

Laboratory Data and Model Comparisons of the Transport of Chemical Signatures From Buried Landmines/UXO

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ABSTRACT

Sensing the chemical signature emitted from the main charge explosives from buried landmines and unexploded ordnance (UXO) is being considered for field applications with advanced sensors of increased sensitivity and specificity. The chemical signature, however, may undergo many interactions with the soil system, altering the signal strength at the ground surface by many orders of magnitude. The chemodynamic processes are fairly well understood from many years of agricultural and industrial pollution soil physics research. Due to the unique aspects of the surface soil environment, computational simulation is being used to examine the breadth of conditions that impact chemical signature transport, from the buried location to a ground surface release. To provide confidence in the information provided by simulation modeling, laboratory experiments have been conducted to provide validation of the model under well-constrained laboratory testing conditions. A soil column was constructed with soil moisture monitoring ports, a bottom porous plate to regulate the soil moisture content, and a top plenum to collect the surface flux of explosive chemicals. The humidity of the air flowing through the plenum was set at about 50% RH to generate an upward flux of soil moisture. A regulated flux of aqueous phase 2,4-DNT was injected into the soil at about ten percent of the upward water flux. Chemical flux was measured by sampling with solid phase microextraction devices and analysis by gas chromatography/electron capture detection. Data was compared to model results from the T2TNT code, specifically developed to evaluate the buried landmine chemical transport issues. Data and model results compare exceptionally well providing additional confidence in the simulation tool.

Keywords: landmine, data, data-model comparisons, soil transport

1. INTRODUCTION

Detection of buried landmines by sensing the chemical signature emanating from the soil surface is believed to be the principal method used by mine detection dogs. This signature could also be measured by vapor sensing technologies for buried landmines or UXO. Chemical movement in soils is impacted by multiple, interdependent environmental factors that complicate transport processes. Understanding and taking advantage of processes that enhance chemical movement to the dog or sensor is critical to improving the detection of buried landmines/UXO. Due to the large number of combinations of individual processes, it is not feasible to use solely laboratory or field experimentation to optimize chemical sensing for buried landmines. Computational simulation methods can more efficiently explore these environmental factors. Caution must be used in making predictions based solely on simulation models. More confidence can be attributed to simulation predictions when experimental data is collected in controlled laboratory experiments and compares well with simulation model results.

The goal of this effort was to collect data in soil column experiments for comparison to simulation model results. The soil column test apparatus was based on systems designed for agricultural pesticide soil emissions (Spencer and Cliath, 1973) and industrial pollutant migration (Petersen et al., 1996). The soil in the column is unsaturated and held at a specified matric potential to control soil moisture content. There is an infinite source of water below the soil that can be transported upward through the soil, which is induced by a low humidity air flowing in a plenum over the soil surface. Soil moisture is measured physically at discrete depth increments by water content reflectometry. A point source of 2,4-Dinitrotoluene (DNT) in water is injected centrally in the column, continuously at a specified depth, and collected daily in the headspace of the plenum by solid phase microextraction (SPME). At experiment termination, the soil column was sacrificed and soil samples were obtained to measure water content and DNT concentration.

The simulation code T2TNT has been used to evaluate the chemical transport in this experiment and has been developed specifically to evaluate the special conditions found with the buried landmine problem (Webb et al., 1999). This code emerged from the TOUGH2 family of codes and has been modified to contain surface boundary conditions and critical phase

partitioning phenomena important to wetting and drying events that vary widely at the ground surface (Webb and Phelan, 2000).

2. MATERIALS AND METHODS

Soil Properties

The soil was obtained in an undeveloped area at Sandia National Laboratories in Albuquerque, NM. The soil was air-dried, sieved to retain the less than 2-mm in diameter fraction, and mixed to obtain homogeneity. Through soil analysis, it was determined that the soil is a sandy-loam with low organic carbon, a moderate electrical conductivity, and a substantial specific surface area (Table 1). The organic matter analysis was performed using the Walkley-Black method (Klute, 1986). Electrical Conductivity was measured using the saturated paste method (Klute, 1986). Particle size analysis was performed by hydrometer (Klute, 1986). Surface area of the soil was determined using the Brunauer-Emmett-Teller (BET) method with a Micromeritics Accelerated Surface Area and Porosimetry 2405 instrument (Klute, 1986). A water retention curve of this soil (Figure 1) was constructed using both the hanging water column and pressure plate (Soil Moisture Corp., Goleta, CA) techniques (Klute, 1986).

Table 1. Soil Properties

Soil Property	Value
Classification	Sandy Loam
Sand	70.4 %
Silt	21.2 %
Clay	8.3 %
Organic Matter	0.8 %
Cation Exchange Capacity	10.7 meq/100g
Electrical Conductivity	0.91 dS/m
Surface Area	23 m ² /g

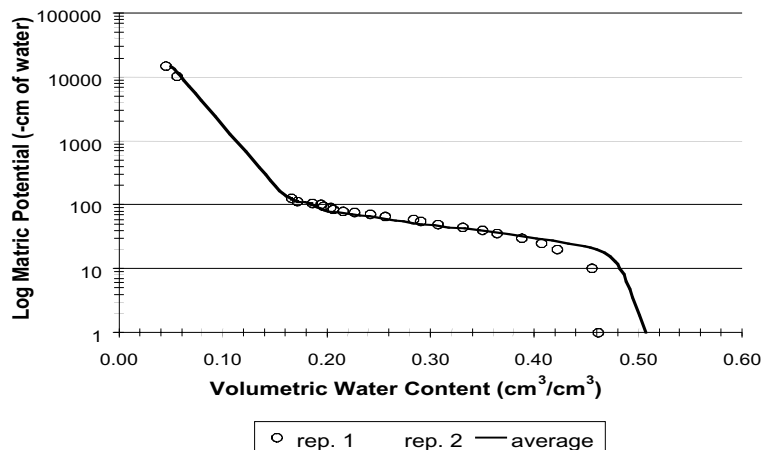


Figure 1. Soil water retention curve

DNT-Soil Interactions

Measurements of the DNT aqueous-solid distribution coefficient (K_d and K_f) were based on the method in 40 CFR 796 and Pennington and Patrick (1990) with aqueous DNT solution concentrations of 0.5, 0.75, 1.25, 1.5, and 2.0 mg/L. A set of higher aqueous concentrations of DNT at 25, 50, 75, 100, 125, and 150 mg/L were also used because the DNT injection in the soil column was set for ~150 mg/L. With both sets of data, it appeared that the DNT sorption behavior on this soil best fit

a Freundlich isotherm. Table 2 shows the fitting parameters for the linear and Freundlich isotherms. The T2TNT code currently uses a linear K_d .

Table 2. Soil Water Partitioning Parameters

Solution Concentration	Linear K_d (mL/g)(R^2)	Freundlich K_f, b (R^2)
Low	1.8 (0.97)	
High	0.72 (0.92)	
Both	0.72 (0.98)	1.7, 0.82 (0.99)

The values shown in Table 2 were derived from batch equilibrium sorption experiments where the soil:water ratio was 1:4. This method is generally accepted and allows for comparison of sorption coefficients between soils and chemicals. However, unsaturated soil systems have soil:water ratios more like 2:1 and a packing density that prevents complete contact of the aqueous chemical solution with all soil particle surfaces. Other work using saturated soil column retardation test methods (Klute, 1986) indicates that the soil water partitioning coefficients are about 3 to 10 times less than from batch equilibration tests. In tests to estimate soil vapor partitioning parameters using these same soils, the data implies a soil water partitioning coefficient (K_d) of ~ 0.4 mL/g (Phelan and Barnett, 2000). The importance of the soil water partitioning coefficient becomes apparent in the data model comparisons discussed later.

Experimental Set-Up

The experiment was conducted in a 34-cm tall and 14.6-cm i.d. acrylic column (Figure 2), which was packed to a dry bulk density of 1.2 g/cm³ by misting fine layers of soil with an aqueous 0.005 M CaCl₂ solution. A 1-bar high flow porous ceramic plate (Soil Moisture Inc.) and a water reservoir were fastened to the lower portion of the column. This reservoir was connected by 0.25 inch (0.64 cm) flexible tubing to a graduated cylinder, providing a hanging water column to control soil moisture content within the acrylic column. Along the length of the column, holes were cut to accommodate the insertion of soil moisture probes and syringe ports. The upper most syringe port served as the injection location for a continuous delivery of aqueous phase DNT into the center of the packed column from a 5-ml syringe connected to a Cole-Parmer (Vernon Hills, IL) model 74900 syringe pump. A stainless steel plenum was fastened to the upper portion of the column providing both sweep-gas flow and a location to sample DNT surface flux.

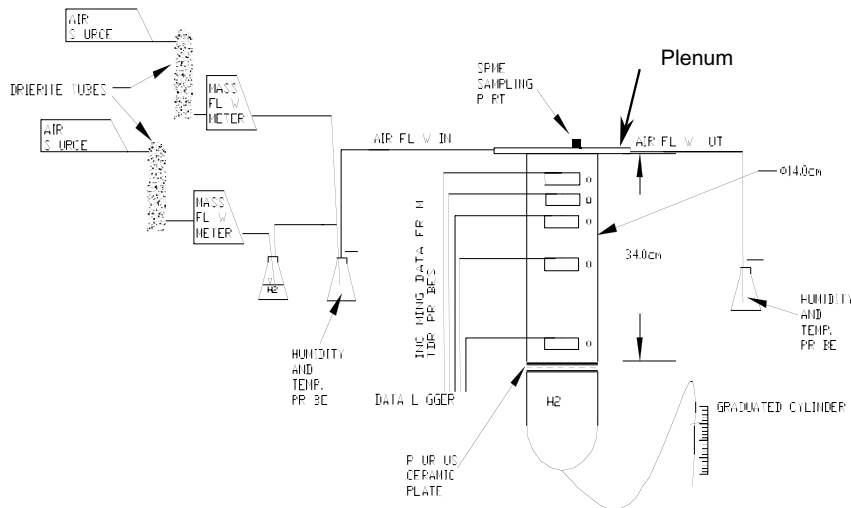


Figure 2. Schematic of Soil Column Test Apparatus

Soil Moisture Measurements

Five Campbell Scientific model CS615 water content reflectometers (WCR) were installed horizontally into the column at depths of 2.5, 5.0, 8.9, 15.2, and 31.0 cm from the surface of the soil. The rods of each probe were cut to a length of 13.2 cm to accommodate the diameter of the column. Each WCR was positioned in a pre-cut hole and sealed with a silicone sealant to prevent liquid and vapor loss. The WCRs were connected to a Campbell Scientific AM-416 multiplexer equipped with a Campbell Scientific model SC32A optically isolated RS232 interface and connected to a Campbell Scientific CR-7 data-logger. Water content was measured every half-hour and collected from the data-logger via a personal computer with Campbell Scientific PC-208 software.

Each WCR probe was calibrated in a 6-inch diameter PVC cell with a height of 5 cm packed with air-dried soil to a dry bulk density of 1.2 g/cm³. An aqueous 0.005M CaCl₂ solution was added to each soil cell and heated by microwave oven to redistribute soil water (Horton et al, 1982). The soil cells were allowed to cool for 2 hours and the cycle was repeated 5 times. The WCR probes were inserted into the soil and measurements of the WCR period and gravimetric water content were obtained. This process was repeated with additional increments of water until 5 water content levels were obtained and then the data was fit to a second order polynomial calibration curve for each probe.

Surface Flux Measurements

Samples of the DNT vapor surface flux were collected through a septum fitted swagelock fitting 2-cm from the center of the plenum in the downstream air flow direction with SPME fibers. These samples were taken once daily for 24 hours for the first 5 days, then twice daily for variable time to collect enough mass to quantify.

The dynamic extraction efficiency of the SPME fibers in the plenum was determined experimentally. An oil-less piston pump pulled an air stream from the plenum through an Orbo-79 tenax cartridge (Supelco). The hole in the bottom of the plenum was sealed with aluminum foil, on which approximately 1 gram of 99% pure crystalline DNT (Aldrich, Milwaukee, WI) was placed. The air stream flowed at a rate of ~1 L/min controlled by a metering valve attached to the piston pump and monitored by a mass flow meter (Aalborg). The system was allowed to equilibrate for two days after which a new tenax cartridge was placed in-line for a period of 30 minutes. During this time, six SPME fibers were sequentially placed in the sampling location for a 30-second duration. This process was repeated three times over the course of three days. Extraction efficiency was calculated as:

$$\% \text{Efficiency} = \frac{(\text{Mass 2,4-DNT on SPME})/(\text{Minutes of Sampling})}{(\text{Mass 2,4-DNT on Tenax})/(\text{Minutes of Sampling})} * 100 \quad (1)$$

The SPME extraction efficiency was determined to be 1.475% of the total DNT mass flowing past it with a standard deviation of 0.18%.

The SPME fibers and tenax cartridges were analyzed using a Varian 3400CX gas chromatograph (GC) equipped with a Varian 1078 split/splitless injection port, an electron capture detector (ECD), a 6-m long 530 micron o.d. DB-5 column (J&W Scientific, Folsom, CA), and a personal computer. Furthermore, a Varian 530 µm flash and on column insert (Supelco) for the injection port was utilized to optimize the entry of the analyte into the analytical column. The GC operating conditions were as offered in the proposed EPA method SW846-8095 (EPA, 1998). The tenax cartridges were extracted using Occupational Safety and Health Administration (OSHA) method 44 (OSHA, 1983).

Experimental Methods

To initiate the experiment, two Badger model 180-22 (Franklin Park, IL) oil-less piston pumps each forced an air stream through drierite traps (Cole-Parmer). One of the air streams passed through an air-sparging tube into an Erlenmeyer flask filled with deionized water. The dry and humid air streams were combined to produce a single air-stream flowing at ~1 L/min with ~50% relative humidity (RH). The combined air stream then passed into the plenum and acted as a sweep gas. The RH (%), temperature (°C), and absolute humidity (g/m³) of the air both entering and exiting the plenum were measured through a Vaisala HMP-36B relative humidity sensor and displayed on a Vaisala HMI-36 humidity data processor.

The column was equilibrated with the soil matric potential set at -20 cm and the sweep gas for 2 weeks. This induced an upward water flux of ~ 14 mL/day. The aqueous DNT solution was injected at a continuous flow of ~ 10% of the upward water flux (1.44 mL/day). Twenty-nine days after injection began, the column experiment was concluded and soil samples were collected for both gravimetric water content and DNT analysis using a 7/16-inch polycarbonate tube. Nine boreholes were made with five equidistantly centered between the WCR rods, and four more made perpendicular to the WCR rods and coincident with the air stream axis (Figure 3). One-half inch (1.3 cm) deep surface samples were taken at all 9 holes, while samples were taken from the center and four adjacent bore holes every half inch to a depth of 6 inches (15 cm).

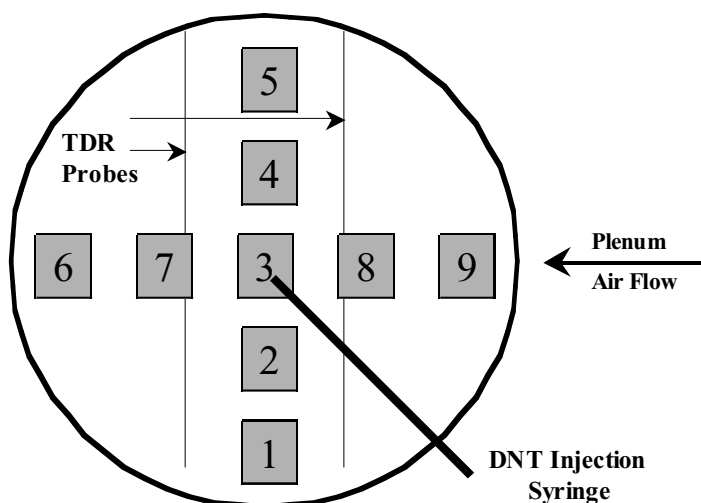


Figure 3. Soil Sampling Array

The soil samples were extracted using the SW846-8330 method excluding the salting-out procedure. These extracts were analyzed using a Hewlett Packard 6890 series GC with a thin coat 530- μm o.d 6-meter long RTX-225 column attached to a micro ECD. The GC was further outfitted with a Hewlett Packard 7683 series refrigerated autosampler. Operating conditions were as mentioned for SPME and tenax analysis.

3. EXPERIMENTAL RESULTS

In the design of this test, a soil moisture of about $0.25 \text{ cm}^3/\text{cm}^3$ was desired to maintain the soil pore space at about 50% saturation. From the soil moisture characteristic curve (Figure 1), this would indicate a matric potential of -50 to -70 cm. However, in the test equilibration period, a matric potential of -20 cm provided the desired water content as indicated by the WCRs. The soil moisture content at each WCR depth over the duration of the experiment is shown in Figure 4. This shows that the soil moisture distribution remained relatively constant over the test duration and shows an expected gradient with higher moisture contents at the bottom of the soil column.

The flux of DNT in the plenum increased by about 10^4 over the duration of the test (Figure 5). The early time period measurements have significant uncertainty due to the long (24 hour) SPME sampling periods. At the end of the test, the SPME sampling periods were about 1 minute in order to stay within the linear range of the GC/ECD system. The last SPME plenum sample was obtained on day 26.

The experiment was sacrificed on day 29. The results of the soil samples obtained for soil moisture content are shown in Figure 6. For holes 2, 7 and 8, the variation in soil moisture contents is a maximum of about 20% at any depth. These moisture contents compare favorably with the average WCR values at each depth. Holes 3 and 4 both show significantly lower moisture contents that can not be explained by variations in loading the soil column or airflow directions in the plenum relative to the soil core locations. The best explanation is that the sampling method caused compaction of the soil resulting in a loss of water.

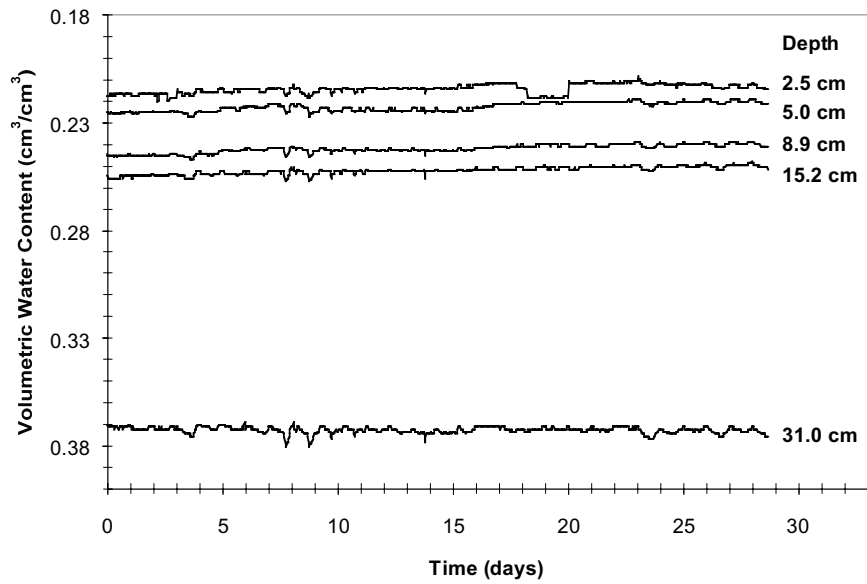


Figure 4. Water Content Reflectometry Measured Soil Moisture Content in Soil Column

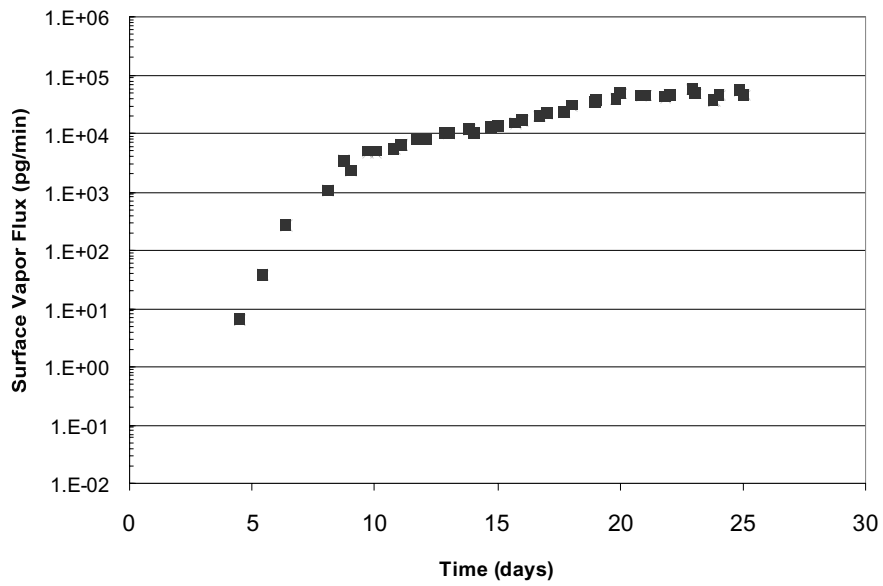


Figure 5. 2,4-DNT Surface Flux

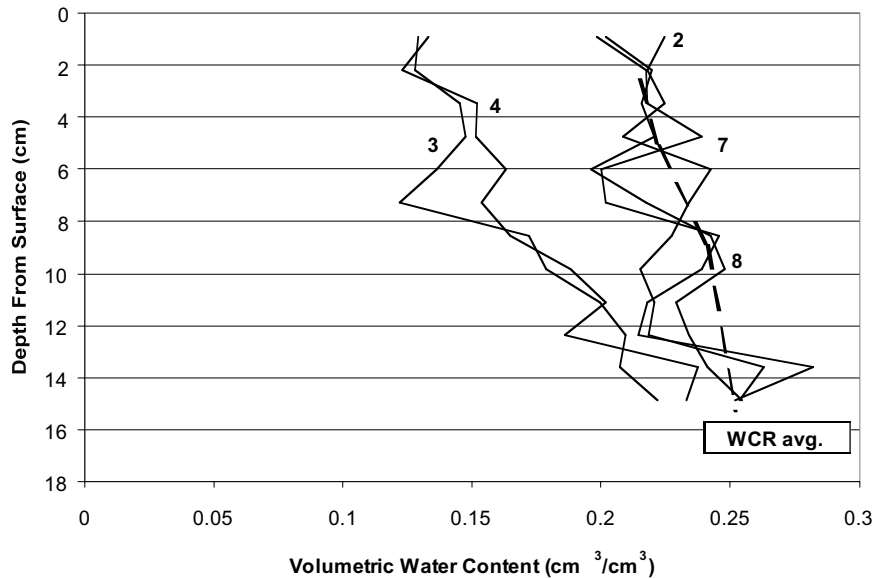


Figure 6. Soil Moisture Content at Test Conclusion

4. SIMULATION PARAMETERS

The experiment described in this paper has been modeled with the T2TNT code (Webb et al., 1999) using a two-dimensional model (R-Z). The various parameters in the simulation are discussed in more detail below.

Problem Definition

The problem under consideration is a soil column with a porous plate boundary condition at the bottom and forced flow of air at the top. Based on the expected large radial variation in the DNT concentration, a two-dimensional R-Z model with 0.73-m radius and 0.333-m depth was developed as shown in Figure 7. The mesh spacing is 9 elements in the R-direction and 34 elements in the Z-direction for a total of 307 elements including one for the atmosphere. The bottom boundary condition, which represents the porous plate, is constant saturation and pressure, while the top boundary condition in the plenum is constant pressure, temperature, and relative humidity. The boundary layer thickness at the top of the column had to be specified because the algorithm in T2TNT is only applicable to a free boundary layer, not a confined one as in this experiment. The boundary layer thickness was varied to match the steady-state evaporation rate for water from the column, which resulted in a steady flow of water up the column from the lower porous plate boundary condition. The final calibrated boundary layer thickness was 2.1 cm.

The DNT source rate, including the water addition, is added to the center radial element 0.35 m from the surface. The characteristic curves based on the soil water retention curve were fit to a van Genuchten form using the Webb (2000) extension as discussed in more detail by Webb and Phelan (2000); the parameters are given in Table 3. As mentioned earlier, the measured soil characteristic curve and the data from the soil column did not match. Therefore, the saturation at the porous plate boundary condition was varied in order to achieve the desired saturation in the top of the column. A comparison of the measured and predicted moisture contents in the top of the column are presented in Figure 8.

A constant half-life of 30 days for DNT was assumed. Other parameters used in the model are summarized in Table 4. Because the saturation never got below about 0.40, enhanced vapor-solid sorption was not a factor (see Webb and Phelan, 2000). As will be shown later, the value of K_d was varied in order to match the data. Future tests, which are already underway, will involve drying and rewetting of the surface of the column so that vapor-solid sorption will become an important factor.

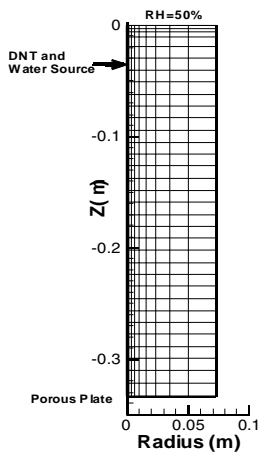


Table 3. Two-Phase Characteristic Curve and Other Parameters

Permeability ^a	$1.0 \times 10^{-12} \text{ m}^2$
Porosity	0.504
Fully-Saturated Conditions ^a (S_S)	0.999
Liquid/Residual Saturation ($S_{l,r}$)	0.1194
Matching Saturation (S_l^* - see Webb, 2000)	0.1325
Air Entry Pressure Parameter ($1/\alpha_G$)	3410. Pa
n	2.14
m (= $1 - 1/n$)	0.533

^a — assumed

Figure 7. Model Nodalization

Table 4. 2,4-DNT Chemical Properties

K_H (25°C)	9.8E-6
K_d (ml/g)	varies
A_0 ($\log_{10} K_d$ (w=0.))	13.3
Alpha	44.0
D_l (cm^2/day)	0.632
D_g (cm^2/day)	5790.
MW	182.14
$t_{1/2}$ (days)	30.

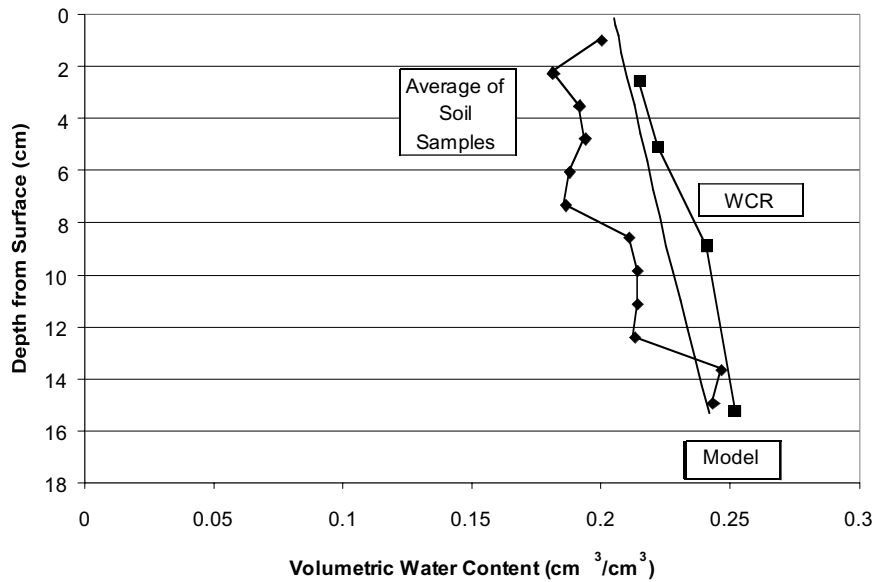


Figure 8. Soil water content data-model comparisons

5. DATA-MODEL COMPARISONS

Surface Vapor Flux

The surface flux of DNT was measured in the experiment using SPME fibers. Based on the data discussed earlier, a value of the low range liquid-solid sorption coefficient, K_d , of 1.5 mL/g was selected for the initial simulations to compare to the data. As shown in Figure 9, while the surface flux as a function of time has the right shape, the values are an order of magnitude or more below the experimental data.

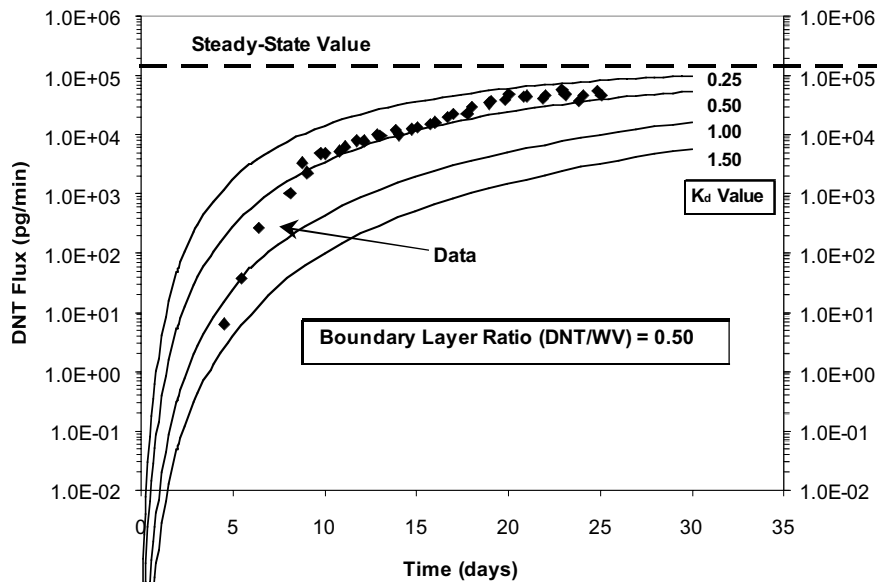


Figure 9. Data Model Comparisons - Effect of K_d Value

The sensitivity of the K_d factor is readily apparent in Figure 9. The data model comparisons can be improved by incorporating a Freundlich soil water partitioning isotherm into T2TNT (planned). Another factor causing the discrepancy in values obtained from measured K_d versus data-model comparisons is in how the measured K_d is obtained. The low soil:water ratio in the batch equilibration K_d method allows for near complete contact of the soil particle surface to the DNT in the water and allows for migration into secondary porosity of soil minerals. In a soil column test this is not the case. Some proportion of soil surface area is not in contact with the water (and DNT) due to partial saturation and surface to surface contact of soil particles. Because of the uncertainty in the value of K_d in the soil column test it was decided to vary the value of K_d until a reasonable match to the data was found as given in Figure 9. The final value of K_d that matches the data reasonably well is ~ 0.5 mL/g. The low concentration batch equilibrium linear K_d value is about a factor of 3 to 5 greater than the best match from this column test. Data from a headspace vapor solid partitioning test also indicates a K_d value of ~ 0.4 mL/g (Phelan and Barnett, 2000), further supporting the lower value indicated in this test.

As discussed by Webb and Phelan (2000), the chemical to water vapor boundary layer ratio is also uncertain. The effect of this variable is shown in Figure 10. For a large variation in the ratio (0.50 to 0.01), the effect on the surface flux is minimal. The final values of K_d and the boundary layer ratio derived from the surface flux data are 0.50 mL/g and 0.25, respectively.

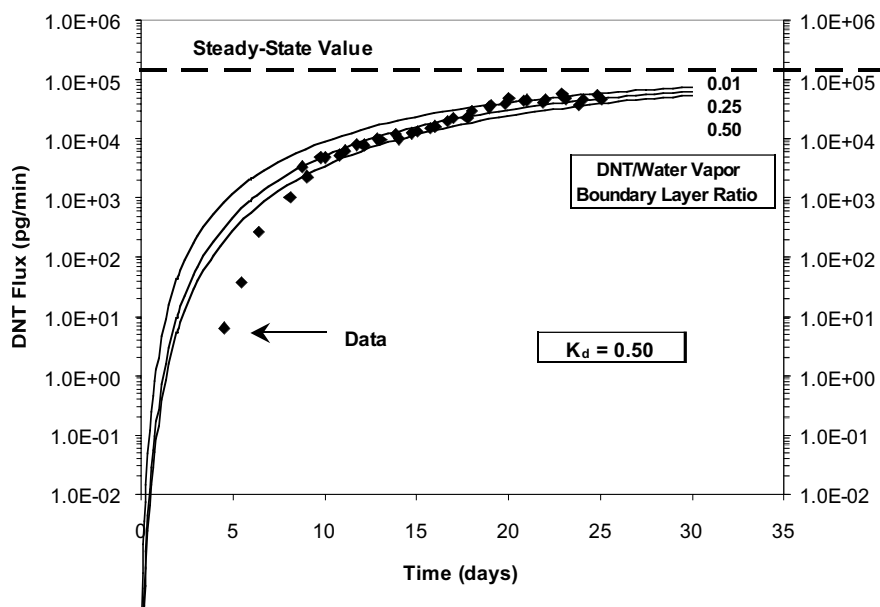


Figure 10. Data Model Comparisons - Effect of Boundary Layer Ratio

Using these values, a comparison was made of the measured and predicted DNT concentrations in the soil. Predictions for the centerline and first radius borehole are compared to the data as a function of depth in Figure 11. The centerline predicted concentrations show reasonable agreement with the data. The shapes of the curves are very similar, while the predicted concentrations are a factor of 2-3 high. A number of factors could contribute to this difference. First, the effective value of K_d effectively decreases with increasing concentration (Table 2). Therefore, the sorbed mass will be over predicted at higher concentrations, such as near the centerline, if a linear K_d value is used. This problem will be addressed when the Freundlich isotherm is incorporated into T2TNT. Second, the concentration decreases significantly with increasing radius. If the location of the injection is not exactly at the center of the column, or if the center soil borehole location is a little off center, the concentrations would be lower. The main factor is probably the Freundlich isotherm. Nevertheless, the data-model comparison for the centerline concentrations is reasonable.

The comparisons for the first radius are very good. The predicted concentrations essentially go through the middle of the four data curves as given in Figure 11. The variation with depth is very good at depths down to 7 cm. Note that the injection location is 3.5 cm and the concentrations above this point decrease slightly due to dispersion. Below the injection point the concentrations decline rapidly as this is against the direction of water flow. Below about 7 cm, the data and model predictions diverge. The method detection limit of DNT in this soil is about 10 ng/g. Many of the values shown below 7 cm are at or below this threshold and may be artifacts. In addition, the soil samples were collected by repeated insertion of the sampling tube. If contaminated soil particles from upper horizons fell to the bottom and were sampled, trace levels may be indicated but were not representative of that horizon.

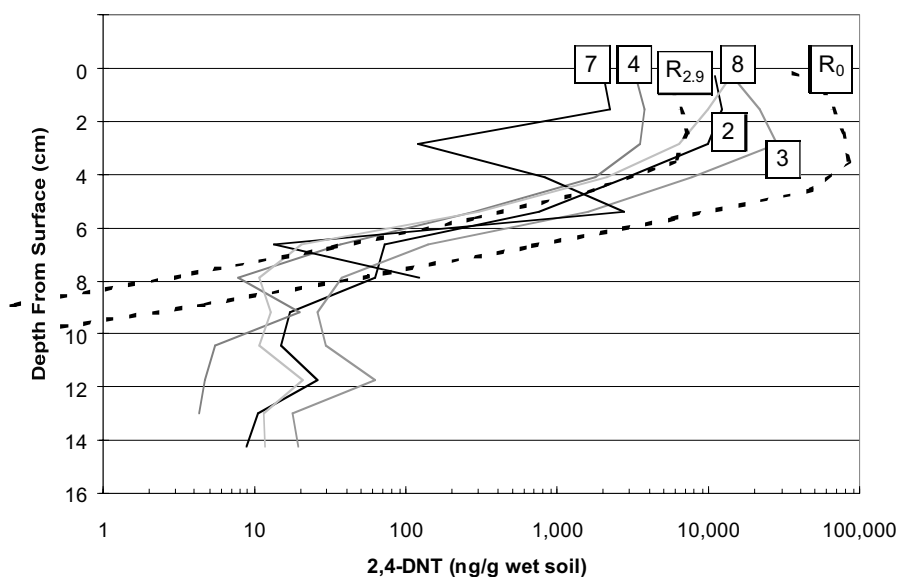


Figure 11. Soil Profile DNT Data Model Comparisons

6. SUMMARY AND CONCLUSIONS

An experiment was designed and completed to evaluate the dynamic transport of 2,4-DNT in a soil column for comparison to simulation model results. The test apparatus was designed to induce an upward flow of water that is valuable in movement of DNT to the soil surface. Surface flux measurements were completed with solid phase microextraction devices that eliminated the need for complex sample preparation procedures. Careful control and measurement of the water transport also allowed data model comparisons of water movement in the soil. Computational simulation was performed using the T2TNT code and showed excellent comparison to the surface flux and soil concentration data. Under these well controlled conditions, the T2TNT code very closely simulated the chemical transport of DNT in near surface soils. Experiments are currently in progress where, after establishing a steady state flux, the soil will be dried by passing dry air through the plenum and then rewetted with humid air. This will evaluate enhanced vapor solid sorption at low soil water contents in a dynamic system.

7. ACKNOWLEDGEMENTS

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