Volatile Organic Compounds in Headspace over Electrical Components at 75 to 200°C Part 2. Analytical Response with Gas Chromatography-Differential Mobility Spectrometry for Airborne Vapor Monitoring

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Abstract

Volatile organic compounds (VOCs) emitted into headspace over discrete electronic components including resistors, capacitors, diodes, transistors, and transformers were identified and quantitatively determined in Part 1 of this series using gas chromatography/mass spectrometry. Emissions of VOCs were characteristic of each component and headspace concentrations increased with temperature and persisted with time in most instances. Technology suitable for routine continuous monitoring of air quality based on gas chromatography/differential mobility spectrometry (GC/DMS) was evaluated with the same electronic components here, Part 2 of this series. Distinctive patterns in plots of ion peak intensity, retention time, and compensation voltage were obtained from VOCs from resistors, capacitors, and insulation from wires of a transformer held at 200°C for ten minutes. Intensity of response and analytically rich information produced by GC/DMS suggest further utility also as an air quality monitor or smart smoke alarm with electronics-dense habitats in spacecraft or offices and industrial venues.

Keywords: Volatile organic compounds; Electronic components; Headspace vapors; Differential Mobility spectrometry

Introduction

Concern has existed for more than 30 years on the loss of quality in ambient air of homes and offices through the release of volatile organic compounds (VOCs) from building materials and consumer products [1-3]. Efforts have been made to associate these to sick building syndrome with expansive investigations, in one instance for 1159 common household products, 31 volatile organic compounds distributed among 65 product categories [1]. In others, studies were directed either towards specific substances of toxicological significance or toward emissions from particular materials such as carpet, paints, common household fluids, and electronics or computer components [4-6]. Substances such as organophosphate flame retardants, phthalate esters, and formaldehyde are often prominent in such targeted studies. An interest which parallels these concerns is the composition of airborne vapors within enclosed atmospheres or re-circulated air such as found in aircraft, spacecraft, and submarines where there is limited or no exchange with natural fresh air and where the accumulation of impurities may reach levels of toxicological concern or those affect human performance. While analytical methods for exploratory studies have been based on measurements by gas chromatography with mass spectrometry, air quality monitoring on the International Space Station makes use of small, low-power technology based on gas chromatography with differential mobility spectrometry [7].

An interest in this laboratory has been the source apportionment of VOCs arising from common discrete components of electronics such as resistors, capacitors, and circuit boards and any specificity of detection of VOCs entering ambient air. This arises from interests in human exposure and also in the development of sophisticated and ultra-sensitive smoke alarms able to identify a source of a fire from monitoring airborne vapors. In a recent study, the composition of VOCs in headspace over electronics parts were quantitatively determined at temperatures simulating electronics at temperatures from 50°C to over 200°C, well-before near combustion conditions [8]. Emission rates for VOCs increased from a low of 0.001 ng/g per min for nonanal from transformer wire at 100°C to a maximum of 2.5 ng/g per min at 150°C for isophorone from a resistor. Findings suggested that VOCs arose from impurities in plastics and not from thermal decomposition of materials. Emissions usually contained between 20 and 40 VOCs above background and were characteristic of each component including resistors, capacitors, diodes, transformer materials, and circuit board. These mass loadings in an electronics dense space without much dilution of vapors are compatible with picogram levels of response of ion mobility spectrometry, a method that is both small and analytically rich compared to simple metal oxide or e-nose sensors.

In differential mobility spectrometry (DMS), a specific embodiment of ion mobility spectrometry, the advantages of ion chemistry found with IMS are combined with micro-fabricated drift tubes (15 mmx5 mmx0.5 mm) and an additional advantage of simultaneous detection of ions in both negative and positive polarity. Gas phase ions derived from individual VOCs are characterized in DMS using high frequency asymmetric oscillating electric fields providing ion spectra which can be used to assign identification, particularly in conjunction with chromatographic pre-separation. The lack of significant fragmentation during ionization and operation at ambient pressure provides low detection limits of 10-100 pg and response to a broad range of substances. Performance of DMS as a detector for gas chromatography has been established in development and application [9,10]. The critical uncertainty is whether substances of interest will form stable ions sufficient for ion mobility characterization. In previous studies with GC/DMS, analytical response to a broad range of substances was obtained from pyrolysis of bacteria [11] and from combustion of...
natural materials [12]. The behavior of ions in DMS is reasonably well-established and based on mobility (K), or speed (vE) of an ion swarm normalized to electric field strength (E) or K=vE/E. Electric field strength (or ratio of E as gas number density, N, increases) K becomes non-linearly dependent on E/N which is described by an alpha function in Equation 1:

K (E/N)=K_o [1 + α (E/N)] (1)

The alpha function of field dependence allows ion characterization by differences in mobility (AK) based on mobility at low field strength (here -600 V/cm) and high field strength (here 15000 V/cm). The molecular and structural understandings for AK are largely undeveloped though a general pattern is decreased AK with increased ion mass in a purified gas atmosphere. DMS ions are passed using gas flow at ambient pressure through a planar structure and exposed to a perpendicular asymmetric electric field (the separation field) and a superimposed DC field, the compensation field. In manner analogous to quadrupole mass spectrometers, a DMS analyzer filters ions continuously and a sweep of the compensation field provides a measure of all ions in the analyzer. Spectra are plots of ion current versus compensation field at fixed separation field.

The principles governing the compensation field where an ion appears are sufficiently undeveloped and the practice of DMS is semi-empirical. The objective for this work was to determine if vapors from components from headspace over electronic components produced analytical information sufficiently distinct to evaluate GC/DMS as an advanced smoke alarm or as a monitor for emission from electronics in a further development. Headspace vapors drawn near the surface of a complete electronics board, heated externally in air at ambient pressure, were analyzed using a commercial GC/DMS similar to those used on the International Space Station for air quality monitoring. The objective was to determine if a vapor profile could be recognized over background with a simulated over-heating of electronics from a power module.

Materials and Methods

Instrumentation

A Hewlett-Packard 5890 gas chromatograph was equipped with split-splitless injection, a RTX-50 column, and 30 m long by 0.25 mm ID with 0.25 µm film thickness (Restek Corp., Bellefonte, PA) interfaced to a differential mobility spectrometer. Other parameters included carrier gas, helium at 1.0 mL/min; injector temperature of 230°C, initial column temperature, 0°C for 5 min; program rate, 6°C/min to 100°C and then 10°C /min for a final temperature of 200°C. The gas chromatograph was equipped with a cryogenic valve for sub-ambient cooling with liquid carbon dioxide. The inlet of the gas chromatograph with a heated (120°C) 4 port gas switching valve (model 4C4 UWT, VICI Valco Instruments Co. Inc., Houston, TX) placed into the carrier gas tubing before the injection port. Attached to the injection port through Swaglok fittings and heated 1/16” Silco steel tubing (Restek Corp.) was a headspace chamber designed to receive electronic components through a removable cap. This chamber was a glass cylinder (12.7 cm x 1.6 cm) with a volume of 25 cm³ and was thermo coated with a resistive element applied to the outside of the chamber and a model CT15 temperature-controller (Minco, Minneapolis, MN, USA). The chamber was attached to the heated valve using 2 to 3 cm long transfer lines of Silco-steel tubing maintained at 120°C.

The gas chromatograph and the inlet system including the sample container were the same used in the studies described above in the GC/MS determination of vapors in headspace above electronic components [8]. The detector was a differential mobility spectrometer made at New Mexico State University. The differential mobility spectrometer contained a planar micro-scale drift tube made from ceramic plates with gold plate copper base electrodes [10]. The separating electrodes were 4 mm wide x 6 mm long and a width of 0.52 mm was used for the gap between the electrodes. Electrodes were fixed in a metal body to which a ~1 mCi 63Ni ion source was attached. The ion source was connected to a transfer line into the GC oven. Air at 0.5 L/min was heated and passed into the transfer line for gas flow through the DMS analyzer. The transfer line and DMS analyzer were maintained at the same temperature. Air was provided using a pure air generator (Mode 737, Add co Corp, and Miami, FL) and was further purified through beds of 13x molecular sieve. Moisture in the carrier gas was monitored using a model MIS-2 meter (Panametrics, Inc., Waltham, MA) and was 30 ppm or below. The drift tube was operated using in-house built electronics containing an RF waveform generator, a sweeping voltage generator, and an electrometer. The waveform generator was based on a soft-switched, semi-resonant circuit that incorporated a fly-back transformer and allowed variable peak-to-peak amplitudes of the asymmetric waveform from 200 V to 1600 V without altering the waveform shape. The operating frequency of the RF generator was 1.3 MHz. A compensation voltage ramp was synchronized with the data collection system and provided a scan of compensation voltage from –10 V (or -5 V) to +30 V (or 24 V) at a frequency of 1 scan every 3.8 (or 2.8) s. Signals were processed using a model 6024E National Instruments (Austin, TX) board; spectra were digitized and stored for every scan using software derived from Labview (National Instruments).

An aluminum-clad capillary column was used to join the analytical column to the DMS analyzer since polyimide coatings on conventional columns released vapors over 60°C altering the chemistry in the ion source of the DMS. A sheath of stainless steel tubing at 100°C was used as a transfer line and preheated at 500 ml/min was added to the sheath so effluent from aluminum clad column was swept rapidly into the DMS ion source, joined to the transfer line using compression fittings. The pressure of the DMS analyzer was ambient conditions and drift tube was under temperature control at 80°C.

A second GC/DMS instrument, an Air Quality Monitoring (AQM) system was utilized for sampling vapors off a complete circuit board, the Air Quality Monitor (Sionex Corp, now Draper Laboratory, and Boston, MA). In the AQM, a sample pump acquires a sample for 5 to 10 s at ~140 mL per min and vapors are pass through a sorbent bed composed of 60/80 mesh Carbopack B (3.7 mg) and Carboxen 1000 (4.0 mg). The trapped VOCs are flash desorbed from the pre-concentrator to the GC column which is 15 mx 0.25 mm, bonded DB5 (1 µm 5% phenyl polydimethylsiloxane (Agilent, Santa Clara, CA). Carrier gas flow through the column was ~1 mL/min with ~400 ml/min make up flow into the DMS analyzer. Only air is used for the carrier gas and detector make-up gas. An on-board computer is a PicotTX with Windows XP operating system and manufacturer provided software (Expert, Sionex Corp.) to controlling the instrument parameters (such as temperatures and pressures) and acquires data.

Materials and Reagents

Standards: Standard solution for individual chemicals was made in hexadecane solvent at concentrations of 0.01 to 1000 ng/µL for all.
compounds identified by GC/MS [8]. Chemicals were obtained from Sigma-Aldrich Co. (St. Louis, MO) and from Chem Services, Inc. (West Chester, PA). The purity of the standards was certified as >98% and were used within expiration dates. All standards were free of detectable levels of impurities and were confirmed by identification of mass spectra through GC/MS analyses [8].

Components: The resistor was 1.3 Ohm 1 watt from Xicon Passive Electronics (part no 249-1.0, Mouser Electronics, Mansfield, TX). The capacitor was 470 µF and 25 V (Siemens Corporation, New York, NY). The transformer was Hewlett Packard Part No 9100-30310-1 5200A (Palo Alto, CA) and was disassembled to wires with plastic insulating layers.

Procedures

Analysis of standard solutions: Two types of analyses were made with standard solutions of VOCs in hexadecane. A manual injection with a 10 microliter syringe (model 1701RN, Hamilton Company, Reno, NV) was used to inject 1 microliter of standard with split less time of 30 s. The relative standard deviation was <10% from the manual injection. In a second method, 1 microliter of a standard solution was placed into the headspace chamber for components and allowed to volatilize. The vaporized constituents were transferred with the gas switching valve to the GC/DMS for analysis in order to determine or confirm instrument performance. The septum in the GC was replaced after twenty injections.

Off-gassing and influence of temperature: Electrical components were weighed and then placed in the headspace chamber at room temperature; the chamber was swept with purified nitrogen, isolated with a switching valve, heated to constant temperature to 200°C, and held for one hour. After the 1 hour period, headspace vapors in the chamber were transferred through a heated switching valve into the GC column at 10°C for five minutes. During this time, carrier gas was supplemented with additional flow of nitrogen at 150 mL/min to ensure rapid transfer of VOC mass from the chamber; this constituted a 30X exchange of volume in the chamber for best quantitative transfer. Studies were made in triplicate or more using fresh samples in each heating sequence. Blank analysis between replicates confirmed any residual cross-contamination with these procedures. Authentic substances and standards were analyzed under identical conditions to confirm identifications of unknowns by retention time and DMS spectra.

Data processing: The software used to obtain DMS spectra was written in-house with Lab-View software (National Instruments, Austin, TX) and operated with a NI model 6024e data acquisition card. The columns of the matrix from a measurement were configured to exact values for compensation voltage (CV) and retention time (RT) with the y dimension was scaled for -10 to 20 V in compensation voltage and the x dimension scaled from 0 to 35 min for retention time. In Origin software (Origin Lab, Northampton, MA) a template was created to process the data as a contour figure in black and white and processed in a 3-D format.

Heating of a complete circuit board: In addition to measurements with individual components a complete electronic board, a Power Supply board from on a Hewlett Packard Gas Chromatograph (model 5880A, Series II), was heated externally from 25-180°C to simulate overheating of power electronics on a commercial electronic device. The board was placed on a large hot-plate in air at ambient pressure and equipped with a thermocouple, placed on the board, pressed between a resistor and the board. Power applied to the hot plate caused heating of the board and vapors were drawn from the surface by placing the inlet of the AQM near the board surface. Parameters for the AQM were: sample time, 10 s; isothermal period of 120 s at 35°C column temperature; column temperature ramp to 140°C in 120 s. The separation voltage on DMS was 950 V or as field normalized to number density, 120 Td. Approximately 500 scans were collected per measurement. The DMS analyzer was 80°C.

Result and Discussion

GC/DMS characterization of VOCs from a resistor

Results obtained from GC/DMS characterization of VOCs in headspace over a resistor are shown in Figure 1 (bottom) as a plot of ion abundance (z axis), retention time (x-axis) and compensation voltage (y-axis). The reactant ion peak is seen at 5.8 V in the y-axis and is seen as a band of constant ion intensity through the elution profile except where substances elute from the GC column. The reactant ion peak is a hydrated proton (H\(^+\))(H\(_2\)O)\(_n\)) in positive polarity with two or three hydration number at this temperature and moisture level. At those elution or retention times when a substance elutes from the column, a loss in intensity of the reactant ion is seen and ion peaks appear between 5.8 and 0 V. These are Product ions which appear as protonated monomer, or MH\(^+\)(H\(_2\)O)\(_n\)+ and are formed from ionization of neutral constituents principally through displacement reactions as shown in Equation 2:

\[
M + H^+ + (H_2O)_n \rightarrow MH^+ + (H_2O)_{n-1} + H_2O (2)
\]

Since there are, as of now, no precise quantitative models to relate ion structure and CV in a purified gas atmosphere, comparisons of DMS spectra to authentic standards and retention times are reliable measures to identity constituents. Consequently, authentic standards for major substituents detected in headspace over the same resistor and identified in Part 1 of the Title work [8] were analyzed by GC/DMS; results are shown in Figure 1 (top). Chemical identities from prior studies with GC/MS of some substance, along with retention times and CV values are shown in Table 1. Major constituents from the resistor included 2-butoxy-1-ethanol, 1,3 dimethyl-benzene, benzaldehyde, and isophorone. These substances each had distinctive DMS spectra (two of these four are shown in Figure 2) and these provides another level of selectivity in addition to the selectivity of ionization and that of chromatographic retention.

Another pattern seen in the contour plots and very well known in IMS and DMS is the appearance of a second peak during the elution of a substance. This arises from a second displacement when the concentration of vapor is sufficient high that reaction in Equation 3 occurs:

\[
M + MH^+ + (H_2O)_n \rightarrow M_2H^+(H_2O)_{n-1} + (H_2O) (3)
\]

Forming a proton bound dimer, M\(_2\)H\(^+\)(H\(_2\)O)\(_{n-1}\). In the instance of a chromatograph elution peak, ion patterns can be complex for a single substance where a protonated monomer appears at the front of the elution (Equation 2), the protonated monomer is converted to proton bound dimer (Equation 3) near and at the maximum of the elution profile, and then a protonated monomer appears on the falling edge of the elution profile. This is seen clearly with the authentic standards for 2-n-butoxy-1-ethanol at 23 min and for benzaldehyde at 25 min. Since the proton bound dimer is greater in ion mass than the protonated monomer, the CV is closer to zero. This formation of protonated
monomer and proton bound dimer can be seen also in Figure 2 for butoxy-1-ethanol. Not all substances form stable proton bound dimers above room temperature and thus, this pattern is not universal providing additional analytical information for a spectrum.

| Retention Time (min) | Compensation Voltage (V) | Peak Intensity (V) |
|----------------------|--------------------------|-------------------|
| Resistor             |                          |                   |
| 1,3 DMB              | 21                       | 1.47              | 6.21              |
| 2-n-Butoxy-1-Ethanol  | 22.5                     | 0.145             | 8.02              |
| Benzaldehyde         | 25.2                     | 0.186             | 8.05              |
| Isophorone           | 26.3                     | 0.372             | 10                |
| Capacitor            |                          |                   |
| Alpha methyl styrene  | 24.2                     | -0.591            | 10                |
| Benzaldehyde         | 25.2                     | 0.186             | 7.58              |
| Acetophenone         | 28.4                     | 0.309             | 7.65              |
| Transformer          |                          |                   |
| Phenol               | 14.9                     | 2.28              | 8.06              |
| 3-Methylphenol       | 16.4                     | 1.63              | 6.77              |
| 2,4 Dimethylphenol   | 17.4                     | 0.397             | 5.04              |
| 3-Ethylphenol        | 17.6                     | 1.11              | 4.96              |

Table 1: Chemical identities from prior studies with GC/MS, of some substances, along with retention times and CV values.

In a general analysis of Figure 1, more than twenty chromatographically resolved substances can be detected in the topographic plot and these are not yet identified. Still, such complex patterns are useful for chemometric pattern recognition methods such as multivariate analysis used elsewhere with complex GC/DMS data sets [11]. Such computational power was not applied immediately here since parameters for the DMS here were chosen for maximum sensitivity and not for high resolving power. Thus, distribution on the CV axis is over a relatively narrow range from -5 to 7 V and simple inspection was applied. For example, there is a slight and general trend in peak location toward 0 V (or decreased ΔK) with increased elution time and this is consistent with increased ion mass from substances with longer elution time and lower vapor pressures (increased mass). Also, the patterns of protonated monomer and proton bound dimer disclose concentrations and this can be advantageous for quantitative determinations. The emission level determined was calculated to be between 0.03 and 1 ng/g (component) per minute from Part 1 of the title work [8].

Figure 1: Contour plots from GC/DMS analysis of headspace vapors over a resistor (bottom) and from authentic standards of major constituents (top). Constituents were 1,3 dimethylbenzene ▲, 2-n-butoxy-1-ethanol △, benzaldehyde □, and isophorone ■.

GC/DMS characterization of VOCs from a capacitor

Findings from GC/DMS analysis of vapors over a capacitor are shown in Figure 3 (bottom) where the topographic plot of ion intensity, retention time and compensation voltage can be compared directly to that for the resistor (Figure 1). Similarities and differences can be seen compared to the findings with the resistor. There are differences in distribution and intensity of peaks in the retention time scale as well as placements on the CV scale. The reactant ion peak is present in both as expected and there is a stronger vapor loading of VOCs and this is seen as several extended regions of retention time where the reactant ion is completely depleted. This constitutes a condition of saturation of the ionization region. More than thirty constituents were detected in Figure 3 (bottom).

Figure 2: Spectra from DMS analyzer for vapors of a. benzaldehyde, b. 2 butoxy-1-ethanol c. 2,4 DMP, and d. 3-ethylphenol. Reactant ion peak present near 7 CV, with monomer and dimer formation between -5 and 5 CV.

As with patterns for VOCs released by the resistor, a few components were seen at significantly higher response than other substances and were for the capacitor alpha-methylstyrene and acetophenone. Emission levels of these compounds were determined to
be 0.3 to 0.6 ng/g for this sampling period. Benzaldehyde was common to both resistor and capacitor.

**GC/DMS characterization of VOCs from transformer materials**

Parts of a disassembled transformer was analyzed in a composite mixture with amounts approximately proportional to non-metallic materials and findings from GC/DMS analysis of vapors are shown in Figure 4 (bottom) also as a contour plot. Comparison can be made to Figures 1 and 3, although the plot is turned 90 degrees to the former. The visually striking result is the appearance of only a few intense peaks comprised of phenol and substituted phenols. There was favorable agreement between retention times for the phenol standards and also good agreement on CV values for ion peaks. In the top frame of Figure 4, DMS spectra of four phenols are shown with ion peaks between 0 and 2.5 V compensation voltages with the reactant ion peak at 5.8 V.

![Figure 3: Counter plots of GC/DMS analysis of headspace vapors over a capacitor (bottom) and from authentic standards from major constituents (top). Constituents were alpha-methylstyrene ▲, benzaldehyde △, and acetophenone ■.](image)

The spectra appear to be only single ion peaks without an additional product ion from a proton bound dimer and this is seen also in Figure 2 for 2,4 DMP and 3-ethylphenol. The strong presence of phenols was associated to the amount of electrical wiring found in a transformer. Although the standards are not shown in contour plots, retention times of unknown peak matched those for the authentic standards, and findings were consistent with results from GC/MS analyses (Part 1 of title work, 8). Emission levels of these phenolic compounds were determined to be 0.7 to 1.5 ng/g.

The combination of separations from a gas chromatograph and the analysis of vapors with a mobility spectrometer or differential mobility spectrometer used here is more than simply a second dimension of analytical information, although the second dimension is valuable. For example, the spectra in Figure 2 illustrate the analytical response of this generation of DMS analyzer with resolution values of between 10 and 20. The benefit of ion characterization with CV, however, can be obtained with little more complexity than a simple ionization detector. The distinctions in the DMS spectra can be seen by inspection of spectra in these figures and even more chemical information is available if the spectra are processed and analyzed used advanced computational methods. Nonetheless, the benefit of combined technology is more than a sum of the parts and the pre-fractionation of the VOC mixture with gas chromatograph results in the introduction of single or simple mixtures to the ion source of the DMS analyzer.

In this, the ionization chemistry is simplified over what would occur if the sample of VOC was entered into the ion source as a mixture. The uncertainty of charge exchange and competitive reactions, part of API methods, would lead to unclear response, perhaps un-interpretable results. The differences in patterns in topographic plots between electrical components are due not to variations in the method and can be attributed preliminarily to differences in the composition of the electrical components and verified via GC/MS. In addition the DMS retention times match the authentic standards in the GC/MS methods, and that actual DMS monitoring can detect samples in milliseconds.

![Simulated over heating of power board and response with commercial GC/DMS](image)
vapors from such a board may be found in headspace near a board during an over-heating episode and well before the board flames.

Figure 5: Control data (left frames) from analysis of headspace over circuit board at 25°C with plot of ion current, retention time, and compensation voltage (at bottom) and spectrum for reactant ion peak (at top); at right were results from analysis of headspace vapor from the same circuit board at 180°C with spectra composited at top and topographic plot at bottom. The GC/DMS was an Air Quality Monitor.

Figure 6: Ion chromatograms from the intensity of the reactant ion peak inverted and plotted with retention time from GC/DMS measurements of a complete circuit board at three temperatures using an Air Quality Monitor.

When samples of air are drawn from an instrument rack, these findings suggest that the AQM could provide a first alert to overheated electronics. The plausibility of detecting these vapors, once diluted into a larger volume of air in a room, has not been established and may depend upon pre-concentration, sampling location, and patterns of flow of air in the room.

Conclusions

Findings from GC/DMS analyses of headspace vapors over heated electronic components illustrated that response to VOCs was intense and distinctive for each electronic component. The findings were consistent with results determined in Part I by GC/MS analyses [8] and demonstrated response by GC/DMS was suitable as a detector of vapors released in the low ng/g levels with components at elevated temperature. These findings suggest that the Micro analyzer, GC/DMS instruments on-board the International Space Station for air quality monitoring should exhibit response to vapors emitted from instrument racks with overheated circuits or electronics.

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