A Modified Direct Sample Analysis (DSA) Ionization Source for the Direct Analysis of Thin-Layer Chromatography (TLC) Plates

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1 Introduction

Presented here is a DAPCI source for the direct analysis of TLC plates and other surfaces. The source was modified from a commercial DSA (Direct Sample Analysis) source made by PerkinElmer. Modifications were made to the heater and nozzle assemblies as well as the capillary extension. While only one application is shown, this source configuration has far reaching utility.

While TLC plates offer rapid analyte separation, detection can often be time consuming and non-specific. Strategies include retention factor comparison with a standard, specific reagent binding or spectroscopy. Additionally, spots of interest can be scraped off the plate and dissolved in a solvent for separate analysis. Ambient mass spectrometry has the potential to improve the analysis of TLC plates by offering direct sampling strategies. **The goal of this project was to create an automated system to analyze TLC plates using a modified DSA source**.

2 Previous Strategies

Mass spectrometry has been applied previously to the analysis of TLC plates. The techniques are divided into two categories, direct and indirect. Indirect techniques involve the removal of the spot in question for separate analysis. Ambient mass spectrometry falls into the direct category where the plate remains intact.

Indirect	Direct
MALDI	MALDI
FAB	AP-MALDI
ESI-MS	LD-APCI
APCI-MS	LIAD-ESI-MS
GC-MS	DESI
LC-MS	DART

3 Commercial Setup

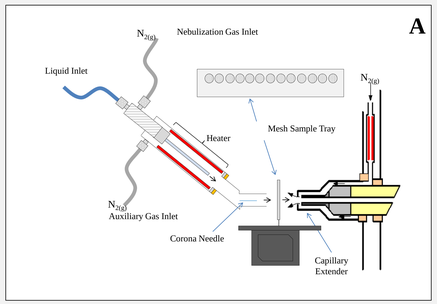


Figure 1. A) Schematic of the DSA ionization source. **B)** Overall view of the DSA attached to the AxION TOF. **C)** Close up view of the DSA with covers off **D)** Close up view of the DSA sample tray inlet to the AxION TOF.

4 Schlieren Study

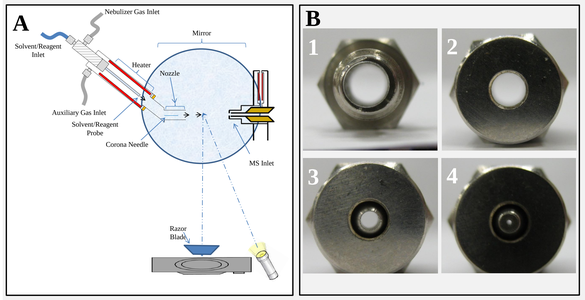


Figure 2. A) Schlieren experimental setup
B) Nozzle internal diameter 1) 4.8 mm 2) 3.2 mm 3) 1.5 mm 4) 0.5 mm

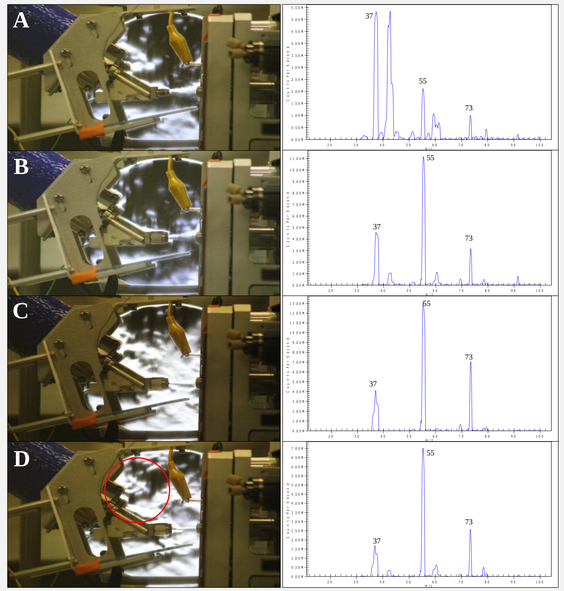


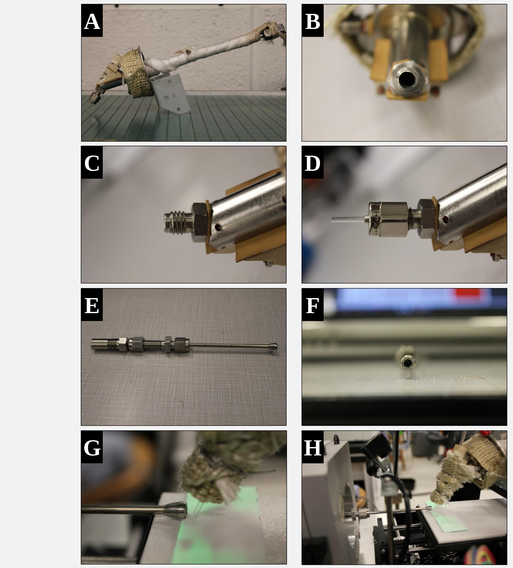
Figure 3. Schlieren photographs and corresponding water cluster mass spectra produced by each nozzle. A) Nozzle 1 B) Nozzle 2 C) Nozzle 3 D) Nozzle 4

Decreasing nozzle internal diameter produced higher intensity water clusters. Despite this trend the intensity decreased with the smallest nozzle. This decrease is attributed to gas leaks within the system (circled in red above).

Modifications made to Commercial Source

- New more rugged heater assembly
- Heater provides more gastight fit
- Smaller nozzle
  - Improved spatial resolution
  - Made of glass to prevent electrical arching
- Automated sample xy stage
- Manual vertical source adjustment
- Camera to monitor sample position
- 254/365nm UV lamp
- External high voltage and nitrogen control
- Longer capillary extension

5 Modified Source



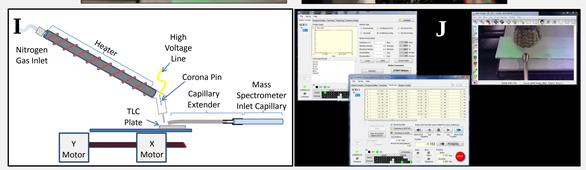


Figure 4. A) Modified source **B)** Nozzle adapter front view **C)** Nozzle adapter side view **D)** Adapted glass nozzle **E)** Capillary extender side view **F)** Capillary extender front view **G)** Capillary extender, nozzle and TLC plate on sample carrier **H)** Capillary extender, nozzle, TLC plate, camera **I)** Instrument schematic **J)** Camera view and computer software control

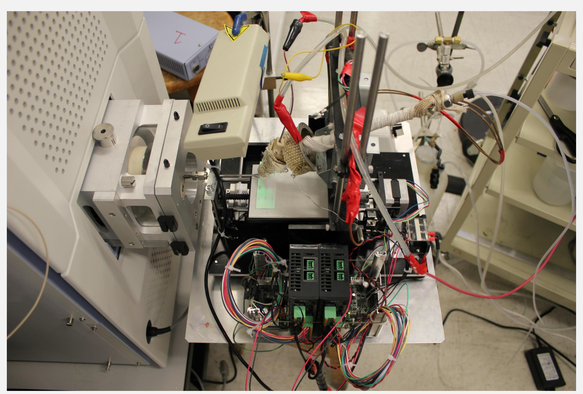


Figure 5. Complete assembly

6 TLC Plate Analysis

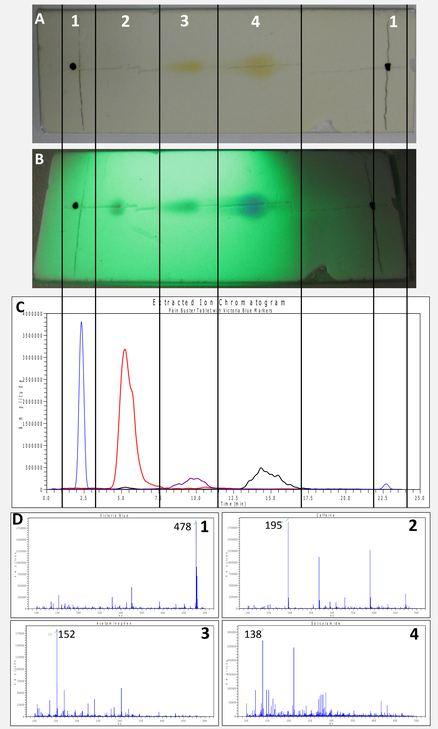


Figure 6. A) TLC plate under white light **1)** Victoria Blue dye from a blue Sharpie **2)** Caffeine (spot not visible under white light) **3)** Acetaminophen **4)** Salicalymide **B)** TLC plate under UV (254nm) light **C)** Extracted ion chromatogram of analyzed TLC plate. **D)** Mass spectra of each compound from the TLC plate

7 Conclusion

The initial desire to better understand the DSA led to a study using schlieren photography. This study illustrated how nozzle size can affect reagent ion spectra and gas flow patterns. The knowledge gained from the study assisted us in constructing a multidimensional platform for the analysis of TLC plates.

The new prototype platform successfully combines an automated sample stage with the modified source. TLC plates as well as other samples have successfully been studied using this configuration. The improvements to the heater and nozzle assembly reduce the amount of gas leaks and improves gas flow. The smaller nozzle provides more precise sampling over the stock design.

To fully utilize the potential of this system additional developments are required.

These developments include:

- More efficient heat transfer to the nitrogen gas
- Improve the gas flow path to further reduce leaks
- Develop more compact design
- Develop computer program to integrate all aspects of the software controls
- Improve capillary extension design to better capture desorbed ions

Patent Pending
2014-015WL