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VAPOR PRESSURE DETERMINATION OF VM USING THE DENUDER-LIQUID CHROMATOGRAPHY-MASS SPECTROMETRY TECHNIQUE

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14. ABSTRACT The vapor pressure of <i>O</i> -ethyl <i>S</i> -2-(diethylamino) ethyl methylphosphonothiolate (VM) has been determined using a novel, denuder-based liquid chromatography–mass spectrometry (LC–MS) approach designed specifically for low-volatility and thermally unstable materials. This system incorporates a novel, packed-bed, vapor-generation system specifically designed to deliver low-volatility and thermally unstable compounds at low or high flow rates using milligram quantities of the material. The vapor pressure analysis system takes advantage of denuder technology to efficiently transfer the chemical sampled from the airstream into a liquid vehicle for transport to a detector such as LC–MS. Capturing the chemical of interest into a liquid vehicle eliminates the need for heated transfer lines by virtually eliminating line losses. The results of the vapor pressure study of VM are presented.					
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PREFACE

The work described in this report was authorized under project no. WBSR.0014307.20.1. The work was started in June and completed in September of 2012.

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VAPOR PRESSURE DETERMINATION OF VM USING THE DENUDER-LIQUID CHROMATOGRAPHY-MASS SPECTROMETRY TECHNIQUE

1. INTRODUCTION

Limited vapor pressure data has been available for *O*-ethyl *S*-2-(diethylamino) ethyl methylphosphonothiolate (VM) because of its low volatility and thermal instability. A novel vapor-generation system and a new vapor pressure measurement platform for low-volatility materials were developed to address the problems associated with difficult-to-measure species. The system was designed and built by the Chemical, Biological, and Radiological Filtration team of the U.S. Army Edgewood Chemical Biological Center.

Traditional means of generating chemical vapors for filter testing include standard saturator cells and aerosol generators, which can be combined with heat and air to create vapors. These systems are not always suitable for application to compounds that exhibit temperature sensitivity or low volatility or have a high cost of custom synthesis. These types of chemicals are often cost-prohibitive and difficult to obtain in quantities that are large enough to support operation of standard saturator cells or other types of aerosol-vapor generators. Additionally, many of these materials are highly toxic, which makes working with them in large quantities undesirable.

A novel approach was developed to address these issues and create a stable, high-flow vapor generator for low-volatility materials that requires only minimal chemical consumption. The technique was based on the science of static-coating capillary columns.¹ Similarly, a packed bed of inert particles is filled with a solution of the test chemical. As the volatile solvent is stripped away under vacuum, the test chemical remains behind as a film, coating the contacted surfaces. A generator is created when a carrier gas flows through the packed bed. This allows an operator to predetermine the amount of chemical needed to conduct the required testing. Using the well-established engineering principles incorporated in the packed-bed design,² the resulting generator output can be scaled to meet the flow-rate requirements of the test. When an inert carrier gas such as nitrogen is used, and the bed temperature is constant, the vapor-chemical film-coating equilibrium is not exceeded at the outlet end of the generator. Therefore, the vapor generator produces stable output-chemical concentrations that are based on the vapor pressure of the test chemical. The output concentration remains constant until the chemical is either consumed or the dynamic factors change.³

Many analytical methods, such as those commonly used for air monitoring, require that the gases under study be measured in real time using validated analytical procedures. Unfortunately, many of these methods for temperature-sensitive compounds involve the use of impingers or gas-sampling tubes, which have limitations.⁴ Impingers are often used to collect chemicals such as acids and anhydrides. One of the biggest problems associated with impingers involves how to get the air-vapor sample into a liquid vehicle for detection. When using an impinger, a known volume of air is actively drawn through a liquid medium. The liquid chemically reacts with or physically dissolves the chemical being sampled. The resulting liquid

is then analyzed to determine the airborne chemical concentration. In the case of gas-sampling tubes, the air is drawn through a solid medium, such as a resin or carbon, which traps the analyte. As with the impingers, the sample is collected on the medium, extracted using an appropriate solvent, and analyzed to determine the concentration. Many drawbacks are associated with impingers and sampling tubes, including the difficulty of use, the excessive time requirements, and the lack of reproducibility, which tend to make these methods less than optimal.⁴ Most importantly, impingers and gas-sampling tubes do not allow for the real-time detection that is required for breakthrough studies.

Alternatively, instrumentation such as gas chromatography–mass spectrometry (GC–MS) systems do have the capability to detect gas samples in real time. However, not all analytes can be detected using GC–MS, and just getting the sample to the instrument is problematic. For example, vapor sampling through tubing can result in sample losses because of surface adsorption to the tube walls. To mitigate this problem, air samples are typically ported to the instrument via heated transfer lines, and specially coated tubing may be used to reduce chemical reactivity. However, line losses can still be significant. Heated transfer lines are also problematic and must be carefully controlled. If a line is too hot, the analyte can break down, and if it is too cold, the material being sampled will tend to adsorb to the colder regions.

To address the sample transport and adsorption concerns for temperature-unstable, low-volatility chemicals, a new technique that combines denuder sampling technology and liquid chromatography (LC) for near-real-time detection was developed. The denuder air-sampling technique used in this work was adapted from a method developed by Hoke and coworkers to detect acid gases in firefighting applications.^{5–8} Denuders may be likened to continuous impingers that incorporate gas-sampling analysis technology to continuously introduce a gas or aerosol sample into a solvent stream, where the analyte of interest is taken up into a trapping solution. The trapping solution is delivered to the denuder by a chemical pump (e.g., a solenoid, syringe, peristaltic, rotary, or high-performance liquid chromatography [HPLC] pump). Sample flow rates on the order of 1–4 L/min are obtained using a mass flow controller (MFC), rotameter, or critical orifice, with a vacuum source. The vacuum source, which is external to the analyzer, continuously draws sample air into the denuder, where the chemical agent or aerosols are extracted into the liquid phase of the trapping solution to be quantified. The solution can then be discarded, saved, or sampled for further evaluation as needed.

2. EXPERIMENTAL PROCEDURES

2.1 Vapor Generator Preparation

The generator was constructed using a custom-designed, plastic-coated borosilicate glass saturator cell filled with 4 mm borosilicate beads, as shown in Figure 1 (Glassblowers, Inc.; Turnersville, NJ). Once the vapor generator was assembled, the inner walls of the pipe were rinsed several times with dichloromethane to remove impurities. The filling solution was prepared by dissolving 50–100 mg of VM in approximately 60 mL of dichloromethane. The resulting solution was then transferred into one arm of the saturator cell. Repeated solvent rinses were added to wash any residual filling solution into the generator. Solvent was added until the solution level was within 2 cm of the top of the beads.

To remove the solvent, a vacuum was slowly applied to the exit arm of the saturator using a suitable pressure controller operating at an approximately 1 L/min flow rate at 100 torr. The gradual reduction from ambient pressure to 200 torr over 20 min allowed the chemical solution to be mixed during the solvent outgassing. The evaporation was monitored to ensure the bubble rate in the beads was not violent enough to cause splashing on the walls. The entire solvent-stripping process took approximately 4 h.

To prepare the vapor generator for use, the saturator cell was fitted with the chemically inert carrier stream hardware and the corresponding MFC for the ensuing vapor pressure tests. The generator was then conditioned for approximately 24 h under an airflow into a waste-scrubber filter to remove high-volatility impurities and purge residual test chemicals from the arm. Once conditioned, the generator was placed in a water bath (Thermo Neslab; Newton, NH) so that its temperature could be carefully controlled for testing. The generator was then integrated into the vapor pressure test system. The airstream entered the side arm of the generator and passed through the thin-film coating of the chemically wetted surfaces of the glass beads. This produced chemical vapor outlet concentrations equal to the vapor pressure of the test chemical at the temperature of the bed. The chemical stream then exited the center arm of the generator and was delivered to the denuder for vapor pressure analysis.

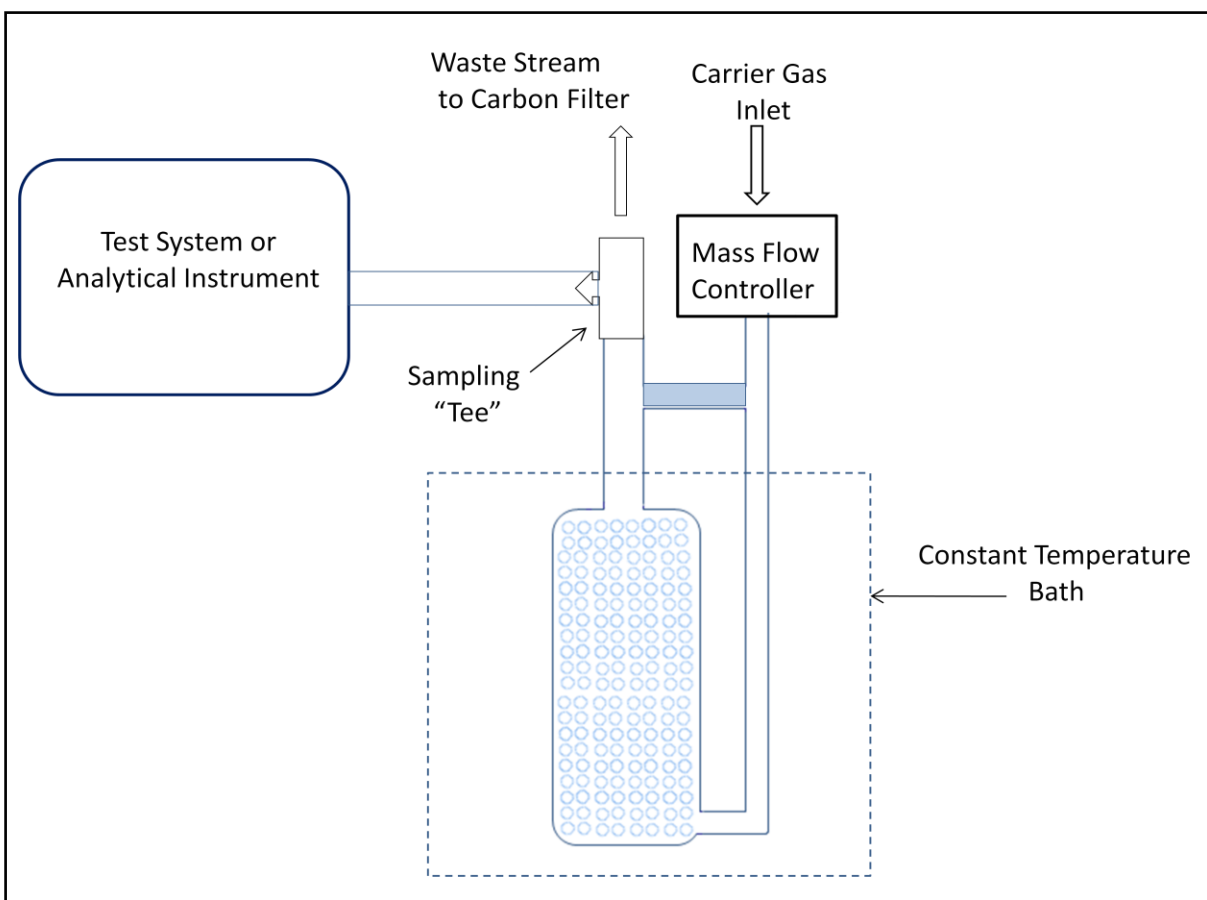
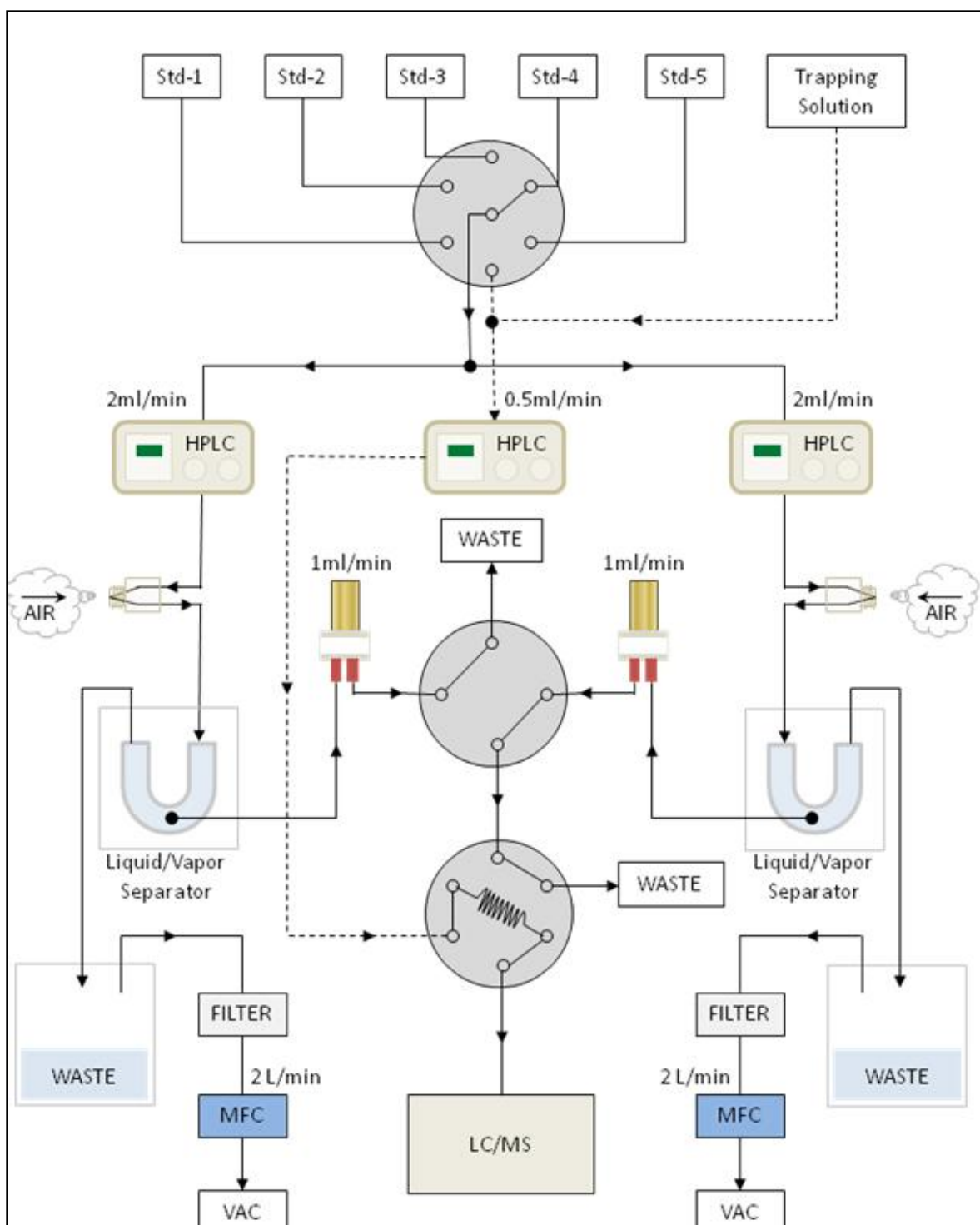


Figure 1. The packed-bed vapor generator.

Once the generator was placed in the water bath and connected to the denuder system, the arm carrying the vapor stream was wrapped in heat tape set to 85 °C. The entire system was then wrapped again in aluminum foil. These precautions were taken to prevent any of the sample from condensing in a “cold spot” before it reached the denuder. LabView software (National Instruments; Austin, TX) was used to control the temperature of the water bath as well as the timing of the collections. The water-bath temperature at every set point was verified by comparison with readings from an external mercury thermometer. The nitrogen flow rate through the generator was 100 sccm/min (standard cubic centimeters per minute) for temperatures of 30 °C and above and 10 sccm/min for temperatures below 30 °C.

2.2 Vapor Pressure Analysis

The vapor sampling shown in Figures 2 and 3 was conducted using A-7 denuders (St. John Associates; Beltsville, MD) with 1.25 cm diameter bodies and 6.4 mm tapered pipe threads. The denuders were fabricated of chemically resistant Kel-F material (3M; Saint Paul, MN). Except where noted, all tubing used to connect this sampling system was 3.2 mm o.d. $\times$ 1.6 mm i.d. polytetrafluoroethylene (PTFE). The trapping solution used for the experimental compound was HPLC-grade acetonitrile (Sigma-Aldrich; St. Louis, MO). The trapping solution was transported to the denuder via a LabAlliance 1500 series HPLC pump (Thomas Scientific; Swedesboro, NJ) set to a flow rate of 2.0 mL/min. The pump delivered the solution to the denuder through an in-line 500 psi back-pressure check valve, which maintained pumping efficiency via 1.6 mm PTFE tubing that was connected directly to the base of the denuder. The vacuum return line of the denuder was connected to the top of a specially designed U-tube bottle (Glassblowers, Inc.) fitted with a three-hole GL45 vacuum cap (Diba Industries; Danbury, CT) to coalesce the returning trapping solution. Vacuum tubing connected to the opposite top end of the U-tube was used to siphon off the excess trapping solution, once the U-tube was filled, and to direct it to a glass waste container that was fitted with a similar two-hole vacuum cap (Diba Industries). The sample vacuum through each of the denuders (1.5 sLpm [standard liters per minute]) was controlled by a suitable MFC protected by an in-line filter. The in-line filter was useful in creating a slight pressure drop between the waste container and the MFC to prevent vapor condensation inside the controller.



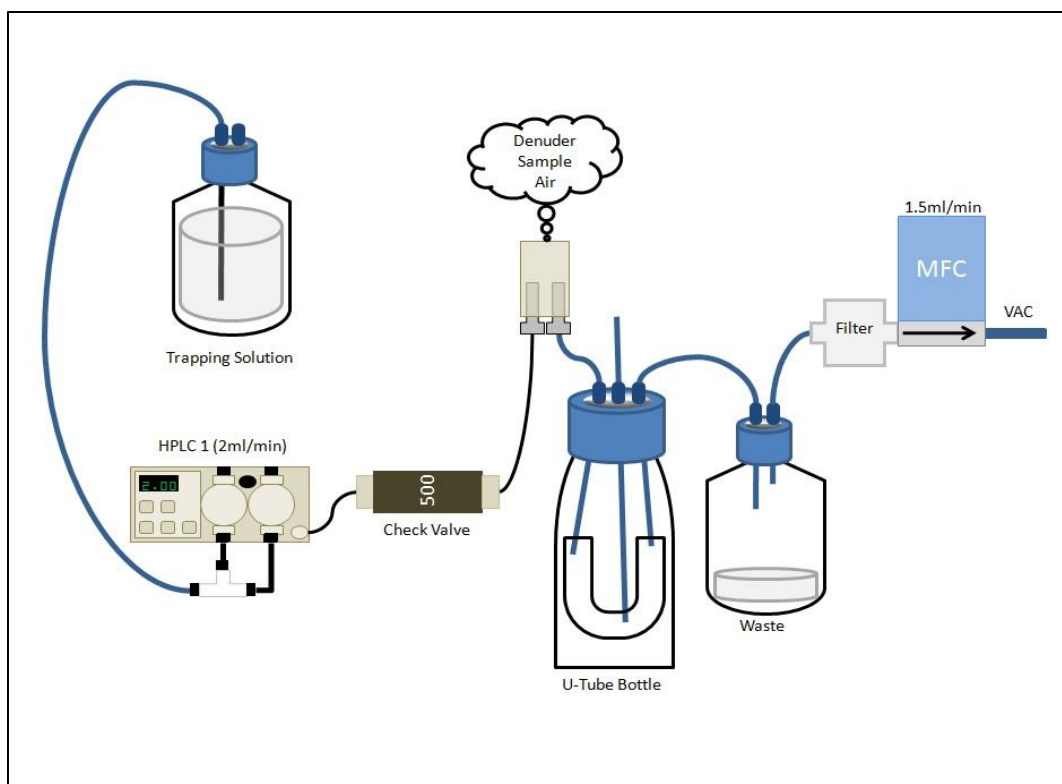


Figure 3. The denuder sampling apparatus.

A 3.2 mm, two-position, six-port 316 stainless steel switching valve (Valco Instruments Company; Houston, TX), shown in Figure 4, allowed for convenient alternating sampling of the two filter effluent streams during testing. Solenoid pumps with 50 μL displacement (model 130SP2450-1TP; Bio-Chem Fluidics; Boonton, NJ), operating on a 3 s cycle, were used to pump a portion of the trapping solution from the respective U-tubes to the steam-selector valve, SV1. The selected denuder stream (output from SV1) was connected to a similar six-port valve, SV2, and configured as shown in Figures 4 and 5. Because of the high pressure of the LC–MS injection system, a separate HPLC pump was used to transfer the sample loop contents (0.5 mL) from the selected denuder sample to the LC–MS injection loop (25 μL). The same trapping solution that was used in the denuder sample collection was used as the mobile phase (0.40 mL/min) for the HPLC pump (Figure 3) to fill the injection loop of the LCQ Advantage Max LC–MS (Thermo Finnigan; San Jose, CA) for analysis. The mobile phase was 0.1% formic acid of $\geq 98\%$ purity (Sigma-Aldrich) in HPLC-grade acetonitrile. All valve-switching, water-bath temperature control, and start signals were provided through an automated LabView process-control program that was written in-house. The filling of the LC–MS analysis loop was timed to receive the center cut of the denuder sampling loop of SV2.

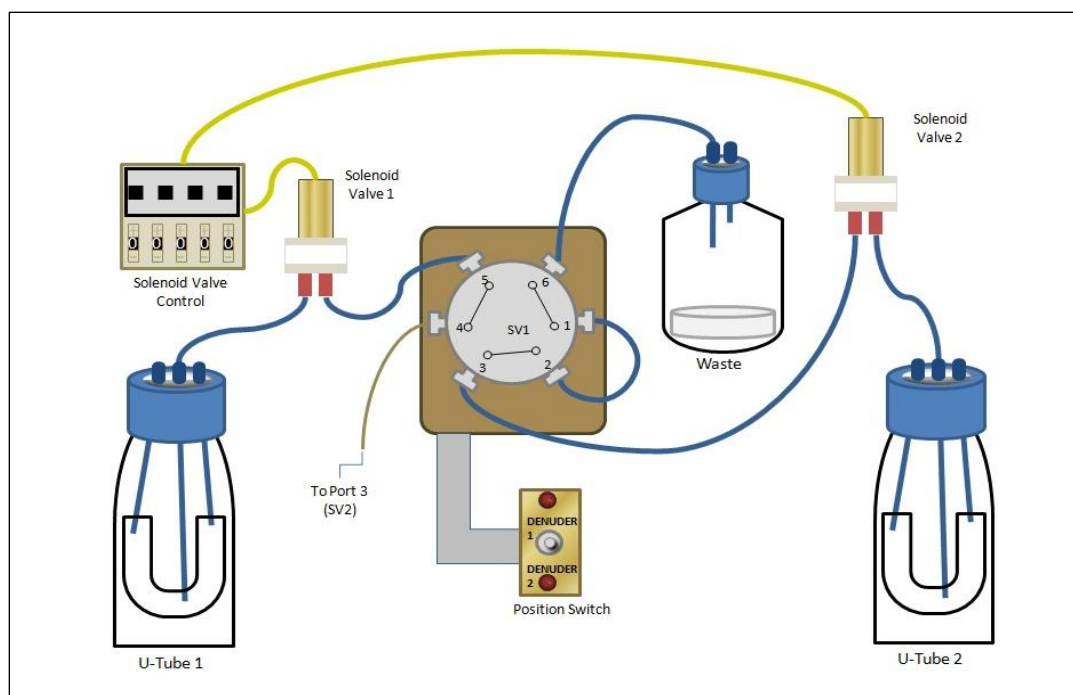


Figure 4. Sample transfer.

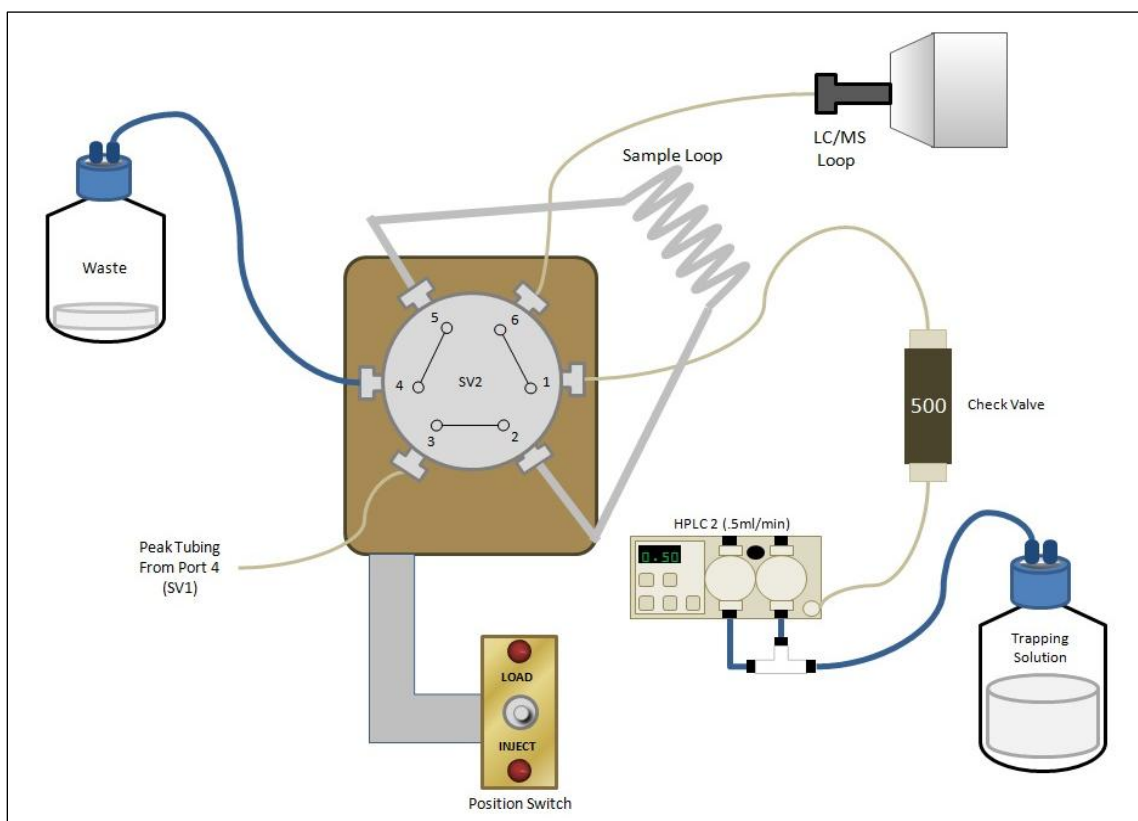


Figure 5. Second stage of the denuder sample-collection system.

2.3 Calibration

Standard calibration using the denuder–LC–MS system was performed before and after each set of vapor pressure measurements. This ensured that instrument reproducibility did not affect the results. Five freshly prepared standards ranging from 1.045×10^{-2} to 2.09 mg/L in acetonitrile were used for each analysis. The standards were made from a 10.45 mg stock solution in acetonitrile that was also prepared fresh daily. The fresh standards were used to prevent any bias that might have occurred from degradation of the sample over time. Table 1 shows the instrument settings used in the experiment. Each standard was measured at least 15 times, and the measurements were averaged for each calibration point. A representative calibration curve is shown in Figure 6.

Table 1. LC–MS Instrument Settings

Parameter	Test Condition
Mobile phase	0.1% formic acid in acetonitrile, run at 400 μ L/min
Run time	4 min
Tune type	Zoom scan; range ± 2 u, centered on the parent ion of the test chemical

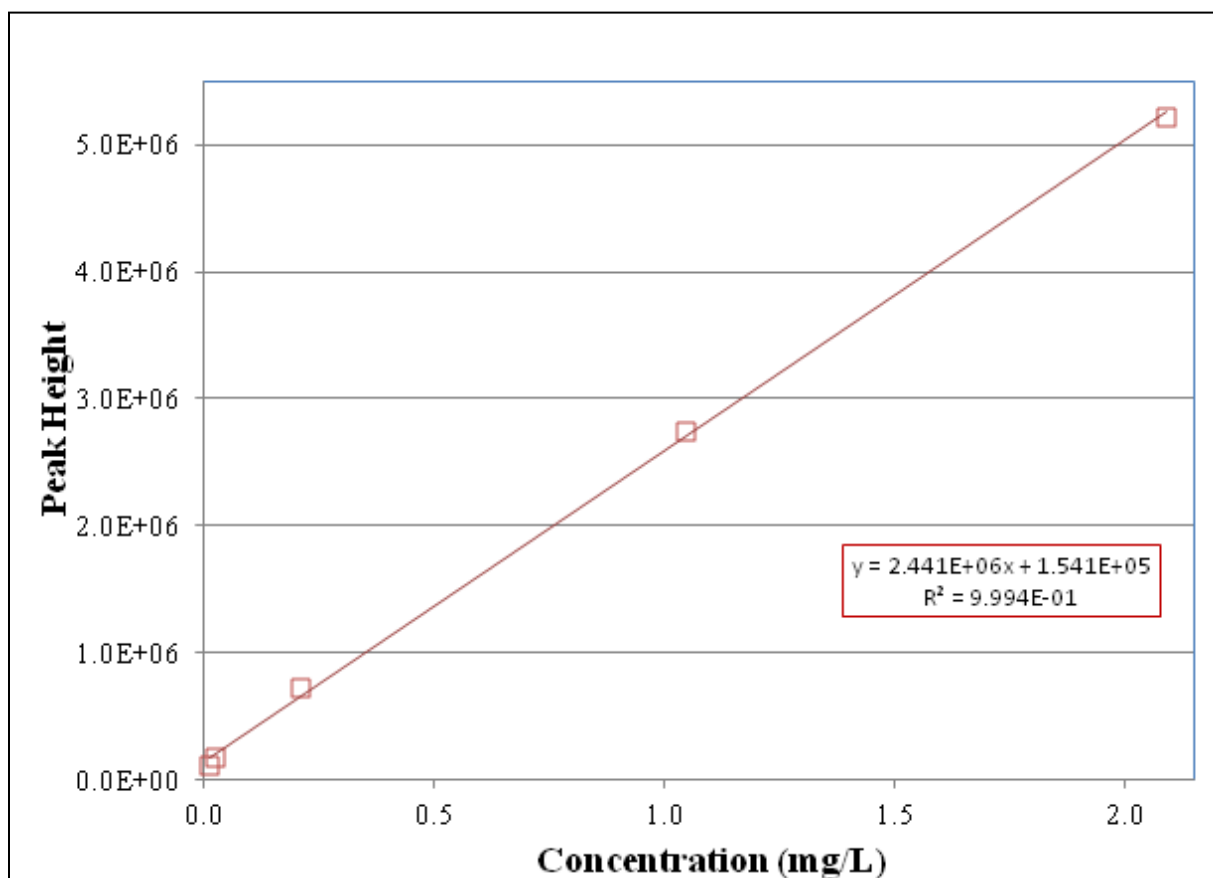


Figure 6. VM calibration data from 12 June 2012.

3. VAPOR PRESSURE ANALYSIS: RESULTS AND DISCUSSION

Vapor pressure measurements were collected four different times on different days. Stock and standard solutions were prepared fresh each day to reduce the error associated with sample decomposition. Calibration was performed before and after each of the vapor pressure runs to eliminate instrument-variability errors in the results. The vapor pressure results are summarized in Table 2. The data showed reproducibility at each measured temperature. When compared with a limited data set that was obtained by another research group using this material, the denuder method agreed within 30%. The denuder data were also compared with vapor pressure curves plotted for materials with similar vapor pressures, as shown in Figure 7. As expected, the curve from the denuder data had a similar slope.^{17,18}

Table 2. Summary of Vapor Pressure Data

Date	Temperature		Pressure (Pa)		Absolute Difference (%)
	°C	K	Raoult's Law	Calculated	
7 June 2012	10.00	283.15	5.96E-02	8.71E-02	31.5
	15.00	288.15	1.41E-01	1.49E-01	5.5
	20.00	293.15	2.85E-01	2.50E-01	14.0
	30.00	303.15	6.23E-01	6.61E-01	5.7
	40.00	313.15	1.62E+00	1.63E+00	0.7
12 June 2012	40.00	313.15	1.19E+00	1.63E+00	26.7
	30.00	303.15	5.11E-01	6.61E-01	22.7
	20.00	293.15	1.70E-01	2.50E-01	31.8
	10.00	283.15	7.06E-02	8.71E-02	18.9
	0.00	273.15	2.78E-02	2.78E-02	0.0
	-9.80	263.35	5.66E-03	8.19E-03	30.9
19 June 2012	-9.80	263.35	1.14E-02	8.19E-03	38.7
	5.00	278.15	6.05E-02	4.98E-02	21.6
	10.00	283.15	6.99E-02	8.71E-02	19.7
	20.00	293.15	2.13E-01	2.50E-01	14.5
	30.00	303.15	7.36E-01	6.61E-01	11.4
20 June 2012	35.00	308.15	1.05E+00	1.05E+00	0.0
	30.00	303.15	7.93E-01	6.61E-01	20.0
	25.00	298.15	5.33E-01	4.10E-01	29.9
	20.00	293.15	2.87E-01	2.50E-01	15.0
	10.00	283.15	9.80E-02	8.71E-02	12.5

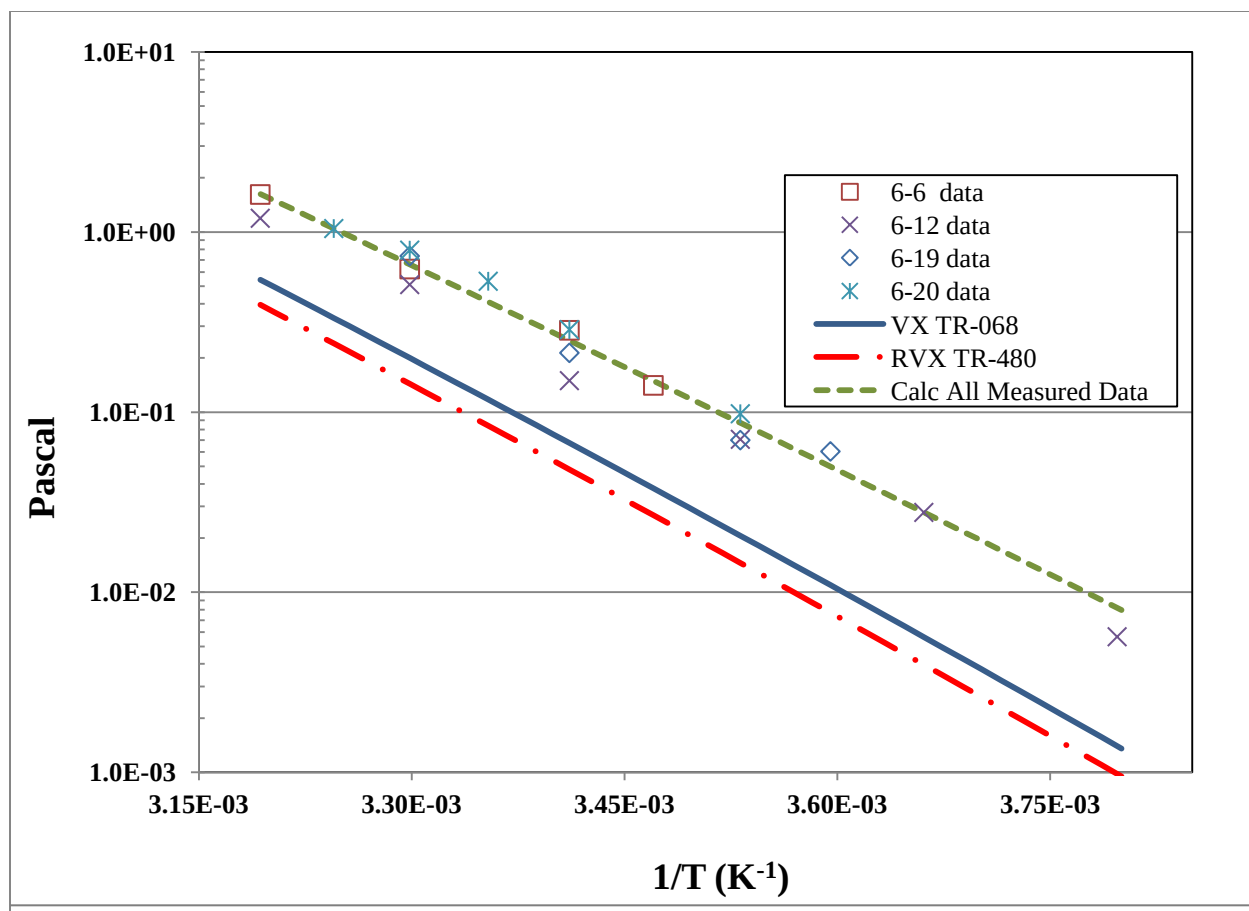


Figure 7. Comparison of VM with *O*-ethyl *S*-(2-diisopropylaminoethyl) methylphosphonothioate and *O*-isobutyl-*S*-(diethylaminoethyl) methyl phosphonothiolate (VX and RVX, respectively).

4. CONCLUSIONS

The denuder–LC–MS vapor pressure method appeared to work reasonably well for the material studied. The relative vapor pressure of the material was slightly higher than that of VX or RVX, as was expected.

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ACRONYMS AND ABBREVIATIONS

GC	gas chromatography
GC–MS	gas chromatography–mass spectrometry
HPLC	high-performance liquid chromatography
LC	liquid chromatography
LC–MS	liquid chromatography–mass spectrometry
MFC	mass flow controller
MS	mass spectrometry
PTFE	polytetrafluoroethylene
RVX	<i>O</i> -isobutyl- <i>S</i> -(diethylaminoethyl) methyl phosphonothiolate
sccm/min	standard cubic centimeters per minute
sLpm	standard liters per minute
SV	selector valve
VM	<i>O</i> -ethyl <i>S</i> -2-(diethylamino) ethyl methylphosphonothiolate
VX	<i>O</i> -ethyl <i>S</i> -(2-diisopropylaminoethyl) methylphosphonothioate

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