Pulse DART: Improving Throughput and Reducing Helium Consumption, Ambient Background and Matrix Interference

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Overview

- Due to the recent global helium shortage, helium reduction was investigated for DART using a pulse gas flow approach.
- Pulse gas flow approach reduced helium usage up to 95% compared to continuous flow.
- Thermal desorption enabled ionization of solids in their native form by ESI and low vapor pressure inorganic salts by DART.

Introduction

Direct Analysis in Real Time (DART) is an ambient ionization technique that traditionally employs a continuous heated gas stream containing metastables for ionization of small molecules. Helium is most commonly used due to the high internal energy of its metastables. Due to the recent global shortage of helium, we investigated pulsing the helium at set durations only when the sample is positioned directly in front of the DART source to reduce helium usage. Numerous other advantages and benefits with regards to spectral peak shape, ambient background, speed and throughput were discovered.

Methods

A DART-SVP ionization source is interfaced to a Waters QDa mass detector and Agilent 6530 QTOF.

- Pulsing of the DART helium gas was achieved using an external solenoid valve configured between the gas exit from the DART controller and the gas in on the DART source.
- The solenoid valve served as a shutter to prevent or permit the flow of helium to the DART source.
- Marlin G-code scripts were written and executed using the Pronterface software interface with an Arduino board to synchronize the timing of sample introduction with the pulsing of the gas.

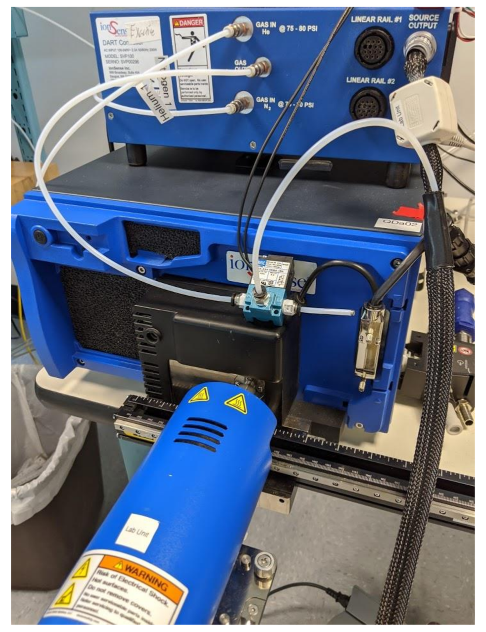

Figure 1: Pulse DART setup with solenoid shutter.

- Row C: 384-well normal DART continuous movement and continuous ionizing gas.
- Row D: 384-well pulse DART discontinuous movement and rapid switching of ionizing gas on when stopped and then off when moving.

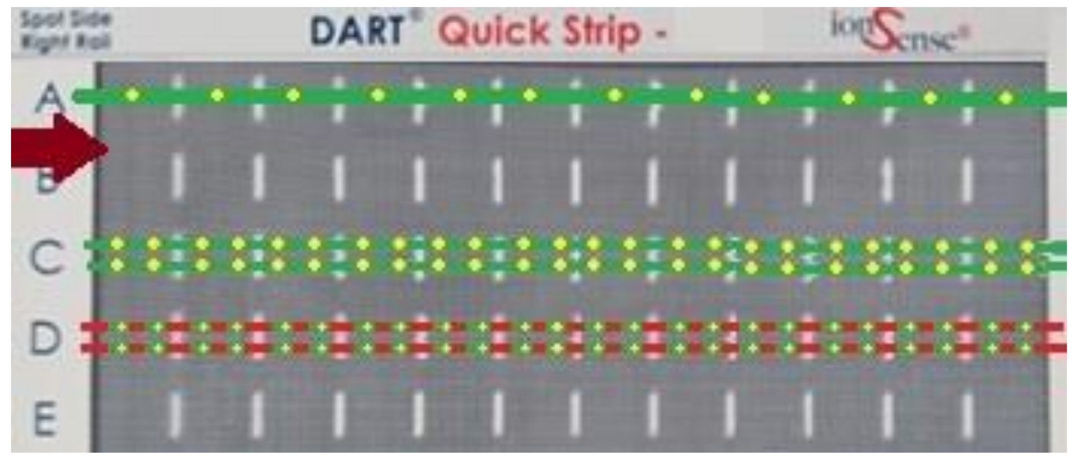

Figure 2: Schemes for sample movement through ionization region and ionizing gas sequence for continuous and pulse DART operation.

Results

Helium Usage Reduction with Pulse DART

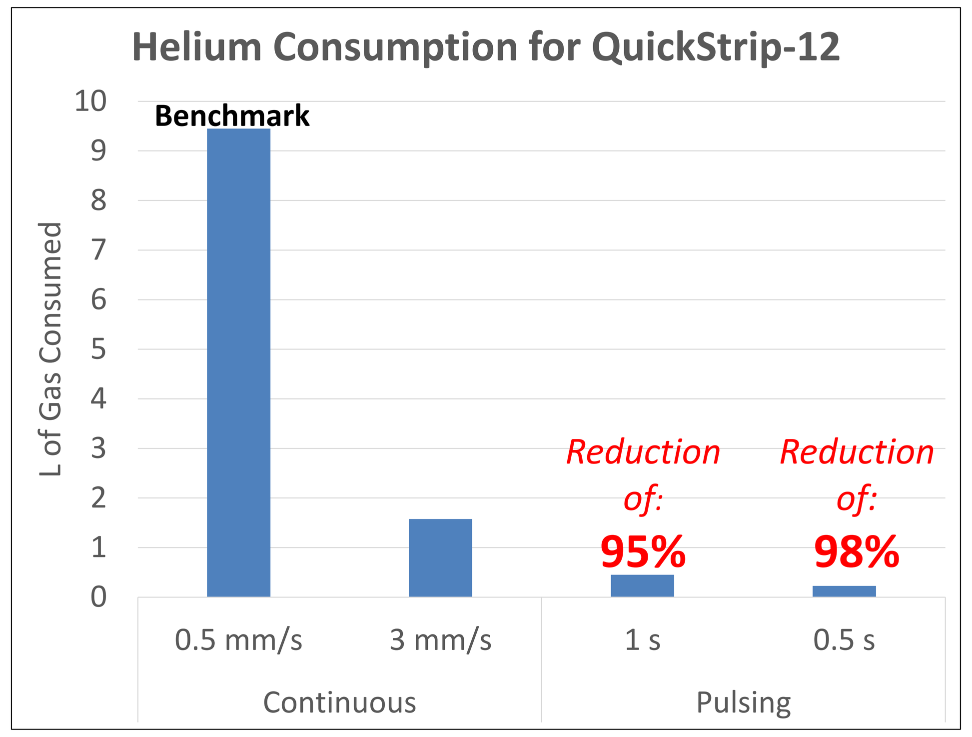

Figure 2: Helium reduction with pulse flow approach for DART compared to a continuous flow of gas.

- Sample introduction rate of 0.5 mm/s is common for continuous flow DART analysis of 12 samples on QuickStrip mesh.
- 3 mm/s is fastest rate while maintaining peak separation.
- A 1 second pulse yields a helium reduction of 95% versus continuous flow with a rate of 0.5 mm/s.

Throughput with Continuous Flow DART

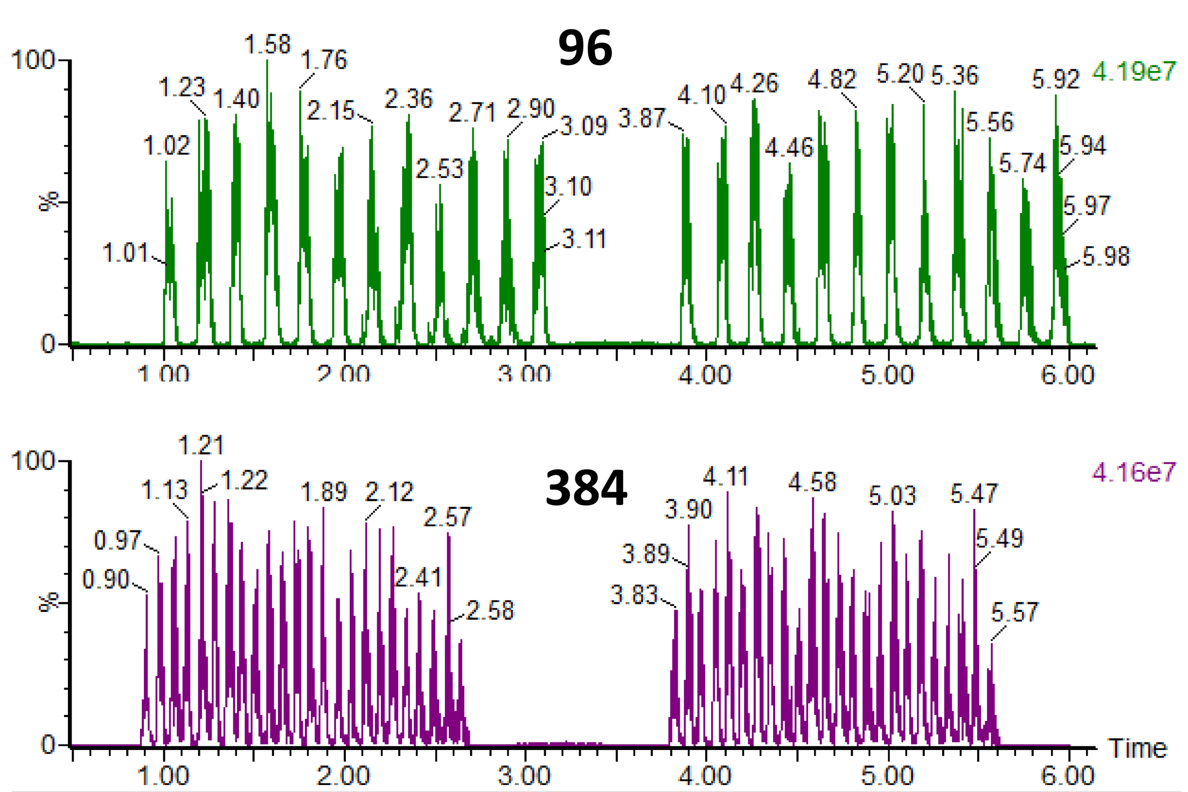

Figure 3: Representative EIC of codeine analyzed in a 96- and 384-sample array. Peaks for two rows are shown.

- 200 nL codeine deposited on QuickStrip mesh
- Sample introduction rate of 1 mm/s.
- Peaks start to overlap for 384
- 96 samples: **20 minutes**
- 384 samples: **40 minutes**

Throughput Improvements with Pulse DART

- Pulse conditions: 1 second pulse, 2 second delay between samples, and sample introduction rate of 7 mm/s.
- Peaks are baseline separated as shown in the EIC.
- **96 samples:** 6.2 minutes
- **384 samples:** 22.3 minutes

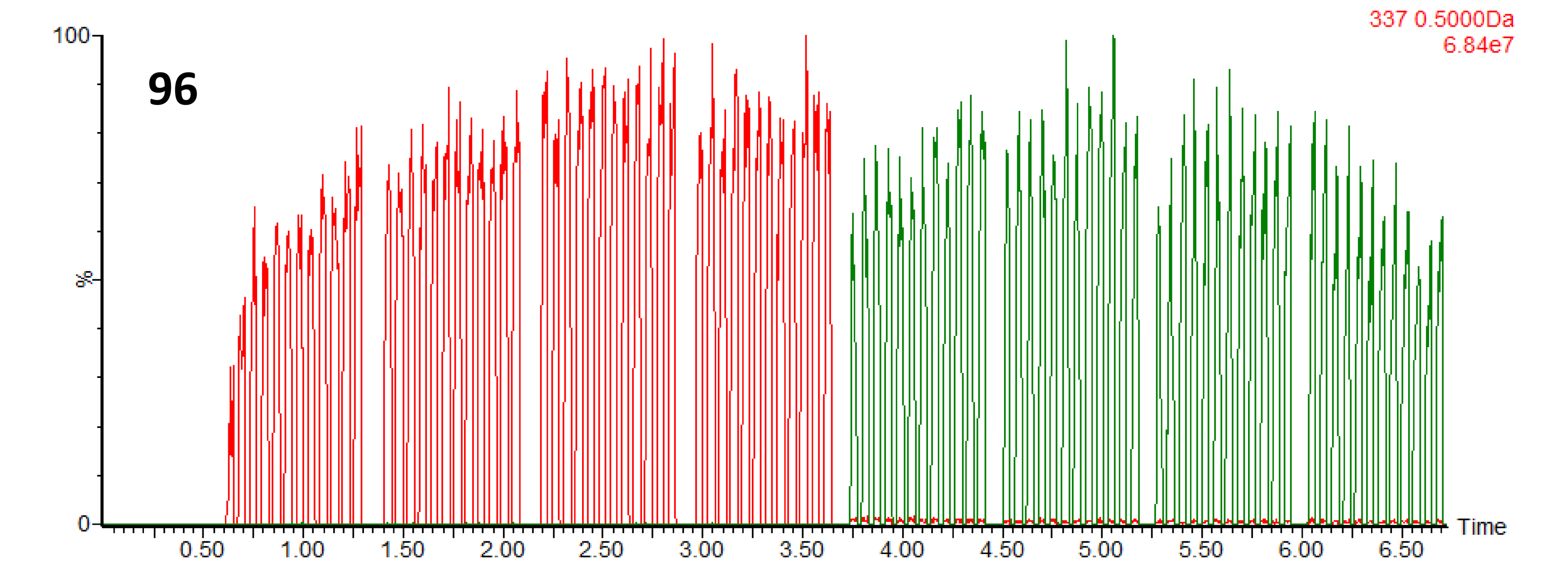

Figure 4: Representative EIC of codeine and fentanyl analyzed in a 96-sample array with pulse DART. Peaks for two rows are shown.

Spectral Improvements

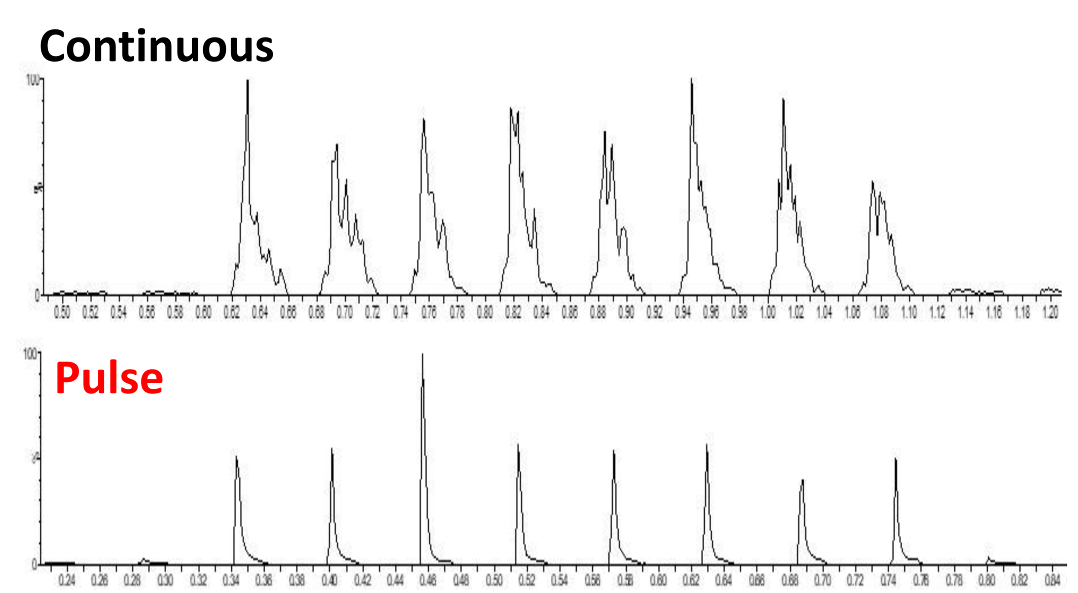

Figure 3: Representative extracted ion chronogram of cocaine obtained using continuous and pulse flow DART-MS

- Pulse DART: 2 second pulses.
- EIC peaks for cocaine with pulse flow DART are sharper and narrower compared to continuous flow DART.

Conclusion

- Helium usage is significantly reduced with a pulse ionizing gas flow.
- Throughput is significant increased with pulse DART with analysis times as short as 6 minutes for 96 samples and 22 minutes for 384.
- Peaks are baseline separated even at these analysis speeds, enabling peak detection for automated processing.