



# Laser desorption-ion mobility spectrometry as a useful tool for imaging of thin layer chromatography surface



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## ARTICLE INFO

### Article history:

Received 11 March 2016

Received in revised form 18 June 2016

Accepted 22 June 2016

Available online 23 June 2016

### Keywords:

Thin layer chromatography

Ion mobility spectrometry

Laser desorption

Explosives

## ABSTRACT

We present a novel method for coupling thin layer chromatography (TLC) with ion mobility spectrometry (IMS) using laser desorption technique (LD). After separation of the compounds by TLC, the TLC surface was sampled by the LD-IMS without any further manipulation or preparation. The position of the laser was fixed and the TLC plate was moved in desired directions by the motorized micro-positioning stage. The method was successfully applied to analyze the TLC plates containing explosives (tri nitro toluene, 1,3,5-trinitro-1,3,5-triazacyclohexane, pentaerythritol tetranitrate, 2,4-dinitro toluene and 3,4-dinitro toluene), amino acids (alanine, proline and isoleucine), nicotine and diphenylamine mixtures and detection limits for these compounds were determined. Combination of TLC with LD-IMS technique offers additional separation dimension, allowing separation of overlapping TLC analytes. The time for TLC sampling by LD-IMS was less than 80 s. The scan rate for LD is adjustable so that fast and effective analysis of the mixtures is possible with the proposed method.

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## 1. Introduction

TLC is a simple, fast and inexpensive chromatography technique which is used for separation of mixtures of chemical and biochemical compounds [1]. Traditionally, the analyte spots on the TLC plate are visualized by chemical and optical methods such as staining techniques and dyeing reagents [2–4] ultraviolet (UV) absorption and fluorescence spectroscopy [5].

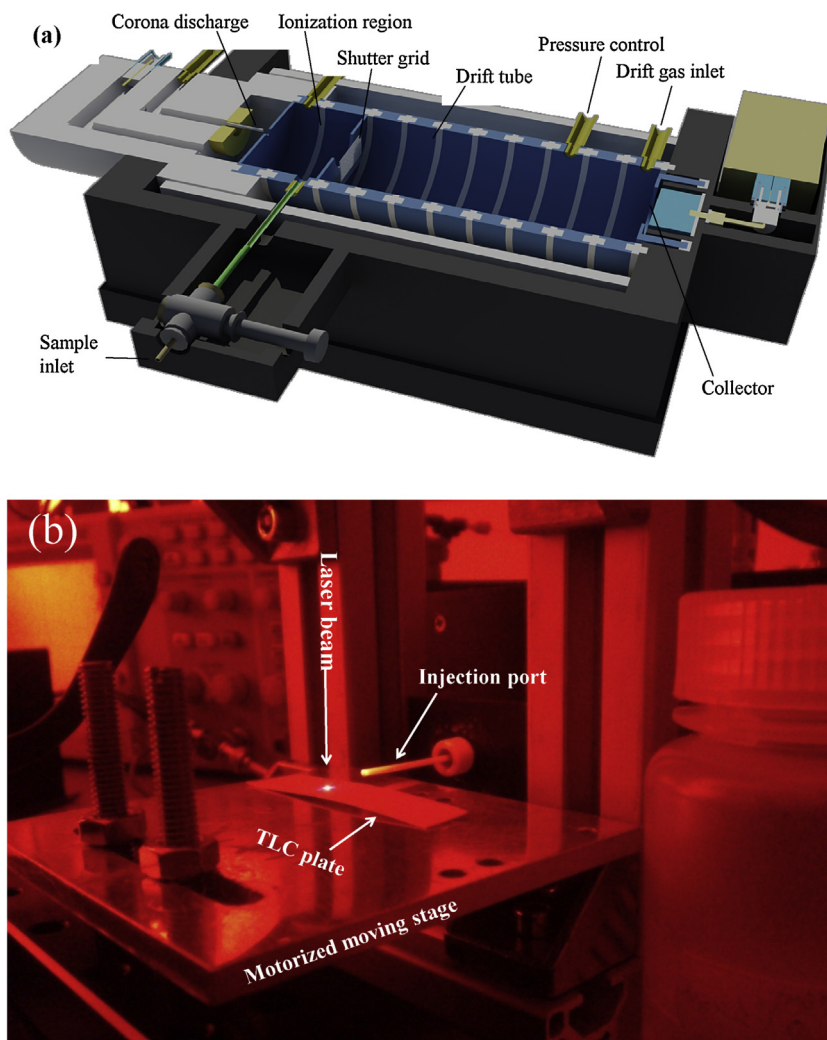
The traditional visualization methods have some limitations in the characterization of the structure of the compounds on the TLC plate. Furthermore, detection of the analyte on the TLC by the traditional methods strongly depends on the optical and chemical properties of the analytes. Therefore, considerable attempts have been made to develop coupling of TLC technique with other standard analytical techniques. Mass spectrometry (MS) with different ionization sources is a powerful technique for detection and characterization of vast spectrum of chemical compounds. In TLC, the analyte is deposited on the plate, therefore, the ana-

lyte should be sampled from the TLC surface and gasified in order to be detected by MS. Coupling of TLC with MS has been carried out using different techniques and ionization sources such as laser desorption ionization (LDI) [6–9] secondary ion mass spectrometry (SIMS) [10] matrix-assisted laser desorption ionization (MALDI) [11–15] surface-assisted laser desorption ionization (SALDI) [16,17] electrospray ionization (EI) [18–21] electrospray laser desorption ionization (ELDI) [22,23] laser-induced acoustic desorption electrospray ionization (LIAD/ESI) [24] laser desorption/inductively coupled plasma (LD/ICP) [25,26] and so on. Also, different mass spectrometers including time of flight (TOF) ion trap and quadrupole are used to detect and analyze the analytes sampled from the TLC surface. Usually, the mass spectrometry methods are expensive and require vacuum generation systems.

IMS is a fast, cost-effective and simple technique with high sensitivity which works at atmospheric pressure. Furthermore, a vast spectrum of compounds can be detected by IMS [27–32]. The development of coupling methods of TLC with IMS is of high interest due to the simplicity and sensitivity of IMS technique. We reported in our previous work [33] coupling of TLC with ion mobility spectrometry (IMS) by a variety of schemes such as electrospray, scraping or cutting pieces of TLC into the IMS injection port. They were successfully applied for separation and detection of alkaloids. However

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**Fig. 1.** (a) 3D model of IMS instrument used in this work and (b) the photograph of the experiment set up for the TLC-IMS coupled by LD.

special handling of TLC plates was required in above mentioned techniques.

In this work, we will for the first time introduce and explain coupling of TLC with IMS using LD [34]. This technique is suitable for a fast and reliable TLC analysis without any special TLC handling. We demonstrate the performance of the method for separation and detection of explosives, amino acids and narcotics. The results are very promising and we believe that the technique has potential to be implemented in medical, biological and chemical applications where the TLC is widely used.

## 2. Experiment

### 2.1. Ion mobility spectrometer

The Corona Discharge (CD) ion mobility spectrometer (IMS) used in this work was developed at Department of Experimental Physics Comenius University in collaboration with MaSa Tech. Company. Fig. 1a shows schematically different components of the IMS. The detailed description of the instrument can be found elsewhere [35]. The CD ion source has capability to work in both negative and positive modes. In the positive mode, the CD ion source produced reactant ions such as  $\text{H}_3\text{O}^+$ ,  $\text{NO}^+$  and  $\text{NH}_4^+$  (with dopant gas) and the analyte is ionized via proton transfer from

the reactant ions to the analyte (M). In the negative mode,  $\text{O}_2^-$ , or  $\text{Cl}^-$  (with dopant gas) reactant ions were generated in CD and  $\text{M}^-$  or  $\text{M.Cl}^-$  ions were produced after injection of the sample into the ionization region. The produced ions were pulsed into the drift tube by a shutter grid with a pulse width of  $30\ \mu\text{s}$  and frequency of 50 Hz. The ions were separated in the drift tube on the basis of their ion mobilities; therefore, the ions reach the collector at different drift times. Purified air was used as a drift gas at flow rate of 700 mL/min. The IMS is able to work at atmospheric as well like at subatmospheric pressure. During this experiment it was operated at the pressure of 0.5 bar in order to suck the sample into the IMS through the sample inlet capillary. The sample gas flow was set to 600 mL/min and the IMS cell was operated at  $73^\circ\text{C}$ .

### 2.2. Materials and method

Tri nitro toluene (TNT), 1,3,5-trinitro- 1,3,5-triazacyclohexane (RDX) and pentaerythritol tetranitrate (PTEN) were obtained from Ministry of defense (Bratislava, Slovakia). 2,4-Dinitro toluene (2,4-DNT), 3,4-dinitro toluene (3,4-DNT (97%)), proline and isoleucine were purchased from Sigma-Aldrich. Methanol (99.5% v/v), n-hexane, ethyl acetate and chloroform were Sigma products and used as the solvent and the mobile phase.

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