Robotic Sampling for Applications in Direct Analysis in Real Time (DART®)

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Overview

Utilization of Direct Analysis is Real Time (DART®)-based ionization for high throughput analysis is facilitated by the combination of a microprocessor driven linear guide and CTC Analytics HTC PAL robot. This customized robotic sample introduction system provides the means to inject liquid samples onto a solid surface and subsequently present that surface to the DART equipped MS for analysis. The advantages of this system are that it permits the use of a wider range of robotic sampling tools in combination with ambient ionization.

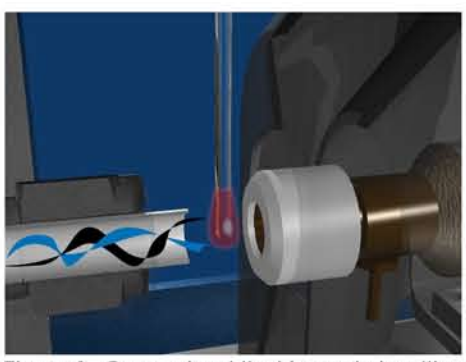

Figure 1. Conventional liquid sample handling with customized closed end glass capillary tubes (DIP-it Samplers®).

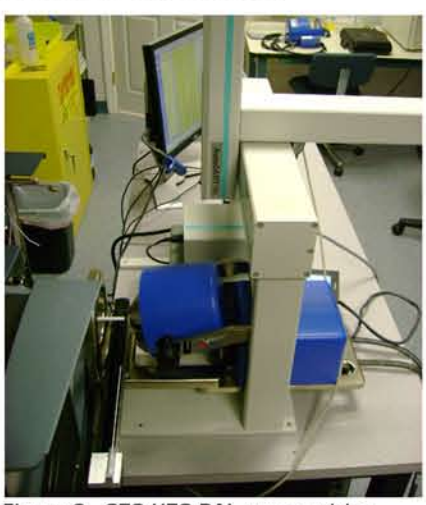

Figure 2. CTC HTC PAL set up with a DART ionization source in combination with a motorized linear guide.

Figure 3. LC/DART.

Figure 4. Spotting with 1 cm spacing.

Introduction

Analysis of samples using metastable atoms at ambient pressure with a direct analysis in real time (DART) source equipped LC/MS has been completed by manual presentation of material for analysis in most cases. We have implemented a robotic sample presentation system to facilitate high throughput analysis of liquids using conventional robotics and syringe-based injections commonly implemented for LC/MS. In prior experiments we had sampled liquids directly onto a glass rod by dipping the end of the rod into the liquid. The difficulty with this configuration was that it required the top of the sample vial to be open to air during sampling. In order to sample from capped vials we recognized the need to use a syringe-based sampling system. The combination of two robotic units, one for syringe control and one for moving the analyte from the injection point to the DART ionization region for analysis was envisioned to provide a means for either sampling from vials in seconds, or prepping samples for rapid off-line analysis at a later time.

Results & Discussions

Samples were dissolved in solvents and sealed in vials as typical for LC/MS analysis. The vials were positioned on the deck of the CTC HTC PAL equipped with a 10 µL volume syringe. A holder capable of maintaining the position of a solid rod up to 12 inches in length was positioned so that the sampling rod could be pushed through the DART ionization region for desorption ionization MS analysis. Results from desorption of two analytes from three different sample rod materials including; glass, ceramic and metal are shown below.

DART is a very rapid desorption method often requiring only a few seconds for complete sampling. Analysis of complex samples can require more time. To permit analysis over a longer period of time we used the application of sample onto the solid rods and slow movement of the sample through the ionization region. The device is shown to facilitate desorption of sample ions with good signal to noise that is sufficient for experiments such as optimization of the mass spectrometer tuning parameters critical for MS/MS experiments, and MS/MS of multiple peaks in a single sample.

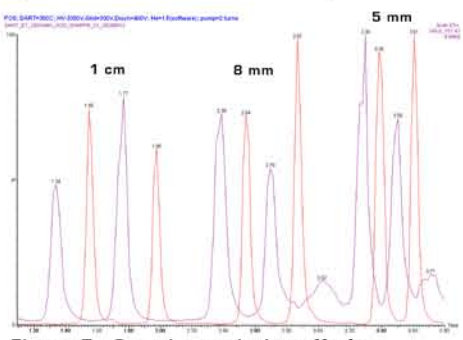

Figure 5. Sample resolution off of an ALUMINA CERAMIC rod. Expo Vis-à-Vis Wet Erase black marker (purple) and Sharpie blue permanent marker (red).

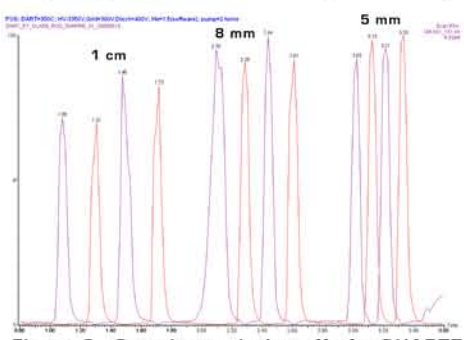

Figure 6. Sample resolution off of a QUARTZ GLASS rod. Expo Vis-à-Vis Wet Erase black marker (purple) and Sharpie blue permanent marker (red).

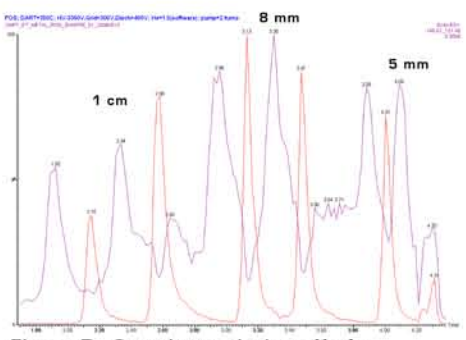

Figure 7. Sample resolution off of a STAINLESS STEEL METAL rod. Expo Vis-à-Vis Wet Erase black marker (purple) and Sharpie blue permanent marker (red).

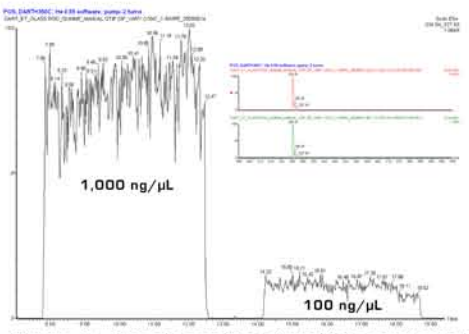

Figure 8. Steady analyte signal for compound tuning using the LC/DART set up with a smooth glass rod. Quinine 1,000 ng/µL and 100 ng/µL in 1:1 MeOH:H_2O.

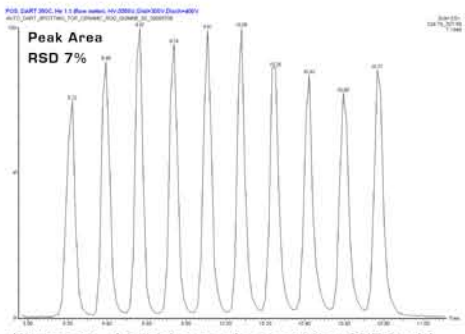

Figure 9. Liquid sample spotting (250 nL) onto an alumina ceramic rod. The ceramic rod was guided into the DART beam with the linear rail at 0.8 mm/sec.

Figure 10. Liquid sample handling with CTC PAL syringe and direct spotting onto ceramic rod.

Conclusions

It has been demonstrated in this work that robotic sample handling has enabled a more efficient and reproducible means of analysis for DART/MS experiments. The sampling time for the analysis of liquids has been improved from 40 sec/sample to 12 sec/sample with spotting onto the sampling rods. Future experiments include investigation of a tablet scanner and a TLC plate scanner.

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