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# Enhancing capabilities of aspiration-type Ion Mobility Spectrometer using a Pulsed Sampling System and a heated transfer line

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ABSTRACT

An aspiration-type Ion Mobility Spectrometer (IMS) was connected to an in-house developed Pulsed Sampling System (PSS) through a heated capillary column acting as transfer line, after being modified to a closed loop IMS device. Performance of the whole system was evaluated using acetone gas standards in the concentration range of 200–2000 ppb. Tests were carried out using an in-house developed chemical environment generator for producing acetone concentrations. The system presented long-term stability, linearity, sensitivity and low minimum detectable level; less than 286 ppb for acetone with a closed loop that circulates 1300 mL/min of purified air. Repeatability (RSD) was less than 15% for all concentrations. All data were produced in dry atmosphere inside the IMS; presence of moisture (e.g. when saturation of molecular sieves occurs) seems to severely reduce performance.



## 1. Introduction

Ion mobility spectrometry (IMS) is an established field (on-site) method for detection of chemical warfare agents, explosives and drugs [1]. Applications also exist in environmental monitoring, health, industrial and biomedical areas [2–7]. IMS is based on the ionization of the sample, the formation of ion swarms at ambient pressure and temperature and their separation based on their different velocities under a voltage gradient and a drift gas [1]. It is a highly sensitive technique with fast response. IMS instruments are lightweight, operate at ambient pressure, they have small size, low cost and portability and can be used for real-time monitoring. In order to overcome certain limitations, such as, limited selectivity, low resolving power, cross sensitivities, saturation of signal, matrix effect, low reproducibility due to variation of moisture and temperature, as well as, complicated logistics, paper work and licenses due to radioactive sources, various IMS designs have been proposed over the last two decades [8].

Various types of IMS exist (e.g. conventional IMS, Field Asymmetric IMS, aspiration IMS) [1,8]. These allow for different levels of performance, efficiency and suitability for specific applications. In aspiration IMS ionization occurs at ambient pressure, temperature and humidity, usually through a radioactive source ($^{241}Am$). Ions travel through an orthogonal electric field and they are deflected to multiple plates (channels). Depending on the polarity of the electric field which alternates continuously, positive and negative ions can be detected simultaneously. Ions with higher mobility deflect to the first plates. Signals of the plates produce characteristic pattern for each compound [8]. Aspiration-type IMS is known for simplicity and high speed of analysis, but also for limited resolution [9,10].

In the particular aspiration-type IMS design used in this work ambient air is sampled and ionized and the ions are deflected by an electric field into a tube with 16 plates; 8 upper and 8 lower plates acting as detectors (Fig. 1). The advantages of the aspiration-type IMS include: ion mobilities are recorded constantly because there is no shutter, there is usually no use of membranes as inlet systems, there is continuous operation, detection levels are as low as ppb or ppt, there is very high sensitivity (the sensitivity is easily adjusted because its ionization efficiency is flow rate dependent) and potentiality for miniaturization. Limitation of aspiration-type IMS is the low resolution due to inlet geometries, number and width of channels and diffusion broadening. Reproducibility and sensitivity can be affected by the lack of control of sample moisture.

One important component of field setup is the sampling system as a front-end part of the IMS. This component might affect sample integrity, it may protect from moisture, dust and saturation, it may also affect performance and efficiency, especially

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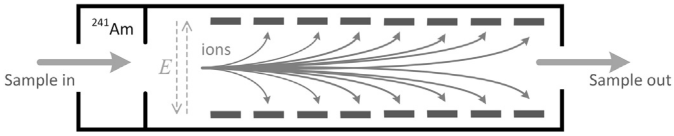


**Fig. 1.** The principle of operation of the Aspiration-type IMS.

dynamic response. More broadly speaking, the inlet system can allow for sample pre-concentration and separation. Different types of inlet systems include membrane-based systems and exponential and dynamic dilution [11–13]. Membrane-based systems have the advantage of strongly limiting amount of water in the sensor but they have limitations such as reduced sensitivity, low dynamic response and poor selectivity for non-polar compounds.

In this paper a commercial aspiration-type IMS that was originally used with a direct interface with ambient air has been connected with an in house developed Pulsed Sampling System (PSS) through a heated transfer line [14–18]. The key idea for this new set-up was to enhance sampling capabilities of the aspiration-type IMS with the scope of providing protection against saturation and very fast dynamic sampling together with the potentiality of chromatographic separation; use of heated, short (up to 2 m) chromatographic capillary column as transfer line. This enhancement can address the limited resolution. Additionally, a closed loop was used with the aspiration-type IMS that is expected to significantly control moisture and through that the chemistry of the ion-molecules collisions. With a closed loop it is expected that moisture inside the IMS cell is very low and stable favoring both sensitivity and spectral stability. PSS can control the amount of sample in the IMS cell and consequently the dynamic range. Moreover, PSS is a universal inlet system compared to the membrane inlets which are selective. This paper focuses on presenting the performance of the system in terms of time of response, minimum detectable level, linearity, reproducibility and sensitivity.

## 2. Materials and methods

### 2.1. Materials

The following materials were used: Acetone, Analytical reagent grade by Fisher Scientific, Molecular Sieves 13X in the form of beads (8–12 mesh) by Sigma–Aldrich and zero air by Air Liquide.

### 2.2. Instrument and devices

The whole system consists of an in-house developed Pulsed Sampling System (PSS) connected with Environics aspiration-type IMS (ChemproPD) through a heated transfer line (Fig. 2).

#### 2.2.1. Pulsed Sampling System

The following terms are used in this work:

*Sampling time*: Time period during which the sampling system allows the intake of ambient air to the detector; *Idle time*: Time period during which the sampling system cuts off the intake of ambient air and introduces a flow of purified air or other barrier gas in the detector; *Cycle*: The sum of sampling time and idle time; *Cycles repetitions*: How many times a cycle is repeated; *Measurement session*: Time period of repetitions.

The PSS principles of operation are described in detail in [17,18]; it periodically introduces air samples (pulses) into the IMS. Between two air samples there is a period of introduction of purified air that provides the necessary time for running the analysis. The schematics of the whole system in the two modes of operation (sampling and idle mode) are presented in Fig. 2. The main body of the PSS consists of three coaxial tubes which are connected through ports A, B and C to a pneumatic system including pumps, flowmeters and valves. It should be noted that PSS was modified for the current work by adding a casing of molecular sieves in pneumatic system that allows for purified ambient air to be introduced into the IMS during the idle mode operation. The inner tube is a heated capillary column which introduces a portion of the sample into the IMS device. The outer tube is continuously subjected to negative pressure through Port A for forcing the sample into the system. The intermediate tube has ports B and C, which are activated according to the mode of operation; in idle mode port C is activated and allows purified air to fill the intermediate tube, in sampling mode port B is activated and subjected to negative pressure in order to force the sample into to intermediate tube. Hence, in sampling mode the sample is forced into the outer and the intermediate tubes and a portion of it travels across the transfer line and enters the IMS. In idle mode, purified air entering via port C, forces the sample out of the intermediate tube and travels across the transfer line into the IMS. The sampling and idle time can be adjusted for each application. The alteration between idle and sampling mode can be either manual or automated according to a sampling protocol.

#### 2.2.2. Closed loop IMS

Environics aspiration-type IMS (ChemproPD) was modified to become a closed loop system by using two valves, a casing with

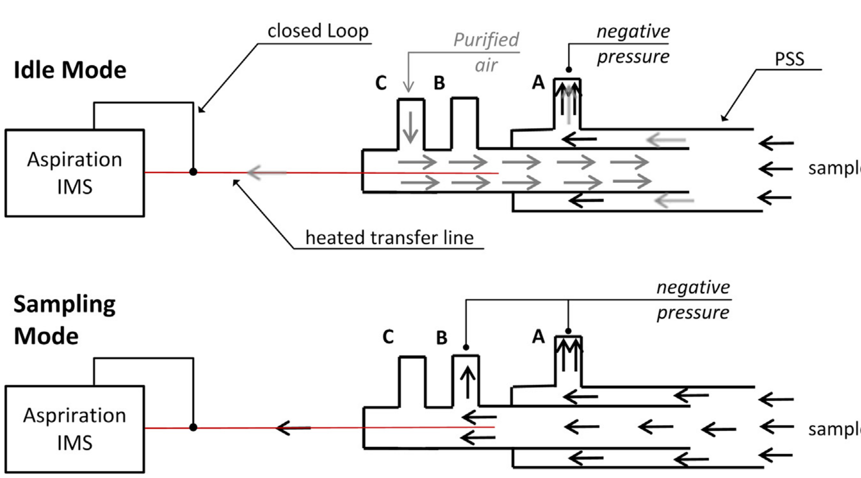


**Fig. 2.** System set-up with PSS in idle (upper diagram) and sampling (lower diagram) mode of operation.