



Temperature-programmable low thermal mass silicon micromachined gas chromatography and differential mobility detection for the fast analysis of trace level of ethylene oxide in medical work place atmospheres[☆]

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ABSTRACT

A portable, fast gas chromatographic method for the direct measurement of part-per-billion level of ethylene oxide in medical work place atmospheres is introduced. Ethylene oxide is a chemical of significance in life science for its critical role as a highly effective sterilizing agent for heat sensitive surgical instruments. However, ethylene oxide is highly flammable, a suspected human carcinogen, is mutagenic, and henceforth, requires close monitoring. The approach incorporates a temperature-programmable silicon micromachined gas chromatograph for the fast separation of ethylene oxide from airborne organic interferences. The separation approach is hyphenated to a micromachined differential mobility detector, improving targeted analyte detection, and enhancing the overall selectivity obtained. A complete analysis can be conducted in less than 60 s. Further, ethylene oxide in the matrix mentioned can be measured directly with low possibility of false positives and without the need for any sample pre-treatment, such as pre-concentration or derivatization. A relative precision of less than 5% RSD ($n = 20$) over a range from 5 parts per billion (v/v) to 500 parts per billion (v/v) was obtained.

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1. Introduction

Ethylene oxide (CAS-75-21-8) is critical in the production of detergents, thickeners, solvents, plastics, and various organic chemicals such as ethylene glycols, polyglycol ethers, and ethanolamines to name a few. As a highly potent disinfectant gas that leaves no residue on items it contacts, ethylene oxide is also widely used in hospitals and the medical equipment industry to replace steam in sterilization of heat sensitive tools, and equipment such as disposable plastic syringes or sutures [1–3]. Despite being a vital raw material with diverse applications, ethylene oxide (EO) itself is a hazardous substance. It is a highly flammable, carcinogenic, mutagenic, irritating, and anaesthetic gas, oftentimes, with misleadingly pleasant aroma like diethyl ether, a chemical with much lower level of toxicity [4–6]. As a result, close monitoring of EO is of significant importance.

Industrial emissions of EO are strictly regulated and inventoried by environmental and health agencies. Non-inventoried EO emissions are a result of uncontrolled fugitive emissions or venting

with other gases. Other common sources of atmospheric EO include losses during disinfections of hospital equipment, combustion of hydrocarbon fuels, bacterial degradation products, photochemical smog and cigarette smoke [7,8]. There are several analytical approaches available to monitor the presence of sub part-per-million level of EO based on passive sampling devices such as badge or passive diffusive samplers reported in the literature and as proposed by the American Society of Testing Material (ASTM) or the Occupational Health Safety Association (OSHA) [9–13]. Due to the fact that EO is not well retained by various commercially available adsorbents, a common analytical strategy for both badge and passive tube sampling involves reaction gas chromatography, followed by extraction prior to analysis [10–12]. The adsorbents used for the trapping of EO, such as cellulose fibers, organic porous polymers, or carbon molecular sieves are impregnated with hydrobromic acid. EO readily reacts with hydrobromic acid at ambient temperature to quantitatively form 2-bromoethanol, which has a substantially higher breakthrough volume on the adsorbent used. For instance, with a 100 mg of activated charcoal as an adsorbent, at a concentration of 15 ppm (v/v) of EO, a temperature of 25 °C and a relative humidity of 70% and a flow rate of 50 mL/min, 2-bromoethanol has a breakthrough volume of 39 L as compared to ethylene oxide which has a breakthrough volume of 2.6 L. 2-Bromoethanol in the spent adsorbent is subsequently extracted with an organic solvent like

[☆] Honouring Professor Dr. Milton Lee's 65th Birthday.

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benzene, carbon disulfide, or dimethyl formamide, and the extract is analyzed by gas chromatography with a selective detector such as electron capture [10,11].

While the techniques described are adequate and convenient for the passive monitoring of EO, especially over time periods ranging from 15 min to 8 h for time weighted average measurements, there are several important constraints to this methodology, such as the lack of near real-time data, inadequate selectivity delivered by the detector used, extensive sample preparation, industrial hygiene issues with regulated solvents like benzene and carbon disulfide, spent solvent disposal cost, and hardware that is not field-deployable. With the shortest sampling time of 15 min, analysis times including steps like sampling, extracting and analysis itself, even when an on-site analytical instrument is available, can be as long as 30 min. The delay in data availability can have negative consequences on personnel who continue to be exposed to the chemical should a release occur. While the typically used electron capture detector has an adequate sensitivity for the detection of the brominated derivative of EO, without sufficient chromatographic separation it can be constrained by interferences in the presence of other chlorinated and oxygenated compounds like chloroform, formaldehyde, acetaldehyde, diethyl ether, acetone, iso-propanol, and methanol, compounds commonly found in healthcare environments. The sampling scheme also adds to the overall cost of ownership. Increased complexity, such as additional sampling pumps, non-reusable adsorbents for passive sampling, and the impact of moisture in the atmosphere on conversion and trapping efficiency also contribute to analysis costs [10,11]. A portable or benchtop GC–MS operating in selective ion monitoring mode can be used for the application described, however instrument availability for large-scale deployment, the need for specialized analytical skill to operate the instrument, and the overall cost of ownership makes this option much less practical.

A portable, fast gas chromatographic approach based on a temperature-programmable low thermal mass silicon micromachined gas chromatograph and differential mobility detection is introduced here. Successful development and implementation to provide direct measurement of EO at the low part-per-billion level without sample enrichment and derivatization in less than 60 s is discussed.

2. Experimental

An Agilent CP-4900 micromachined gas chromatograph (Agilent Technologies, Middelburg, The Netherlands) was used as the analytical platform. The original isothermal temperature controlled column module was removed and replaced with a locally fabricated temperature-programmable low thermal mass column module as described by Mustacich and Everson [14]. The module was equipped with a 5 m × 0.25 mm i.d. × 1 μm 5% phenylmethyl polydimethylsiloxane column, manually coiled to a diameter of 2.54 cm for the separation of analytes of interest. The column was interfaced with a micromachined silicon injector (Agilent Technologies, Middelburg, The Netherlands) via the existing inlet interface manifold using viton o-rings and compression fittings taken from the standard micromachined isothermal temperature module. The inlet temperature was 120 °C and an injection time of 250 s was used throughout. Helium carrier gas was supplied at an inlet pressure of 5 psig (constant pressure). The outlet of the analytical column was connected to a custom, locally fabricated metal manifold which interfaced to the micromachined differential mobility detector (Sionex, Bedford, USA) via a heat traced 40 cm × 0.25 mm i.d. deactivated, uncoated fused silica tubing with viton o-rings and compression fittings similar to those used to interface the column inlet to the injector. The fabrication and

configuration of the portable analytical system were described elsewhere [15]. Temperature-programming of the low thermal mass column unit was conducted using a standard TC-4 low thermal mass temperature-programming controller (Agilent Technologies, Delaware, USA). The temperature was programmed from 50 °C (1 s) to 150 °C @ 3.5 °C/s, and maintained at 150 °C for 10 s. On-board pump sampling duration was 20 s and transport gas used was filtered, carbon dioxide free air with a flow rate of 500 mL/min.

Detector control and data handling capability were handled by Sionex Expert version 2.15, a software suite provided by the manufacturer. The micro differential mobility detector temperature was 90 °C. The radio frequency value (RF) was 1100 V and the compensation voltage value (V_c) was 0.5 V. Deconvolution of three-dimensional data to the familiar two-dimensional data for quantitative analysis was handled by Expert integrated with Varian Maitre Elite chromatographic data system version 3.2. A Hewlett-Packard notebook computer equipped with a Pentium Quad-Core 2.5 MHz processor, 4 Gb of RAM, and 500 Gb hard drive with Windows XP Professional SVP-3 as operating system was used to host the software and process the data obtained.

Certified 10 ppm (v/v) and 100 ppm (v/v) EO in air standards were acquired from Air Liquide (Edmonton, Canada). Secondary standards over the range from 2 ppb to 500 ppb (v/v) were prepared from the primary standards by serial dilution for calibration purpose with an EnviroNics 2014 Computerized Gas Dilution and Blending System. A typical approach for gas sampling is to obtain a representative sample with either an intrinsically safe, battery driven electrical pump or a hand-pump into a Tedlar bag. As reported earlier, oxygenated compounds such as ethylene oxide can be quite adsorptive and best recovery of greater than 90% was found with a new Tedlar bag, with the content of the bag analyzed within the first 3 h at ambient temperature after sampling is completed [16]. A potential alternative to improve sample stability over time involves the use of industry standard electroplated Summa canister or Siltek treated stainless steel sampling cylinders. This approach typically involves substantially high cost of ownership. For the application described, Tedlar bags are convenient, economical, and adequate for the intended application. Air samples in various local medical facilities and veterinary clinics were taken with an inert hand sampling pump and new 200 mL Tedlar bags. All samples were analyzed within the first hour of sampling to ensure the integrity of the samples was not comprised. The bags were discarded after use to minimize cross-contamination or the potential for adsorption/absorption which can cause low recovery for the analyte of interest.

3. Results and discussion

3.1. Temperature-programmable micromachined gas chromatography with low thermal mass technology

Despite being available for more than three decades, the use of silicon micromachined gas chromatography has been mainly restricted to fixed gases and very volatile organic compounds [17–19]. One of the constraints associated with this technique is the lack of temperature-programming capability to extend the range of applicability. Applying low thermal mass gas chromatography to deliver temperature-programming capability is an appropriate choice since the technology offers low power consumption – in the order of watts when compared to kilowatts observed in conventional heated bench top gas chromatographs – over the same temperature-programming range. The low power consumption feature of the technology is conducive for use with portable equipment such as silicon micromachined gas chromatographs. One of the attractive features of the low thermal mass gas chromatography

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