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Vapor Sensing Performance of the University of Missouri Rolla Explosives Vapor Sensor

James M. Phelan, James L. Barnett and Dayle R. Parker

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Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550

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James M. Phelan
Environmental Technology Department

James L. Barnett and Dayle R. Parker
Explosive Materials and Subsystems Department

Sandia National Laboratories
P.O. Box 5800
Albuquerque, NM 87185-0719

Abstract

Sensing the vapors from explosives has many applications for military, humanitarian demining and homeland security applications. The University of Missouri at Rolla has developed a trace chemical detector system that is based on a dual electron capture detector method. The system's vapor sensing performance was evaluated with headspace vapor standards from aqueous solutions of TNT and DNT, and with soil standards using DNT. Replicate measurements showed that the UMR Vapor Sensor could detect low concentrations of TNT and DNT. However, the system showed large variability and a narrow linear range. Preliminary tests with natural plant odors indicated that the vapor sensor response to nitroaromatic explosive compounds was not specific.

Acknowledgements

This work was sponsored by the University of Missouri, Rolla through Dr. Robert Mitchell and Dr. Shubhen Kapila. Dr. Paul Nam, University of Missouri Rolla, is gratefully acknowledged for instrument operation and data collection for this effort. James Barnett and Dayle Parker are gratefully acknowledged for preparation of the test standards and operation of the chemical analysis systems for the performance tests. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

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1.0 Introduction

Trace chemical detection of explosive signatures is valuable technology with military (Jenkins et al, 2001), humanitarian demining (Phelan and Webb, 2002) and homeland security applications (NRC, 2002). Analyte specificity and sensitivity are key factors necessary for successful systems. Detectors need a selective response to specific target compounds to minimize false alarm indications. Detectors must be very sensitive because of the ultra low concentrations of target analytes typically found in field situations.

The University of Missouri at Rolla (UMR) has been developing a trace chemical detection system for several applications. One configuration has been developed for application to explosive vapor sensing ("UMR Vapor Sensor"). This instrument was brought to Sandia National Laboratories to evaluate the vapor sensing performance (sensitivity and selectivity to 2,4,6-trinitrotoluene [TNT] and 2,4-dinitrotoluene [DNT]) and complete an initial screen on the affect of natural odors that might impact chemical sensing for buried landmines.

2.0 Performance Test Standards

2.1 Introduction

The estimated vapor-sensing threshold of the UMR Vapor Sensor was reported by UMR to be about 250 fg following a sample collection and preconcentration of 50 mL/min for 4 seconds, which corresponds to a vapor concentration of about 0.075 ng/L (10 ppt). The best comparison headspace vapor sampling and analysis tool is solid phase microextraction (SPME) with gas chromatograph/electron capture detection (GC/ECD). With a minimum 10 pg ECD signal, a 100 min sampling time and a SPME sampling rate of 10 mL/min, the lowest detectable vapor concentration is about 0.01 ng/L (1 ppt).

In order to quantify the detection threshold of the UMR Vapor Sensor, we prepared DNT soil headspace vapor standards and both TNT and DNT water headspace vapor standards. Soil and water standards were prepared to produce varying levels of TNT and DNT vapors as a headspace in closed containers. The containers were filled half full with the source material leaving half the volume for headspace vapor sensing. The containers were opened just long enough for sample collection and then closed to re-equilibrate for future testing. Headspace standards were prepared to bracket the expected sensing threshold of the UMR Vapor Sensor and were verified (above 1 ppt) using SPME sampling and GC/ECD quantification measurements.

2.2 Water Headspace Vapor Standards

Headspace vapor standards created with water solutions of TNT and DNT are modeled after the water-air partitioning process described by Henry's Law. Henry's Law constant is a relative measure of the amount of the chemical that exists in the gas phase to that in the aqueous phase, at equilibrium, and is defined as

$$K_H = \frac{C_G}{C_L} \qquad [1]$$

where K_H is the Henry's Law constant (unitless) and C_G is the concentration in gas phase (g/cm^3 headspace) and C_L is the concentration in the liquid (aqueous or water) phase (g/cm^3 water). Henry's Law constant is also a function of temperature because both C_G and C_L are functions of temperature. Several groups (Dionne, 1986; Pella, 1977) have collected vapor pressure data for TNT and DNT. The data from Pella (1977) have been used in this work and are shown in Table 1.

Table 1. Vapor Pressure versus Temperature Relationships (Pella, 1977)

Chemical	Vapor Pressure Equations
TNT	$\log_{10}(p/\text{Torr}) = (12.31 \pm 0.34) - (5175 \pm 105)\ K/T$
DNT	$\log_{10}(p/\text{Torr}) = (13.08 \pm 0.19) - (4992 \pm 59)\ K/T$

Water solubility data for DNT and TNT was used from Phelan and Barnett, 2001. An empirical relationship of water solubility as a function of temperature was determined and is shown in Table 2.

Table 2. Aqueous Solubility Empirical Correlation [$y = a+b(T/°C)^c$]

	A	b	c
DNT	135.59	0.0064382	2.8569
TNT	86.045	0.0034874	2.9131

Using the information from Tables 1 and 2, a relationship for K_H as a function of temperature was developed and is graphically shown in Figure 1.

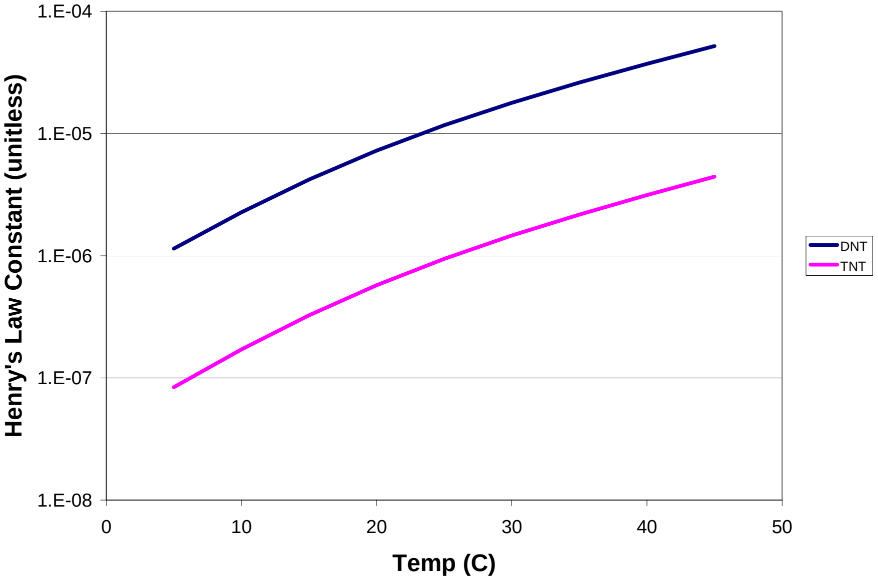

Figure 1. DNT and TNT Henry’s Law Constant as a Function of Temperature

To achieve the desired headspace vapor concentrations for testing at the limit of detection, serial dilutions of stock aqueous solutions of TNT or DNT were prepared to target the values shown in Table 3 and 4. The stock solutions of TNT and DNT were 10 mg/L. The detection limit of a direct aqueous injection for the HPLC is about 50 μg/L. From Table 3 and 4, the water solutions will be near this detection limit. If lower solution concentrations become necessary, validation of solution concentrations would require preconcentration using Solid Phase Extraction (SPE) with Gas Chromatography/Electron Capture Detection (GC/ECD), which extend the detection limit down to 0.05 μg/L. However, at these ultra trace levels, the variability of the measurements using SPE will increase causing more uncertainty in the headspace vapor concentration estimates. Thus, at the lowest solution concentrations we will extrapolate the value from the lowest measured solution concentration and will verify headspace concentrations with SPME sampling/analysis.

Figure 2 shows the headspace concentrations of TNT and DNT as a function of aqueous phase concentration with the HPLC and SPE-GC/ECD detection limits. Another method using headspace vapors derived from contaminated soils will also be used and is described in Section 2.3.

Table 3. DNT Headspace Vapor Concentration from Aqueous Solution (23°C)

Water Concentration	Headspace Concentration	
ug/L	ng/L	ppt
1	0.015	1.7
5	0.053	8.5
30	0.32	50.8
50	0.52	84.7
100	1.05	169
500	4.78	767

Table 4. TNT Headspace Vapor Concentration from Aqueous Solutions (23°C)

Water Concentration	Headspace Concentration	
ug/L	ng/L	ppt
10	0.010	1.3
50	0.051	6.6
300	0.306	39.7
500	0.510	66.2
1000	1.02	132
5000	5.09	662

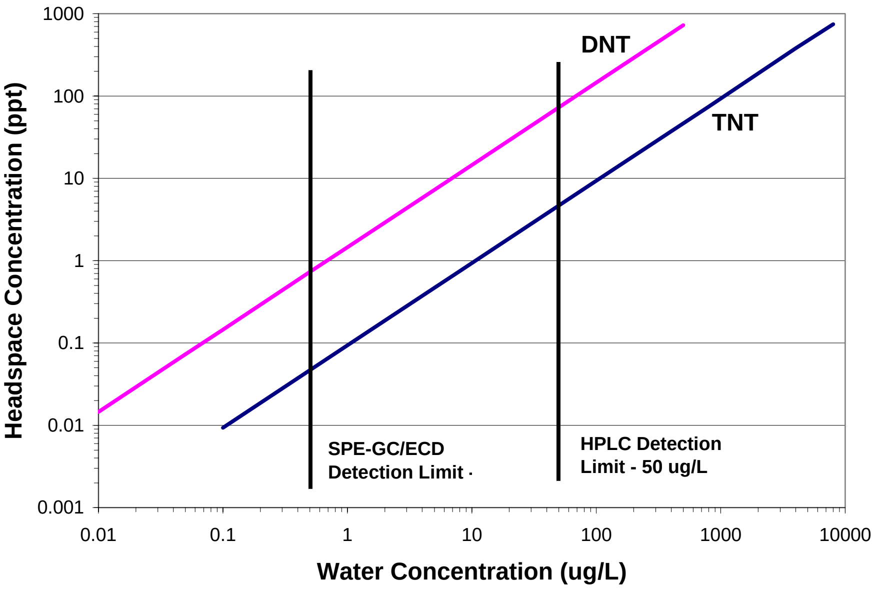

Figure 2. Headspace Vapor Concentrations of TNT and DNT with Aqueous Analytical Method Detection Limits (23°C)

2.3 Soil Headspace Vapor Standards

Soils can also produce discrete vapor headspace values that are a function of total soil concentration and soil moisture content. This phenomenon combines three phase partitioning processes: water-air partioning (K_H), soil-water partioning (K_d) and soil-vapor partioning ($K_{d'}$). Using methods developed previously in our lab, we are able to prepare known headspace vapor concentrations over a wide dynamic range (Phelan and Barnett, 2001).

Soil standards were prepared by mixing crystalline chemical into Sandia loam soil (sieved to retain < 2 mm fraction) in a one-gallon paint can on a rotating tumbler for about 24 hours. The container was then placed into an oven at 100°C for four hours, and then placed on the rotating tumbler for 24 hours. Serial dilutions of the stock material were prepared using clean soil in one-quart paint cans. Stock soil standards were prepared to target ~ 7,000 ng/g for DNT. Five replicate samples of each soil were obtained, extracted with acetonitrile and quantified on the GC/ECD. Mean and standard deviation values for each soil are shown in Table 5. The method detection limit for DNT in soil is about 5 ng/g.

Headspace vapor concentrations for soil standards at soil moisture contents up to 11% (wt/wt) are also shown in Table 5. The average (std dev) soil moisture contents of the dry DNT soil standards were measured to be 0.5% w/w (0.07) (n=3). At this low moisture content, the vapor concentrations for even the highest soil residues were too low for the UMR Vapor Sensor to measure. Thus, the soil was wetted to a target soil moisture content to bracket the expected UMR Vapor Sensor performance. One complication for the wet soil headspace testing is the uncertainty in time to reach equilibrium vapor concentrations. In previous preliminary and very limited kinetics testing, equilibrium was reached in 24 hours for TNT, but required about 6 days for DNT (Phelan and Barnett, 2001). However, after about two hours, the DNT had reached about 50 percent of the equilibrium maxima.

Table 5. DNT Soil Standards [mean (std dev) n=5] and Estimated Headspace Vapor Concentrations (23°C) as a Function of Soil Moisture Content

	D-1		D-2		D-3		D-4		D-5		D-6	
Moisture Content	Soil Residue 6599 (370) ng/g		Soil Residue 779 (30) ng/g		Soil Residue 79 (3) ng/g		Soil Residue 8 (0.5) ng/g		Soil Residue 1 (0.1) ng/g		Soil Residue 0.1 ng/g estimated	
g/g	headspace (ng/l)	headspace (ppt)	headspace (ng/l)	headspace (ppt)	headspace (ng/l)	headspace (ppt)	headspace (ng/l)	headspace (ppt)	headspace (ng/l)	headspace (ppt)	headspace (ng/l)	headspace (ppt)
0.001	1.1E-06	1.6E-04	1.3E-07	1.9E-05	1.3E-08	2.0E-06	1.4E-09	2.1E-07	1.7E-10	2.5E-08	1.7E-11	2.5E-09
0.010	4.4E-04	6.6E-02	5.2E-05	7.7E-03	5.3E-06	7.8E-04	5.7E-07	8.4E-05	6.7E-08	9.9E-06	6.7E-09	9.9E-07
0.015	5.1E-03	7.5E-01	6.0E-04	8.9E-02	6.0E-05	9.0E-03	6.5E-06	9.7E-04	7.7E-07	1.1E-04	7.7E-08	1.1E-05
0.020	3.6E-02	5.4E+00	4.2E-03	6.3E-01	4.3E-04	6.4E-02	4.6E-05	6.9E-03	5.4E-06	8.1E-04	5.4E-07	8.1E-05
0.025	1.7E-01	2.6E+01	2.0E-02	3.1E+00	2.1E-03	3.1E-01	2.2E-04	3.3E-02	2.6E-05	3.9E-03	2.6E-06	3.9E-04
0.030	6.1E-01	9.2E+01	7.3E-02	1.1E+01	7.4E-03	1.1E+00	7.9E-04	1.2E-01	9.3E-05	1.4E-02	9.3E-06	1.4E-03
0.035	1.7E+00	2.5E+02	2.0E-01	3.0E+01	2.0E-02	3.0E+00	2.2E-03	3.3E-01	2.6E-04	3.8E-02	2.6E-05	3.8E-03
0.040	3.8E+00	5.7E+02	4.5E-01	6.7E+01	4.6E-02	6.8E+00	4.9E-03	7.4E-01	5.8E-04	8.7E-02	5.8E-05	8.7E-03
0.050	1.2E+01	1.9E+03	1.5E+00	2.2E+02	1.5E-01	2.2E+01	1.6E-02	2.4E+00	1.9E-03	2.8E-01	1.9E-04	2.8E-02
0.070	4.3E+01	6.4E+03	5.1E+00	7.6E+02	5.1E-01	7.7E+01	5.5E-02	8.2E+00	6.5E-03	9.7E-01	6.5E-04	9.7E-02
0.090	7.1E+01	1.1E+04	8.3E+00	1.2E+03	8.5E-01	1.3E+02	9.1E-02	1.4E+01	1.1E-02	1.6E+00	1.1E-03	1.6E-01
0.110	8.6E+01	1.3E+04	1.0E+01	1.5E+03	1.0E+00	1.5E+02	1.1E-01	1.6E+01	1.3E-02	1.9E+00	1.3E-03	1.9E-01

Using the values from Table 5, Figure 3 shows the dynamic range of DNT headspace vapor concentrations as a function of total soil concentration and soil moisture content, respectively.

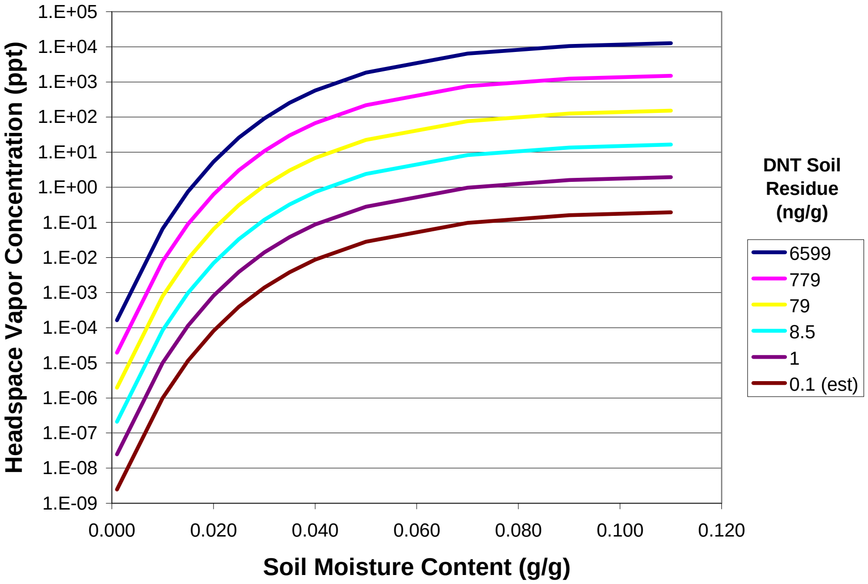

Figure 3. DNT Soil Headspace Concentrations as a Function of Soil Moisture Content

Pre-test calculations showed that the sensor threshold testing could be accomplished with most aqueous solutions above the HPLC detection limit of 50 μg/L. Most of the work was focused on use of the aqueous solutions. The dry soil standards prepared at the six levels (about 10x apart) were going to be much too low in headspace vapor concentration. Therefore, the DNT soil standard D-3 was selected, wetted up to about 7% (w/w) and allowed to equilibrate for ~2 hours.

2.4 Headspace Verification Sampling and Analysis

To verify the headspace vapor concentrations of TNT and DNT, solid phase microextraction (SPME) fibers were used to sample the headspace vapors, which are then quantified using a GC/ECD. The polydimethylsiloxane–divinyl benzene (65 μm PDMS/DVB) coated SPME fibers (Supleco) were used for this application. The SPME fibers sample TNT and DNT at a constant rate, which allows one to measure the sampling rate and derive an effective volumetric sampling rate using a saturated headspace vapor concentration, as follows (Jenkins et al., 2001):

$$EVSR_{SPME} = \frac{MSR_{SPME}}{VD_T} \qquad [2]$$

where, the $\mathrm{EVSR}_{\mathrm{SPME}}$ is the Effective Volumetric Sampling Rate (mL/min) for the SPME, the $\mathrm{MSR}_{\mathrm{SPME}}$ is the Measured Sampling Rate (pg/min) for the SPME in the headspace volume, and the VD_{T} is the assumed vapor density (pg/mL) at the measured temperature. For both DNT and TNT we use the vapor pressure data from Pella, 1977 (Table 1). About 100 mg of DNT or TNT was placed into a 40 mL amber septa top vial and left to equilibrate in a temperature controlled chamber. SPME fibers were allowed to equilibrate at the same temperature as the vial, then placed into the headspace for 1 minute and quantified on the GC/ECD. The vial and SPME calibration temperatures ranged from 10 to 30°C.

Once the $EVSR_{SPME}$ is determined, unknown headspace vapors can be quantified by collecting a sample for a known time and calculating the vapor concentration as follows:

$$VC_T = \frac{MSR_{SPME}}{EVSR_{SPME}} \qquad [3]$$

where, VC_T (pg/mL) is the vapor concentration in the unknown headspace at a specific temperature. An example of the $EVSR_{SPME}$ for TNT and DNT at near lab temperatures is shown in Figure 4.

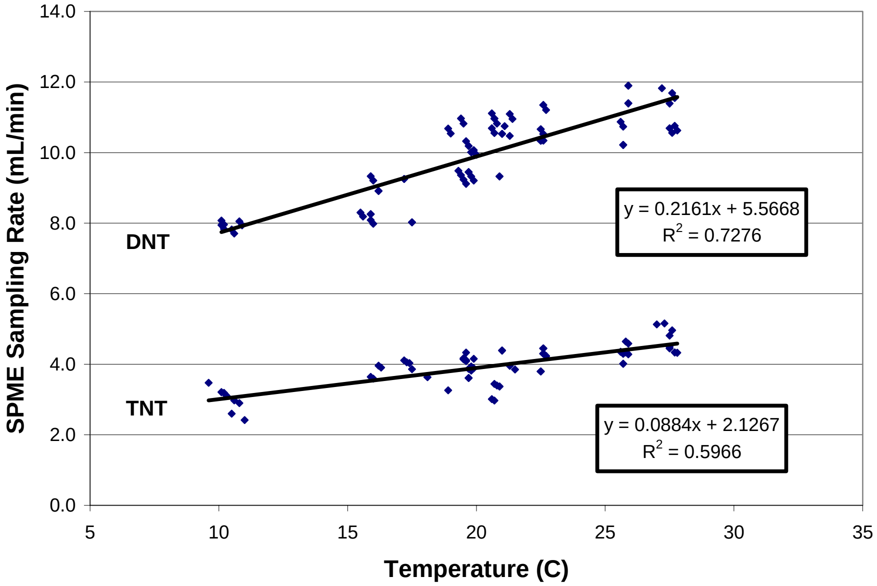

Figure 4. Effective Volumetric Sampling Rate for TNT and DNT Using a 65µm PDMS/DVB SPME Fiber

Headspace vapor concentrations of aqueous solutions and soil standards were measured during the sensor performance tests. Sampling intervals ranged from 5 to 180 minutes. Verification analysis of the aqueous solutions by HPLC showed a bias of 0.93 for TNT and 0.88 for DNT. Actual solution concentrations used are shown in Table 6.

Table 6. Actual Aqueous Solution Concentrations Used in Vapor Performance Tests

Water Concentration	
TNT (ug/L)	DNT (ug/L)
9300	440
465	44
279	26
47	4.4
4	0.4
0.4	

Figure 5 shows the comparison of estimated TNT headspace vapor with measured values for each using only the aqueous solutions (TNT soil standards were not available for this test session). There was very good correlation for the aqueous solutions over a range of about 3 orders of magnitude ($r^2 = 0.98$). Figure 6 shows the estimated DNT headspace vapor concentration compared to the measured values for both aqueous solutions and the D-3 soil standard. This correlation was also very good ($r^2 = 0.99$), indicating that air-water partitioning (Henry's Law) and soil-water-air partitioning coefficients are valid over the range of values tested.

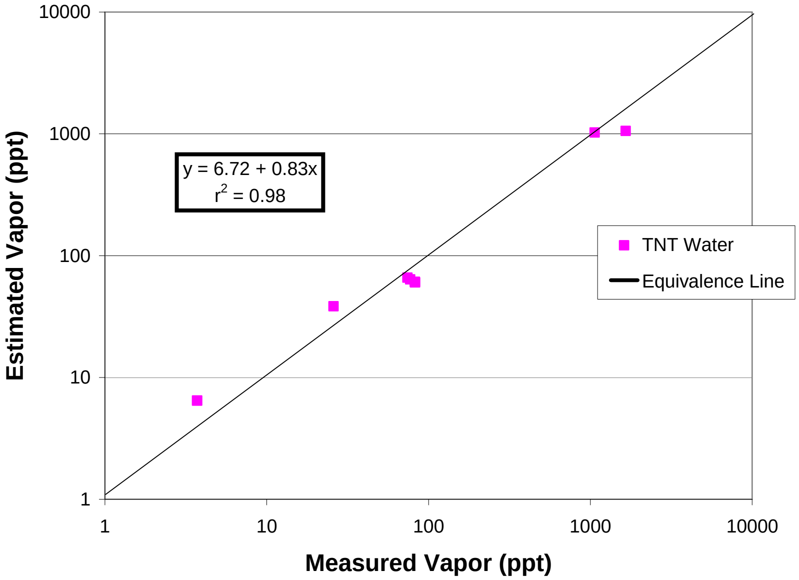

Figure 5. Comparison of Measured Versus Estimate Headspace Concentrations of TNT

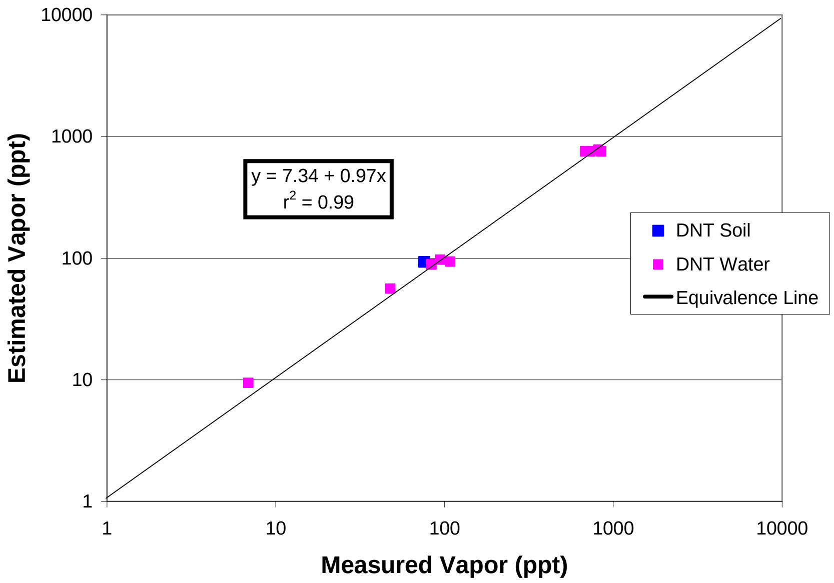

Figure 6. Comparison of Measured Versus Estimate Headspace Concentrations of DNT

3.0 UMR Vapor Sensor Threshold Tests

3.1 Test Methods

The UMR Vapor Sensor was operated by UMR personnel at Sandia National Laboratories from July 24- 26, 2002. The device consisted of a tabletop instrumentation package (~60 cm x ~60 cm x ~30 cm tall) and a laptop computer for data acquisition. Aqueous solution and soil standards were placed on the lab bench top and temperature was monitored with an adhesive thermocouple on the exterior of the headspace sample container. The top to each jar was carefully removed and the vapor inlet was placed midway into the headspace region.

The UMR Vapor Sensor sample collection controller was set to sample at 50 mL/min for 4 seconds. For each test run, the system samples the headspace vapors once, then samples room air for two cycles. The vapor samples are preconcentrated in a short (~10 cm) section of glass capillary GC column (uncoated). The preconcentrator is then moved to a heated zone and the sample is desorbed into the detector train. An entire sampling, desorption and analysis sequence takes about 15 seconds.

The detector consists of a micro electron capture detector (ECD), followed by an electron attachment reactor (tungsten wire), followed by a second micro ECD. Data collection software collects ECD response as a function of time. Figure 7 shows a typical data trace for both ECD detectors for the vapor headspace sample and the two room air samples.

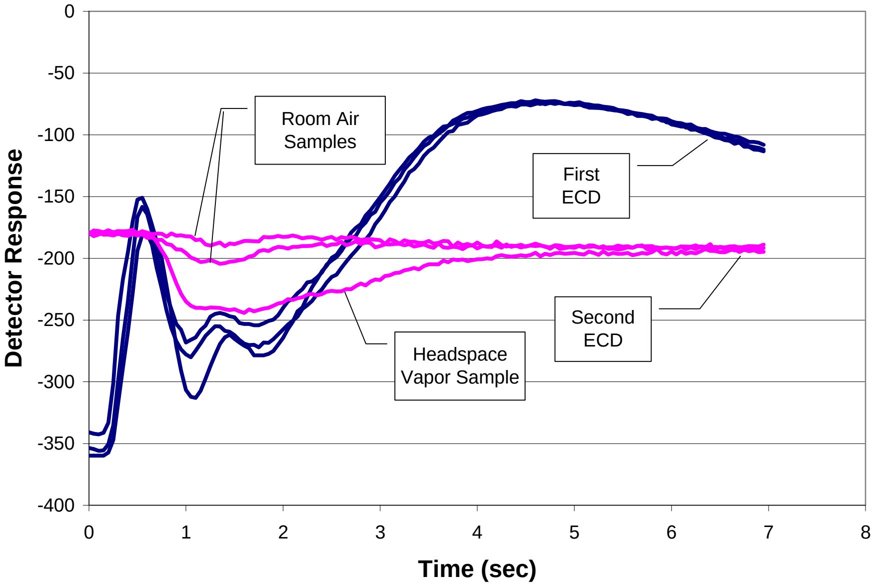

Figure 7. Test Run Results, Typical (TNT 465 µg/L aqueous, headspace)

Integration of the second ECD response over time provided a summation of the total detector response from the preconcentrated vapor sample. Headspace vapor integrated areas were determined using a software package - Peak Fit, Version 4.06 (SPSS, Inc.) .

High concentration aqueous solutions were used to verify initial operations of the sensor. Seven vapor sampling/data acquisition runs were completed to assess variability of the second ECD peak area. Since the headspace samples contained a significant water component, samples from a water blank made with deionized water were used to assess this background response. Figure 8 shows seven replicates with minimal response to water vapor. The average (std dev) response area was 5.23 (2.31).

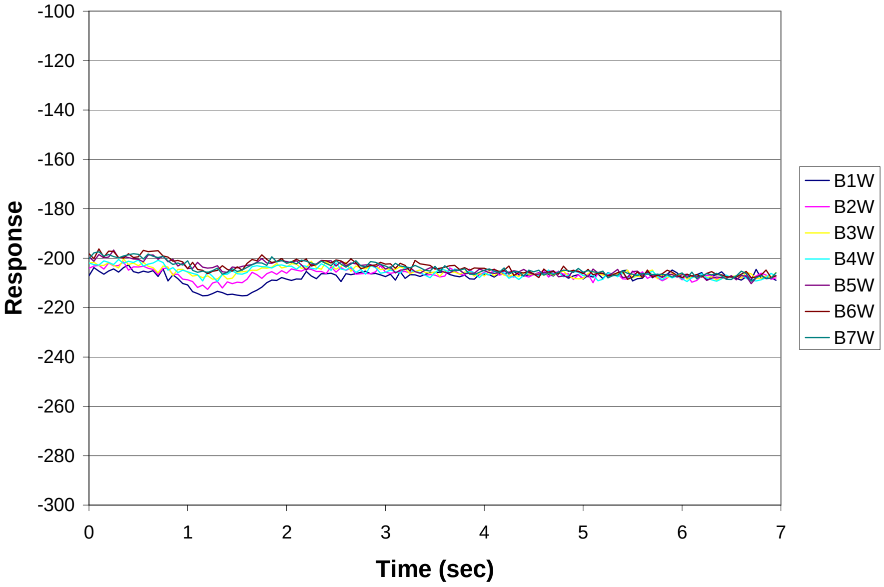

Figure 8. Deionized Water Response, 7 replicates

3.2 TNT Results

Figure 9 shows an average response (n=7) of the UMR Vapor Sensor from TNT headspace vapors over aqueous solutions of various concentrations. These results indicate an initial rising detector response; however, the response plateaus at the higher headspace concentrations. This response has been observed with laboratory GC/ECD detectors, which also exhibit a similar narrow linear range.

Figure 10 shows a summary of the instrument response as a function of sequential run. This shows an initial high response for the first sample for all but the lowest headspace vapor concentration. This indicates that re-equilibration of the headspace vapors may not have occurred after the first sample. This is expected because inter sample times of only 2-3 minutes are unlikely to allow for a full re-equilibration of the headspace with the aqueous solution. The trends do not show continued depletion over the sequence. Thus, the vapor levels may not have been at the equilibrium maxima, but maybe about a factor of two below.

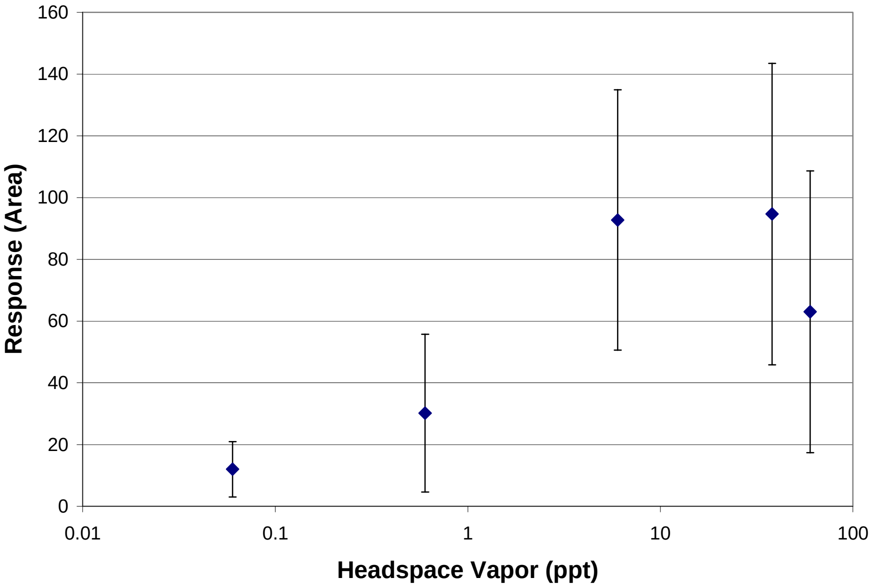

Figure 9. TNT Headspace Vapors Over Aqueous Solutions – Average (1 std dev error bars) Response (n=7)

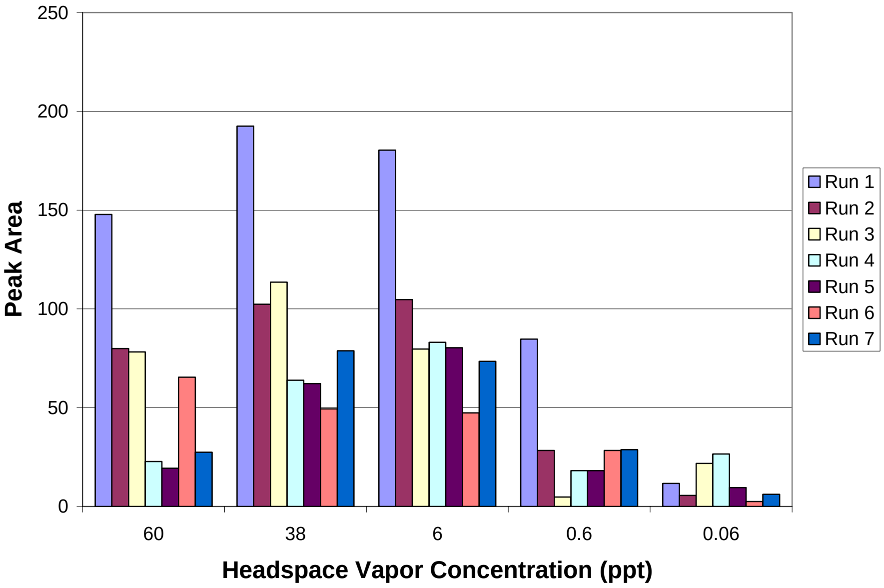

Figure 10. TNT Headspace Over Aqueous Solution - Sequential Runs

Figure 11 shows the variability of the detector response over the seven sequential trials for the 60 ppt TNT headspace over aqueous solution sample.

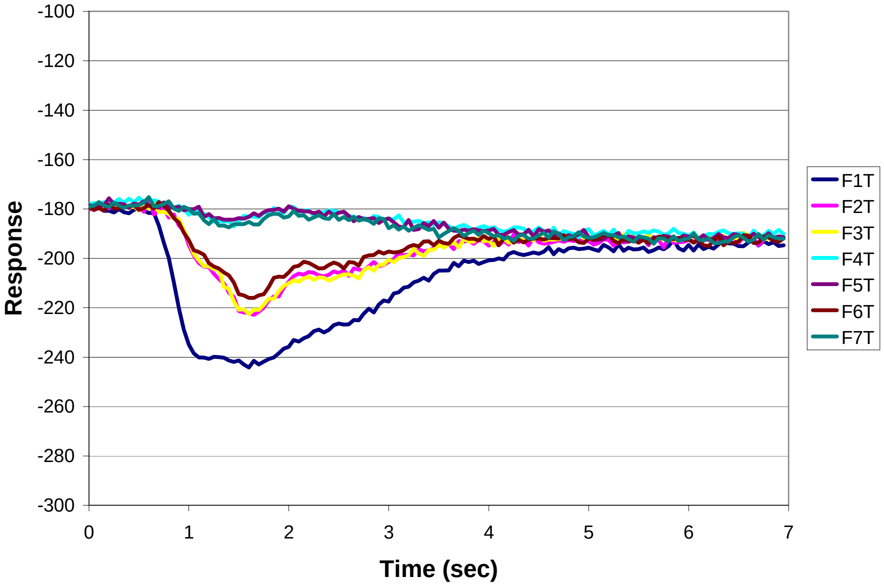

Figure 11. TNT Headspace over Aqueous Solution, 60 ppt – 7 Replicates

3.3 DNT Results

Data was collected for DNT in the same manner as for TNT using both aqueous solution, and then also with the D-3 soil standard. Figure 12 shows the DNT average and standard deviation detector response from to 100 ppt. The DNT showed the same trend as the TNT with a rising response to increasing vapor level with a leveling off at higher concentrations. The D-3 soil sample showed a large negative bias. We recognize the potential for more uncertainty with the soil headspace standards. Therefore, a SPME sample was obtained just after the UMR vapor sensor sample and the measured headspace concentration was very close to the estimated equilibrium vapor concentration (Figure 6), indicating the D-3 soil standard was accurate.

Figure 13 shows the peak areas for the seven sequential runs. As with the TNT, the first sample was notably larger for the higher headspace concentrations (about 50 to 100% greater). Figure 14 shows the variability of seven trials for DNT headspace over aqueous solutions at 90 ppt.

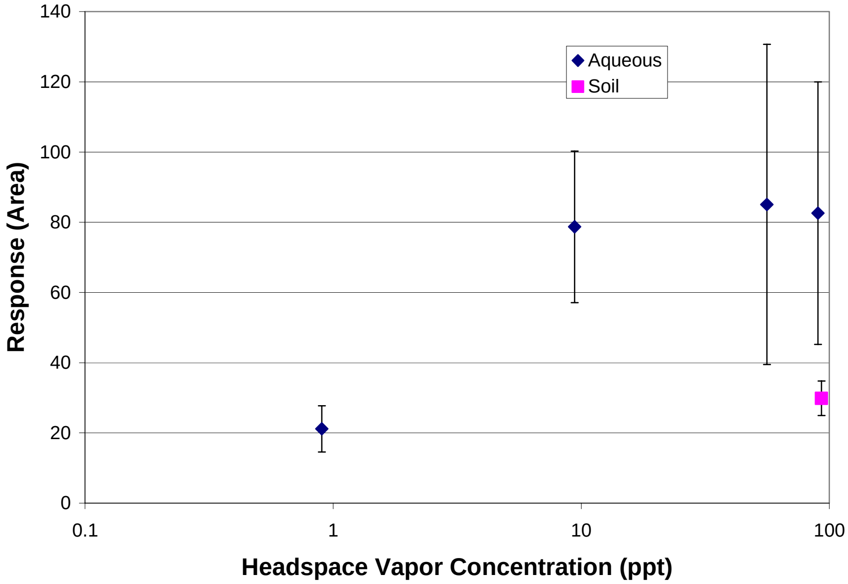

Figure 12. DNT Headspace Vapors Over Aqueous Solutions and Wet Soil – Average (1 std dev error bars) Response (n=7)

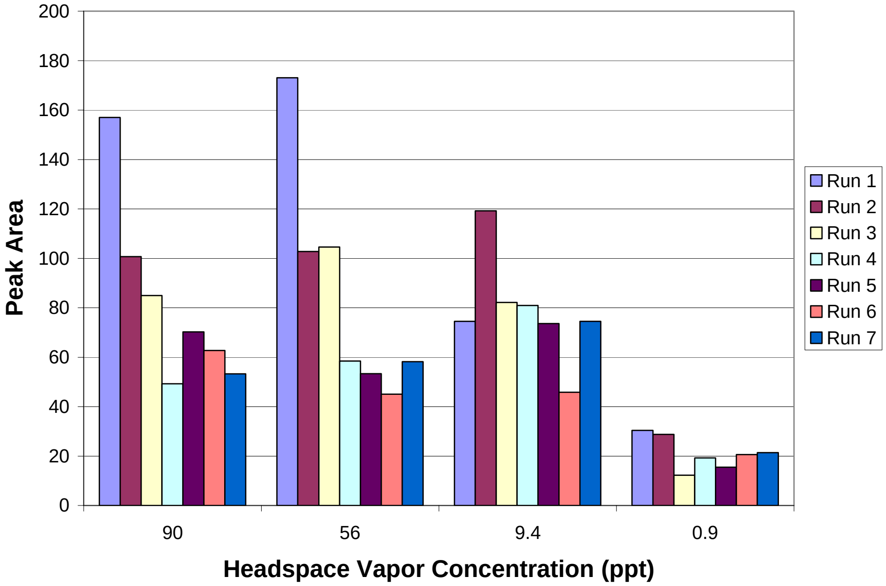

Figure 13. DNT Headspace Over Aqueous Solution - Sequential Runs

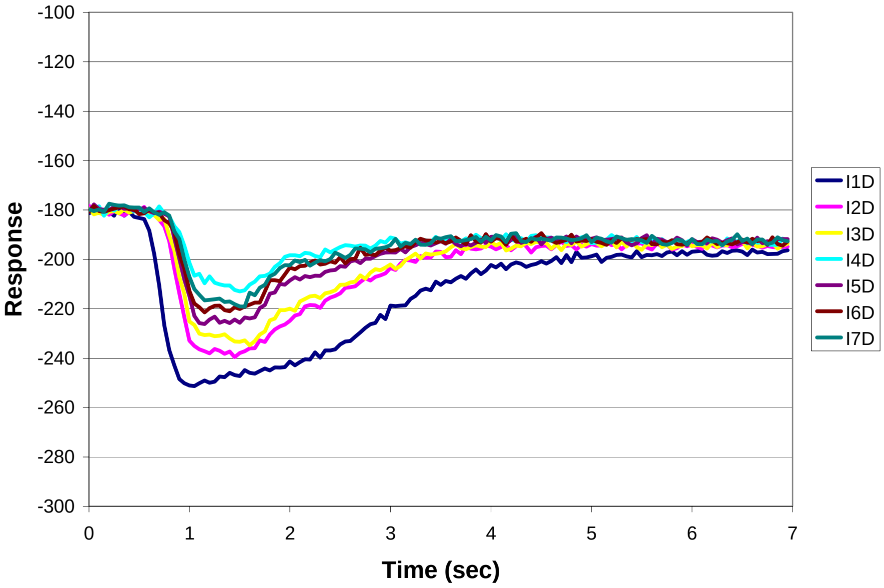

Figure 14. DNT Headspace over Aqueous Solution, 90 ppt – 7 Replicates

3.4 Interferent Results

For an initial screen of interfering odors, two plants were selected from the landscaping outside the building. A small branch from a flowering purple sage plant and needles from a fir tree were collected and placed into separate 250 mL glass jars. The headspace vapors were allowed to generate for about 1-2 hours prior to testing. Figure 15 shows the results from headspace of the flowering purple sage plant. Significant peaks were recorded by the second ECD in a similar fashion as the TNT and DNT response.

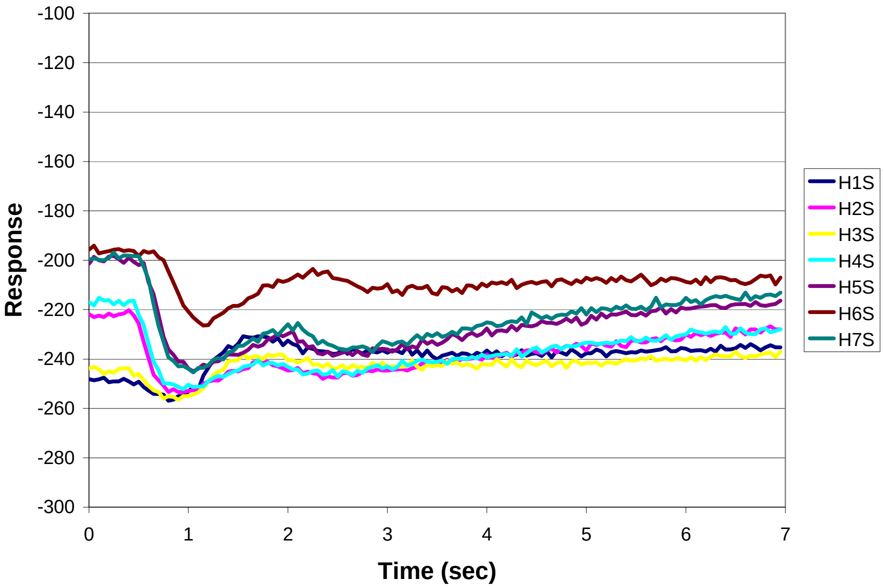

Figure 15. Headspace over Purple Sage Leaves and Flowers – 7 Replicates

Figure 16 shows the response to the headspace vapors of the fir tree needles. The instrument response was barely perceptible in this case. From this preliminary screening, it appears that more work is needed to evaluate natural compounds that might interfere with the detection of explosive vapors.

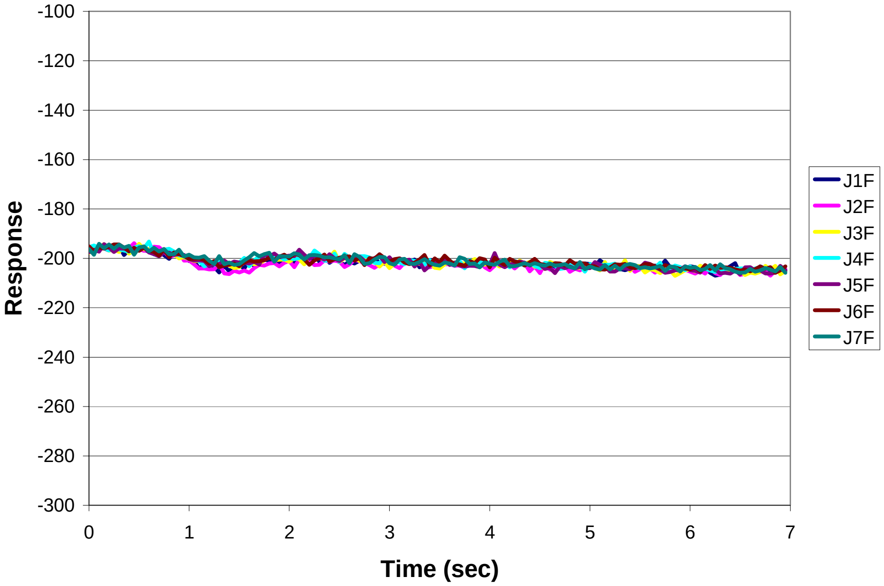

Figure 16. Headspace over Fir Tree Needles – 7 Replicates

3.5 Results Summary

The UMR Vapor Sensor displayed similar narrow response linearity and high variability for both TNT and DNT aqueous solutions and DNT soil standards. From this data, it is very difficult to estimate the vapor-sensing threshold. Typical methods that require high confidence in a regulatory environment use the U.S. Environmental Protection Agency (EPA) protocols that specify a minimum detection limit (MDL) determined statistically as a one-sided t-test at a desired confidence level (EPA, 1992). This is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. This is calculated by multiplying a 99% t-statistic times the standard deviation from replicates of samples at 3 to 5 times the instrumental signal to noise ratio.

Figures 9 and 12 show the large variance in the second ECD response for seven replicates. The EPA t-statistic method would reveal extremely large minimum detection limits that do not serve a useful purpose at this time.

Another method more typically used to quantify the performance of a sensor that is in the developmental stage is to determine the minimum detectable concentration as a function of signal to noise ratio. This method is also challenged in that the total response is an integral of the response over time.

Baseline noise could be determined as a root mean square of the peak height variation just prior to the sample entering the detector. However, the shapes of the TNT and DNT peaks are very broad, indicating that peak height is not a good indicator of the total integrated response.

In summary, the UMR Vapor Sensor appears to show good sensitivity down to about 1 ppt TNT and DNT; but, the variability is much too great for use as a quantitative device and linearity of response is probably less than 100. Response to two naturally occurring fragrances from plants (purple sage leaves/flowers and fir tree needles) showed minimal response to one (fir tree needles) and a significant response to the other (purple sage leavers/flowers).

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