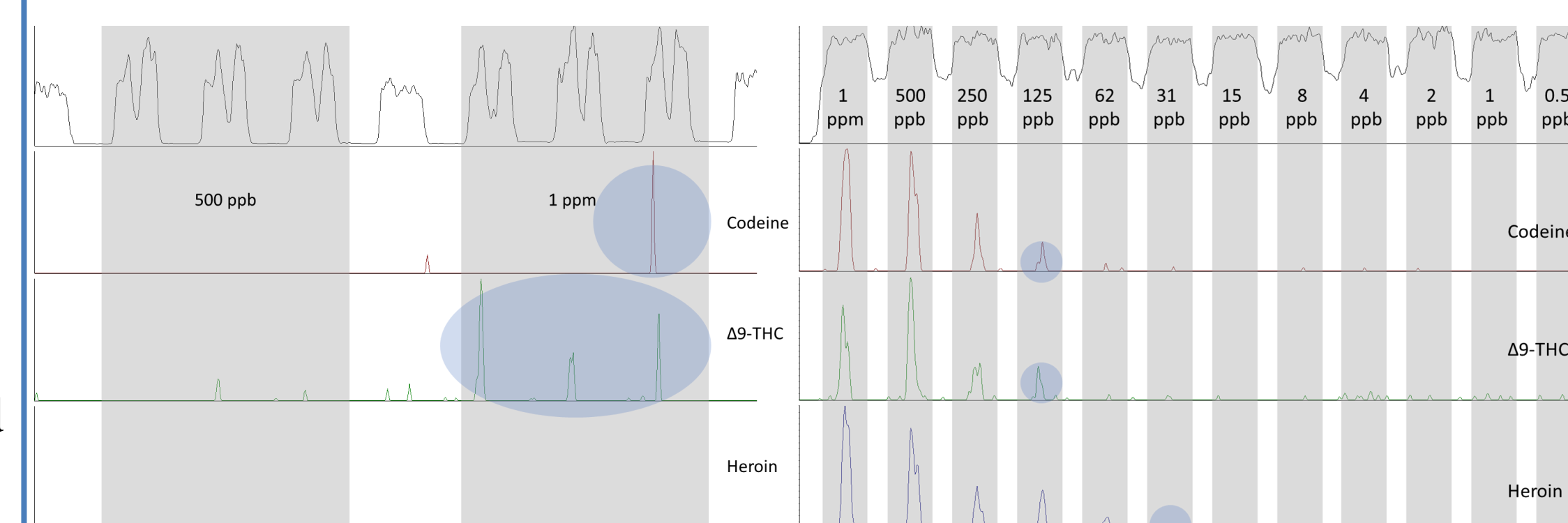


Figure 3 – Urine Direct Analysis LOD

Figure 4 – SPE-it Extraction LOD

Conclusion

Current Workflow

- Manual transfer of solvent to 96 wells for conditioning and washing, and of samples, which can take up to half an hour
- Manual transfer of 1 box of SPE-its onto 8 12-sample racks can require up to 5 minutes
- Analyzing 8 racks of SPE-its require manually loading every 5 minutes
- Time requiring operator for one box of SPE-its (96 samples):
 - 35 minutes
 - Changing out SPE-it racks every 5 minutes (8 total)

High Throughput Workflow

- High throughput workflow does not require manual transfer of solutions
- High throughput workflow does not require manual transfer of each SPE-it individually
- Analyzing eluent requires manually changing out QS-HT every 40 minutes
- Time requiring operator for one box of SPE-its (96 samples):
 - <1 minute
 - Change out QS-HT cards once every 40 minutes