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13. ABSTRACT *(Maximum 200 Words)*

Military test and training ranges generate scrap materials from targets and ordinance debris. These materials are routinely removed from the range for recycling; however, energetic material residues in the range scrap has presented a significant safety hazard to operations personnel and damaged recycling equipment. The Strategic Environmental Research and Development Program (SERDP) sought proof of concept evaluations for monitoring technologies to identify energetic residues among range scrap. Sandia National Laboratories teamed with Nomadics, Inc. to evaluate the Nomadics FIDO vapor sensor for application to this problem. Laboratory tests were conducted to determine that the vapor-sensing threshold to be 10 to 20 ppt for TNT and 150 to 200 ppt for DNT. Field tests with the FIDO demonstrated the proof of concept that energetic material residues can be identified with vapor sensing in enclosed scrap bins. Items such as low order detonation debris, demolition block granules, and unused 81-mm mortars were detected quickly and with minimum effort. Conceptual designs for field-screening scrap for energetic material residues include handheld vapor sensing systems, batch scrap sensing systems, continuous conveyor sensing systems and a hot gas decontamination verification system.

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Abstract

Military test and training ranges generate scrap materials from targets and ordnance debris. These materials are routinely removed from the range for recycling; however, energetic material residues in this range scrap has presented a significant safety hazard to operations personnel and damaged recycling equipment. The Strategic Environmental Research and Development Program (SERDP) sought proof of concept evaluations for monitoring technologies to identify energetic residues among range scrap. Sandia National Laboratories teamed with Nomadics, Inc. to evaluate the Nomadics FIDO vapor sensor for application to this problem. Laboratory tests were completed that determined the vapor-sensing threshold to be 10 to 20 ppt for TNT and 150 to 200 ppt for DNT. Field tests with the FIDO demonstrated the proof of concept that energetic material residues can be identified with vapor sensing in enclosed scrap bins. Items such as low order detonation debris, demolition block granules, and unused 81-mm mortars were detected quickly and with minimum effort. Conceptual designs for field-screening scrap for energetic material residues include handheld vapor sensing systems, batch scrap sensing systems, continuous conveyor sensing systems and a hot gas decontamination verification system.

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1.0 Introduction

The Strategic Environmental Research and Development Program's Exploratory Development (SEED) Program issued a statement of need seeking proof-of-principal work for analysis, characterization and treatment of energetic residues on scrap materials at military training/testing installations. The objectives of this effort included:

- Develop/apply state-of-the-art, field-portable monitoring technologies to analyze and characterize the energetics residues found on the wide variety of scrap materials that are exclusive of unexploded ordnance (UXO) but which include munitions casings, training targets, and other scrap/fragmented materials found at testing/training ranges on DoD installations. Methods to detect buried UXO or remove scrap materials from ranges are not a part of this statement of need.
- Develop new, innovative, and cost effective technologies and processes to effectively treat energetics residues typically found on these scrap materials so as to render the scrap materials non-hazardous and suitable for recycling/recovery. Treatment technologies should consider the potential range of sizes of scrap pieces, range of energetic material residues, need for size reduction of the scrap for recycling, and the control and/or monitoring of any effluent (liquid or gaseous) from the treatment process.

Proposed efforts must address the diversity of scrap materials and energetics residues found at all types of DoD testing/training ranges for all military service branches. The results of this work, if successful, should lead to further development or demonstration and eventually be applicable to multiple military sites for a wide variety of scrap materials and the various energetics residues.

Sandia National Laboratories teamed with Nomadics, Inc. (Stillwater, OK) to show how the Nomadics FIDO sensor could be used for vapor sensing of explosive residues. This report provides background on vapor sensing for TNT based explosives among military scrap, describes the FIDO sensor detection threshold tests and FIDO sensor screening tests for explosives among scrap materials, then closes with a description of conceptual deployment opportunities.

2.0 Vapor Sensing for TNT Based Explosives Among Military Scrap

2.1 Problem Definition

Military scrap is an ambiguous term that describes a wide variety of materials found on test and training ranges that can be loosely grouped into target objects and ordnance debris. The target objects can include vehicles such as trucks and tanks, buildings made from concrete block and/or wood, and soil structures made with berms. Ordnance debris includes remnants of both inert-filled and high explosive filled projectiles (UXO). Debris from high-order ordnance detonation includes metal fragments (colloq. frag) in various sizes and shapes. This detonation debris can include low order detonation residues that contain large amounts of high explosive fill material. Current military range practice is to manually or mechanically collect this wide array of scrap material in accumulation areas in preparation for recycling operations. Figure 1 shows the type of scrap collected on the former Naval firing range at Kaho'olawe Island, Hawaii.

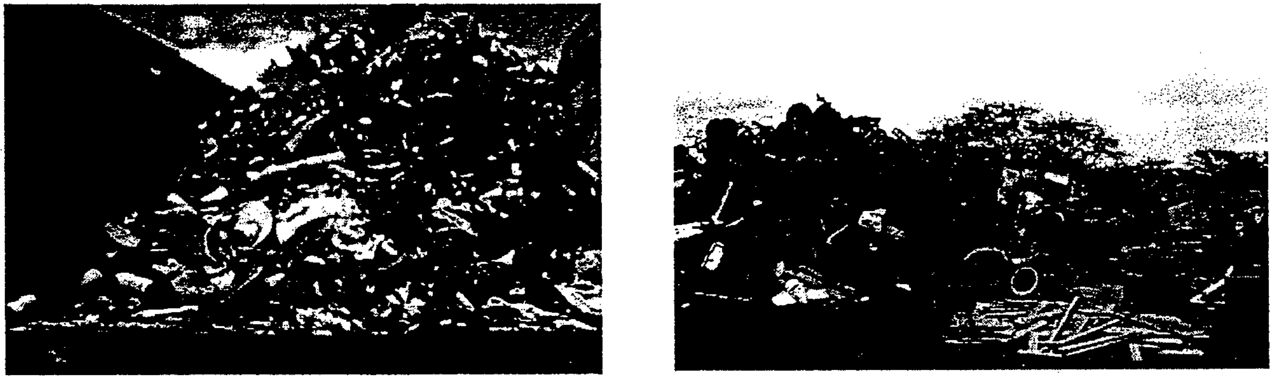


Figure 1. Range Scrap from Kaho'olawe Island Firing Range

If sufficient explosive residue is present on or among the scrap, the recycling operation is at risk for a detonation that could harm personnel and damage equipment. This project proposed to evaluate a proof-of-concept method for screening military scrap using an explosives vapor sensor. The concentration of explosive chemical vapors in air can range from saturated conditions to values below instrument detection limits. This vapor concentration is dependent on the evaporation rate from scrap material surfaces and solid phase energetics, and interactions of the vapor with scrap materials. The scrap materials include bare steel of various states of oxidation, painted surfaces, wood, plastic, and tires. With rapid vapor deposition onto adjacent scrap and dilution into air streams, the headspace vapor concentration will be much less than saturated vapor density demanding a very sensitive vapor sensor.

The hypothesis in our proposal stated that discrimination of detonable and non-detonable quantities of energetic material residues might be possible by sensing differences in vapor concentrations. After completing this work, we now recognize that this is unlikely because the process of vapor release from these two types of sources are similar and other factors have a greater impact on the steady-state concentration than detonable versus non-detonable quantities of explosive residues. Nevertheless, the ability to discriminate any scrap showing explosive residues may still be valuable as a method to minimize the amount of scrap requiring more careful, time-consuming and costly sorting. Both handheld vapor sniffers and automated scrap sorting systems can be considered for use in this application.

2.2 Acceptable Limits for Explosive Residues on Surfaces

Regulatory and military requirements for scrap and energetic material residues on military testing and training ranges have not been fully defined. Regulatory aspects of solid wastes generated from range training and testing is a complicated management problem. The majority of the scrap from ranges is likely to be metal. It appears that the best regulatory position is to use the scrap metal exclusion of RCRA (Webb, W., 1999). If this position can be institutionalized, the range recycling practices will require inert certification procedures. Inert certification is principally a safety issue to prevent detonation of explosive materials during processing of the scrap. Chemical residues from energetic materials that are insufficient to sustain detonation may still be an environmental contamination issue if released to the environment (soils or groundwater). Scrap items containing these non-detonable surface residues may still require segregation, depending on the recycling objectives of the range programs. For chemical sensing, there are three levels for which to discriminate: scrap containing no detectable energetic material, scrap containing detectable non-detonable energetic material, and scrap containing detonable energetic material. As the range recycling program matures, there may be different treatment processes for different levels of energetic materials.

Potentially applicable military specifications that define acceptable residues on surfaces can be found for decontamination of buildings and equipment. These include DOD 5160.65M, *Single Manager for Conventional Ammunition*; NAVSEA OP 5, *Ammunition and Explosives Ashore: Safety Regulations for Handling, Storing, Production, Renovation and Shipping*; and the guidelines established in IOCP 385-1, *Classification and Remediation of Explosive Contamination*. Rigorous testing with friction, impact and electrostatic discharge methods was completed to establish explosive hazard thresholds (EHTs) that may be regarded as de-minimus and can be considered safe (Caris et al., 2001). Explosive hazard thresholds (EHT) have been reported as follows: porous material, 35 mg of an explosive per kg of sampled material (35 mg/kg); Non-porous surface, 3.5 mg of an explosive per 100 cm2 of surface area (3.5 mg/100 cm2 wipe); and TNT at 46 mg/kg (unspecified material) (Anderson and Ricks, unk).

The decontamination explosive hazard thresholds for porous materials seem to be in conflict with environmental restoration soil safety guidelines. Non-detonable amounts of explosives in soils has been determined through various testing methods and an upper limit has been prescribed as 10% (w/w) or 100,000 mg/kg by the Department of Defense Explosive Safety Board (DDESB) (Balasco, 1987). This is in sharp contrast to the 35 mg/kg noted above for porous material, a term also used to describe soils. At Los Alamos National Laboratory, the explosives safety group has defined the soils threshold to be 5% (w/w) or 50,000 mg/kg based on testing results in their laboratories.

Another measure for safe explosive residuals ma be found in the detonation physics of energetic materials. Mass detonating quantities of energetic materials are of principal concern for safety during treatment. An important feature of energetic materials is that a minimum critical diameter is necessary for detonation to propagate through the material (Cooper, 1996). For most military explosives (TNT, RDX and HMX) the critical diameter is about 1 to 10 mm. The failure thickness is defined similarly for surface coatings and ranges from 0.5 to 2 mm for the same group of explosives (Cooper, 1996).

2.3 Volatilization of Solid Phase Energetics

Many explosives exist as solids at normal temperatures. Sublimation is a special case of evaporation phenomena where a substance passes directly from a solid to a vapor without appearing in the intermediate liquid state. Both sublimation and liquid state vaporization can be described by diffusional mass transfer where the rate of mass loss is determined by the rate at which the molecules migrate from the region of saturated vapor (solid phase surface) to a lower concentration in the bulk gas. At steady-state, the rate of vaporization can be expressed in terms of the amount of solid material lost per unit time and per unit surface area by Fick's first law:

$$\frac{\partial m}{A \cdot \partial t} = M \cdot D \cdot \left(\frac{C_S - C_b}{L} \right) \qquad [1]$$

where m is mass, A is surface area of the solid phase, t is time, M is molecular weight, D is the diffusion coefficient in air, C_S and C_b and the molar concentrations of vapor at the solid surface and the bulk gas, respectively, and L is the length of the concentration gradient. The latter is also referred to as the diffusion layer thickness, which is affected by the velocity of air flowing past the material.

Volatilization of explosive vapors from other surfaces (scrap metal, painted surfaces, soil) is a more complex process due to interactions with the host material. In most cases, the vapor concentration at the material surface will be much less than saturation (C_s) causing a lower concentration gradient and a lower vaporization rate. The difference in volatilization rates between solid phase energetics and surface residues is too complex to assess through this effort, but the results from the bin tests indicates the difference may not be that great, and more importantly, could be overshadowed by the total surface area available for volatilization. Equation [1] includes a surface area term that shows a direct relationship with the volatilization rate per unit area. Thus, a material with a low volatilization rate over a large surface area may exceed the total mass transfer into air than a high volatilization rate over a small surface area.

2.4 Project Approach

Since there is no definition of an acceptable deminimis amount of explosive residue on scrap, even small quantities exceeding the explosive hazard thresholds or failure diameter/thickness need to be identified. These small amounts may be indistinguishable from larger surface area materials containing non-detonable surface residues. Re-deposition of volatilized explosive vapors complicates the process in a way that estimating the vapor concentration with numerous sources and sinks of variable nature using theoretical methods is impractical. Therefore, a project was defined to assess whether there is sufficient vapor from various sources and use sensing threshold determinations to evaluate the utility of vapor sensing for explosives among scrap.

This project was organized into three tasks as follows:

Task 1: Sensor Performance

Operational sensitivity of the latest Nomadics sensor was measured in the laboratory with comparison to headspace vapors derived from water solutions and contaminated soils verified with solid phase microextraction vapor sampling devices.

Task 2: Bin Tests

Scrap typical of what is found on training and test ranges was loaded into 1 m^3 wooden boxes until about half full. Several control boxes and several trial boxes were set up for background evaluations. Various size (mass, surface area) pieces of explosive materials (TNT demo blocks, low order detonation debris, live 81 mm mortars) were be placed into the bin for various times sufficient to detect a measurable concentration. The vapors inside the closed bin were sampled and analyzed with the Nomadics sensor.

Task 3: Conceptual Design

After completion of the bin tests, the data were evaluated to determine the best use the Nomadics AFP sensor in a handheld or automated scrap sensing system.

3.0 Sensor Performance Tests

3.1 Introduction

The estimated vapor-sensing threshold of the FIDO was reported by Nomadics to be in the low femtograms (fg) range. This was determined by delivery of a known concentration emitted by a vapor generator and measurement with the FIDO sensor. Using 1 fg with a ~ 50 mL/min sampling rate for ~ 5 seconds, this corresponds to a vapor concentration of about 0.0002 ng/L (0.03 ppt). The best comparison headspace vapor sampling and analysis tool is solid phase microextraction (SPME) with gas chromatography/electron capture detection (GC/ECD). With a minimum 10 pg ECD signal, a 100 min sampling time and a SPME sampling rate of 8.5 mL/min, the lowest detectable vapor concentration is about 0.01 ng/L (1 ppt).

In order to quantify the detection threshold of the Nomadics FIDO, we believed we would need to extrapolate the headspace vapor concentration from soil headspace vapor standards and 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 the FIDO to obtain a good signal, and then closed to re-equilibrate for future testing. Headspace standards were prepared to bracket the expected sensing threshold of the FIDO and were verified (above 1 ppt) with measurement using SPME sampling and GC/ECD quantification.

3.2 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

$$K_H = \frac{C_G}{C_L} \qquad [12]$$

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

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 Figure 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 2.

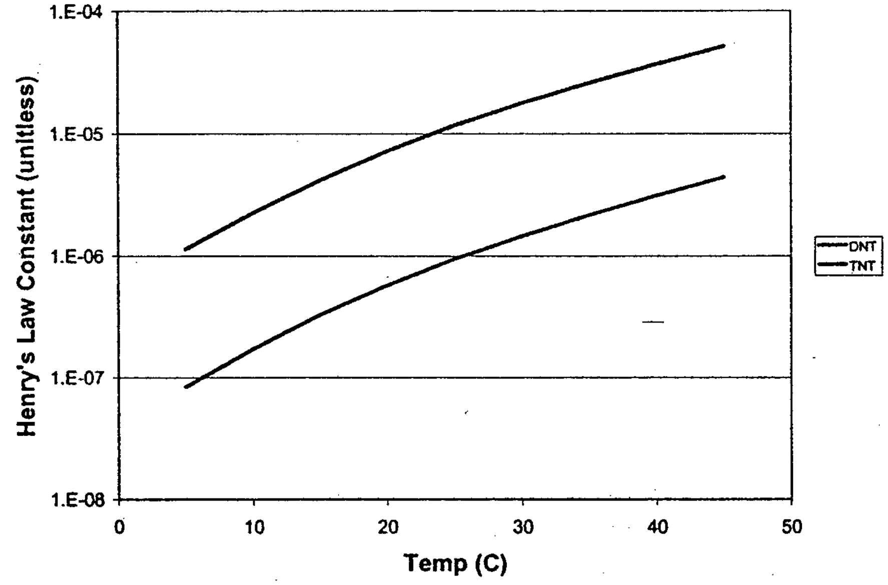


Figure 2. DNT and TNT Henry's Law Constant as a Function of Temperature

To achieve the desired headspace vapor concentrations to test FIDO at the limit of detection, serial dilutions of stock aqueous solutions of TNT or DNT were prepared as shown in Table 3 and 4. The stock solution of TNT was 81 mg/L and for DNT was 100 mg/L. The detection limit of a direct aqueous injection for the HPLC is about 50 μg/L. Since all of the water solutions will be at or below this limit, validation of solution concentrations will require preconcentration using Solid Phase Extraction (SPE) with Gas Chromatography/Electron Capture Detection (GC/ECD), which will extend the detection limit down to 0.05 μg/L. This will suffice for all but the lowest concentration for DNT. However, at these ultra trace levels, the variability of the measurements using SPE will increase causing more uncertainty in the headspace vapor concentration estimates. Figure 3 shows the headspace 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 3.3.

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

Water Concentration	Headspace Concentration	
ug/L	ng/L	ppt
0.01	0.0001	0.015
0.05	0.0005	0.073
0.1	0.0010	0.145
0.5	0.0049	0.727
1.0	0.0097	1.454
5.0	0.0487	7.270

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

Water Concentration	Headspace Concentration	
ug/L	ng/L	ppt
0.1	0.0001	0.007
0.4	0.0003	0.037
0.8	0.0007	0.075
4.0	0.0035	0.373
8.0	0.0070	0.747
80.0	0.0624	7.461

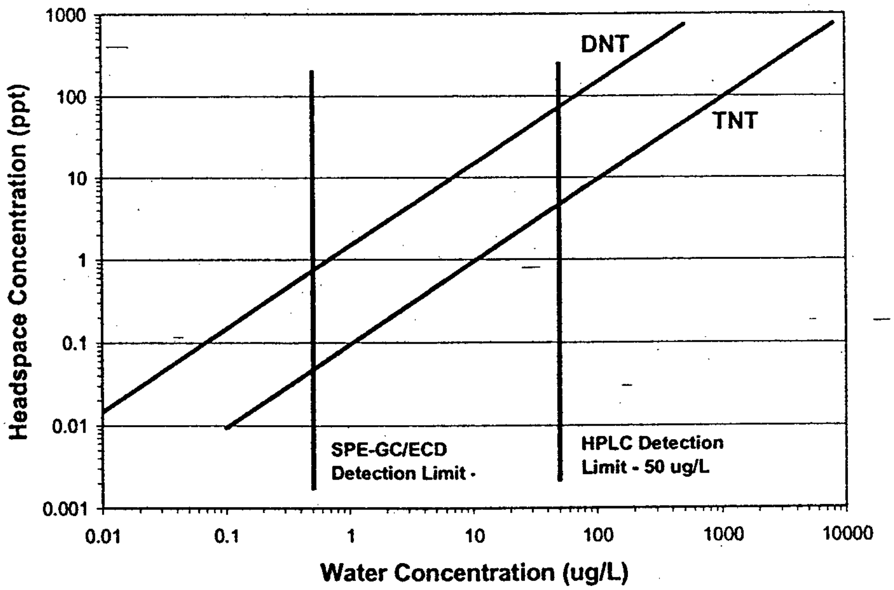


Figure 3. Headspace Vapor Concentrations of TNT and DNT with Aqueous Analytical Method Detection Limits (23°C)

3.3 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 ~ 100,000 ng/g for TNT and 7,000 ng/g for DNT to produce equivalent headspace vapor concentration for each compound. 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 Tables 5 and 6. The method detection limit for TNT and 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 Tables 5 and 6. The average (std dev) soil moisture contents of the dry DNT and TNT soil standards were measured to be 0.5% w/w (0.07) (n=3) and 0.2% w/w (0.04) (n=3), respectively. At this low moisture content, the vapor concentrations for even the highest soil residues were too low for the FIDO to measure. Thus, the soil was wetted to the target soil moisture content to bracket the expected FIDO 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).

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

Table 6. TNT Soil Standards [mean (std dev) n=5] and Estimated Headspace Vapor Concentrations (23°C) as a Function of Soil Moisture Content.

	T-1		T-2		T-3		T-4		T-5		T-6	
Moisture Content	Soil Residue 93,250 (4900) ng/g		Soil Residue 9,705 (210) ng/g		Soil Residue 1,190 (60) ng/g		Soil Residue 120 (7) ng/g		Soil Residue 11 (0.7) ng/g		Soil Residue 1 (0.2) ng/g	
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	1.3E-07	1.6E-05	1.3E-08	1.6E-06	1.6E-09	1.9E-07	1.6E-10	1.9E-08	1.5E-11	1.8E-09	1.9E-12	2.3E-10
0.010	2.4E-04	2.6E-02	2.4E-05	2.6E-03	2.9E-06	3.1E-04	2.9E-07	3.1E-05	2.8E-08	3.0E-06	3.5E-09	3.7E-07
0.015	4.3E-03	4.6E-01	4.3E-04	4.6E-02	5.3E-05	5.7E-03	5.3E-06	5.7E-04	5.0E-07	5.4E-05	6.2E-08	6.7E-06
0.020	4.0E-02	4.3E+00	4.0E-03	4.3E-01	4.9E-04	5.3E-02	4.9E-05	5.3E-03	4.7E-06	5.0E-04	5.8E-07	6.2E-05
0.025	2.3E-01	2.4E+01	2.3E-02	2.4E+00	2.8E-03	3.0E-01	2.8E-04	3.0E-02	2.6E-05	2.8E-03	3.3E-06	3.5E-04
0.030	8.6E-01	9.2E+01	8.6E-02	9.2E+00	1.1E-02	1.1E+00	1.1E-03	1.1E-01	1.0E-04	1.1E-02	1.2E-05	1.3E-03
0.035	2.4E+00	2.6E+02	2.4E-01	2.6E+01	3.0E-02	3.2E+00	3.0E-03	3.2E-01	2.8E-04	3.0E-02	3.5E-05	3.7E-03
0.040	5.4E+00	5.8E+02	5.4E-01	5.8E+01	6.6E-02	7.0E+00	6.6E-03	7.1E-01	6.3E-04	6.7E-02	7.8E-05	8.3E-03
0.050	1.6E+01	1.7E+03	1.6E+00	1.7E+02	2.0E-01	2.1E+01	2.0E-02	2.1E+00	1.9E-03	2.0E-01	2.3E-04	2.5E-02
0.070	4.5E+01	4.8E+03	4.5E+00	4.8E+02	5.5E-01	5.9E+01	5.5E-02	5.9E+00	5.2E-03	5.6E-01	6.5E-04	7.0E-02
0.090	6.4E+01	6.9E+03	6.4E+00	6.9E+02	7.9E-01	8.4E+01	7.9E-02	8.4E+00	7.5E-03	8.0E-01	9.3E-04	9.9E-02
0.110	7.2E+01	7.7E+03	7.2E+00	7.7E+02	8.8E-01	9.5E+01	8.9E-02	9.5E+00	8.4E-03	9.0E-01	1.0E-03	1.1E-01

Using the values from Tables 5 and 6, Figures 4 and 5 show the dynamic range of TNT and DNT headspace vapor concentrations as a function of total soil concentration and soil moisture content, respectively.

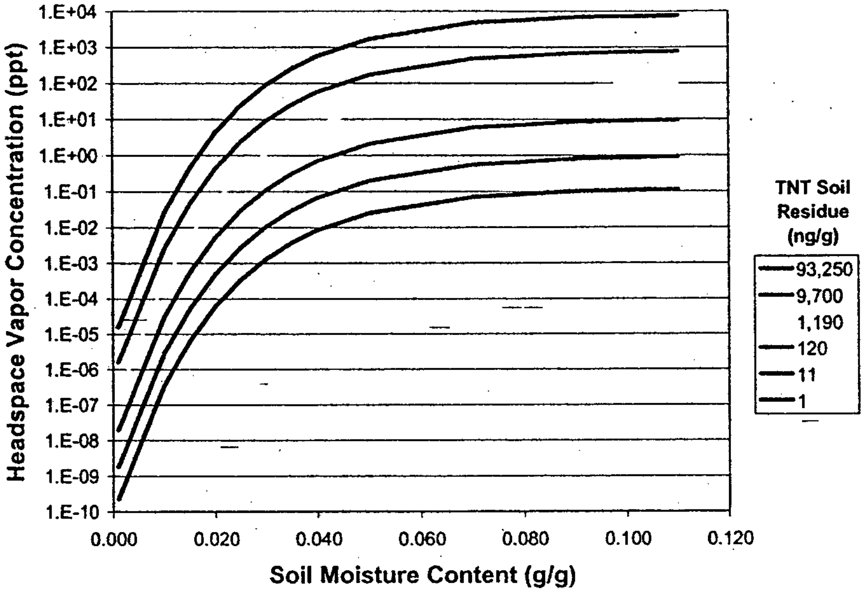


Figure 4. TNT Soil Headspace Concentrations as a Function of Soil Moisture Content

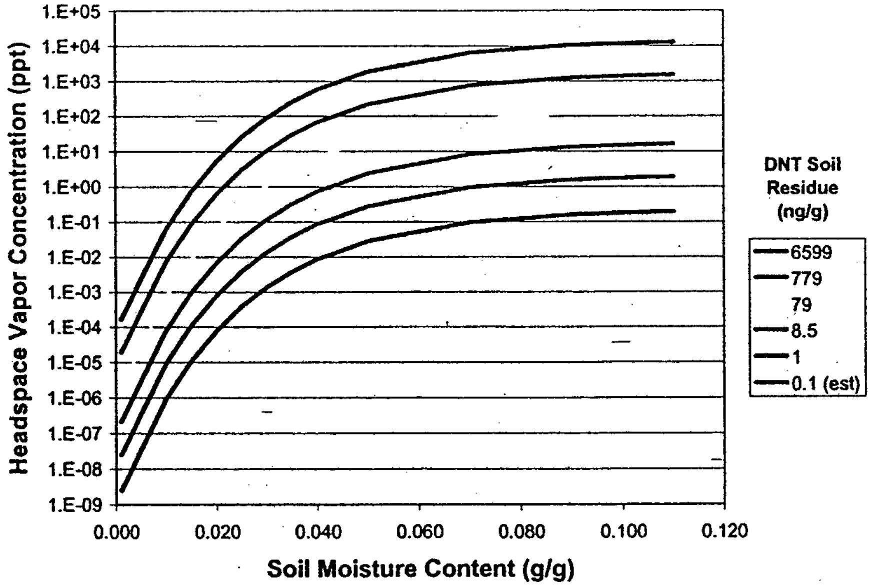


Figure 5. DNT Soil Headspace Concentrations as a Function of Soil Moisture Content

As the testing began with the FIDO 4E sensor, we found that the threshold for detection was near 10 ppt for TNT and 200 ppt for DNT. Sensor threshold testing could easily be accomplished with aqueous solutions above the HPLC detection limit of 50 µg/L. The dry soil standards prepared at the six levels (about 10x apart) were going to be much too low in headspace vapor concentration for the FIDO. Therefore, the soil standards of the three highest levels (T-1, T-2 and T-3; D-1, D-2 and D-3) were wetted up to about 4% (w/w) and allowed to equilibrate for 24 hours. Gravimetric soil moisture content results for DNT showed an average (std dev) value of 4.5% w/w (1.7). The soil moisture content for the TNT soils were not measured but can be safely assumed to be 4.2% (w/w) based on the dry moisture content and the added moisture.

3.4 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 $EVSR_{SPME}$ is the Effective Volumetric Sampling Rate (mL/min) for the SPME, the MSR_{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 at near lab temperatures is shown in Figure 6.

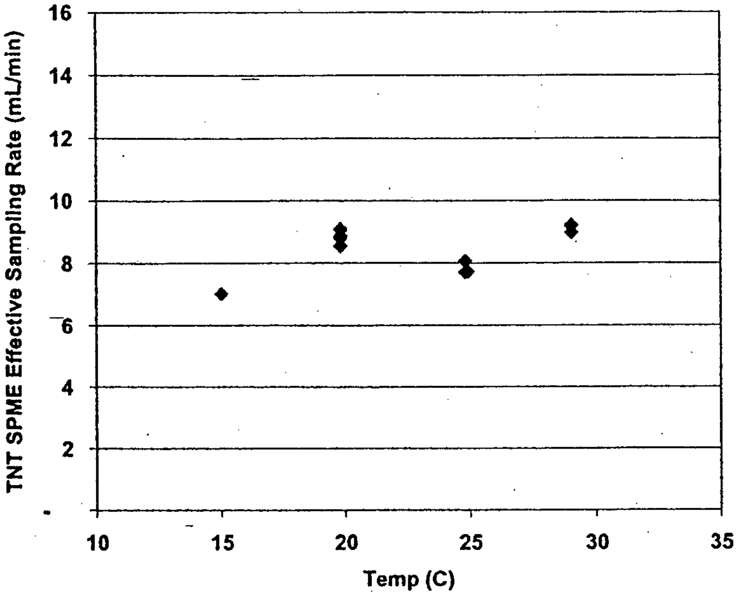


Figure 6. Effective Volumetric Sampling Rate for TNT Using a 65μm PDMS/DVB SPME Fiber

Headspace vapor concentrations of aqueous solutions and soil standards were measured during the Nomadics FIDO sensing threshold tests. Sampling intervals ranged from 0.5 to 194 minutes. Figure 7 shows the comparison of estimated DNT headspace vapor for both aqueous solutions and soil standards with measured values for each. There was very good correlation for the aqueous solutions, however, there were both over and underestimates when compared with the soil standards. One of the problems recognized with this method is that the headspace vapor levels vary dramatically with small changes in soil moisture content. We have learned that we might have been better off using the three lowest soil standards and raising the soil moisture content above 10% (w/w) to be in a region where small variation in soil moisture content has less impact on the headspace vapor levels (see Figures 4 and 5).

Figure 8 shows the estimated TNT headspace vapor concentration compared to the measured values for both aqueous solutions and soil standards. This correlation was much improved when compared to the DNT results (with no good explanation). One phenomena noted in our limited vapor release kinetics testing was that TNT rose above steady-state quickly (~3x in 20 minutes), returned to expected steady state values within 24 hours, and remained there for four days. However, DNT was slow to reach steady-state values, requiring about 5 days to reach expected values. More work is needed to define these release kinetics and correlation with expected values before use in more definititve sensor threshold testing.

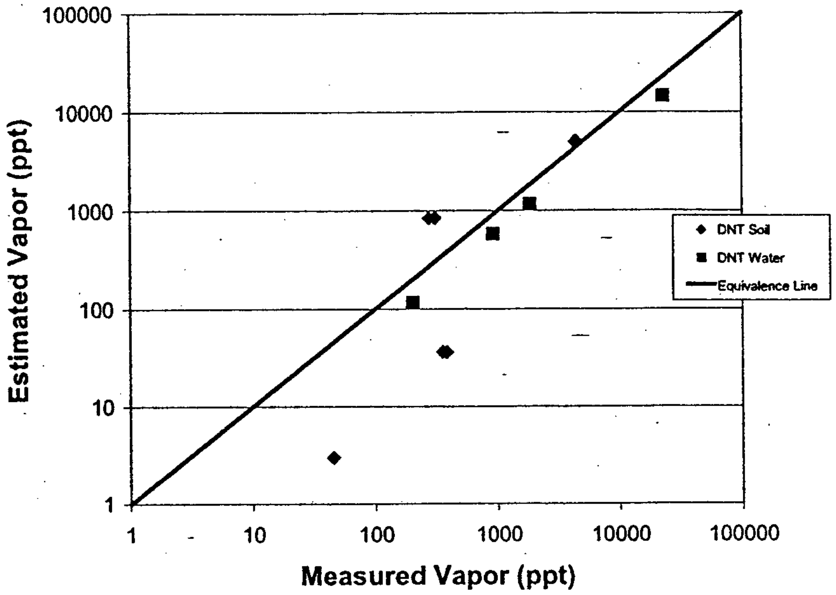


Figure 7. Comparison of Measured Versus Estimate Headspace Concentrations of DNT

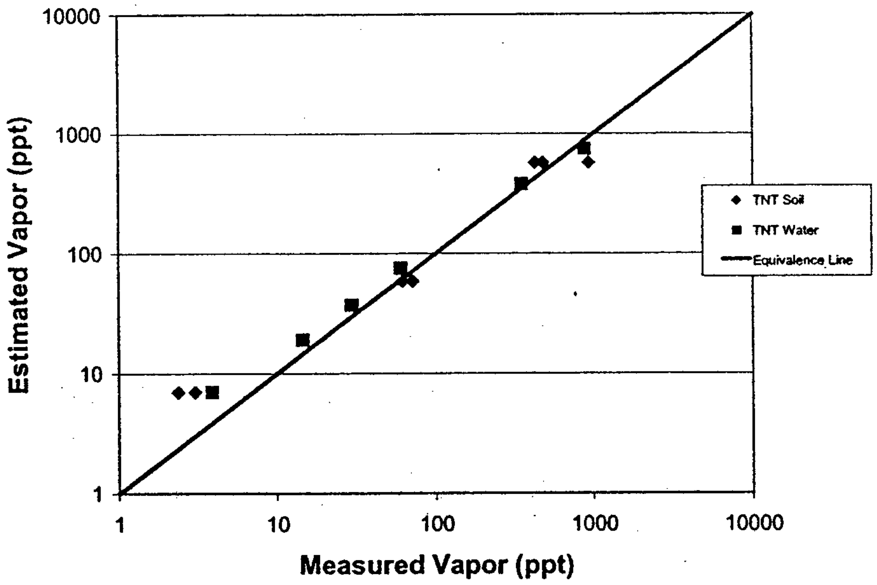


Figure 8. Comparison of Measured Versus Estimate Headspace Concentrations of TNT

3.5 Nomadics FIDO Vapor Sensing Threshold Tests

The Nomadics FIDO 4E sensor was operated by Nomadics personnel at Sandia National Laboratories from May 15 –17, 2001. The device consisted of a small handheld sensor/electronics package and a laptop computer for data acquisition. Aqueous solution and soil standards were placed on the lab bench top. The top to each jar was carefully tipped to the side and the FIDO vapor inlet was placed midway into the headspace region.

High concentration aqueous solutions were used to verify initial operations of the sensor. After initial screening found the sensing threshold to be around 10 ppt for TNT and 200 ppt for DNT, more careful replicate vapor sampling/data acquisition runs were completed. Seven vapor sampling/data acquisition runs were averaged into a single data file for sensing threshold determinations. This FIDO version showed a small response to water vapor. This was believed to be water deposits on the sensing lens and not a polymer response. All the averaged replicate data were corrected for a blank water vapor response. The average percent quench and the root mean square (RMS) noise from the five seconds prior to vapor inlet were calculated for each aqueous solution and soil standard. The ratio of the average water corrected percent quench to the RMS noise was used to calculate the signal to noise (S/N) ratio.

3.5.1 TNT Results

Data collected from the FIDO response is quantified as a normalized response that indicates a quench in the fluorescence of the excited polymer. About 10 seconds of baseline was recorded, the FIDO was placed into the headspace vapor for 5 seconds, then removed until baseline was re-achieved. Figure 9 shows an average response (n=7) of the FIDO from TNT headspace vapors over aqueous solutions of various concentrations. The time to maximum fluorescent quench was about 5 seconds with a recovery of 10 to 20 seconds after removal from the headspace vapor source.

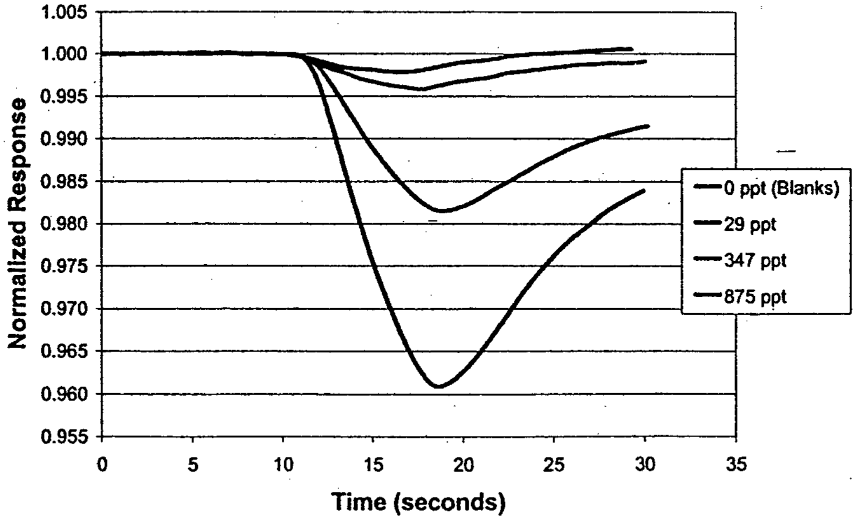


Figure 9. TNT Headspace Vapors Over Aqueous Solutions – Average Time Response (n=7)

Figure 10 shows a summary of the maximum percent quench as a function of measured headspace vapor level. This shows a very linear response over a range of vapor from ~30 to 900 ppt (30X).

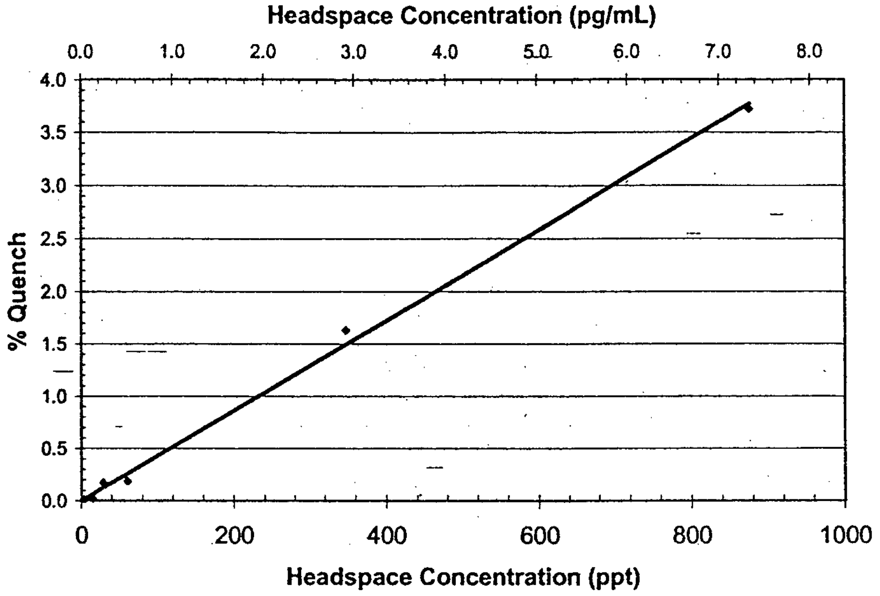


Figure 10. TNT Headspace Over Aqueous Solution, Summary – Percent Quench

An average signal to noise (S/N) was developed using a ratio of the average response from the seven tests at each vapor concentration to the average noise in the last 10 seconds before presentation to the headspace vapors. The S/N as a function of TNT headspace vapor concentration is shown in Figure 11.

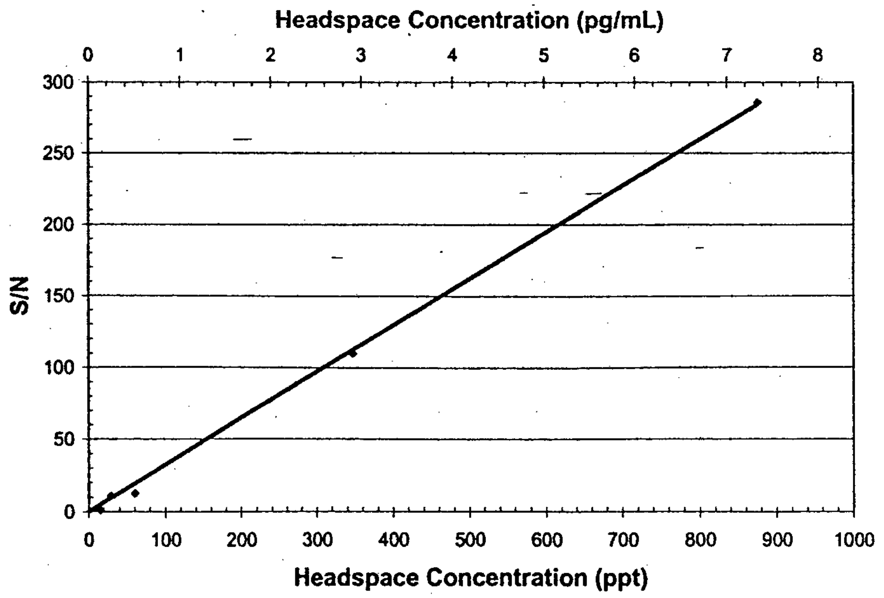


Figure 11. TNT Headspace Over Aqueous Solution, Summary – Signal to Noise

The same information as for TNT headspace vapors over aqueous solutions is shown for TNT headspace vapors over soil standards is shown in Figures 12 – 14.

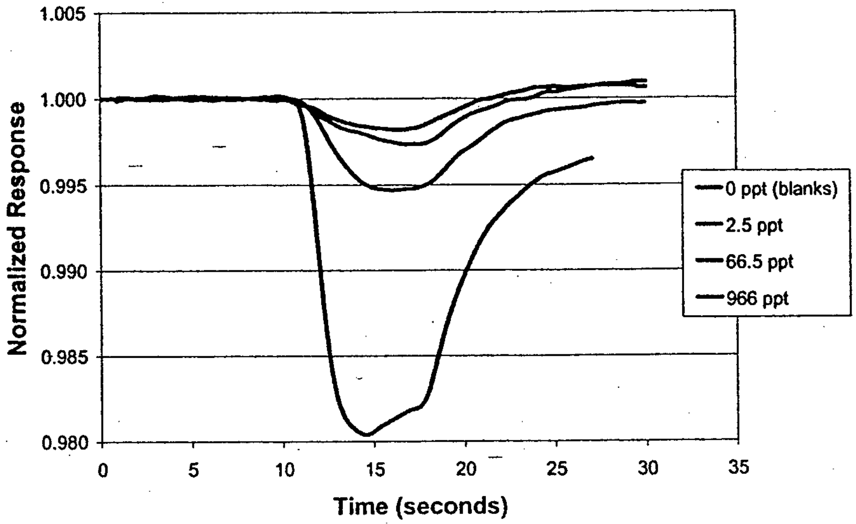


Figure 12. TNT Headspace Over Soil Standards - Time Response

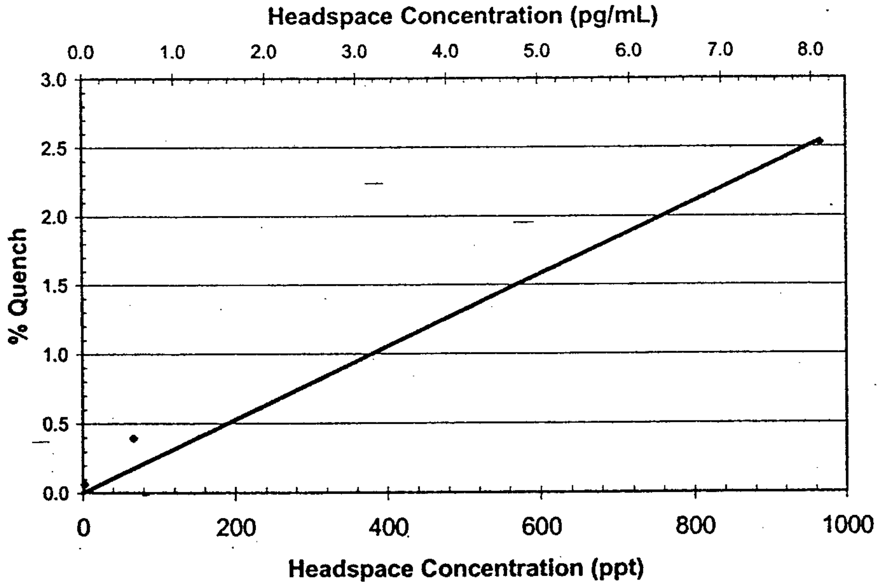


Figure 13. TNT Headspace Over Soil Standards, Summary Results – Percent Quench

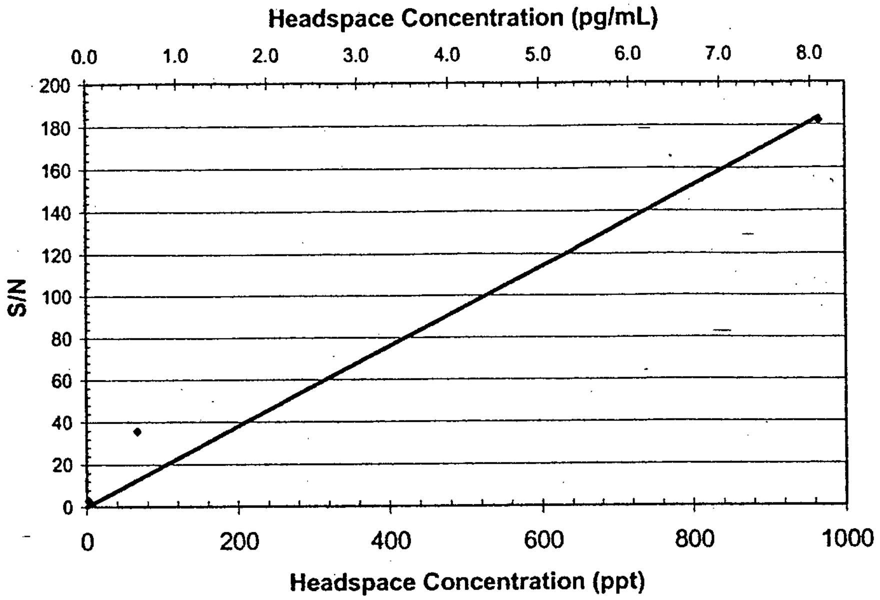


Figure 14. TNT Headspace Over Soils Standards, Summary Results – Signal To Noise

3.5.2 DNT Results

Data was collected for DNT in the same manner as for TNT using both aqueous solution (Figures 15 – 17) and soil standards (Figures 18 – 20).

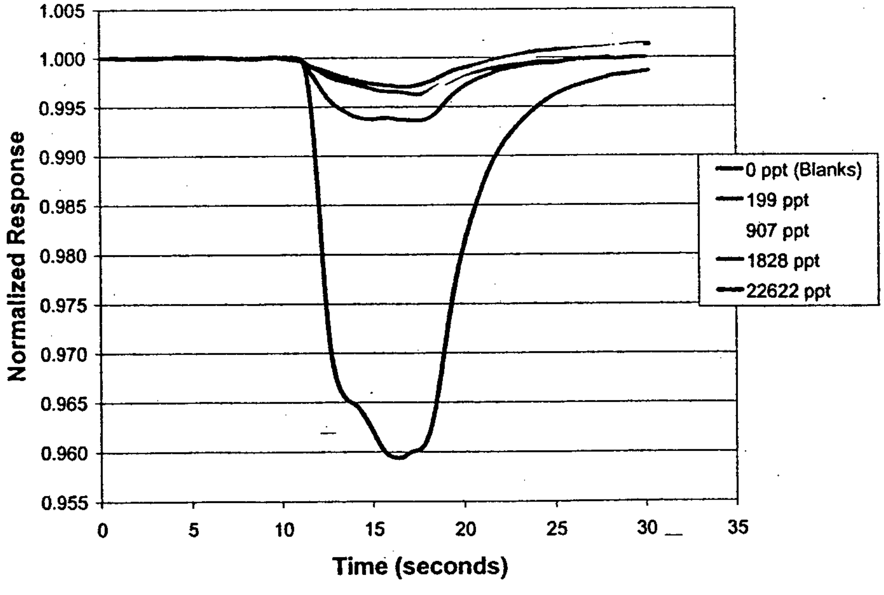


Figure 15. DNT Headspace Over Aqueous Solution – Time Response

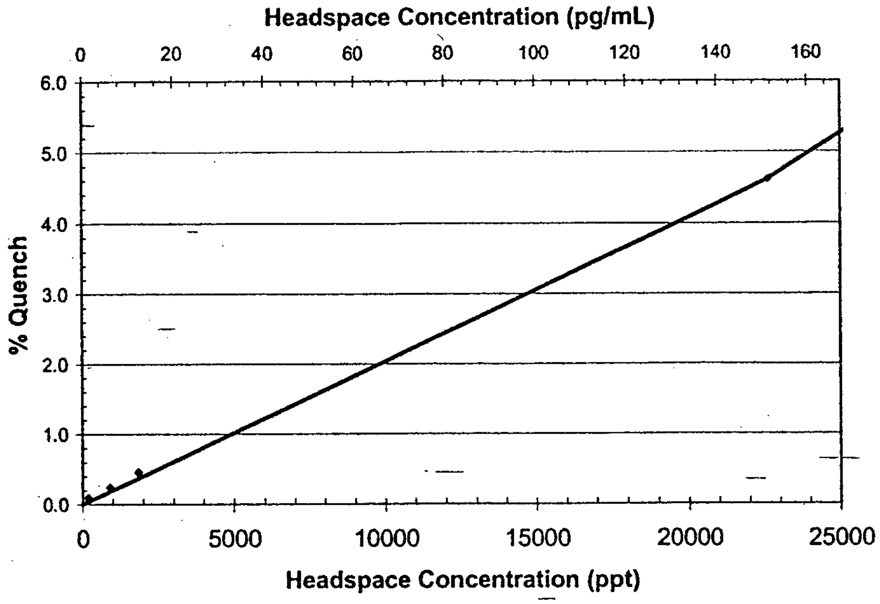


Figure 16. DNT Headspace Over Aqueous Solution, Summary – Percent Quench

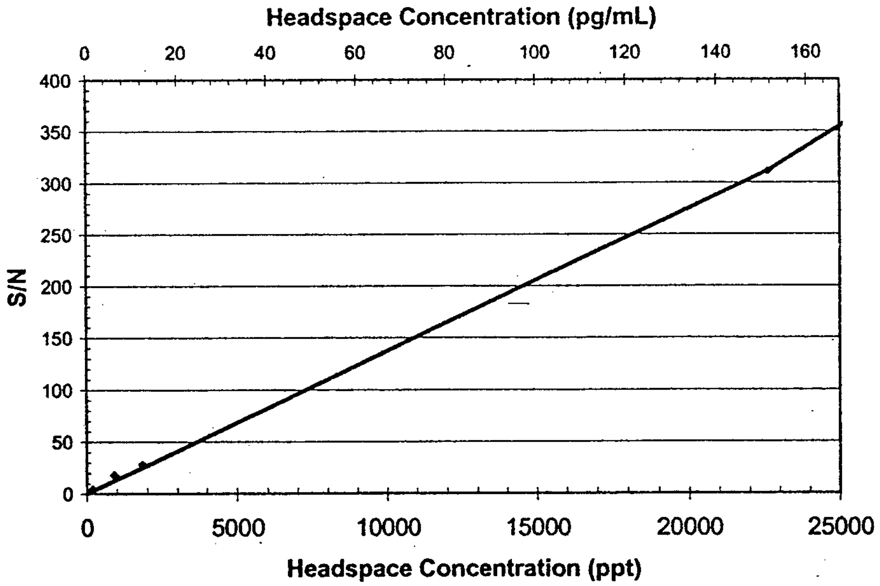


Figure 17. DNT Headspace Over Aqueous Solution, Summary – Signal To Noise

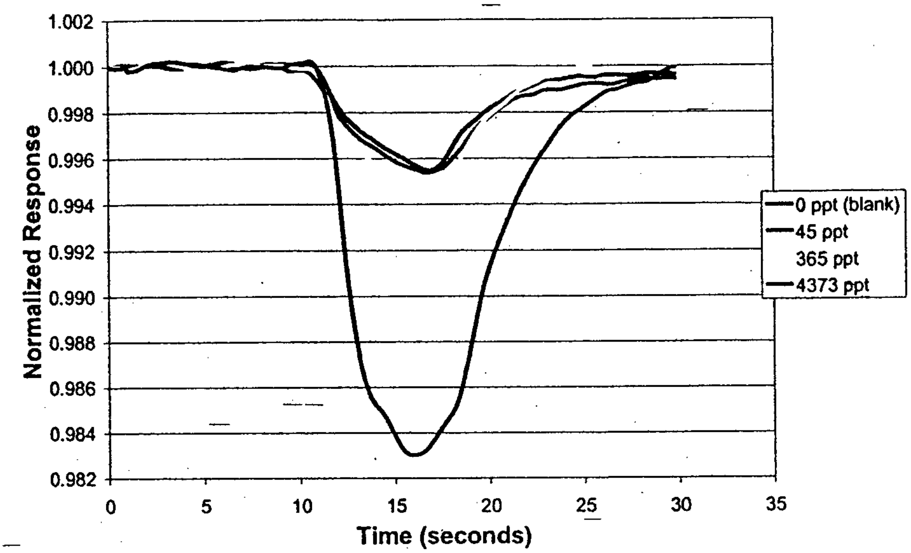


Figure 18. DNT Headspace Over Soil Standards – Time Response

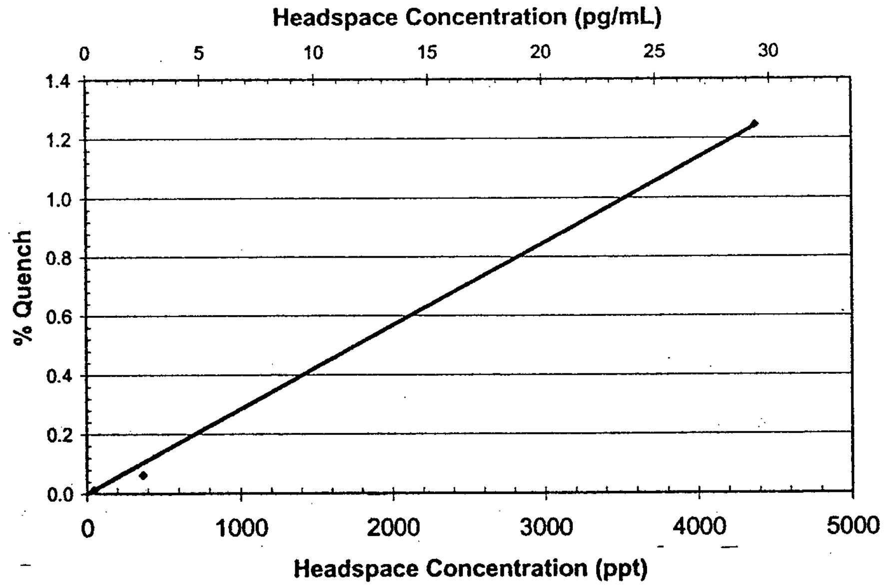


Figure 19. DNT Headspace Over Soil Standards, Summary – Percent Quench

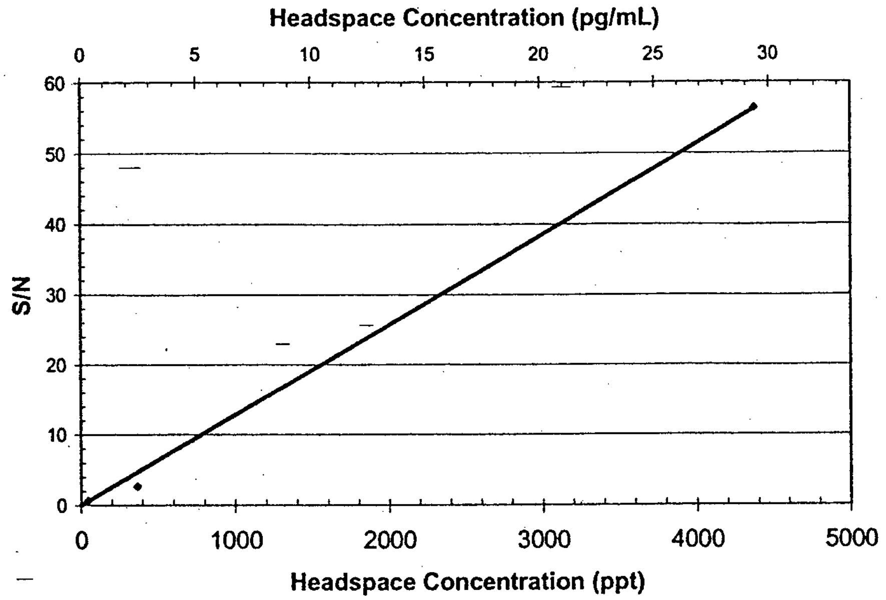


Figure 20. DNT Headspace Over Soil Standards, Summary – Signal To Noise

3.5.3 Results Summary

The Nomadics FIDO 4E sensor was shown to have a good linear response to headspace vapor samples from TNT and DNT aqueous solutions and soil standards. From this data, we can estimate the vapor-sensing threshold in several ways. For situations requiring high confidence in a regulatory environment, the U.S. Environmental Protection Agency (EPA) protocols 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.

An example of the variance in the quench in replicate samples is shown in Figure 21 for TNT headspace vapors (30 ppt) over aqueous solution. To calculate the statistical MDL, the data was processed in several steps. There was a small response of the FIDO to water vapor (see Figure 8), so the average of the peak response to the water vapor (n=7) was first calculated. Next, for all sample runs the root mean square of the first nine (9) seconds prior to placing the FIDO sampler into the headspace vapor was calculated to estimate the instrumental noise. Next, the maximum response for each trial was identified. Then, the maximum quench for each trial was corrected by subtracting the average response due to water vapor.

The standard deviation for the seven trials of the maximum quench corrected for water was determined and is shown in Table 6 for the three lowest TNT headspace over water standards. The regression line for the signal to noise for TNT headspace over water standards (Figure 10) was used to determine the S/N values at each concentration and are shown in Table 7. Table 7 also shows the 99% 95% and 90% confidence minimum detection limits in percent quench and headspace vapor concentrations (ppt), using the t-statistic of 3.14, 1.94 and 1.44, respectively.

We have presented three levels of statistical confidence detection limits because the requirements for vapor sensing of explosive vapors among scrap have not been established. In addition, the time between sampling events for the replicates was short, about three minutes, which was not based on a rigorous assessment of the time needed to reestablish equilibrium headspace concentrations. However, the patterns and maximum quench for the replicate series did not show a declining trend indicative of inadequate time to re-equilibrate the headspace vapor concentrations. Since the response of FIDO to DNT was much less than for TNT, a rigorous statistical analysis of the MDL for DNT was not performed.

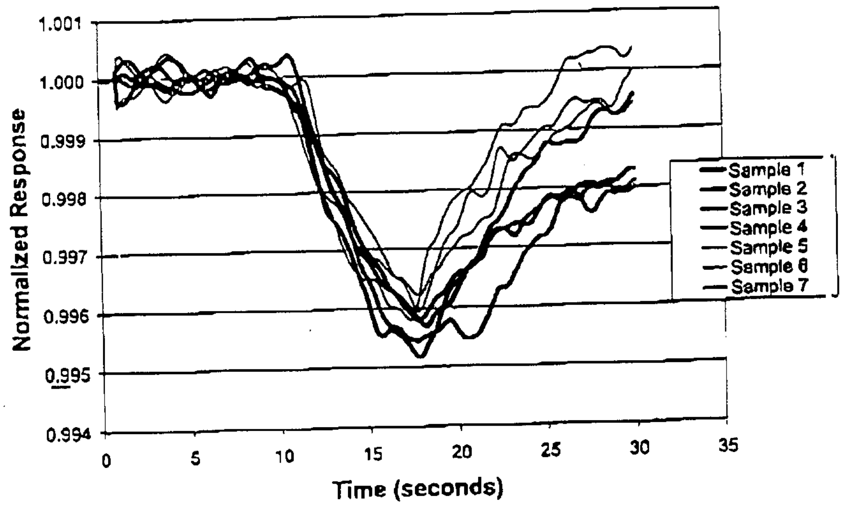


Figure 21. TNT Headspace Vapors (30 ppt) Over Aqueous Solutions – Variability of Replicates

Table 7. Minimum Detection Limits for TNT Over Aqueous Solutions

			99% MDL		95% MDL		90% MDL	
Water Conc (µg/L)	Measured Vapor Conc (ppt)	Signal to Noise	std dev, %Quench Corrected for Water (n=7)	Headspace Conc (ppt)	std dev, %Quench Corrected for Water (n=7)	Headspace Conc (ppt)	std dev, %Quench Corrected for Water (n=7	Headspace Conc (ppt)
400	30	0.025	0.078	18	0.05	11	0.04	8
200	15	0.060	0.188	44	0.12	27	0.09	20
80	4	0.053	0.166	39	0.10	24	0.08	18

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. The regression lines for S/N versus headspace concentration were determined for TNT and DNT for both soil and aqueous standards (Figures 11, 14, 17 and 20). The slopes for these regression lines were used to calculate the minimum detectable vapor concentrations for TNT and DNT at various S/N ratios and are shown in Table 8 and 9, respectively.

Table 8. TNT Sensing Threshold Test Results Summary

Signal to Noise Ratio	Soil Standards	Aqueous Solution	Units
2:1	11	6	ppt
3:1	16	9	ppt
4:1	21	12	ppt

Table 9. DNT Sensing Threshold Test Results Summary

Signal to Noise Ratio	Soil Standards	Aqueous Solution	Units
2:1	156	145	ppt
3:1	234	218	ppt
4:1	311	291	ppt

In summary, the absolute detector threshold provided by a clean matrix (vapor generator effluent) of the FIDO is very low, in the low femtograms range for TNT, which corresponds to an equivalent vapor sensing threshold of 0.03 ppt. However, the actual vapor sensing threshold is more like 10 to 20 ppt for TNT and 150 to 200 for DNT. These differences (~ 150 to 300x) are due to sample entry losses and matrix effects (e.g. water vapor and other airborne chemical interferences). We also found that the FIDO had a very linear response from detection threshold up to testing limits (~100x) with a fast response time (~ 5 seconds to maximum quench) and return to baseline (~15 to 20 seconds).

4.0 Bin Tests

The Nomdics 4D sensor was operated by Nomadics personnel at Sandia National Laboratories from August 28-30, 2001. This sensor package contained a slightly different inlet, optical and data collection/storage system compared to that used in the vapor sensing threshold tests. Figure 22 shows two of the 1 m^3 bins at the Sandia National Laboratories field test location.

Figure 22. Bin Test Field Test Location

4.1 Field Calibration

As described in the sensor threshold testing effort (Section 3.2) TNT aqueous headspace solutions were developed as positive controls during the bin tests. Solutions were prepared gravimetrically for a stock solution at 10 mg/L followed by serial dilutions to make 1 mg/L and 0.25 mg/L solutions. These solutions were measured by RP-HPLC and determined to be 9 mg/L, 0.7 mg/L, 0.07 mg/L after completion of the tests (September 7, 2001). There was a significant decline in the middle and lowest standards compared to the gravimetric basis.

The measured values were used to define the headspace vapor levels. The FIDO 4D sensor did not have a response to water vapor (compared to the FIDO 4E used in the sensor threshold tests). Figure 23 shows single quench traces (no averaging) for each of the calibration solutions. Figure 24 shows a calibration plot of percent quench compared to estimated headspace vapor concentration. A S/N ratio was calculated for each calibration test (not shown), plotted with the estimated headspace vapor concentration, and the slope of this line was used to define the signal to noise ratio detection limits shown in Table 10. The FIDO 4D unit did not demonstrate as low a sensitivity as the FIDO 4E unit used in calibration testing by a factor of 10 (Table 8); however, the sensitivity to water vapor was eliminated.

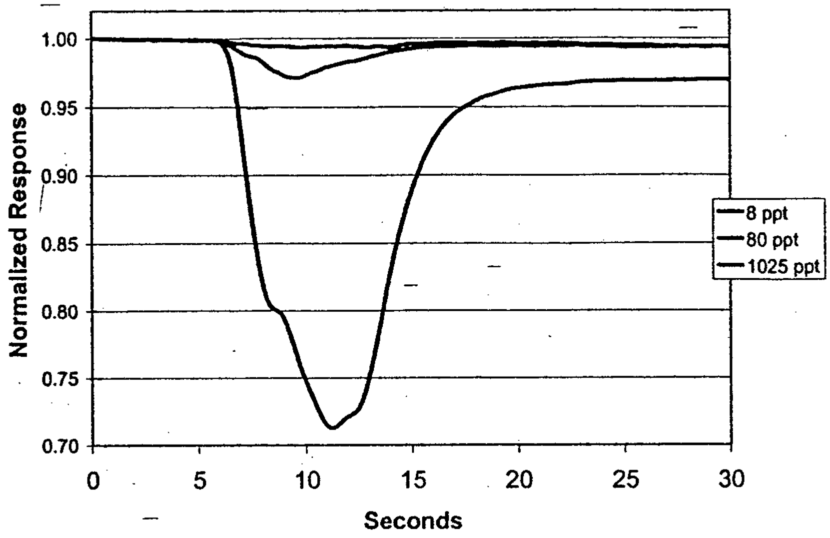


Figure 23. Field Calibration of FIDO 4E

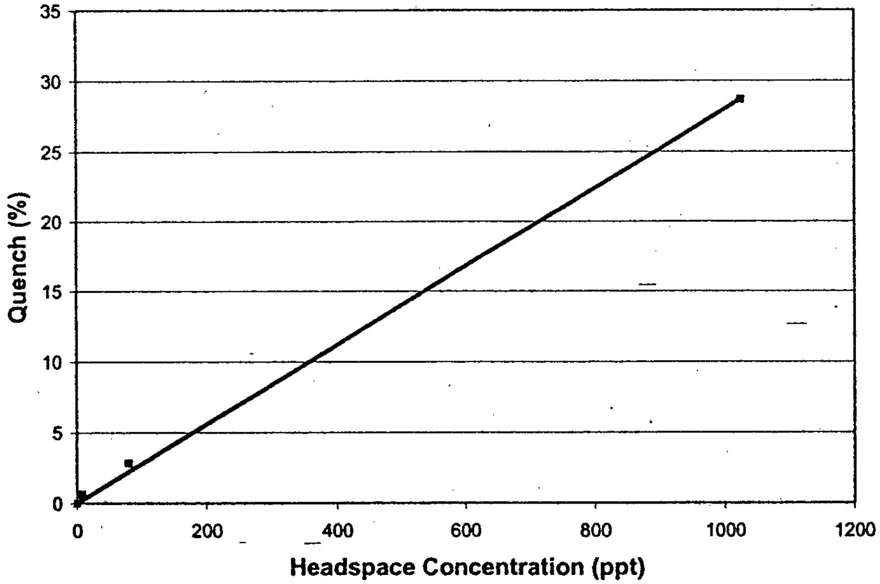


Figure 24. Field Calibration, Quench versus TNT Headspace Vapor Over Aqueous Solution

Table 10. Field Calibration TNT Sensing Threshold Test Results Summary

Signal to Noise Ratio	Aqueous Solution	Units
2:1	71	ppt
3:1	107	ppt
4:1	143	ppt

4.2 Bin Test Plan

Six bins were constructed of plywood to be 1 m^3 in volume. Scrap was obtained from Sandia National Laboratories locations and included wood, metal, plastic and one tire in each bin. Figure 25 shows typical contents in these bins. A 2.5 cm diameter hole was drilled into the top of each bin. The FIDO was placed into the hole such that the tip of the FIDO inlet protruded just into the bin. To improve presentation of the explosive vapors to the FIDO instrument a small portable fan was placed into the bin (Figure 26).

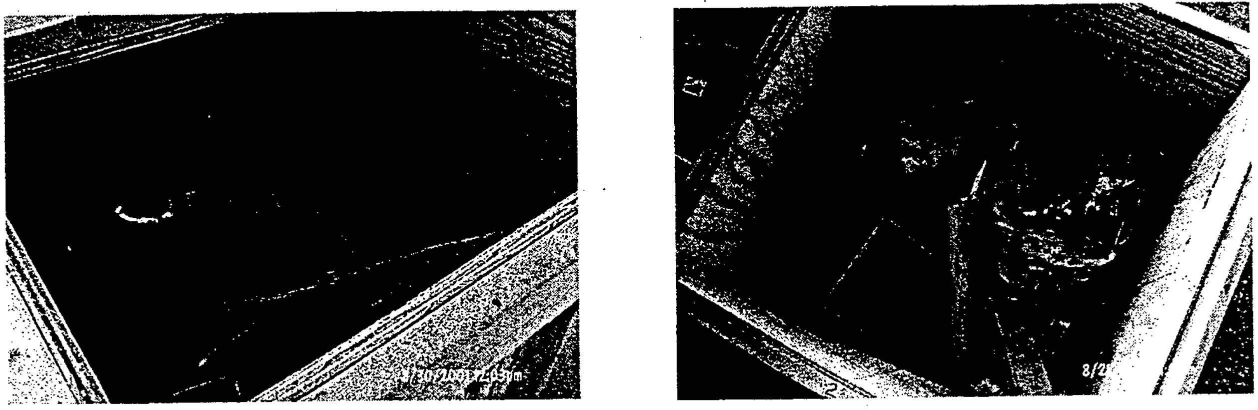


Figure 25. Typical bin contents

Figure 26. Recirculation Fan Inside Bin

4.3 Bin Trials

The following sections describe the type of explosive containing material placed into the bin, the time prior to measurement with the FIDO, the temperature, a chart of the time-quench response of the FIDO and the maximum quench response.

4.3.1 Low Order Detonation Debris

Test conditions
Time prior to sensing- 7 minutes
FIDO sampling duration - 2 seconds
Bin temperature - 23°C inside bin

Test Results
Background max quench = 0.94 %
Low order debris max quench = 5.8 %

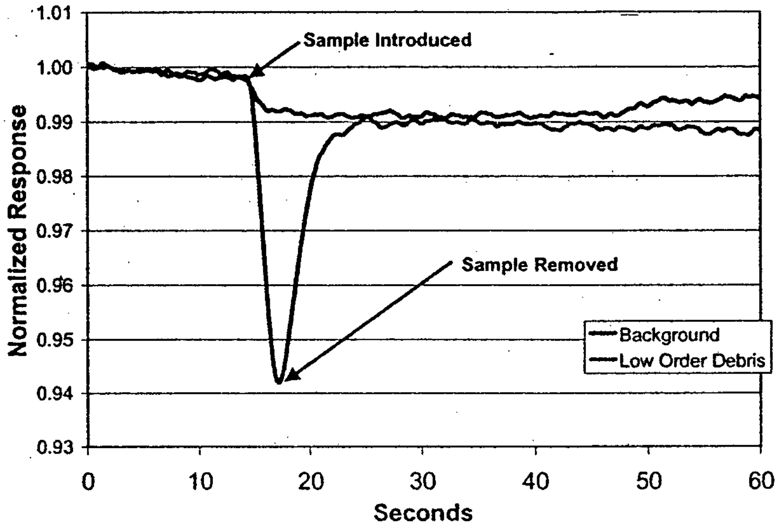
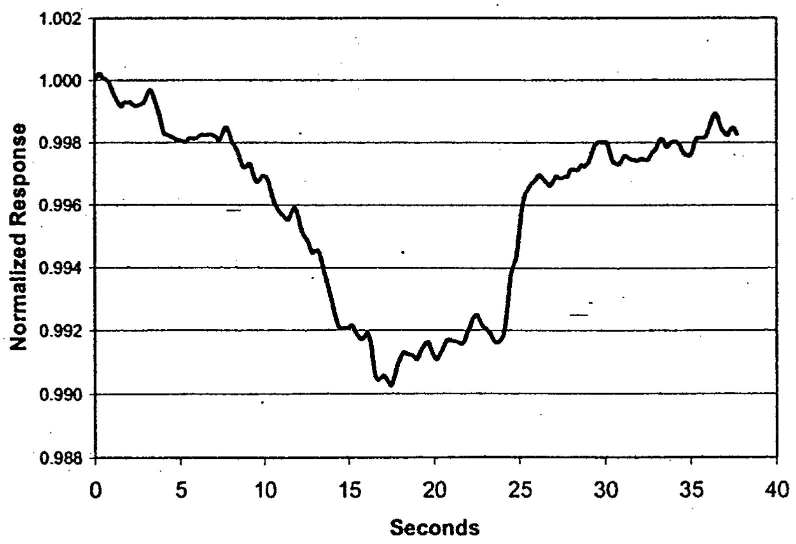


Figure 27. Low Order Debris Response

4.3.2 Demolition Block Crumbles (1 gram in plastic cup)

Test conditions
Time prior to sensing - 7 minutes
FIDO sampling duration - 2 seconds
Bin temperature - 23°C inside bin

Test Results
Background max quench = not measured
Max quench = 0.98%



No Picture

Figure 28. Demolition Block Response

4.3.3 81 mm Mortar in Pan

Test conditions
Time prior to sensing - 3 minutes
FIDO sampling duration - 2 seconds
Bin temperature - 23°C inside bin

Test Results
Background max quench = not measured
Max quench = 1.99%

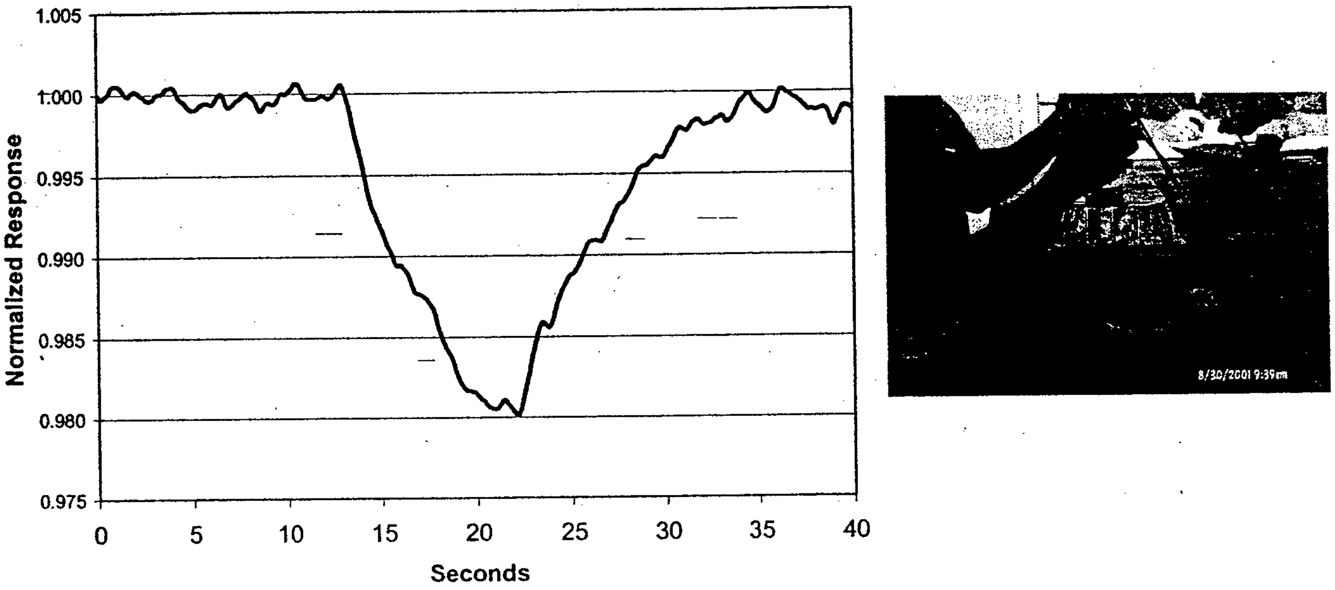


Figure 29. Mortar in Pan Response

4.3.4 81 mm Mortar Shipping Tube (not in bin)

Test conditions
Time prior to sensing – N/A
FIDO sampling duration - 2 seconds
Bin temperature - 23°C inside bin

Test Results
Background max quench = not measured
Max quench = 0.81%

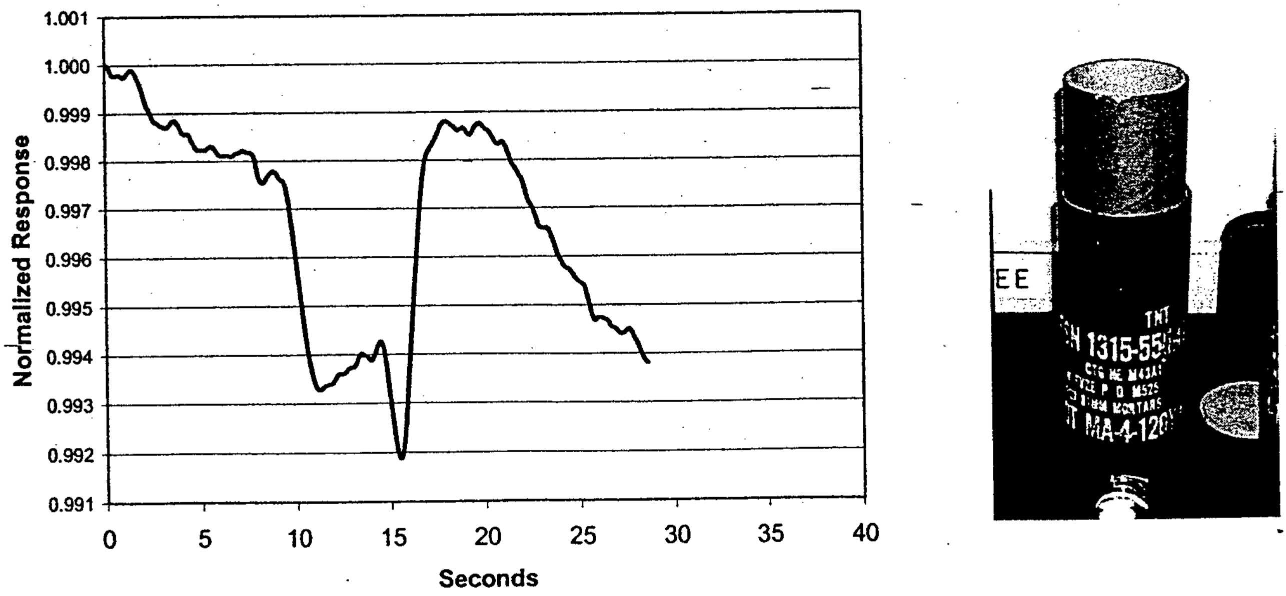


Figure 30. Mortar Shipping Tube Response

4.3.5 81 mm Mortar in Bin

Test conditions	Test Results
Time prior to sensing – N/A	Background max quench = not measured
FIDO sampling duration - 2 seconds	Max quench = 0.44%
Bin temperature - 23°C inside bin	

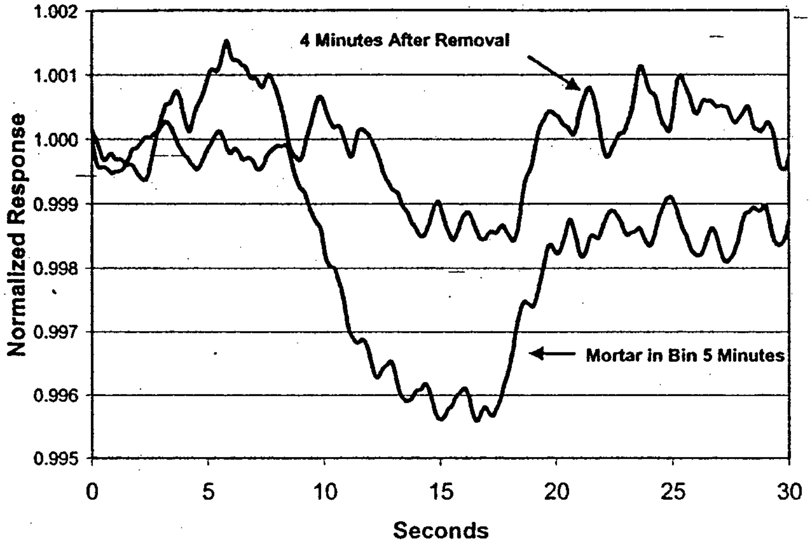


Figure 31. Mortar in Bin Response

4.3.6 81 mm Mortar in Bin with Fuse Removed

Test conditions	Test Results
Time prior to sensing – N/A	Background max quench = not measured
FIDO sampling duration - 2 seconds	Max quench = 1.12%, 0.72%, 0.21%
Bin temperature - 23°C inside bin	

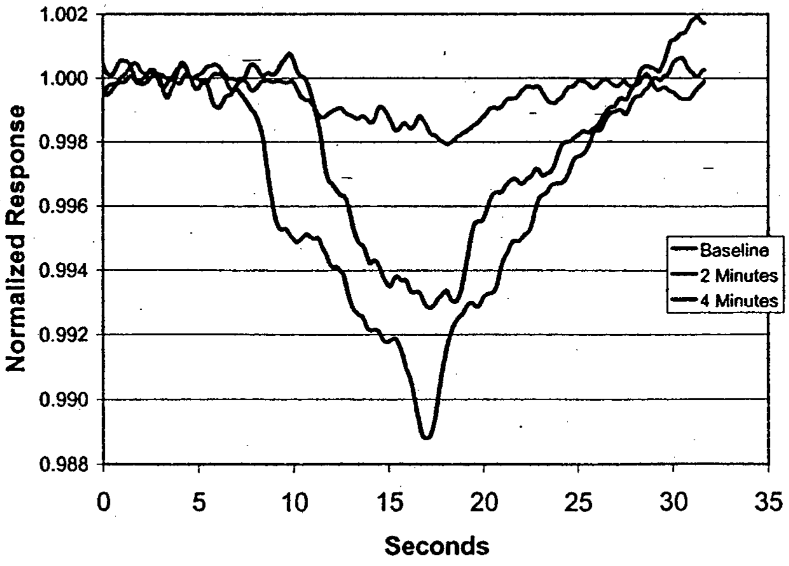


Figure 32. Mortar in Bin with Fuse Removed Response

4.3.7 Long Term Monitoring of Low Order Debris in Bin

Test conditions	Test Results
Time prior to sensing – N/A	Background max quench = not measured
FIDO sampling duration - 2 seconds	Max quench =
Bin temperature - 23°C inside bin	

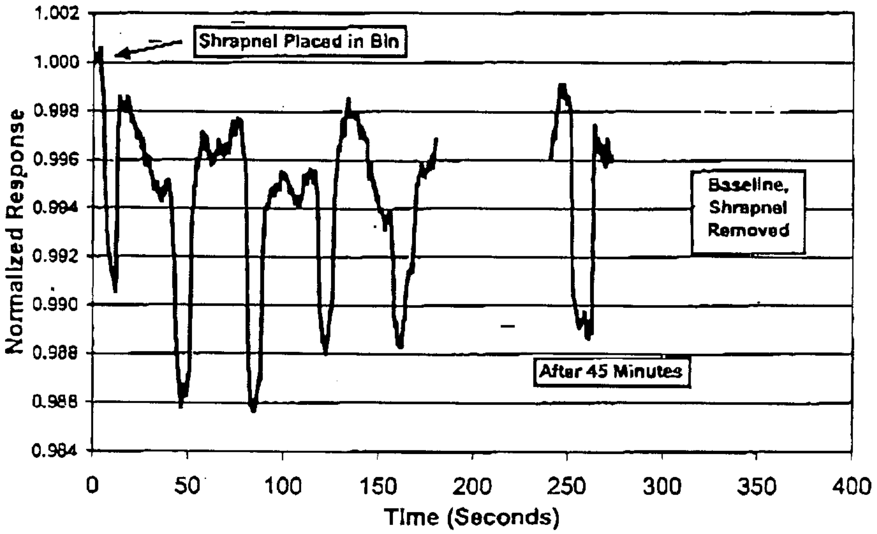


Figure 33. Long-Term Monitoring of Low Order Debris in Bin Response

4.3.8 Summary

Of the item surveyed with in the bin tests, the low order debris showed the highest FIDO quench. Table 11 shows the maximum quench and estimated vapor concentration derived from the calibration plot shown in Figure 24. These results show discrimination of explosive vapors derived from various items among scrap is possible. Some of the values in Table 11 are below the derived 2:1 S/N ratios (Table 10). Therefore, the assignment of a S/N ratio for a sensor threshold could be less than 2:1 for this device.

Table 11. Bin Test Summary Results

Item	% Quench	ppt
low order debris	5.8	207
demo block	0.98	35
mortar in pan	1.99	71
shipping tube	0.81	29
mortar in bin	0.44	16
mortar in bin, no fuse	1.12	40

5.0 Conceptual Design

5.1 Conceptual Scrap Sensing Systems

The following four conceptual scrap sensing systems have been envisioned:

1) Handheld Manual Inspection Method. A handheld survey tool can be constructed for use in manually interrogating suspect items in scrap piles on the range. Both a low volumetric rate sampling system and a high volumetric rate sampling system have been envisioned.
2) Batch Bin Method. The simplest approach for a small volume of range scrap is to place the material into a bin, place a fan inside or use an external air source (ambient or heated) to circulate the air, and sample with the FIDO at multiple locations through penetrations in the bin sides or top. These bins can be small or as large as shipping containers.
3) Continuous Conveyor Method. For large volumes of scrap, a continuous conveyor can be constructed with scrap items placed onto a conveyor belt that passes into a sensing chamber. The conveyor can either stop for a short period or run at a slow rate through the sensing chamber. Internal or external air sources (ambient or heated) can be used to circulate air in the sensing chamber. Again, the FIDO sensors can be placed at multiple locations on the sides or top. If there are infrequent indications by the FIDO, the system can be stopped and scrap items can be inspected on the conveyor belt. If there are frequent indications, a segregation system can be employed that automatically transfers this material to a pile for later manual inspection.
4) Hot Gas Decontamination Quality Assurance. SERDP has expressed interest in using the FIDO vapor sensor to verify adequate decontamination of scrap that has been processed by a hot gas system. This system involved organizing the scrap into a pile, covering the pile with insulation, and passing hot air from one end through the scrap to an exit at the opposite end. The gas exiting the system could be monitored for explosive vapors and when the concentrations reach a minimum determined value, the scrap can be considered safe for traditional recycling.

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