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Characterization and validation of ion mobility spectrometry in methamphetamine clandestine laboratory remediation

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ABSTRACT

This project evaluated the efficacy of ion mobility spectrometry (IMS) as a tool for determining remediation success at clandestine methamphetamine laboratory sites. Specifically, limits of detection (LOD), limits of quantitation (LOQ), and matrix effects were investigated as relevant to typical remediation sites and situations. The recoveries of pseudoephedrine and methamphetamine from a range of various surfaces likely to be found in a clandestine laboratory were examined. Portable IMS instruments with thermal desorption were found to be a reliable tool for evaluating the degree of remediation if sufficient procedural and instrumental controls are put into place. In general, detection limits were in the same range as state guidelines as well as laboratory methods using GC/MS and LC/MS. Direct vapor sampling can be used to detect high levels of methamphetamine and potential interferences, but cannot approach the detection limits needed for evaluation of remediation efforts. IMS cannot be used alone to determine the efficacy of remediation efforts; final confirmation using laboratory instrumentation is essential. For the purpose of this study, typical field settings of the IMS were used and the conditions were not optimized.

1. Introduction

The immediate and long-term hazards associated with clandestine methamphetamine laboratories are well known and significant efforts have been directed towards developing reasonable standards for the remediation of clandestine methamphetamine sites [1–15]. Regardless of the synthetic method used to produce methamphetamine, the clandestine laboratory site is typically highly contaminated and requires either demolition or extensive remediation. A recent Federal statue has addressed some of the issues related to remediation and how to gauge if a clean-up has indeed been successful and if a site is safe for re-habitation [13]. In 2005, the United States Drug Enforcement Administration (DEA) published a manual entitled *Guidelines for Law Enforcement for the Clean-up of Clandestine Drug Laboratories* that describes protocols and procedures, but not specific clean-up methodologies [14]. The Environmental Protection Agency (EPA) has drafted guidelines for clean-up, but this document has not yet been released in final form. Various states have adopted different acceptable levels of residual methamphetamine which range from 0.1 to 0.5 μg/100 cm^2 as summarized in the DEA manual [13]. According to the California Study, levels in some states may be as high as 1 μg/100 cm^2 [16]. The quantities refer to the area of a given surface that has been sampled, typically by swiping followed by field or laboratory analysis.

Ion mobility spectrometry (IMS) is frequently used for rapid field evaluation for a variety of compounds [17]. IMS has been deployed as a rugged and reliable field sensing system for chemical warfare agents since the 1980s [18]. It has also found significant use for the detection of explosives, monitoring of environmental compounds, and a drug detection system [19]. More recent applications of IMS include pharmaceutical quality control, verification of the cleaning of pharmaceutical equipment surfaces, pharmaceutical process analysis, and determination of active pharmaceutical ingredients [20–23].

IMS operates at atmospheric pressure and separates ionized analytes as ions and ion/molecule clusters (Fig. 1). With thermal desorption instruments, such as used here, samples are deposited on a Teflon® membrane filter, which are then vaporized by the desorber heater. Ionization occurs from thermal electrons emitted from a ^{63}Ni beta-ray source. The product ions are then gated into a drift region for mobility analysis. Under the influence of an electric field gradient and against the counterflow of a drift gas, the ions move toward the collector plate.

The ion mobility constant, K ($cm^2\ V^{-1}\ s^{-1}$), is used to identify the analyte from the observed ion peaks. Ion mobility constants are calculated according to Eq. (1) [29]:

$$K = d/tE \quad (1)$$

where d is the distance an ion will travel in the measured time (t) under the electric field (E).

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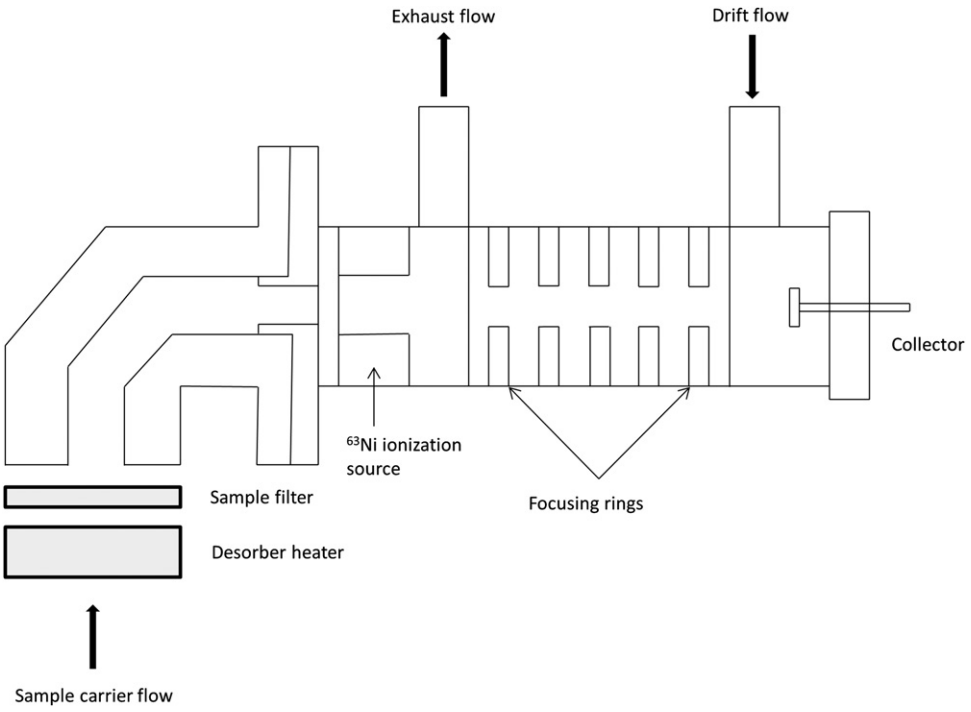

Fig. 1. Schematic representation of IMS, adapted from [24].

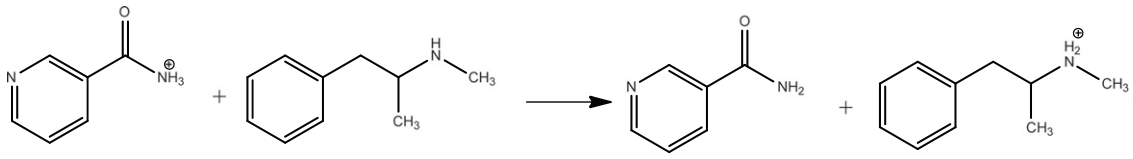

Fig. 2. Formation of protonated methamphetamine from NTA and methamphetamine.

The drift times required by the ions to reach the collector electrode are generally proportional to their masses, but inversely proportional to their reduced ion mobilities K_o ($cm^2\,V^{-1}\,s^{-1}$). The reduced mobility constant compensates and standardizes for pressure and temperature towards standard conditions, as shown in Eq. (2) [29]:

$$K_o = (d/t_d E)\,(273/T)\,(P/760) \quad (2)$$

where d is the length of the drift region (cm), t_d is the time it takes the ion to travel the distance d (s), E is the applied electric field ($C\,cm^{-1}$), T is the temperature of the buffer gas (K), and P is the pressure in the drift region (Torr).

IMS can be operated in the positive or negative mode. For this study, IMS was operated in the positive mode, which is the mode used for drug detection. In this mode, the drift gas contains nicotinamide (NTA) used as both a calibrant and a reactant. In the reaction region, the protonated NTA transfers a proton to the sample molecule, M, as shown in Eq. (3) [29]:

$$[NTA]H^+ + M \rightarrow NTA + [M]H^+ \quad (3)$$

This reaction only proceeds if the proton affinity of the sample molecule is greater than that of the NTA. Methamphetamine responds in a similar fashion, as shown in Fig. 2. The principles and background of IMS has been extensively described elsewhere [17–29].

IMS instrumentation offers many advantages for field use including atmospheric pressure ionization, small instrument size (many commercial hand-held units are available), and low power requirements. Field units can be programmed to respond to the appearance of drift peaks in given drift time windows corresponding to the mobility peaks of target compounds. However, such responses are not unique in that these mobility channels correlate to drift times, cross-sectional areas, and mass-to-charge ratios and not to specific compounds. This can generate false positives, which may result from a number of factors including poor desorption from substrates, low concentration, or competing ion/molecule reactions. A key goal of this study was to identify the strengths and limitations of IMS on specific, but critical field deployment. Lessons learned here can be extended to other field applications.

IMS is frequently used for screening at clandestine laboratory sites and for the detection of methamphetamine [17,25]. Several papers have demonstrated methods of detecting methamphetamine in the presence of nicotine and cigarette smoke which are common interfering compounds seen at clandestine laboratory sites [26]. Accordingly, there is a strong theoretical and practical basis for employing IMS in the context of clandestine laboratory remediation. The goal of this work is to determine the performance limits of detection for residual methamphetamine at remediated clandestine laboratory sites.

2. Materials and methods

2.1. Reagents

For sample preparation (standards of methamphetamine and pseudoephedrine), LC/MS-grade methanol (Fluka/Sigma Aldrich,