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Transport of Nitrotriazolone (NTO) in Soil Lysimeters

Washington Braida¹, Mahmoud Wazne¹, Adebayo Ogundipe², Gulsah Sen Tuna¹, Julius Pavlov¹, Agamemnon Koutsospyros³, and Vanessa Balok⁴

¹ Stevens Institute of Technology, Center for Environmental Systems/Civil Environmental and Ocean Engineering Department, Castle Point on Hudson, Hoboken, NJ, 07030, USA, ²James Madison University, School of Engineering, Harrisonburg, VA 22807, USA, ³Department of Mechanical, Civil and Environmental Engineering, University of New Haven, West Haven, CT, 06516, USA, ⁴ US Army Environmental and Demilitarization Technology Division, RDECOM/ARDEC, Picatinny Arsenal, NJ, 07806, USA.

ABSTRACT

NTO is a high aquatic solubility constituent of new insensitive munitions (IM) formulations. This study addresses transport of NTO in the vadose zone in pure and IM formulations in a variety of soils representative of testing installations across the USA. NTO is highly mobile in all the soils tested and advective water transport linked to the rainfall pattern appears to be the controlling mechanism on NTO mobility. The NTO dissolution rate is hindered when NTO is part of IM formulations containing low aqueous solubility components. The presence of low solubility products appears to force NTO to diffuse through the organic matrix before reaching the aqueous phase. Despite its ionic character in solution, substantial amounts of NTO are taken up by ryegrass. This likely is the result of the high concentration of NTO in the aqueous phase and the high water uptake of ryegrass.

Keywords: NTO, lysimeters, vadose zone, transport, dissolution, uptake

1.0 INTRODUCTION

The U.S. Defense establishment is developing and testing a variety of candidate explosive compounds and formulations to provide insensitive munitions (IM) to the warfighter. A promising candidate compound for IM formulations is 3-nitro-1,2,4-triazol-5-one (NTO) (CAS #932-64-9). NTO formulations are already in production, and fielded munitions are expected soon (Fung et al. 2009). NTO was first reported in 1905 (Manchot and Noll, 1905), with further studies into its chemistry being undertaken at various points (Chipen et al., 1966, Katritzky and Ogretir, 1982). NTO performs comparably well to RDX, though it is much less sensitive to shock. This property makes it an attractive candidate for military munitions. The U.S. Army is currently developing and manufacturing insensitive munitions formulations based on NTO with full scale production planned for the near future (Fung et al. 2009). This paper addresses the leaching of NTO, given a defined source of residual energetic material (pure NTO and IM formulation), in the vadose zone upon interaction with the environment (i.e., simulated rain, soil, plants).

2.0 EXPERIMENTAL METHODS

2.1 Selection of munitions items and soils

This study used two forms of NTO: a) NTO (pure compound) and b) OSX (an explosive formulation containing NTO, HMX, DNAN). Furthermore four different types of soils were chosen for the experiments; Site 1, Site 2, Site 3, and Site 4 representing typical conditions from various ranges around the continental USA.

2.2 Experimental approach

The experimental setup consisted of ten lysimeter columns (Figure 1 and Figure 2) allowing use of the two target formulations and two soils at a time. Experiments were performed using replicate soil columns for each product tested (NTO and OSX) and one control column per soil type. Four soils were tested, referred to as Site 1-4 (ST1-ST4). The lysimeters were constructed using 20.3 cm internal diameter white schedule 40 PVC pipe, 40-50 cm long, are sealed at the bottom with a schedule 40 PVC white end cap. A small drainage hole was drilled on the bottom of each column for sample collection. A typical layer profile of the lysimeters is shown in Figure 2. Four ceramic Rhizon mini-lysimeters (5 cm in length, 2 mm in diameter) placed along soil profile (2, 9, 16 and 23 cm below the surface) were used to collect water samples during the transient stage of the study. Soils were collected from various sites representative of the typical range of soils across the continental USA. The average mass of soils added to the lysimeters was; 6.7 kg for ST1, 10.6 kg for ST2, 8.16 kg ST3 and 7.8 kg ST4, respectively. Soil characterization results are presented in Table 1. (i.e., specific gravity, percent fines, unified soil classification, lost of ignition, water content, cation exchange capacity and pH).



Figure 1. Ten lysimeter set up.

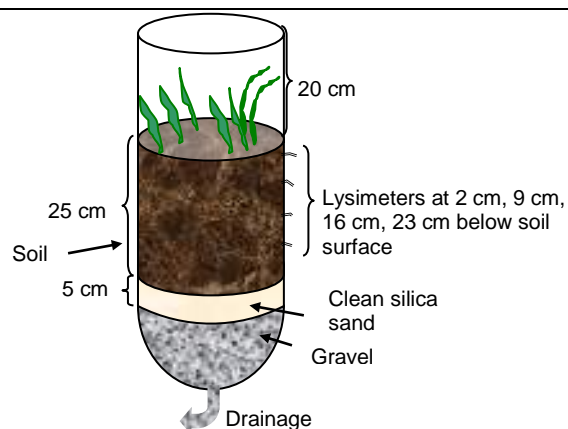


Figure 2. Lysimeter schematic.

Once the soils were in place, each of the lysimeter columns were watered with 250 mL of tap water daily for a week to allow for settling and equilibration. Ryegrass seeds (200-250) were planted into the soils in each column and watered with approximately 250 mL of tap water until germination. After ryegrass plants were fully developed, the MCs (NTO, OSX) were placed on top of the soil layer at the center of the column (NTO: 1 g for ST1, 1.03 g for ST2, and 0.76 g for ST3 and ST4; OSX: 0.76 g for all soils).

Table 1. Selected soil properties

<i>Property</i>	<i>ST1</i>	<i>ST2</i>	<i>ST3</i>	<i>ST4</i>
Specific gravity	2.57	2.60	2.62	2.62
Percent fines	0.54	0.44	0.66	5.71
Percent sand	58.42	44.46	98.64	93.98
Unified soil classification	SW	GW	SP	SP-SM
Percent LOI (lost of ignition)	11.04	7.15	4.84	3.65
Water Content (%)	1.29	1.07	0.29	0.31
Cation exchange capacity (CEC) meq/100g	21	11	4	4
pH	7.55	5.65	3.70	8.31

2.3 Rainfall patterns

A plastic watering can with a spray nozzle was used for watering the soils simulating daily rainfall events. The amount of simulated rainwater used was based on a 30 year average of the National Oceanic and Atmospheric Administration’s National Weather Service’s NOAA Data (<http://www.weather.gov/climate/>). Data for total precipitation (rainfall and snow) averages were available for the vicinities of the areas of interest. Rainfall depth from snowfall data is generally estimated 10 % of snowfall depth. Rainwater pH for each period was obtained from the National Atmospheric Deposition Program website (<http://nadp.sws.uiuc.edu/sites/ntnmap.asp>). The total yearly average rainfall and pH of rainwater for each region are summarized in Table 2. The chemical make up of the synthetic rainwater is given elsewhere (AFRL, 1998). The pH of the synthetic rainwater was adjusted accordingly with nitric acid.

Table 2. Total estimated average rainfall (in) and pH of rainwater.

Soil Sample	Rainfall, in	Snowfall, in	Total, in	pH
ST1	48.28	1.90	48.47	4.54
ST2	45.82	14.70	47.29	4.63
ST3	37.07	8.10	38.88	5.03
ST4	3.80	0.00	3.80	5.21

The columns were watered once a day with the specific amount of synthetic rainwater leaving weekends to normally dry out. The volume of synthetic rainwater required per day to simulate 6 months rainfall over ninety-day period was evaluated (Table 3).

Table 3. Rainwater volume characteristics.

Soil Sample	Yearly total, in	Area, in ²	Yearly volume, in ³	Volume/day, in ³	Volume/day, mL
ST1	48.47	50.27	2436	10.1	166
ST2	47.29	50.27	2377	8.80	144
ST3	38.88	50.27	1954	7.24	119
ST4	3.80	50.27	191	0.71	11.6

Due to the low volumes of leachate collected from ST2 and ST1 lysimeters, the daily amount of simulated rainwater added after day 9 increased to 216 mL and 174 mL for ST1 and ST2, respectively. This resulted in a rainwater addition equivalent to 1.3 years of rainfall for the ST1 soil, 1.1 years for

ST2 soil, and 1.3 years for ST3. Given the low rainfall values for ST4 and to mimic environmental conditions, the research considered watering this soil twice a week rather than on a daily basis. Moreover, the amount of water using in each watering event was increased to 122 mL to assure the collection of leachate from the bottom of the column. Under these conditions the amount of rain added to the ST4 throughout the 6 months of the test roughly represents 7.3 years of rainfall.

The duration of each experimental run was 3 months for all soils except to ST4. Ryegrass plants were harvested at given times depending on their size to monitor the energetic materials uptake. The quantity of leachate collected daily for mass balance analysis and the sample aliquots were analyzed to assess the release of MCs. Energetic materials (NTO and OSX) were analyzed by HPLC following approved analytical method. Daily pH of the effluent from each column was recorded. The samples from the mini lysimeters were taken every 20 days or when a sufficient volume accumulated in the syringe for ST4 soils. At the end of the experiment, all ryegrass plant was removed from the lysimeter and the soil profile was removed in 5 cm incremental layers for a detailed inspection. Soils, plant's shoots and roots were solvent extracted and the concentrations of NTO and OSX components were measured by HPLC.

3.0 RESULTS

The total volume of rainwater added to the amended columns (four columns) during the study was 51.86 L for ST1, 42.78 L for ST2 soils, 42.50 L for ST3, and 22.94 L for ST4. The amounts of leachate collected from the amended lysimeters were 43.49 L from the ST1, 37.7 L from ST2 soils, 32.08 L from ST3 soils, and 9.81 L from ST4 soils, respectively. The volumes of water samples collected by the mini lysimeters were 0.62 L for ST1, 0.45 L for ST2, 0.39 L for ST3 and 0.37 L for ST4, respectively. These values suggest a water unbalance due to accumulation in the soil and/or evapotranspiration of 15%, 11%, 23%, and 56% for ST1, ST2, ST3, and ST4 soils, respectively, over the length of the study. These values are reasonable considering the size of surface area of the top of the lysimeters and that the experiments were run at a fairly constant room temperature ($21 \pm 1^\circ\text{C}$).

The pH of the daily collected leachate from each soil column was measured throughout the duration of the study. Figure 3 shows the variation on the pH of the effluent leachate over time of the lysimeters amended with NTO and OSX and control lysimeters for ST1-ST4 soils. As it can be seen, for the NTO and OSX amended lysimeters, the pH values did not deviate significantly from the pH of the control non-amended columns for ST1 soil. ST2 soils showed a bigger variation between and control lysimeters compared to ST1 soils. For the NTO amended columns the pH values of the leachates from the first day of the experiment were 6.34 and 5.73 for ST1 and ST2 soils, while the final pH values were 7.97 for ST1 and 5.55 for ST2 columns respectively. The pH values of the leachates from the first day of the experiment were 6.66 and 5.31 for OSX amended ST1 and ST2 soils, while the final pH values were 8.22 and 6.25 for ST1 and ST2 soils, respectively. In all cases (amended columns and controls) the pH of the leachate was higher than the pH of the influent rainwater as a result of the buffering capacity of the soils. This was quite evident for the ST1 soil. The pH of the influent rainwater was 4.63 for ST1 soil columns and 5.03 for ST2 soil columns. For ST2, the control column shows a higher pH of the effluent leachate for both NTO and OSX amended lysimeters. The interaction between the pH of the rainwater and the soil minerals (i.e., buffer capacity) determines the final pH of the effluent. There were not significant differences between the pH of the control non-amended columns and the pH of the leachate from ST4 soil during the experiment. The pH of the leachate from the ST4 lysimeters is similar to the pH of the soil itself which appears to neutralize the

acidity of the simulated rainwater. Conversely, for the ST3 columns the starting pH value of the leachates was higher than the pH of the soil and the rainwater (ranging from 5.5 to 6.6) and gradually descended to a final pH that mimics the pH of the simulated rainwater. Amended columns show a slightly lower pH than the control lysimeter.

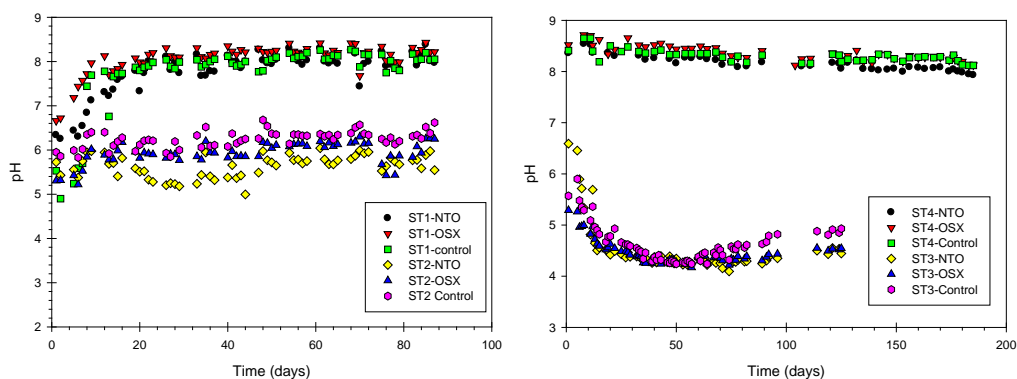
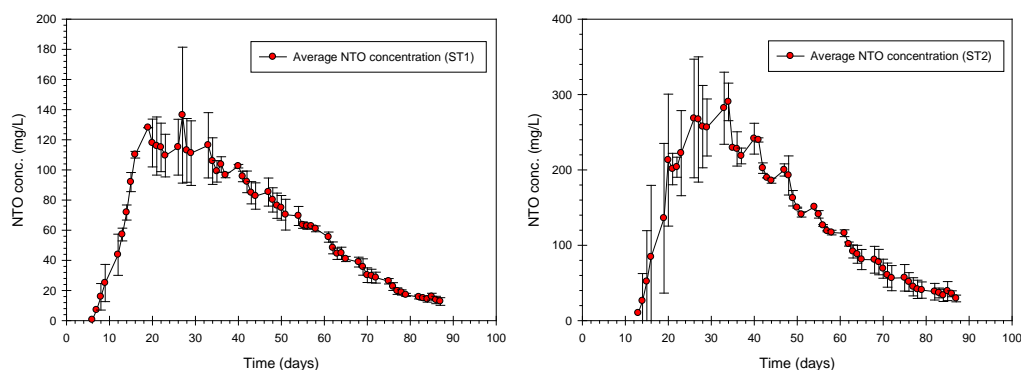


Figure 3. Average (2 lysimeters) pH of the leachate collected at the bottom of the NTO and OSX amended ST1 and ST2 lysimeters (left); ST3 and ST4 lysimeters (right). Soil pH: ST1 7.55, ST2 5.65, ST3 3.70, ST4 8.31. Rainwater pH: ST1 4.64, ST2 5.03, ST3 4.54, ST4 5.21.

Figure 4 shows a typical concentration profile of dissolved NTO in the leachate from the soil lysimeters amended with NTO powder. Figure 5 presents a concentration profile of dissolved NTO from in the leachate collected from the OSX amended lysimeters. In all cases the values presented in the figures are the average from two replicates (i.e., the two soil lysimeters) along with the calculated standard deviation of the experimental measurements. DNAN and HMX were not detected in the leachate from the OSX amended lysimeters.



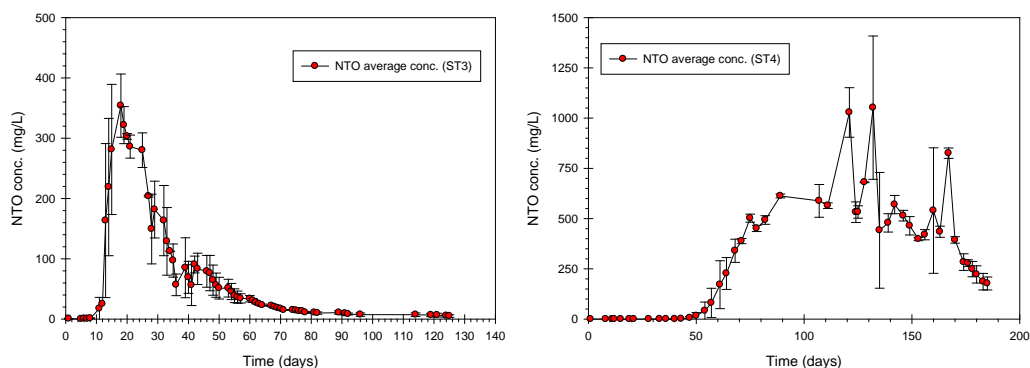


Figure 4. Dissolved NTO concentration (mg/L) in the effluent leachate from the all soil lysimeters amended with pure NTO.

The leaching curves of NTO from NTO-amended lysimeters presented similarities for soil samples ST1, ST2 and ST3 and it is very different for ST4 soil. NTO started to leach out from ST1 lysimeters on day 6 and reached its maximum concentration (136 mg/L) on day 27 (Figure 4, upper left). For ST2 soil, NTO leaching out of the lysimeter started to leach on day 13 and reached its maximum concentration (290 mg/L) on day 34 (Figure 4 upper right). For ST3 soil, NTO was detected at ppb levels in the leachate after one day and it reached its maximum concentration (353 mg/L) after 18 days. ST3 lysimeters were watered for 96 days and watering was resumed for 9 more days starting in day 114 (18 days pause) after the lysimeter set up (Figure 4, bottom left). The objective of this activity was to check for any potential concentration rebounding effect, however, this effect was not observed.

The picture was substantially different for ST4 soils where NTO was not detected in the leachate at the 100 ppb level until day 31 and at the mg/L level until day 39 (Figure 4, bottom right). The maximum concentration was achieved after 132 days (1052 mg/L). NTO leaching curves for ST1, ST2, and ST3 soils present similar shapes and the difference resides in the maximum concentration leached out and in the width of the maximum concentration peak. The three curves show significant tailing; being ST3 soil the one that shows the smallest tailing effect. The smaller rainwater additions used for the ST4 soil resulted in a delayed elution of NTO and a long period of NTO concentrations in the leachate above 500 mg/L.

Figure 5 presents two examples of the leaching of NTO from OSX amended soil lysimeters. As expected and likely due to the retardation effect in the dissolution of NTO resulting from the presence of 47% of other low solubility products (i.e., HMX and DNAN) in the OSX formulation, the leaching curves of NTO from OSX-amended lysimeters are flatter and with peak concentration values smaller than the NTO-amended counterparts. The concentration profiles for NTO are similar in values and shapes for ST1 and ST2 soils. The NTO concentration-time curve shows a Gaussian shape with considerable tailing for ST3. Maximum dissolved concentration of NTO leached out from the OSX amended lysimeters was 33 mg/L, 15 mg/L, 43 mg/L and 553 mg/L for ST2, ST1, ST3, and ST4 soil columns, respectively. The trend of the results is in agreement with the previous results for NTO amended lysimeters. The large difference in the duration of the concentration plateau suggest the interplay of the different factors controlling the leachability of NTO from the columns: i) dissolution kinetics (slower for formulations containing less soluble products that hindered NTO dissolution), ii) rainfall patterns (largest amount of rainfall result in higher rates of transport and iii) soil

characteristics; ST4 soil shows less adsorption affinity for NTO resulting in larger values of the peak concentration.

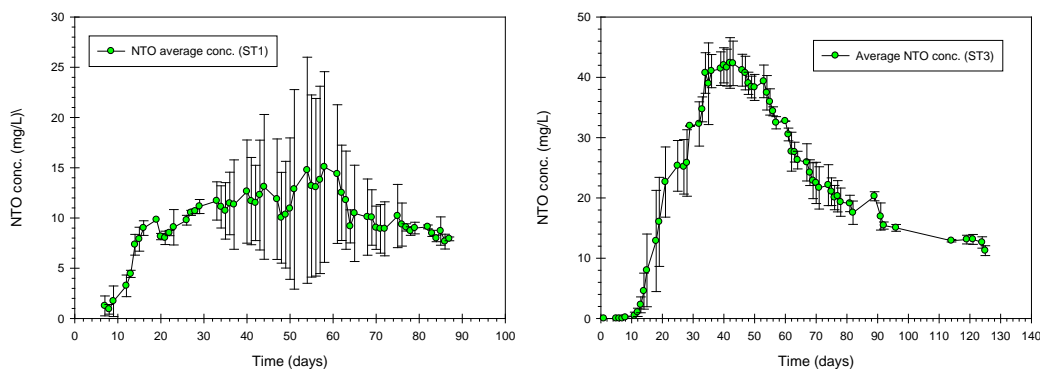


Figure 5. Dissolved NTO concentration (mg/L) in the effluent leachate from the ST1 and ST3 soil lysimeters amended with OSX.

Grass biomass from the columns was harvested twice during the duration of the experiment, after 42 and 87 days for ST1 and ST3 and twice during the test for ST4 and ST3 soils. Grass samples were dried and extracted with an acetonitrile:DI water mixture. The energetic materials of concern, namely NTO and OSX components, were measured in the extract and the uptake of energetic materials was computed for each soil.

After the test was terminated, the soil columns were disassembled and soil samples were taken using a scoop. Soil profiles were extracted at every 5 cm to derive a better transport profile of the munitions through the column (2.5 cm for the first layer). NTO and OSX pieces were placed in the center of the soil columns, and before disassembly of the column, this area was checked for residuals of solid energetic materials. Residuals of OSX were found in the ST3 and ST4 columns while they were not observed in ST1 and ST2 soils but this may be due to the smaller size of OSX particles used in the test. The residuals were carefully separated from the soil and weighted. The amount of OSX residuals recovered were 369 and 469 mg for the two ST4 columns and 460 and 221 mg for the two ST3 columns. Aliquots were dissolved in acetonitrile and analyzed by HPLC to determine the NTO:DNAN:HMX ratio and check for preferential dissolution. After the aliquots of soil were taken at specific depths, they were homogenized and extracted with an acetonitrile:DI water mixture. Finally the energetic materials (NTO and OSX components) were analyzed by HPLC. Results from soil and plant determinations are presented in Table 4. The soil layers in which energetic components were not detected are not included in the Table.

NTO was not detected across the soil profile for ST1 and ST2 lysimeters amended with pure NTO and this agrees with the mass balance calculations that show that all NTO leached out from the columns. For ST3 80+% of the NTO was recovered in the bottom leachate from the lysimeter and the adsorbed concentration in the first 2.5 cm accounts for roughly another 1%. For the ST4 lysimeters the total amount of NTO recovered in the bottom leachate accounts for 90% of the NTO added and a concentration profile of NTO throughout the column can be appreciated. In the case of OSX amended columns, for ST4 and ST3 roughly 50% of the NTO added was recovered in the bottom leachate from the lysimeters, while those percentages drop to 40% for ST2 and 25% for ST1. This may be related to

the partial dissolution of the OSX particles (due to the presence of less soluble DNAN and HMX that hindered the dissolution of NTO). Residuals of OSX were found after the experiment in the ST4 and ST3 lysimeters as it was described previously but they were not observed in the other two soils. HMX and DNAN were observed in the top 2.5 cm of the OSX lysimeters and the largest concentrations correspond to ST4 soil. Grass did not grow well in the amended ST4 and ST3 soils. In the NTO- and OSX- amended ST1 and ST2 lysimeters substantial uptake of NTO was observed in the grass plants. HMX and DNAN were not detected in any of the biomass samples. The analysis of the OSX particles found in the ST4 and ST3 columns at the end of the experiment reveals a DNAN:HMX ratio of 3.15 and 3 for ST4 and ST3 soil respectively. The original ratio in the formulation is 3, which suggest that for ST4 some preferential HMX dissolution occurred. Similarly, the NTO:HMX ratio for the particles recovered from the ST4 columns was 1.5 and 1.6 for the ST3 recovered particles. Both of those figures are much smaller than the 4.4:1 ratio in the original formulation which confirms the experimental leaching results and suggest the preferential dissolution of NTO from the OSX formulation. The particles found still are not able to account for all the NTO added to the column; thus at this stage abiotic or biotic mediated processes that can transform NTO cannot be ruled out. However, addressing in detail those processes was beyond the scope of this project.

Table 4. Energetic concentration in biomass and along the soil profile after the experiment. NA-Non Applicable; ND-Non Detected.

Lysimeter description	Sample Description	NTO (mg/kg)	DNAN (mg/kg)	HMX (mg/kg)
ST1-OSX	Top layer	ND	4.1	12
ST1-OSX	1-2 cm	ND	2.7	7.5
ST2-OSX	Top layer	ND	3.7	12
ST2-OSX	1-2 cm	ND	0.8	9.4
ST1-NTO	Grass-41 days	78560	NA	NA
ST1-OSX	Grass-41 days	18750	ND	ND
ST1-control	Grass-41 and 87 days	ND	ND	ND
ST2 Control	Grass-41 and 87 days	ND	ND	ND
ST2-NTO	Grass-41 days	22790	NA	NA
ST2-OSX	Grass-41 days	7149	ND	ND
ST3-NTO	Soil 0-2.5 cm	2.2	ND	ND
ST3-OSX	Soil 0-2.5 cm	36.6	30.5	4.8
ST4-NTO	0-2.5 cm	1.2	NA	NA
	20-25 cm	1.1	NA	NA
	25-30 cm	6.9	NA	NA
ST4-OSX	0-2.5 cm	33.7	61	19
	2.5-5 cm	4	ND	ND
	5-10 cm	4	ND	ND
	10-15 cm	4.8	ND	ND
	15-20 cm	5.4	ND	ND
	20-25 cm	7.5	ND	ND
	25-30 cm	7	ND	ND
ST3 Control	Grass-12/10	ND	ND	ND
ST4 Control	Grass-12/10	ND	ND	ND
ST4-NTO	Grass-02/11	19.8	NA	NA
ST4-NTO	Grass-02/11	110	NA	NA
ST4-NTO	Grass-roots-02/11	27.7	NA	NA
ST3-OSX	Grass-12/10	111.7	ND	ND

Samples were also collected from each column by mini-lysimeters using a vacuum provided by a plastic syringe. Four set of samples from different depths (2 cm, 9 cm, 16 cm and 23 cm) were collected from each column for every 15-30 days during the experiment for each soil type. Results presented in Table 5 are the average of two replicates for both soils. NTO was the only component detected in the mini-lysimeters' samples. NTO concentration (mg/L) for the four tested soils present erratic trends with time, depth and also varied depending on the source of NTO (i.e., pure product or OSX). For early experiment times a decreasing trend with depth appears to be the rule but after 30 days the interplay among different factors such as dissolution rate, plant uptake, sorption desorption processes and advective water flow resulted in the erratic profiles observed.

Table 5. Dissolved NTO averaged concentrations from NTO amended columns leached through mini lysimeters in ST1, ST2, ST3, and ST4 soils.

	Depth (cm)	Time period (days)					
Average NTO conc. (mg/L)		1/15	16/34	35 /51	52/69	70/ 87	
ST1 -NTO	2	286	512	287	371	42	
	9	71	80	57	36	18	
	16	82	173	164	103	25	
	23	58	20	68	36	20	
ST2-NTO	2	407	205	88	366	11	
	9	503	238	89	456	66	
	16	21	138	135	44	15	
	23	29	318	286	109	51	
		Collection Date					
		10/25/10	12/02/10	12/22/10	01/20/11	02/04/11	03/09/11
ST3-NTO	2	56	12	1	1038		
	9	161	33	6	19		
	16	198	39	4	287		
	23	70	38	25	7		
ST4-NTO	2	2519	710	188	3005	11	2
	9	500	125	36	21	10	2.5
	16	521	1252	486	0.6	157	35.7
	23	32	607	464	1	307	50

4.0 CONCLUSIONS

This study addressed the vadose zone transport of pure NTO and OSX in four different soils representative of testing installations across the country. The experimental results suggest that NTO is highly mobile in all the soils tested. NTO dissolution rate is hindered when NTO is part of the OSX formulation as a result of the presence of low solubility products forcing the NTO to diffuse through the organic matrix before reaching the aqueous phase. HMX and DNAN were not detected in the column effluents suggesting low mobility of those compounds, which may be due to low solubility and/or uptake by biomass and soil near the source point.

Despite its ionic character in solution, substantial amounts of NTO were taken up by ryegrass. This likely is the result of the high concentration of NTO in the aqueous phase and the high water uptake of ryegrass. Ryegrass plants did not develop well in ST3 and ST4 lysimeters amended with NTO and OSX. Control columns showed a strong growth of healthy plants.

Rainfall patterns appear to have a strong influence in the transport of the more soluble product tested; NTO. Mass balance calculations suggest that some NTO may be abiotically or biotically transformed.

The preferential dissolution of NTO from OSX formulations and the quasi constant DNAN:HMX ratio in the OSX formulation before and after the test makes OSX a nice candidate to test the drop impingement model to assess the dissolution of its components. This is a crucial factor in predicting the fate and mobility of IM components in soil systems.

Moreover, the high volume of data collected in this project may serve as a basis for the development of transport models for IM components in the vadose zone. Research in this direction is highly recommended.

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