Paper 38

STUDIES ON THE VAPOR-PARTICULATE PHASE DISTRIBUTION OF ENVIRONMENTAL NICOTINE BY SELECTED TRAPPING AND DETECTION METHODS

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Slide 1 Title and Names (Introduction)

By definition, environmental tobacco smoke (ETS) is an aerosol composed of sidestream and exhaled mainstream smoke. The mainstream fraction of total ETS can usually be ignored as it is small compared to the contribution of sidestream smoke. An abundent and unique component of ETS is nicotine. Approximately 3-4 miligrams of nicotine are released in the environment by a burning cigarette. Consequently, nicotine has been used by many workers as a marker for ETS exposure.

The exact physical behavior of environmental nicotine ,i.e., the distribution between vapor and particulate phases, is not well understood. A recent review article stated that sidestream nicotine exists primarily in the gas phase as the free base due to the alkaline nature of the smoke. However, there have been other conflicting reports suggesting that environmental nicotine is mainly in the particulate phase or evenly distributed between the two phases.

The research which I will present has been directed toward two objectives: firstly, to develop methods to characterize ETS, and secondly, to better define the physical behavior of environmental nicotine. We have taken the approach of using selective trapping and detection methods to meet these objectives.

Slide 2 List of Selective Traps and Detectors

The selective traps used in this work were: Cambridge filter pads, polymeric adsorbents, denuder tubes, and an electrostatic precipitator. The selective detectors employed were: TAGA MS/MS, GC-NPD, GC-FID, and a piezoelectric mass balance. Traps were connected singly or serially in different configurations to collect vapor phase nicotine and/or ETS. ETS samples were obtained from the environmental chamber or a 2'x2'x3' plexiglas box.

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Slide 3 Comparison of XAD-4 and TAGA MS/MS Sampling of ETS Nicotine

This slide compares ETS data obtained in our laboratory by two different instrumental techniques. The upper plot shows nicotine levels versus time as determined by the NIOSH method. This method employs adsorption on a polymeric resin (XAD-4), desorption with ethylacetate, and subsequent analysis by gas chromatography with a nitrogen specific detector (GC-NPD). A personal sampling pump draws sample at a flowrate of 1-1.5 l/min.

In the lower plot, nicotine was monitored in real-time by the TAGA MS/MS at m/e 163, the protonated parent ion (MH^+). Both techniques sampled ETS from the environmental chamber. ETS was generated by smoking two cigarettes in the chamber under standard smoking conditions (35 ml puff volume and 2 sec puff duration). The nicotine decay curves are very similar and the maximun peak concentrations are within 5%.

In the two previous talks, Dave Heavner and Fred Thome described in detail the environmental chamber and TAGA MS/MS, respectively. Briefly, the chamber is a 18 cubic meter stainless steel room capable of six different modes of operation. All the experiments which I will describe were performed with the chamber in the static mode, i.e., no recirulation or make-up. The major advantages of the TAGA MS/MS are real-time monitoring of gaseous compounds at ppb-ppt levels. It is worth repeating that the TAGA only "sees" compounds in the gas phase; particulate phase materials are not detected.

Slide 4 Nicotine Diffusion Tube Apparatus

This slide shows a thermostated diffusion tube device which provides a source of gas phase nicotine. The output of the diffusion tube was monitored weekly by the NIOSH GC method and a value of 5.48 ug/min gas phase nicotine was determined. The output was also monitored by a piezoelectric mass balance and a condensation nucleus counter (CNC) to detect particle mass and number, respectively. No particles were detected. A standard source of gas phase nicotine was then available to calibrate the TAGA.

Slide 5 Schematic Diagram of Diffusion Tube- Adsorbent-TAGA Experiment

This is a schematic diagram of experiments performed to evaluate several adsorbents which are commonly used to trap environmental nicotine.

Slide 6 TAGA MS/MS Monitoring of Diffusion Tube Nicotine

This slide presents a plot of ion intensity versus time obtained by sampling gas phase nicotine fron the diffusion tube apparatus.

Slide 7 Comparison of Three Nicotine Adsorbents

The results of diffusion tube nicotine trapping of three adsorbents, Uniport-S, Tenax, and XAD-4 are shown in this slide. The tubes were constructed with approximately 70-80 mg adsorbent each. The XAD-4 resin is more efficient at trapping vapor phase nicotine. No breakthrough was observed after 60 min for the XAD sorbent. In contrast, Tenax and Uniport-S showed considerable vapor phase nicotine breakthrough. It is important to note that the Cambridge filter pad will also trap gas phase nicotine and exhibits a breakthrough profile similar to Tenax (not shown in slide). The trapping efficiency of any sorbent is dependent upon flowrate, concentration, and the chemical properties of the compound of interest. However, based on a weight-to-weight basis, XAD sorbent is superior to Tenax, Uniport-S, and the Cambridge filter pad for trapping gas phase nicotine.

Slide 8 Schematic Diagram of ETS- Adsorbent- TAGA MS/MS Experiment

This is a schematic diagram of experiments conducted to evaluate the ETS trapping efficiency of the XAD-4 adsorbent and the Cambridge filter pad. The TAGA samples from the exhaust of a bellows pump.

Slide 9 Schematic Diagram of ETS- Adsorbent Breakthrough Experiment

This slide presents a schematic diagram of experiments which were done to detect if any nicotine breakthrough occurs during sampling of ETS. Interestingly, the XAD resin removes only 20% of the ETS particulate mass and 55% of the number of particles. A Cambridge filter pad or another XAD sorbent tube was placed downstream from the first tube to trap possible particulate phase nicotine, if in fact it exists. No nicotine was detected in downstream traps.

Slide 10 Comparison of Filter Pad and XAD-4 Trapping of ETS Nicotine

A comparison of the trapping efficiency of XAD sorbent and the Cambridge filter pad is shown. Two cigarettes were smoked in the chamber and ETS nicotine was monitored by the TAGA. Approximately 20 min after lighting, nicotine breakthrough occurs from the filter pad. No nicotine breakthrough is observed after two hours from the XAD sorbent.

Slide 11 Borgwaldt Electrostatic Precipitator

The Borgwaldt electrostatic precipitator is shown in this slide. The instrument requires a high voltage power supply and operates at 5-24K volts.

Slide 12 **Block Diagram of Electrostatic Precipitator Coupled to a Plexiglas Chamber**

This is a block diagram of the electrostatic precipitator coupled to a 2'x2'x3' plexiglas box. Both sidestream and mainstream trapping experiments were carried out. Smoking is performed in an enclosed vessel and sidestream smoke is forced by positive pressure into the box. A small fan provides adequate sample mixing.

When the precipitator was connected in series with the mainstream pad, more than 95% of the nicotine was trapped in the tar. This was expected as mainstream nicotine is a salt and is associated with the particulate phase. However, when the precipitator is positioned to sample sidestream smoke (or ETS), the results are completely opposite. The precipitator had no effect on sidestream nicotine. The nicotine trapped by XAD resin tubes (#4) were equivalent for both "on" and "off" experiments.

Slide 13 **Block Diagram of a Vapor Phase Nicotine - Electrostatic Precipitator Experiment**

A block diagram of the electrostatic precipitator in series with the diffusion tube apparatus and the TAGA is presented in this slide.

Slide 14 **Effect of Electrostatic Precipitator on Diffusion Tube Nicotine**

The electrostatic precipitator had no effect on the standard gas phase nicotine.

Slide 15 **Effect of Electrostatic Precipitator on ETS Nicotine**

Runs 1 and 2 are replicate smoking experiments with ETS nicotine being monitored by the TAGA. The electrostatic precipitator remained off for the duration of run 1, but was activated at approximately 18 min in run 2. ETS nicotine as detected by the TAGA was not distrubed by the electric field.

Slide 16 **Block Diagram of Vapor Phase Nicotine - Denuder Tube- TAGA MS/MS Experiment**

A denuder tube is a long, hollow tube coated with some material which efficiently removes a particular vapor phase component from an aerosol as it flows through the tube. Diffusing gas molecules collide with the denuder tube wall and are irreversibly adsorbed, while the aerosol particles pass through undisturbed. This slide shows a schematic diagram of a denuder tube in series with the diffusion tube nicotine source. The denuder tube was constructed from a 1/4'' glass tube and coated with phosphoric acid- UCON 5100 stationary phase. A flowrate of <1 l/min (as predicted by the Gromley-Kennedy equation) was required to remove 99% of the vapor phase nicotine and still allow 99% of the particles to pass through the tube.

Slide 17 **Denuder Tube Trapping of Diffusion Tube Nicotine**

The denuder tube efficiently removes gas phase nicotine as detected by the TAGA.

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Slide 18 Block Diagram of ETS- Denuder Tube Experiment

A similar denuder tube experiment was performed to sample ETS.

Slide 19 Denuder Tube Trapping of ETS Nicotine

The denuder tube traps the majority of environmental nicotine as detected by the TAGA.

Slide 20 Denuder Tube Sampling- Particle Mass Detection of ETS

A similar experiment was performed with a piezoelectric mass monitor replacing the TAGA as the detector. ETS was sampled for 5 min through the denuder tube, the 3-way valve was switched, and the sample passed through the blank glass tube. As shown by the plot of mass versus time, a "normal" ETS curve was obtained. The denuder tube removed the ETS nicotine but allowed the particles to pass unobstructed.

Slide 21 Denuder Tube- XAD Trapping of ETS

A final plot shows the results of XAD trapping of ETS with the denuder tube connected in series. The upper curve is derived from direct XAD sampling of ETS. In the next curve (noted by circles), a blank glass tube was placed in series. The lower curve represents data obtained by sampling through the denuder tube. It is obvious that the blank glass tube behaves like a denuder tube toward nicotine. The denuder tube curve (triangles) is flat over the 120 min run. As in the previous denuder tube experiments, nicotine is essentially 100% removed by the denuder tube. This is further evidence that ETS nicotine is in the vapor phase.

Slide 22 Conclusions (no slide)

I have described several methods which we use to study ETS and detect selective components. I have also presented work directed at studying the physical behavior of environmental nicotine. To summarize these results:

* XAD sorbent efficiently traps gas phase nicotine
* Cambridge filter pads trap gas phase nicotine, but less efficiently than a XAD resin tube
* XAD sorbent efficiently traps ETS nicotine
* Electrostatic precipitator traps MS nicotine but has no effect on vapor phase or ETS nicotine
* Denuder tube traps approx. 100% vapor phase and ETS nicotine

These results support the hypothesis that most of the nicotine (probably 95-100%) is in the vapor phase of ETS.