

Development of a Rapid and Accurate Vapor Generation System for Real-Time Monitoring of a Chemical Warfare Agent (CWA) by Coupling Fourier Transform Infrared (FT-IR) Spectroscopy

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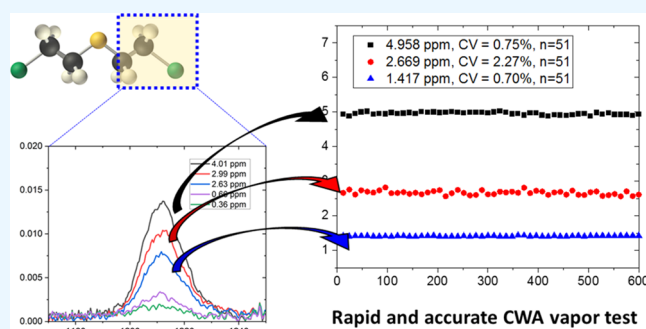
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ABSTRACT: Developing an accurate chemical warfare agent (CWA) vapor generator is critical for homeland security because it enables real-time monitoring of target agent concentration for testing and evaluation. We designed and built an elaborate CWA vapor generator that offers reliable long-term stability and real-time monitoring capabilities by coupling it with Fourier transform infrared (FT-IR) spectroscopy. We evaluated the reliability and stability of the vapor generator using a gas chromatography–flame ion detector (GC-FID) and conducted a comparison between the experimental and theoretical results of sulfur mustard (HD, bis-2-chloroethylsulfide), a real CWA, at concentrations ranging from 1 to 5 ppm. Our FT-IR-coupled vapor generation system showed real-time monitoring ability, which enables rapid and accurate evaluation of chemical detectors. The vapor generation system was able to generate CWA vapor continuously for over 8 h, demonstrating its long-term vapor generation capability. In addition, we vaporized another representative CWA, viz., GB (Sarin, propan-2-yl ethylphosphonofluoridate), and conducted real-time monitoring of GB vapor concentration with high accuracy. This versatile vapor generator approach can enable the rapid and accurate evaluation of CWAs for homeland security against chemical threats and can be used in constructing a versatile real-time monitoring vapor generation system for CWAs.



INTRODUCTION

Chemical warfare agents (CWAs) are highly toxic and dangerous substances that can cause mass citizen casualties or be used to assassinate important government officials.^{1–4} To prevent exposure to CWAs, it is essential to develop a rapid and precise chemical warfare agent vapor generator that can measure vapor concentration in real time for various applications, including the testing and evaluation of protection gear,^{5,6} inhalation toxicity,^{7,8} and chemical detectors.^{9–11} Due to their liquid phase at room temperature, numerous vapor generation techniques for CWAs have been investigated. However, regulations from the Organisation for the Prohibition of Chemical Weapons (OPCW) prohibit the handling of CWAs outside designated research laboratories. Developing a reliable and efficient CWA vapor generator is crucial for test and evaluation of protecting gears and chemical detectors for public health and national security. By accurately measuring CWA vapor concentration in real time, a concrete database for CWA protection equipment can be taken to prevent and respond to chemical attacks. Investigations are needed on developing safe and effective methods for generating CWA vapors while adhering to strict safety protocols and regulations.

Over the years, various techniques have been developed for generating CWA vapors by international government facilities

authorized to handle CWAs via registration with the OPCW. The Edgewood Chemical Biological Center (ECBC) in the United States has developed multiple vapor generation methods, including the delta tube saturation method, large saturation method, and spray atomizer method.^{12,13} These methods have been used to achieve targeted CWA vapor concentration, high flow rates, and long-term stability of CWA vapor generation. The ECBC has reported gas chromatographic analyses using flame photoionization detector (FPD) G-series and V-series agents, which are highly sensitive and accurate. However, real-time monitoring of the concentration of vapor generation can still be challenging when researchers try to modify the concentration of vapor immediately.

To address this issue, three different research groups from the Dugway Proving Ground (DPG), the National Institute of Standards and Technology (NIST), and the Pacific Northwest National Laboratory (PNNL) have reported a Fourier

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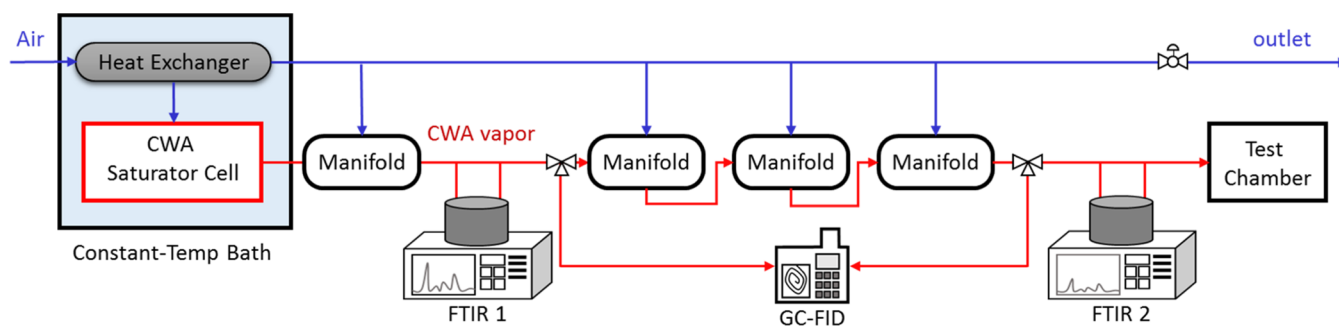


Figure 1. Schematic of the vapor generator coupled with the FT-IR real-time monitoring system.

transform infrared (FT-IR)-coupled monitoring concept of vapor generation by using a syringe pump injection method.¹⁴ They first demonstrated that quantitative FT-IR spectra of isopropyl alcohol (IPA) vapor can be recorded by coupling an FT-IR system, which has an optical path length of 5.6 m, with a vapor generator system. The data measured by the three different research teams were compared by integrating the FT-IR spectroscopic band areas of IPA vapor over the wavenumber ranges of 2750–3050 and 760–1545 cm^{-1} . From this interlaboratory comparison, they reported an error rate of under 0.8% for the vapor generator system with IPA. Their experiments proved that an FT-IR monitoring system can be used to measure the concentration of chemical vapor rapidly with high linearity in the dynamic concentration range. This advancement in CWA vapor generation and monitoring is critical for achieving effective and timely responses to the chemical exposure test and evaluation of chemical protection equipment. By accurately and concretely measuring CWA vapor concentration in real time, the false alarm rate of the chemical detector can be minimized. This implies that there is an urgent need for improving the real-time monitoring capability of the CWA vapor generation system as well as expanding their applicability to a broader range of CWAs.

Herein, we present a real-time monitoring vapor generation system for chemical warfare agents (CWAs) coupled with FT-IR spectroscopic monitoring devices to achieve high stability, accuracy, and rapidity. Figure 1 provides a brief overview of the overall instrumental diagram. The system was used to conduct CWA vapor generation of sulfur mustards (HD, bis-2-chloroethylsulfide), which are vesicants that cause blistering of the skin and mucous membranes on contact. To ensure the long-term stability of the vapor generator, a pressure regulator and a dust filter were installed prior to the heat exchanger. The temperature of the air was controlled by using a heat exchanger to provide a constant air flow to the CWA saturator cell and four manifolds using a mass flow controller (MFC). The pipeline for vapor generation and measurement consisted of 1/4" stainless steel (SUS), and the initial part of the CWA vapor generator was coated with inert nonreactive silicon (SilcoNert 2000) to improve the reliability by reducing adsorption of high-concentration CWA. The air stream carrying the vaporized HD molecules from the heat exchanger to the CWA saturator cell gently diluted the CWA vapors at the first manifold before analysis owing to the high concentration of primary vapor. The CWA vapors were measured in high and low concentrations using the FT-IR real-time monitoring system. FT-IR 1 and 2 were used to measure the difference in vapor concentration by linearly connecting manifolds. Three manifolds were connected linearly to provide rapid and

predictable control of HD vapor concentration, enabling the dilution or increasing of concentration. Two vapor ports that enable the collection of vapor concentration using solid sorbent tubes were located before and after the linear manifold system for analysis with gas chromatography with a flame ionization detector (GC-FID). The stability of the system and its compatibility with theoretical predictions were confirmed by collecting HD vapor from each port.

RESULTS AND DISCUSSION

Figure 2 illustrates the stability of our vapor generation system, which we evaluated by analyzing high and low concentrations

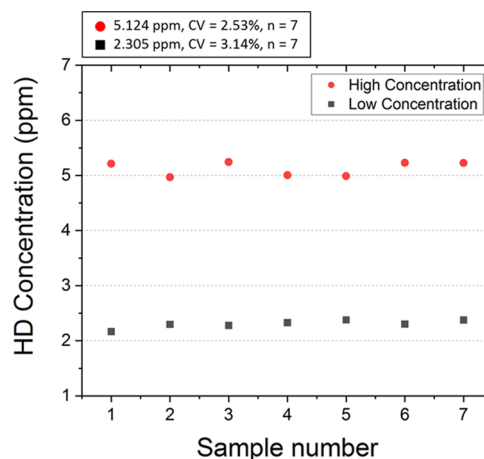


Figure 2. Results of the stability test for customized vapor generation at high concentration (5.124 ppm; red circular dots) and low concentration (2.305 ppm; black rectangular dots).

of HD vapor using a GC-FID. Prior to the experiments, we obtained a standard curve by measuring the GC area of HD solutions diluted in ethyl acetate with different concentrations (see the Supporting Information). The experiments began by providing 2 bar of air stream to the vapor generator and controlling the HD temperature with a bath system set to 20 °C throughout the procedure. After the vapor stream had stabilized, we collected high and low concentrations of HD vapor using a solid sorbent tube (Tenax-TA (PerkinElmer, Waltham, MA)) and the MFC bypass port at a flow rate of 200 sccm (standard cubic centimeter per minute) for 200 s.¹⁵ We measured each concentration seven times ($n = 7$) to verify the system's stability. As shown in Figure 2, the vapor generation system performed very accurately and reliably, with an average concentration of 5.124 ppm and a coefficient of variation (CV) of 2.53% for the high-concentration measurements and an

average concentration of 2.305 ppm and a CV of 3.14% for the low-concentration measurements. These results demonstrate the stability of our customized vapor generation system prior to coupling it with FT-IR for real-time monitoring.

One critical feature of the vapor generator is its custom-built manifold, which has four mixing ports that enable dilution of HD vapor concentration with clean air. As shown in Figure 3a,

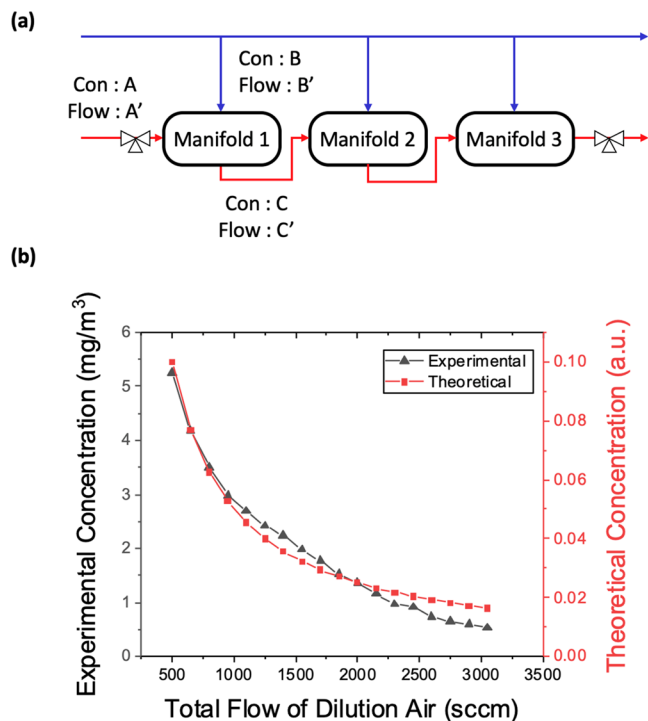


Figure 3. (a) Schematic of the linear manifold system, showing the dilution relationship between the initial HD vapor (red line) and dilution air (blue line) for precise vapor control. (b) Comparison of the experimental HD vapor measurement results (black triangles) and theoretical estimation values (red rectangles) for HD concentration at different total flow rates of dilution air with the linear manifold system.

the manifold is designed with a hemispherical inside structure that facilitates homogeneous mixing of the CWA vapor stream with the clean air at high flow rates, without causing reverse

pressure issues. Additionally, the manifold has a relatively large volume (68 mL), which helps prevent any blocking or reverse pressure issues at interconnection points, including the T-shaped three-way connector.

To assess the accuracy of the customized vapor generation system's dilution performance, we compared the theoretical calculation results with the actual vapor collection results from our experiments. The dilution of HD vapor concentration through the linear manifold system could be estimated using eqs 1 and 2 that describe the relationship between the flow rate and concentration of vapor. Equation 1 assumes an initial concentration of HD vapor as A , with a flow rate of A' , and no CWA in clean air ($B = 0$), with a flow rate of B' . Using these values, we can calculate the final concentration of HD vapor (C) and their combined flow rate (C'). We plotted a comparison graph of the experimental and theoretical estimation by increasing the dilution flow rate with consistent HD vapor concentration, as shown in Figure 3b. Assuming an initial concentration of HD vapor as 1.00 (arbitrary unit), we calculated the relative dilution concentration of HD vapor by total flow increment. For the experiment, we measured the concentration of HD vapor by collecting in Tenax-TA adsorption tubes for 200 s and analyzed it using GC-FID (see experimental details for description). We conducted a total of 19 tests for HD concentration by varying the total dilution flow rate from 500 sccm to 3050 sccm. In our real experiments, we measured the initial concentration of HD vapor as 5.25 mg/m³, which decreased continuously as we diluted the HD vapor with clean air. As shown in Figure 3b (black triangle symbol), the final concentration was 0.52 mg/m³ with a total air flow of 3050 sccm. The theoretical results (red rectangular symbol) also showed an excellent fit with the real experimental results. Overall, the dilution performance of our customized vapor generation system was highly accurate.

$$C = \frac{A \times A' + B \times B'}{A' + B'} \quad (1)$$

$$C' = A' + B' \quad (2)$$

We employed FT-IR spectroscopy to perform real-time monitoring of HD vapor in order to further confirm the system's capability as a CWA vapor generation system. The FT-IR monitoring system (MIDAC corp.) sequentially relays the measured IR spectrum and measurement conditions,

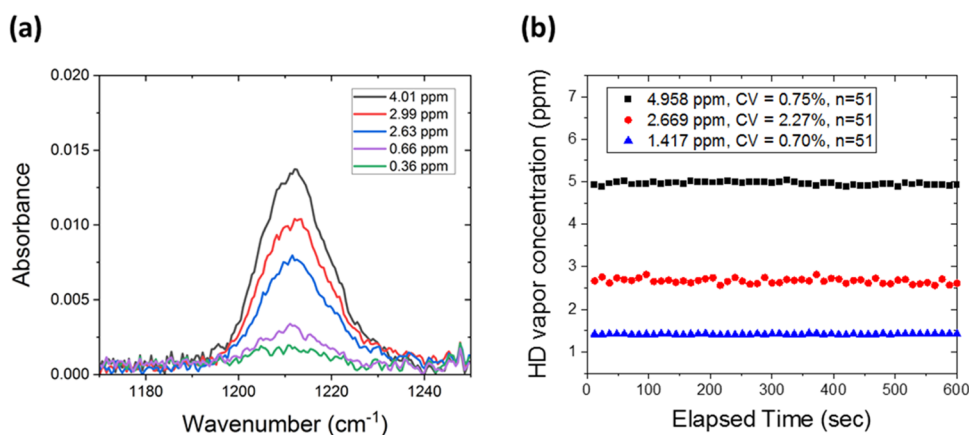


Figure 4. (a) Representative FT-IR spectrum of HD vapor with various concentrations and (b) graph of real-time monitoring results of the FT-IR-coupled vapor generation system for 10 min.

including the optical cell path, cell temperature, and pressure of the vapor stream. All of the information collected on the generated HD vapor was converted into HD concentration using a preset calibration curve that fits Beer's law.^{14,16} The wagging–bending IR spectrum of the C–H bond in the two terminal alkyl halide ($-\text{CH}_2\text{X}$) groups of the HD molecule is typically designated from 1150 to 1300 cm^{-1} .¹⁷ As shown in Figure 4a, the representative FT-IR spectroscopic peak of the HD molecule ($-\text{CH}_2\text{Cl}$) appeared at 1212 cm^{-1} , and the peak intensity also increased as the concentration was increased from 0.36 to 4.01 ppm. To further confirm the real-time capability of the FT-IR-coupled vapor generation system, we fixed three different HD vapor concentrations between 1.4 and 5 ppm. Over a 10 min period, we acquired HD vapor concentration data from the coupled FT-IR devices every 12 s, as shown in Figure 4b. The system exhibited very stable and rapid real-time monitoring capability with HD vapor of all different concentrations. At concentrations of 4.958 and 1.417 ppm, their coefficient of variation (CV) values were measured as under 0.75% for 10 min.

Figure 5 illustrates the concentration of HD vapor obtained from GC and FT-IR analyses (in purple and green,

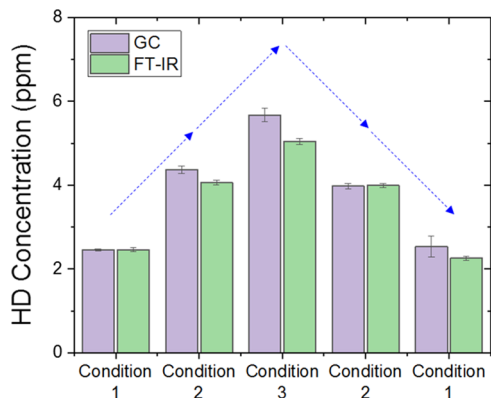


Figure 5. Comparison graph between GC analysis result by collecting HD vapor through Tenax-TA tubes (purple) and FT-IR real-time monitoring results (green) depends on the three different dilution flow rate conditions. While increasing the HD vapor concentration, comparison tests were conducted for each method by returning to the initial condition (blue dotted arrow).

respectively) under three different dilution conditions. The dilution flow rate was increased to elevate the concentration of HD vapor, and after each dilution condition was stabilized, we conducted two rounds of GC analysis to collect HD vapor in Tenax-TA adsorption tubes and obtained real-time concentration data from the FT-IR monitoring system ($n = 20-112$). The concentration data were then used to determine the average tendency and error probability for each dilution condition. The HD vapor concentration measurements obtained by both GC and FT-IR analyses were stable and accurate in the initial dilution condition (condition 1). As the dilution condition increased, the concentration of HD vapor rose sequentially. Under condition 2, both analyses showed an HD concentration of ~ 4 ppm, with low error probabilities. In the final dilution condition (condition 3), there was a similar tendency of concentration increase. However, the average HD concentration obtained from GC analysis and FT-IR analysis differed more significantly under this condition. To ensure the reliability of the customized vapor generator and analysis

methods (GC, FT-IR), we repeated the tests by decreasing the HD vapor concentration from condition 3 to condition 1. The results showed that the repeatability of the vapor generation system and the analysis methods (GC, FT-IR) were very stable and demonstrated excellent real-time monitoring capabilities for HD vapor generation.

To test the long-term stability of the vapor generation system and the FT-IR coupling, we conducted HD vapor generation over an 8 h period. The system consistently generated HD vapor from morning to evening, as shown in Figure 6. Every 12 s, we collected data from the FT-IR device.

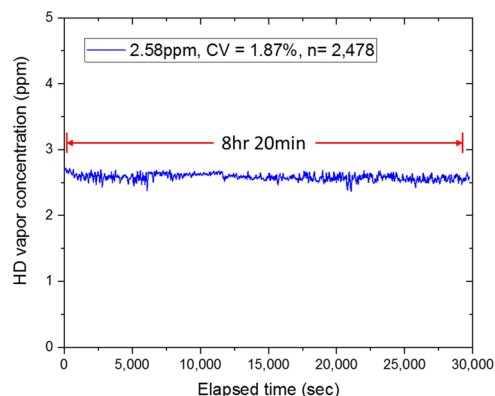


Figure 6. Measurement of the long-term stability of the vapor generation system over 8 h 20 m by using the FT-IR coupling system on the vapor generator. Average concentration of HD vapor was 2.58 ppm, and the coefficient of variation was calculated as 1.87% with 2478 data.

Although the data collection period was short, we obtained 2478 data points, and the coefficient of variation (CV) was 1.87% over 8 h 20 m. These results demonstrate that the system remained stable throughout the day, unaffected by changes in outside temperature or fluctuations in the air feeding system of the facility.

To further expand the capabilities of the vapor generation system with FT-IR coupling, we conducted experiments with a different chemical agent, GB, which is a nerve agent and frequently targeted by portable CWA detectors. Our goal was to investigate whether the FT-IR-coupled system could monitor GB vapor in the same way as it did with HD vapor. To do this, we selected a characteristic FT-IR peak range (880.0–1080 cm^{-1}) of the GB molecule and calibrated the FT-IR system against several concentrations of GB vapor. We then collected data on GB vapor concentrations with three different dilution conditions (1)–(3), as described in Figure 7. As shown in the figure, the vapor generator exhibited a successful concentration recovery behavior from (1) to (3) and vice versa. These results demonstrate that the vapor generation system with FT-IR coupling has advantages not only in real-time monitoring of CWA but also as a universal method for monitoring CWA vapor concentration with reliability and long-term stability.

SUMMARY

In conclusion, we have developed a CWA vapor generation system that allows for real-time monitoring through the coupling of FT-IR, facilitating future testing and evaluation of various types of CWAs (including blister and nerve agents). The dilution system, consisting of three linear manifolds,

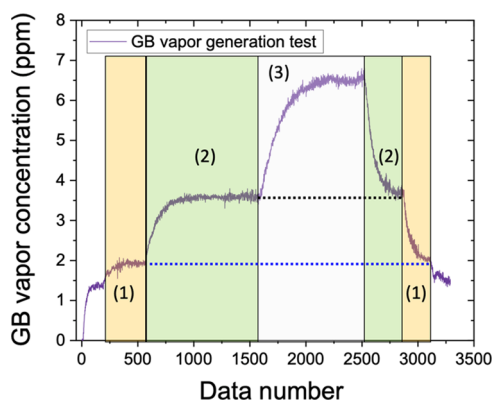


Figure 7. Reproducibility of GB (nerve agent) vapor generation confirmed by real-time monitoring under three different dilution conditions. The vapor generator produced GB vapor under dilution conditions (1)–(3), and the same test method as the previous HD vapor test shown in Figure 5 was repeated. The yellow box indicates the initial and final dilution conditions, which resulted in the same concentration recovery when we decreased the concentration of GB vapor as well as HD vapor. The green box shows results consistent with condition (1).

ensures uniform concentration of the real CWA agent vapor. Our experiments with the real blister agent HD have shown that the concentrations obtained from both GC-FID and FT-IR were in good agreement. Furthermore, dilution estimations based on theoretical calculations exhibited consistent fitted behavior with a realistic collecting concentration of HD. The vapor generation system demonstrated remarkable stability toward three different dilution condition repeating tests. This stability and reliability is attributed to the pressure and temperature control of the prior region of the vapor generation system and the SilcoNert coating of SUS in high-concentration regions. The stable vapor generation system enabled monitoring over 8 h 20 min under a 2.0% CV value in a day. Additionally, we successfully tested another CWA compound, GB, to confirm the versatility of the CWA vapor generation system for every CWA compound. Our results confirm that the FT-IR-coupled CWA vapor generation system is a suitable testing and evaluation equipment for real-time monitoring.

METHODS

Fabrication of the FT-IR-Coupled CWA Vapor Generation System. The FT-IR-coupled CWA vapor generation system is designed to control the flow of CWA vapor into the multiple-pass cell of a commercial FT-IR device (I1815-10-E FT-IR, MIDAC Corp., Costa Mesa, CA) and collect the realistic CWA vapor concentration through a Tenax-TA tube for verification of the entire system. The calibration curve of HD vapor for FT-IR monitoring was plotted by collecting 11 points of stable HD vapor stream (Supporting Information). All of the vapor stream lines for CWA vapor generation and measurement consist of 1/4" SUS. Especially, the initial vapor generation part is coated with SilcoNert 2000 (FITOK GmbH, Offenbach, Germany) to prevent the adsorption of high-concentration CWA. The heat exchanger (shell and tube type) is installed to maintain the temperature of the air stream of the vapor generation system constantly. It is designed with an internal volume of 12.4 L and a coiled tube of length 10 m. The estimated contact area is 0.397 m². The water bath

(RW3-2025, Jeio Tech., Daejeon, Korea) precisely circulates temperature-controlled water to the heat exchanger. A PFU-3 coalescing filter (TPC Mechatronics, Incheon, Korea) that can remove 0.01 μm fine dust and 0.3 μm oil is installed at the front of the vapor generation system to provide filtered air stream into the entire vapor generation system. A RGF-250-400 humidity filter (Labclear) is also installed directly next to the coalescing filter to prevent fluctuations of the humidity of the CWA vapor stream. A humidity controller in the system was installed as a bubbler type. However, in this manuscript, we did not control the humidity parameter for the precise control of flow rate (Supporting Information). All of the air streams are controlled by MC-MFCs (mass flow controllers) (Alicat, Scientific Ind.), including the air stream into the CWA saturator cell and the dilution air stream. All of the MFCs can be opened and closed by communicating with the computer through ethernet/IP simultaneously. A precisely controlled and filtered air stream is passed through a delta tube-shaped glass that contains pure CWA liquid. Gradually, the equilibrium phase is reached in the number of molecules between the evaporated and transferred HD molecules. At the first manifold point, a large amount (1–5 L/min) of clean air and a small amount (less than 1 L/min) of high-concentration CWA vapor are mixed homogeneously. The bypass pipeline allows measurement of the concentration of CWA vapor with FT-IR 1 and collection of the CWA vapor through the Tenax-TA tube for GC-FID analysis. The linear three-manifold system is designed to achieve up to three-times dilution for extremely low concentrations of CWA vapor. Similarly, FT-IR 2 is installed after the dilution procedure for real-time monitoring of low-concentration conditions. A three-way port is provided to collect through a Tenax-TA tube for GC-FID analysis. Real-time monitoring data are measured by AutoQuant Pro provided by MIDAC.

Standard Curve and GC-FID Analysis. To obtain a standard curve for CWA gas concentration, we prepared stock solutions of each CWA and diluted them to obtain five standard curve points. A certain amount of synthesized CWA (with a purity of 99% or higher) was taken and put into a 25 mL volumetric flask, and a stock solution was sequentially prepared from high to low concentration using ethyl acetate (EA, purity ≥99.5%, Sigma-Aldrich Inc., St. Louis, MO) as the solvent. This solution was transferred to a 10 mL vial bottle and diluted within the dynamic range. Next, we removed 2 μL of the prepared standard solution into a 10 μL syringe (Hamilton Company, Reno, NV), added the solution into a Tenax-TA, and pulled the solution for 1 min with an adsorption motor to stabilize it. We then analyzed the stabilized ATD-GC to obtain the concentration standard curve, with measurements taken from low to high concentration.

Gas Chromatography–Flame Ionization Detection (GC-FID). All CWA agents in this study were analyzed using a 7890A gas chromatograph (GC) (Agilent Technologies, Santa Clara, CA) equipped with a Model 650 automatic thermal desorber (ATD) (PerkinElmer, Waltham, MA) front end. The adsorbent tube used in these studies was constructed of stainless-steel Tenax-TA (PerkinElmer, Waltham, MA) and contained 2,6-diphenyl-*p*-phenylene oxide polymer. Before use, the tubes were conditioned under nitrogen at 280 °C for 50 min under a purified nitrogen pressure of 25 psi.

Single tubes were analyzed by capillary gas chromatography (GC). The Tenax was heated to 250 °C over a period of 5 min

and maintained at 250 °C for an additional minute. During this time, the nitrogen carrier gas line was flushed with nitrogen at 70 mL/min. The GC conditions of analysis were as follows: the GC column was an HP-5 fused silica capillary (30 m × 0.25 μm I.D., Agilent); the flame ionization detector (FID) was set at 280 °C; the oven temperature was increased from 40 to 120 °C at 10 °C/min, from 120 to 280 °C at 30 °C/min, and held at 280 °C until the end of the run; and the make-up gas was nitrogen, with hydrogen and air flows of 25, 30, and 400 mL/min.

■ ASSOCIATED CONTENT

SI Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsomega.3c01301>.

Images of the humidity bubbler, temperature and humidity measurement, and monitoring system; flow rate-dependent humidity control; calibration curve for HD vapor generation; and their monitoring with the FT-IR monitoring system (PDF)

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Author Contributions

J.-S.K. designed the FT-IR-coupled vapor generation system and H.S.S. and J.-S.K. conducted vapor generation experiments and wrote the manuscript mainly. H.S.S. and Y.J.K. analyzed the concentration of CWA vapor and optimized the methodology analysis system. H.N. conducted MFC networking and coupling of the dual FT-IR system for real-time monitoring of CWA concentration. All authors discussed the results and commented on the manuscript.

Notes

The authors declare no competing financial interest.

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