Determination of Gas-Phase Carbonyls in E-cigarette Aerosol Using a Sorbent Tube vs. Impinger Collection

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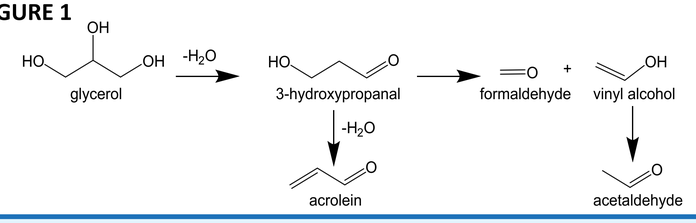
INTRODUCTION

- Low levels of thermal degradation products such as carbonyls (formaldehyde, acetaldehyde, and acrolein) have been reported in e-cigarette aerosols[1-6]
- Glycerol and propylene glycol can form carbonyls under thermal conditions [(oxy)dehydration][7] (Figure 1)

FIGURE 1

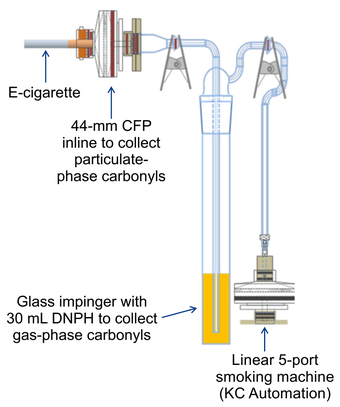


- A rapid, selective, sensitive method using UPLC-MS for measuring carbonyls in e-cigarette aerosols has been developed and validated (Figure 2)
- The use of Cambridge filter pads (CFPs) and impingers are common for traditional cigarette smoke collection techniques

TABLE 1 Puffing Regime

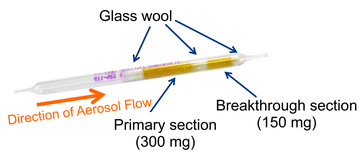
Puff Volume (mL)	Duration (seconds)	Collections	Interval (seconds)	Puff Profile
70	4	Five 20 puff collections	30	Square wave

FIGURE 2
Method 1 Collection Configuration



- Environmental air sampling techniques typically involve use of sorbent tubes for collection of gas-phase carbonyls (EPA method)[8]

FIGURE 3
DNPH SKC Sorbent Tube



OBJECTIVE

To evaluate a sorbent tube collection regime as an alternative to the traditional impinger approach for the collection of gas-phase formaldehyde

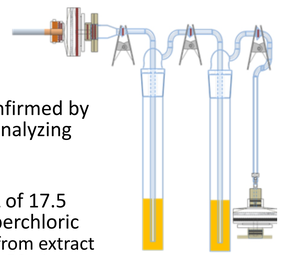
ABSTRACT

The aerosols generated from e-cigarettes are primarily composed of fine particles of liquid and gas phases of the vaporized e-liquid. Low levels of thermal degradation products such as carbonyls (e.g., formaldehyde, acetaldehyde, acrolein, and crotonaldehyde) have been reported in e-cigarette aerosols. A rapid, selective and sensitive method specific to measuring carbonyls in e-cigarette aerosols using UPLC-MS has been developed. This method was optimized for aerosol collection using a 44-mm Cambridge filter pad (CFP) followed by an impinger containing acidified 2,4-dinitrophenylhydrazine (DNPH) to capture both liquid and gas phase carbonyls, respectively. While the use of CFPs and impingers are common for traditional cigarette smoke collection techniques, environmental air sampling techniques typically involve the use of sorbent tubes (e.g., DNPH impregnated silica) for the collection of gas phase carbonyls as described in the U.S. Environmental Protection Agency (USEPA) Compendium Method TO-11A. Therefore, this collection regime was evaluated as an alternative to the traditional impinger approach for gas phase collection. It was demonstrated that both methods are suitable for the collection of gas phase carbonyls in e-cigarette aerosols and they show equivalent trapping efficiencies. For 20 puff collections, it was observed that approximately 70% of the formaldehyde is trapped in the liquid phase on the CFP and approximately 30% is trapped in the gas phase by either the sorbent tube or the impinger. The sorbent tube collection had one major limitation. The tubes had inconsistent packing densities which could restrict air flow, thereby altering the puff volume. While there are no puff volume issues using the impinger method, sorbent tubes must be pre-selected based on packing density prior to aerosol collection.

METHOD 1: CFP + Glass Impinger + DNPH

- Aerosol was collected using 44-mm CFP in front of glass impinger containing 30 mL of 17.5 mmol/L DNPH in acetonitrile with 27.3 mM perchloric acid (Figure 2)
- Mass changes (post-collection mass minus pre-collection mass) were measured to determine device delivery and amount of aerosol collected
- Trapping efficiency of CFP with impinger was confirmed by placing second impinger in collection train and analyzing separately (Figure 4, Table 2)
- Analysis of CFP
  - Place CFP in 40-mL screw cap vial; add 30 mL of 17.5 mmol/L DNPH in acetonitrile with 27.3 mM perchloric acid; vortex 10 seconds; remove 1-mL aliquot from extract and place in 1.5-mL amber autosampler vial; add 50 µL internal standard solution containing pyridine; cap vials and vortex briefly to mix; inject samples on LC-MS system
- Analysis of glass impinger
  - Remove tube insert from bottle; remove 1-mL aliquot from extract and place in 1.5-mL amber autosampler vial; add 50 µL internal standard solution containing pyridine; cap vials and vortex briefly to mix; inject samples on LC-MS system

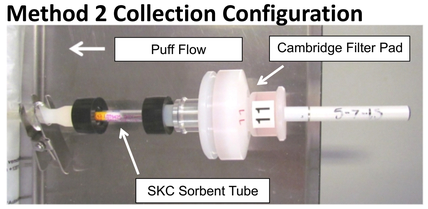
FIGURE 4
Method 1 Trapping Efficiency Collection Configuration



METHOD 2: CFP + DNPH SKC Sorbent Tube

- Aerosol was collected using a 44-mm CFP in front of the SKC sorbent tube (Figure 5)
- Mass changes were measured to determine device delivery and amount of aerosol collected in each of the segments
- Analysis of CFP
  - Same as Method 1
- Analysis of SKC sorbent tubes with DNPH-coated silica gel[8]
  - Use a glass wool puller to remove plugs at either end of both segments and collect the particles with associated plug separately (20 mL glass vials); the breakthrough segment includes the bottommost glass wool plug, and the collection segment includes both plugs on either side of the DNPH-treated silica gel; add 5 mL of acetonitrile to each vial, and cap vial; vortex for 10 sec; place 1 mL of sample into a 1.5 mL amber autosampler vial; add 50 µL internal standard solution containing pyridine; cap vials and vortex briefly to mix; inject samples on LC-MS system

FIGURE 5
Method 2 Collection Configuration



RESULTS

- Formaldehyde was not found in the breakthrough section of the 2nd impinger or the SKC tube
- No visible aerosol was observed in the impinger or in the SKC sorbent tube

TABLE 2 Percent Formaldehyde from Total Formaldehyde Collected

METHOD #1	Rep 1	Rep 2	Rep 3	Average
Cambridge Filter Pad	72.6%	67.9%	74.8%	71.8%
Impinger #1	27.4%	32.1%	25.2%	28.2%
Impinger #2	0.0%	0.0%	0.0%	0.0%

METHOD #2	Rep 1	Rep 2	Rep 3	Average
Cambridge Filter Pad	73.1%	67.7%	74.7%	71.8%
SKC Primary	26.9%	32.3%	25.3%	28.2%
SKC Breakthrough	0.0%	0.0%	0.0%	0.0%

- Inconsistent packing densities are a potential issue when using SKC sorbent tubes for routine analysis
- Differences in packing density result in restricted air flow thereby altering the puff volume
- SKC tubes had to be pre-selected based on a target puff volume of 70 ± 0.5 mL
- There are no puff volume concerns using the impinger method

SUMMARY

- Methods 1 and 2 are suitable for collection of formaldehyde in aerosol samples from e-cigarettes
- Both methods provide equivalent trapping efficiency
- The impinger method (Method 1) would be a more robust method and could be easier to be standardized by multiple laboratories

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