Analytical Detection by Liquid Phase Ion Mobility Spectrometry

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Ion Mobility Spectrometry

OVERVIEW

Purpose To develop a miniaturized liquid phase ion mobility spectrometry as a novel analytical separation technique

Method Liquid phase ion mobility spectrometry with electrodispersion ionization

Results Illustrated the capability of electrodispersion ionization and demonstrated mobility detection by liquid phase ion mobility spectrometry

INTRODUCTION

- Liquid phase ion mobility spectrometry (LPIMS) works on the same principle as gas phase ion mobility spectrometry (IMS), the method of choice for the rapid detection of drugs and explosives.
- In IMS, ions are separated based on the mobility in a medium, driven along by an external electric field. In LPIMS, a non-electrolytic liquid medium replaced the gas medium of IMS.
- Although ions diffuse three orders of magnitude slower in liquids than in gases, real-time ion detection can take place in a miniaturized LPIMS.
- Ionization in LPIMS was achieved by electrodispersion ionization (EDI), a recently developed ionization source.

METHODS

- The EDI Source consisted of a fused silica tubing (25 µm ID), a metal internal reducing connection, a syringe needle and a syringe pump. The EDI was operated in continuous and pulsed mode.
- The LPIMS consisted of a 5mm-long resistive glass tube (Burle Electro-Optics Inc., Sturbridge MA), external resistors and a Faraday plate (Figure 1). A miniature Bradbury-Nielsen gate was fabricated from Macor rings and positioned between two resistive glass tubes (Figure 2).
- Supporting electronics included current-limited high voltage power supply, two-way switches, current-to-voltage amplifier, and data acquisition system.
- Hexane and benzene were used as the liquid medium.
- The sample used was 10 µM Basic Fuchsin, a non-fluorescent dye, dissolved in methanol:water (9:1, v/v).

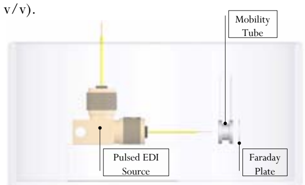


Figure 1. Schematic of pulsed electrodispersion ionization-liquid phase ion mobility spectrometer (pulsed EDI-LPIMS)

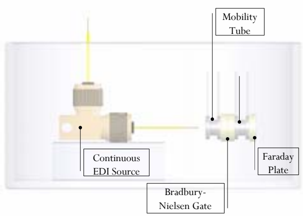


Figure 2. Schematic of continuous electrodispersion ionization-liquid phase ion mobility spectrometer (continuous EDI-LPIMS)

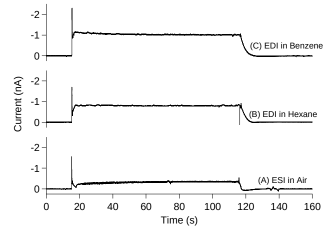
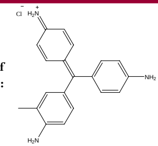


Figure 3. Total ion current response for 10 µM Basic Fuchsin in (a) air by ESI, (b) hexane by EDI and (c) benzene by EDI. ESI/EDI voltage was applied at ~15s and terminated at ~115 s.

Structure of Basic Fuchsin:



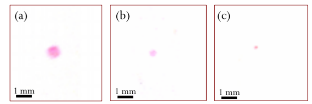


Figure 4. Corresponding photos of ionizing 10 µM Basic Fuchsin (a) by ESI in air, (b) by EDI in hexane and (c) by EDI in benzene.

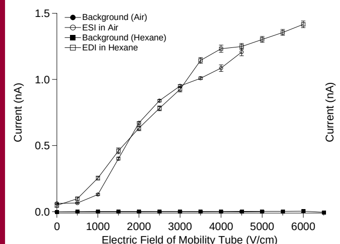


Figure 5. Plot showing total ion current of ESI and EDI increasing with electric field of the mobility spectrometer. The background current is shown for comparison.

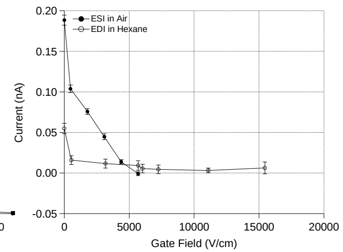


Figure 6. Bradbury-Nielsen Gate could stop ions in air, but not in hexane. The electric field of the mobility tube was 600V/cm.

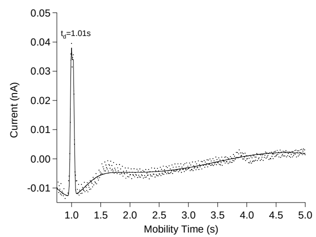


Figure 7. Pulsed electrodispersion ionization-liquid phase ion mobility spectrum of 10 µM Basic Fuchsin.

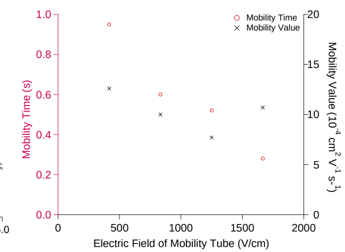


Figure 8. Plot showing that the mobility time of a solvent peak decreased with increasing electric field. The average mobility value was 10.3x10^{-4} cm^2 V^{-1} s^{-1}.

RESULTS

Electrodispersion Ionization

- EDI in hexane and in benzene was compared with ESI in air. It was observed that the total ion current of EDI in hexane and in benzene was relatively higher than that of ESI in air (Figure 3).
- By directing the EDI/ESI onto a piece of paper 5 mm away from the tip of the capillary, it was evident that EDI was capable of transferring ions through the medium used. Figure 4 showed the pink dye of Basic Fuchsin after ionizing by (a) ESI in air, (b) by EDI in hexane and (c) by EDI in benzene.
- The total EDI ion current increased with the electric field, reaching a maximum of approximately 1.4 nA at 6000V/cm in hexane (Figure 5).
- While arcing began at 5000V/cm in air, there was no arcing in hexane; although impurities of hexane underwent electrochemical oxidation at electric field above 6000V/cm.

Liquid Phase Ion Mobility Spectrometry

- While ESI ions in air were stopped by an orthogonal field of ~5500V/cm from the Bradbury-Nielsen gate, EDI ions in hexane leaked through an orthogonal field as high as 15,500V/cm, at which the gate wires showed signs of arcing (Figure 6).
- Figure 7 is the first reproducible liquid phase ion mobility spectrum of 10 µM Basic Fuchsin, obtained with a pulsed electrodispersion ionization source. The electric field was 1000V/cm and the ionization pulse was 500 ms.
- The mobility value of the Basic Fuchsin peak (Figure 7) was 5.08x10^{-4} cm^{2}V^{-1}s^{-1}. The peak width at half height was 62 ms and the resolving power was 16. The resolving power per unit drift length of the 5-mm mobility spectrometer was 32 cm^{-1}. In comparison, a typical 20-cm gas phase IMS has a resolving power of ~100, or a resolving power per unit drift length of 5 cm^{-1}.
- Figure 8 is the liquid mobility spectra of solvent ions at increasing electric field. The mobility time of the most intense peak decreased with increased electric field.

CONCLUSIONS

- Electrodispersion ionization was successfully developed for liquid phase ion mobility spectrometry, to ionize aqueous sample.
- Analytical detection by liquid phase ion mobility spectrometry was demonstrated with Basic Fuchsin and methanol-water.
- The 5-mm liquid phase ion mobility spectrometer had a better resolving power per unit drift length than a typical 20-cm gas phase ion mobility spectrometer.

ACKNOWLEDGEMENT

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