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Sorption of Nitramines to Soil
Final Report of preliminary assessment study, January 2012

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Executive Summary

This nitramine sorption test shows that the bulk of monoethanolnitramine and dimethynitramine are sorbed to the organic rich soils, but that a significant amount remains in the aqueous phase. The test also shows clearly that the studied nitramines have a greater sorption to organic rich soils than to mineral soils. This agrees with the general concept that sorption of organic pollutants to soil is greatly influenced by the organic content in the soil.

The average distribution coefficient at equilibrium, K_d, for organic soils was found to be 16 and 47 for monoethanolnitramine and dimethynitramine, respectively. For the mineral soils, K_d was found to be 2.0 and 3 for monoethanolnitramine and dimethynitramine, respectively. The organic carbon partition coefficient, log K_{oc}, was calculated to be approx. 1.6 and 2.0 for monoethanolnitramine and dimethynitramine, respectively.

Compared with other organic pollutants of environmental concern the studied nitramines are nevertheless to be considered as highly water-soluble and exhibit a comparably low sorption to soil. Based on the relatively low log K_{oc} values and an apparent strong persistence in the environment we can deduce that a significant fraction of the nitramines will likely eventually pass through the terrestrial environment and be leached out into surface waters. Further research is therefore required in order to assess the fate and transport of the nitramines in the environment.

1 Objective and scope of work

The objectives were:

- To determine the sorption of the nitramine to the soil with physiochemical properties characteristic of soils found in the vicinity of the Mongstad area.
- To conduct a screening laboratory study assessing partitioning coefficients or adsorption isotherms on eight soil samples, with focus on organic horizons, from the region around TCM, to assess transport and mobility of nitramines with water through the terrestrial environment.

This task concerns the partitioning of nitramines between water and soil after the deposition to soil. The following activities are included:

- Assessment of partitioning coefficient of nitramines to soils
- Estimation of mobility and theoretical likelihood of nitramines to reach fresh water

2 Health, Safety and Environment

The project involved handling of hazardous chemicals and was conducted in compliance with Health, Safety and Environment (HSE) statutes and regulations. Following the Standard Operational Procedure (SOP) of the University of Oslo (UiO) laboratory all compounds under study, including possible degradation products, are treated as potentially carcinogenic unless other information is available. A Safe Job Analysis has been conducted.
The Department of Chemistry at UiO is a Governmental institution with implemented rules and regulations following our HSE manual[a] in accordance to Norwegian Law. UiO is not required to produce HSE data sheets for compounds synthesized for research purpose.

Work at the National Environment Research Institute (NERI) in Denmark, were conducted in accordance with the SOP operative at NERI, which meets the required regulations according to Danish Law.

3 Sorption theory

The distribution coefficient, K_d, represents the ratio between the concentration of the nitramines sorbed to the soil (C_S in mg kg^{-1}) and the concentration of the nitramines in the solution (C_w in mg L^{-1})(Eqn. 1). The nitramine concentration in the soil phase (C_S) is indirectly calculated as the difference in the concentration of the nitramine in the "blank sample" (nitramine solution without soil) and the concentration in the soil-water solution (C_w).

$$K_d = \frac{C_S\ (\frac{mg}{kg})}{C_w\ (\frac{mg}{L})} \qquad (1)$$

The concentration of a specific sorbed organic species in the soil (C_S) can be empirically modeled as the sum of products of the fraction of organic matter (f_{OM}) and mineral matter (f_{MM}) in the soils with the concentration of the organic species in the organic (C_{OM}) and inorganic phase (C_{MM}), respectively (Eqn. 2).

$$C_S = f_{OM} \cdot C_{OM} + f_{MM} \cdot C_{MM} \qquad (2)$$

Sorbtion to mineral matter is mainly a case of adsorption to the surface of the particles by electrostatic, polar and ionic interactions, while sorption to organic matter is more a case of absorption, which relates to the non-polarity of the organic species. The complexity of these interactions goes beyond the simplicity in Eqn. 2, which does not deal with the role of particle sizes nor the fact that inorganic particles are usually coated with organic matter.
Usually the sorbtion of organic species (especially in their non-charged form) are to a far greater extent influenced by the soil organic content than by the mineral

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[a] See: http://www.mn.uio.no/kjemi/english/about/hse/hse-manual/HSE-manual-department-of-chemistry-english-2011.pdf

content (vanLoon and Duffy, 2005b). This appears also to be the case with the nitramines. For this reason Eqn. 2 is often simplified further to Eqn. 3

$$C_S = f_{OM} \cdot C_{OM} \tag{3}$$

While the K_d represents the distribution coefficient of a specific organic compound to a specific soil type, it can be difficult to relate this value to a variety of soils. As the sorption to soil for organic species is mainly governed by the organic matter content in the soil, the distribution coefficient, K_d, can be derived from Eqn. 3 giving Eqn. 4 or 5:

$$K_d = f_{OM} \cdot K_{OM} \tag{4}$$

Where K_{OM} denote the partitioning coefficient between organic matter and water. In the literature the amount of organic matter is frequently referred to as the organic carbon content. The difference between the amount of organic carbon and organic matter is approximately a factor of 2 (vanLoon and Duffy, 2005a, Veres, 2002, Craft et al., 1991). Henceforth there are numerous papers that compare the partitioning coefficient between organic carbon and water (K_{OC}) for different organic contaminants.

$$K_d = f_{OC} \cdot K_{OC} \tag{5}$$

4 Material and methods

4.1 Sampling locations

Eight soil samples from 6 different locations, within a 30km radius from Mongstad (see Figure 1), were collected for the sorption test: 5 organic forest floor soils, 1 bog soil (organic soil) and 2 mineral soils (Table 1). The locations were selected based on soil cover, mineral soil type, and forest type and growth density (Appendix A). The aim was to collect a set of soil samples that captured the main variation in soil physiochemical characteristics in the regions non-agricultural land.

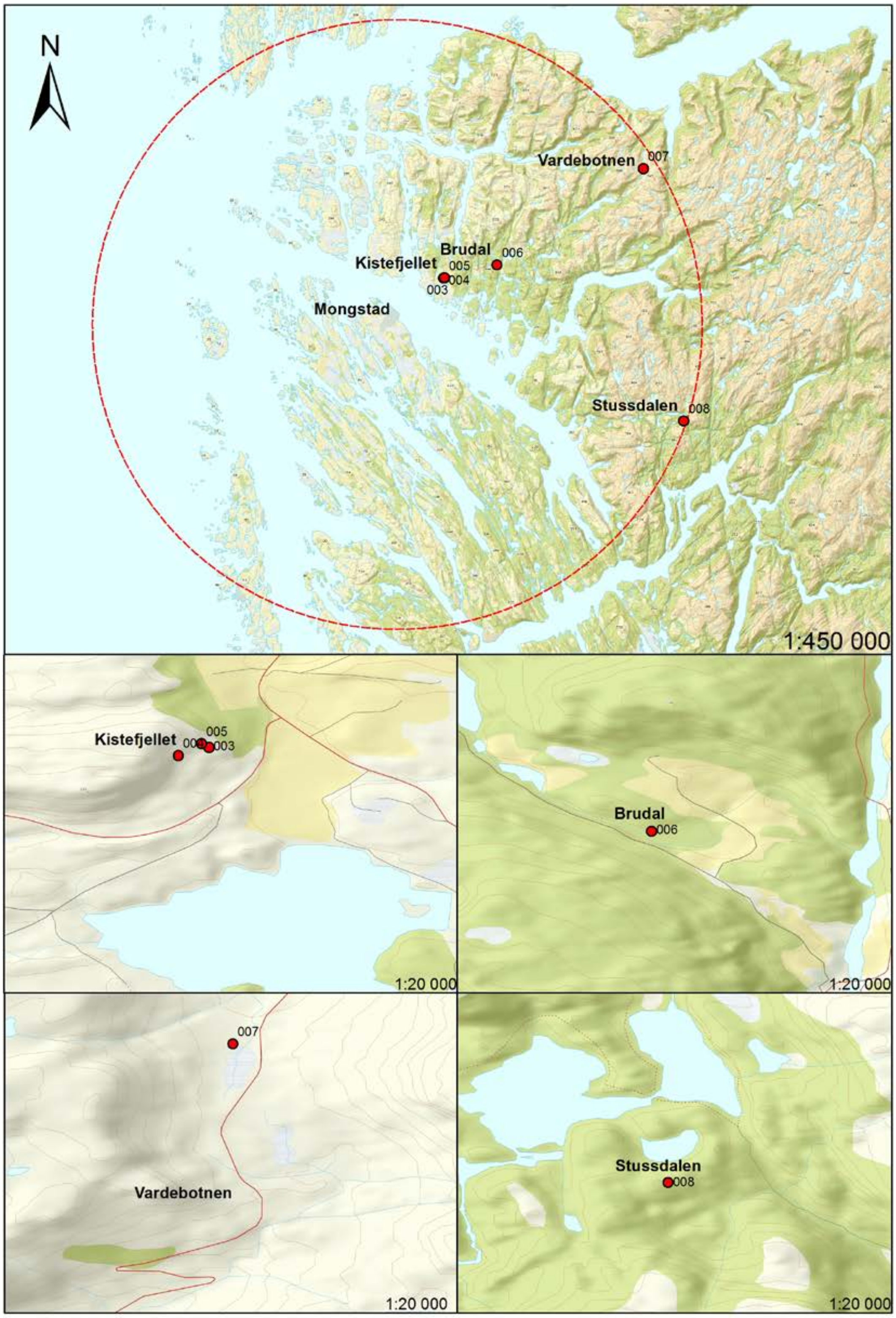

Figure 1: Top map is an overview of the 6 soil sampling sites selected within the 30km radius from Mongstad (indicated by the red dotted circular line). Bottom four maps reveal detailed sampling site location.

Table 1: Soil sampling site location and characteristics.

Sampling point ID	Location	Soil cover	Mineral soil type	Forest type	Soil quality For tree growth in forest
003	Kistefjellet	Thin	Marine and fjord	Deciduous	High
004	Kistefjellet	Thin	Marine and fjord	Deciduous	High
005	Kistefjellet	Thin	Marine and fjord	No trees	Impediment
006	Brudal	Thick	Glaciofluvial	Coniferous	High
007	Varde-botnen	Thick organic	Moraine	No trees (bog)	Impediment
008	Stussdalen	Thick	Moraine	Coniferous	Low

Table 2: Organic matter content measured as Loss on Ignition, and Particle size distribution of mineral soils used in the experiments. Samples are sorted according to decreasing organic content.

Soil Sample	Organic matter (%)	Soil horizon	Particle size distribution (%)		Soil Texture
007-1	95	O			Organic
003-1	89	O			Organic
008-1	86	O			Organic
006-1	78	O/A			Organic
004-1	76	O/A			Organic
005-1	69	O/A			Organic
006-3	9	B	Sand (0.06-2mm) Silt (0.002-0.06mm) Clay (< 0.002 mm)	49 47 5	Sandy loam
008-3	7	B	Sand (0.06-2mm) Silt (0.002-0.06mm) Clay (< 0.002 mm)	59 31 9	Sandy clay loam

4.2 Experimental

The scheme for sample handling, chemical- and physical characterization and sorption experiments are shown schematically in Figure 3. Soil samples were pretreated by oven-drying the samples at 105°C, followed by 2 mm sieving in accordance with ISO 11464:2006. Sorption tests were conducted for two nitramines seperatly: monoethanolnitramine (MEA-NO_2) and dimethylnitramine (DMA-NO_2), see Figure 2 for molecular structure. The nitramines were attained from Prof. Yngve Stenstrøm at the University of Life Science (UMB).

Sorption experiments were conducted on the 8 soil samples listed in Table 1 in accordance with ISO/TS 21268 Part 1 and 2:2007, with a few required modifications [a,b]. The main difference between Part 1 and 2 is regarding the soil to water ratio used during batch sorption. In Part 1 two liters of the aqueous solution is used per kilo soil; In part 2 ten liters solution is used per kilo soil. The rationale for the different methods is to ensure a liquid suspension of sample in water containing the nitramine, accommodating for the large density difference between mineral soil (high density) and organic soil (low density). Part 1 was therefore applied for mineral soils, and part 2 was employed for organic soils.

The initial concentration for the nitramine solutions were approximately 500 µg L^{-1}. 700 mL of the nitramine solution was added to 350 g mineral soil and 70 g organic soil in 1000 mL borosilicate flasks. The soil-liquid suspensions were shaken at 75 rpm in a circular motion. Temporal sampling of the water phase during the sorption experiments were conducted to observe when sorption equilibrium of the nitramines between the soil and liquid is achieved; 10 mL solution aliquots were collected from the sample slurry at increasing time intervals: 0h (i.e. blank sample), 24h, 48h, 96h and 192h. The 10 mL solution aliquots were centrifuged at 14 000 rpm (23700 RCF) for 30 min. The supernate was transferred to brown glass vial, and filtered using 0.2 µm pore size filters before analysis. Additionally, DMA-NO_2 was extracted from water solution using dichloromethane (DCM, CH_2Cl_2) before analysis. Quantitative analysis of MEA-NO_2 and DMA-NO_2 were performed using HPLC-MS and GC-MS, respectively.

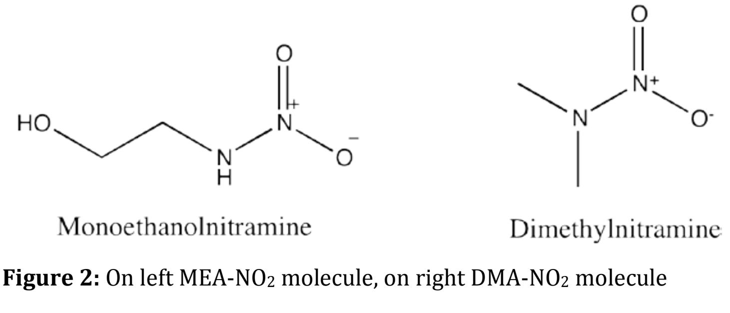

Figure 2: On left MEA-NO_2 molecule, on right DMA-NO_2 molecule

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[a] According to the referenced ISO standard procedure soil is air-dried. However oven-drying at 105°C was employed instead in order to dry the soil faster due to the large amount of soil required for this test and time constraints.

[b] The ISO-standard is setup to be performed within 24 hours (h), however in order to assess whether or not equilibrium is reached within this timeframe the sorption experiment was expanded to include also sampling after 48h, 96h and 192h.

Conductivity, pH and total organic carbon (TOC) concentration were determined in the nitramine solutions before and after the sorption test. pH was measured in samples for all time intervals.

The soil organic matter content, f_{OM}, was determined by method of loss on ignition (LOI) according to Krogstad (1992). The particle size distribution (PSD) for the mineral soil was determined according to ISO11277:2009.

Pre-treatment of soil, LOI and TOC analysis were performed at the Department of Chemistry, University of Oslo. PSD was performed at Eurfins Norsk Matanalyse AS. Sorption test, conductivity, pH measurements and quantitative analysis of MEA-NO_2 and DMA-NO_2 were performed at National Environment Research Institute (NERI), Denmark.

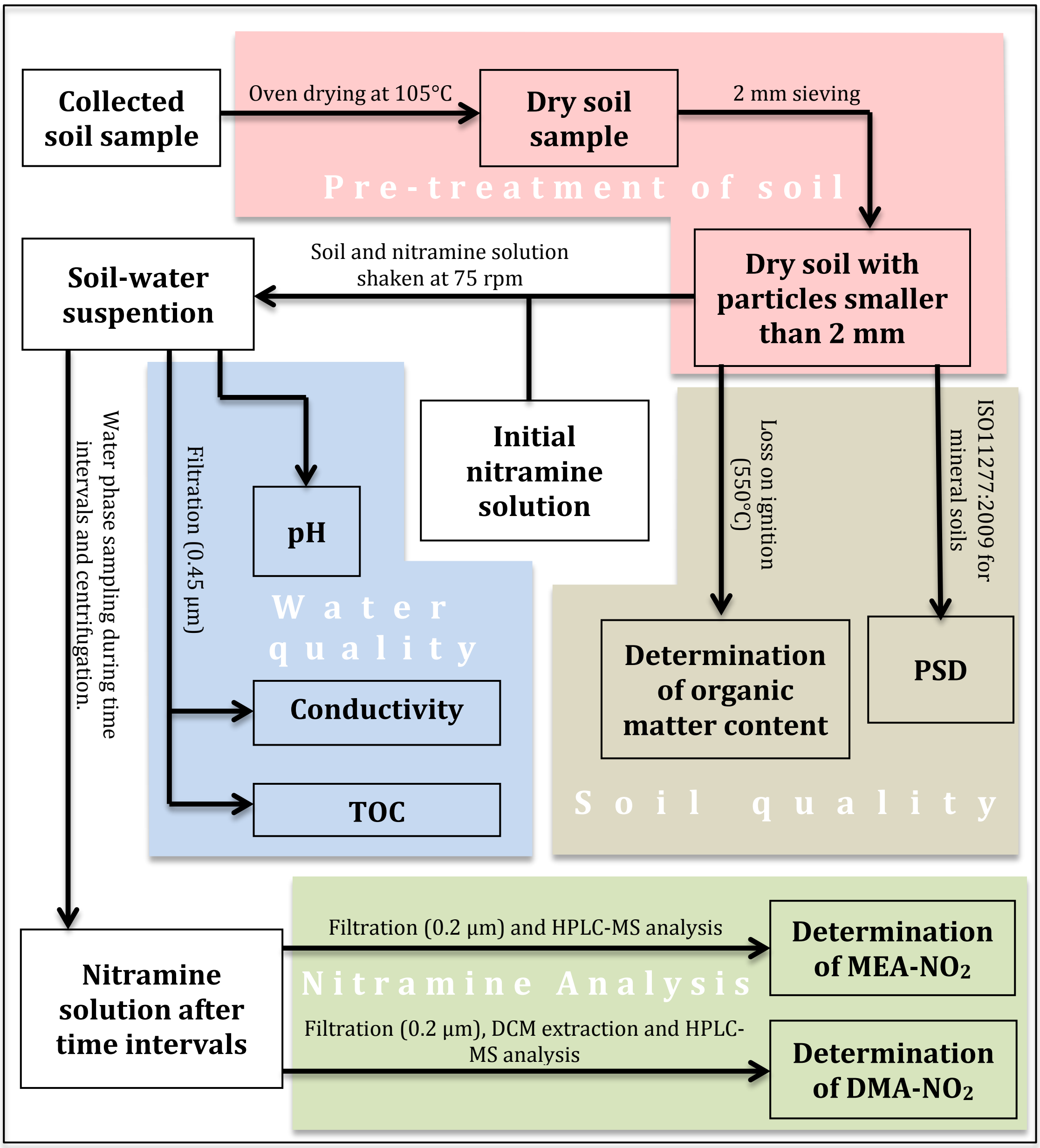

Figure 3: Schematic overview of experimental procedure.

5 Results and discussion

MEA-NO_2 exhibits relatively strong sorption to organic rich soils (O- and A-horizons) compared to mineral soils (B-horizons) (Figure 4).

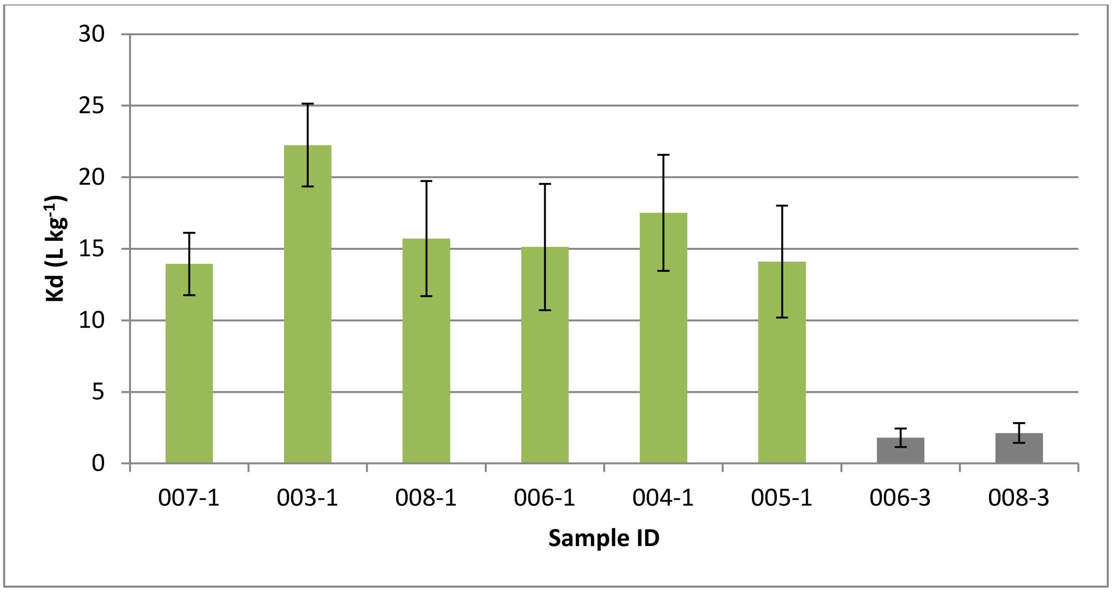

Figure 4: Time averages of the distribution coefficient, $K_d = C_S/C_W$ (L kg^{-1}), for MEA-NO_2 with each soil samples. Organic rich soil samples (O and A horizons) are represented in green, while mineral soils are shown in grey. Samples are sorted according to decreasing organic content. See Table 1 and 2 for sample information.

Sorption of MEA-NO_2 showed no specific trend as a function of time (Figure 5). This is likely due to that sorption equilibrium between MEA-NO_2 in the water-phase and the soil is achieved within the first 24 hours of the test. The observed variation in log Kd is presumed to be mainly due to analytical uncertainty or microbial degradation. Microbial growth was discovered in some of the sample vials after the experiment was completed, though this may not have had any significant effect as the preliminary biodegradation experiment in lake water concludes that the degradation of nitramines appears to be slow [c].

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[c] Part 3.3 of Evaluation of fate of nitramines in soil and freshwater: Biodegradation of nitramines in lake water by Hessen et al.

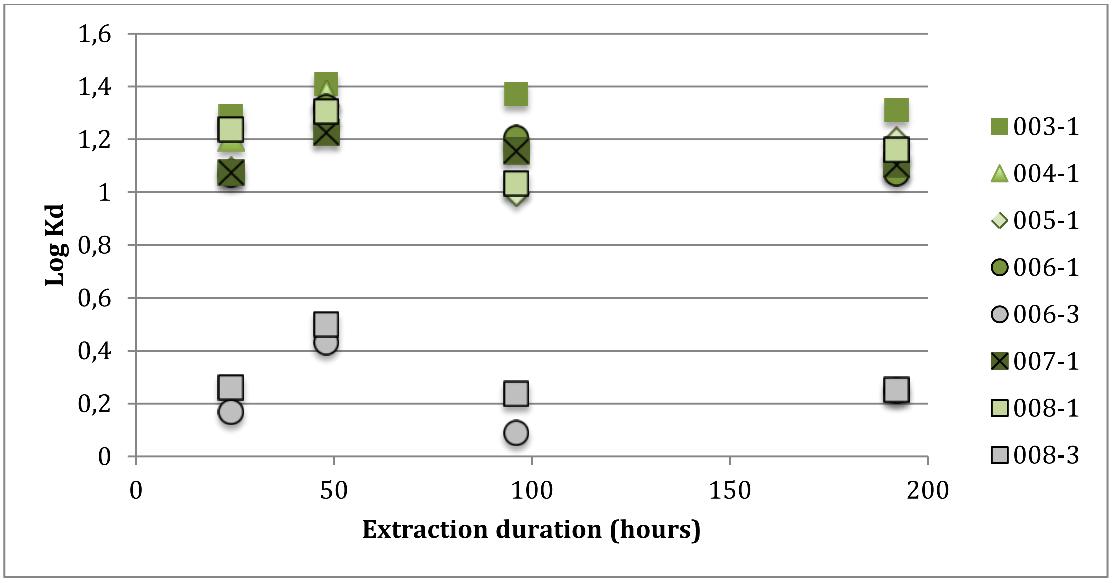

Figure 5: Logarithmical representation of the distribution coefficient, $K_d = C_S/C_W$ (mg kg^{-1} / mg L^{-1}), for MEA-NO_2 with each soil as a function of time. Organic rich soil samples are represented in green, while mineral soils are shown in grey. See Table 1 and 2 for sample information.

DMA-NO_2 exhibit, similar to MEA-NO_2, a significantly larger sorption to organic rich soils than to mineral soils (Figure 6). At equilibrium DMA-NO_2 has stronger sorption to organic rich soil than MEA-NO_2 (compare Figure 4 & 6).

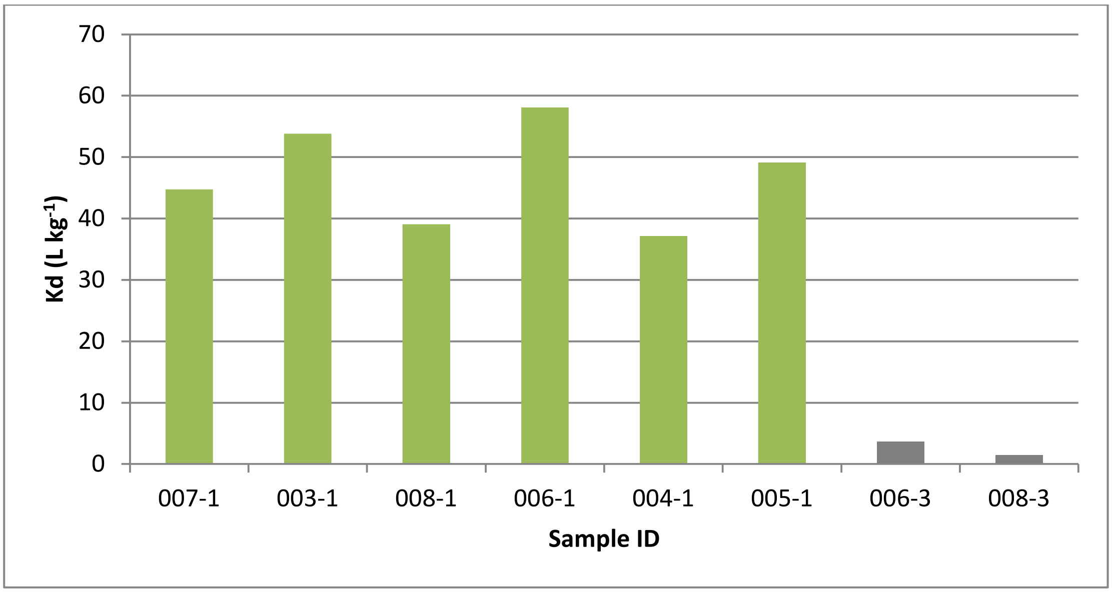

Figure 6: Distribution coefficient, $K_d = C_S/C_W$ (L kg^{-1}) for DMA-NO_2 with each soil samples after 192h. Organic soil samples are represented in green, while mineral soils are shown in grey. Samples are sorted according to decreasing organic content. See Table 1 and 2 for sample information.

DMA-NO_2 appears to have a slower sorption rate than MEA-NO_2, as equilibrium with the organic soil (i.e. LOI>75%) is not reached until after 96 – 192 h (Figure

7). Within the time frame of this test (0 – 192 hours) it is not possible to determine whether the mineral soils have achieved sorption equilibrium with DMA-NO_2 (Figure 7).

Some variation in the log *Kd* values are again noticed. This is the result of a number of uncertainties. Similar to the MEA-NO2 sorption test there are main uncertainties related to the analytical analysis. There is furthermore an additional uncertainty related to preparing the DMA-NO_2 extract solution prior to analysis on GC-MS. Unlike the MEA-NO_2, which can be analyzed directly after filtration, the DMA-NO_2 must be extracted from the water solution using dichloromethane. The extraction is a fairly critical part of the procedure in regards to the recovery of the DMA-NO_2. The average recovery of DMA-NO_2 is reported by the NERI laboratory to be 52%, with an uncertainty of 10. This is greater than the uncertainty of the MEA-NO_2 analysis which is reported to be 5%. Still, negative log *Kd* values, as reported for the mineral soils at 24 and 96h, is not theoretically sound and imply that the accumulated uncertainty in the experiment and quantitative analysis is greater than reported.

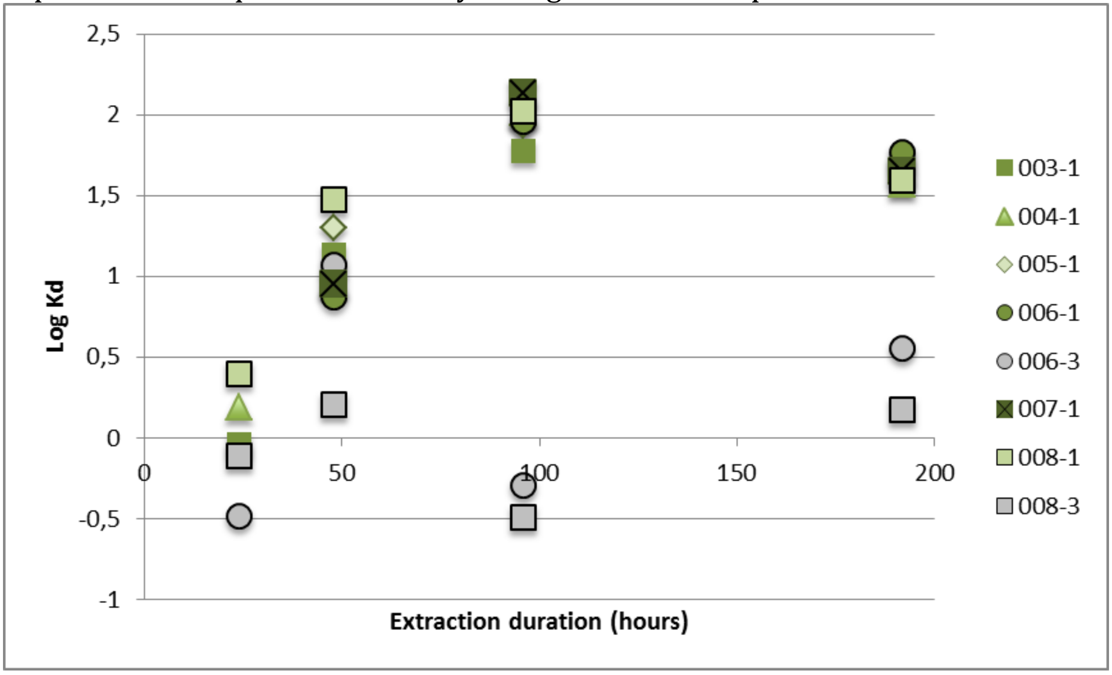

Figure 7: Logarithmical representation of the distribution coefficient, $K_d = C_S/C_W$ (L kg^{-1}), for DMA-NO_2 with each soil as a function of time. Organic soil samples are represented in green, while mineral soils are shown in grey. See Table 1 and 2 for sample information.

The average pH of the soil water suspensions was 3.70±0.09 and 4.07±0.07 for the organic rich and mineral soils, respectively. This implies that all these soils are rather acid due to natural organic acids in thin and poorly weatherable unconsolidated deposits. The low standard deviation indicates that for both the organic rich and mineral soil the pH in the soil/water slurry remained fairly stable throughout the experiment.

The pH may influence the sorption characteristics of the nitramine as they may potentially become protonated and thereby transform from a neutral to charged forms. This would change the polarity of the molecule and thereby alter their sorption relationship to the soil (Appelo and Postma, 2007). However the effect of pH on nitramine sorption is beyond the scope of this experiment.

Table 3: Conductivity (µ*S* cm^{-1}) measured in the water extract after filtration at the end of the experiment. Blank sample represents the conductivity in the solution containing only the nitramines before mixing with soil. Samples are sorted according to decreasing organic content.

Soil sample ID	Conductivity MEA-NO_2	Conductivity DMA-NO_2
007-1	34.3	36.0
003-1	61.4	55.9
008-1	46.6	45.2
006-1	39.0	40.5
004-1	46.9	47.9
005-1	44.2	41.2
006-3	36.0	31.2
008-3	37.1	34.9
Blank	18.0	22.0

The conductivity (Table 3) is a simple proxy for the ionic strength of the solution. The ionic strength is an important factor governing sorption of organic compounds as it influences the thickness of the diffuse double layer around the organic compound. A high ionic strength would therefore favor a greater sorption. The level of conductivity is typical for dilute freshwaters draining non-carbonaceous rock and varies moderately from 61.4 to 34.3 µ*S* cm^{-1} for MEA-NO_2 and between 55.9 to 31.2 µ*S* cm^{-1} for DMA-NO_2.

Calculated f_{OC} *and* K_{OC} values based on K_d at equilibrium and f_{OM} in the soils for the two nitramines are presented in Table 4 and 5.

Table 4: Calculated K_{OC} for MEA-NO_2, based on K_d at equilibrium (time average values) and f_{OC} (f_{OM}). Samples are sorted according to decreasing organic content.

Sample ID	K_d	f_{OM}	f_{OC}	K_{OC}
007-1	14	95	47	29
003-1	22	89	44	50
008-1	16	86	43	37
006-1	15	78	39	39
004-1	18	76	38	46
005-1	14	69	35	41
006-3	1.8	9	4	41
008-3	2.1	7	4	57
Average				42
Standard dev.				8.5
RSD (%)				20

Table 5: Calculated K_{OC} for DMA-NO$_2$, based on K_d at equilibrium (i.e. 192h values) and f_{OC} (f_{OM}). Samples are sorted according to decreasing organic content.

Sample ID	K_d	f_{OM}	f_{OC}	K_{OC}
007-1	45	95	47	94
003-1	54	89	44	121
008-1	39	86	43	91
006-1	58	78	39	150
004-1	37	76	38	98
005-1	49	69	35	142
006-3	3.6	9	4	82
008-3	1.5	7	4	40
Average				102
Standard dev.				35.3
RSD (%)				35

There is unfortunately a rather large relative standard deviation (RSD) for the K_{OC}, especially for DMA-NO$_2$. This may indicate that the sorption of nitramines is not only governed by the organic content of the soil (Table 2). There are large spatial variations in physiochemical properties of natural organic matter. These differences have been shown to plays a major role in governing the K_{OC} of compound in question. E.g. An decreasing H/O-atomic ratio in organic matter appears to result in an decreasing K_{OC} (Grathwohl, 1990). The variation in K_{OC} may also likely be due to uncertainties generated within the experiment and analytical uncertainties in the determination of concentration of nitramines in the water extracts.

Comparing the log of average K_{OC} values of the nitramines with literature values of log K_{OC} of other low molecular weight organic pollutant (Table 6), we see that the nitramines are relatively hydrophilic.

Table 6: Calculated log K_{OC} for the nitramines along with some known organic pollutants of environmental concern (Karickhoff, 1981).

Compound	log K_{OC}
MEA-NO$_2$	**1.63**
Benzene	1.78
DMA-NO$_2$	**2.01**
Tetrachloroethylene	2.56
Naphthalene	2.94
Pyrene	4.83
pp'-DDT	5.38
221,44',55'PCB	5.62

The relative electronegative –OH and -NO$_2$ functional groups of MEA-NO$_2$ and the -NO$_2$ group of the DMA-NO$_2$ (see figure 2) give the nitramines their rather hydrophilic properties, which can explain the relatively low K_{OC} values.

Nitramines will also absorb to dissolved natural organic matter (DNOM). This posed a concern in the interpretation of the data of the batch experiment as measurements of the DOC concentration (a proxy for DNOM) in the solution was more than 10 times higher than concentrations that are commonly encountered in soil-water under natural conditions (Figure 8).

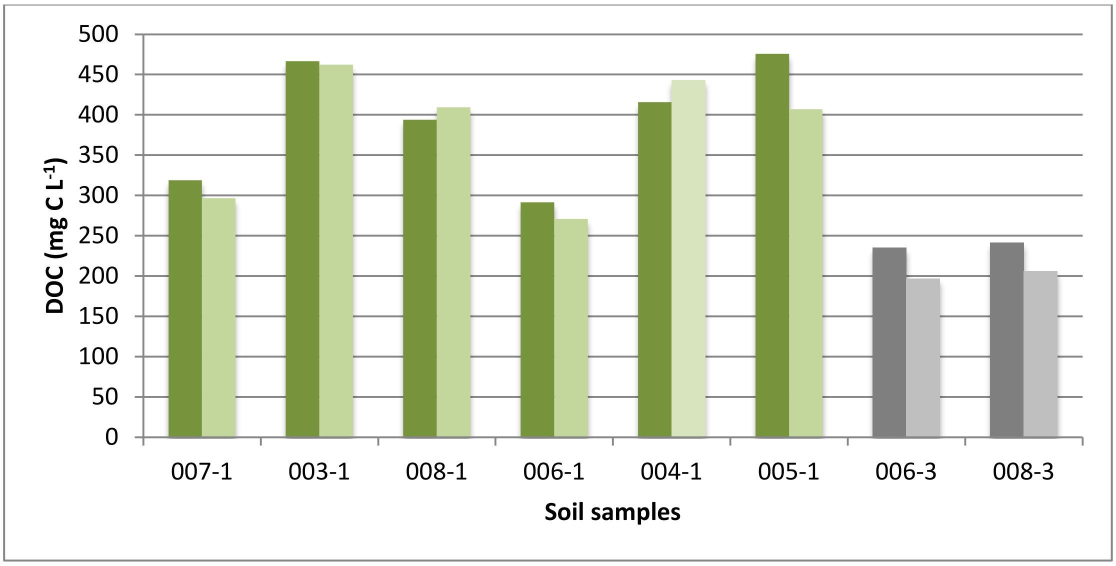

Figure 8: DOC concentration measured after 192h. Dark colors represent flasks with MEA-NO_2, while light colors indicate flasks with DMA-NO_2. Samples are sorted according to decreasing organic content. Soils rich in organic material are shown in green, while mineral soil are shown in grey.

However the amount of organic carbon leached from the soil in this experiment accounts for only approx. 1% of the total organic carbon in the soil (Figure 9). This implies that the amount of nitramines sorbed to the organic matter in the soil is most likely far greater than the amount lost to DNOM.

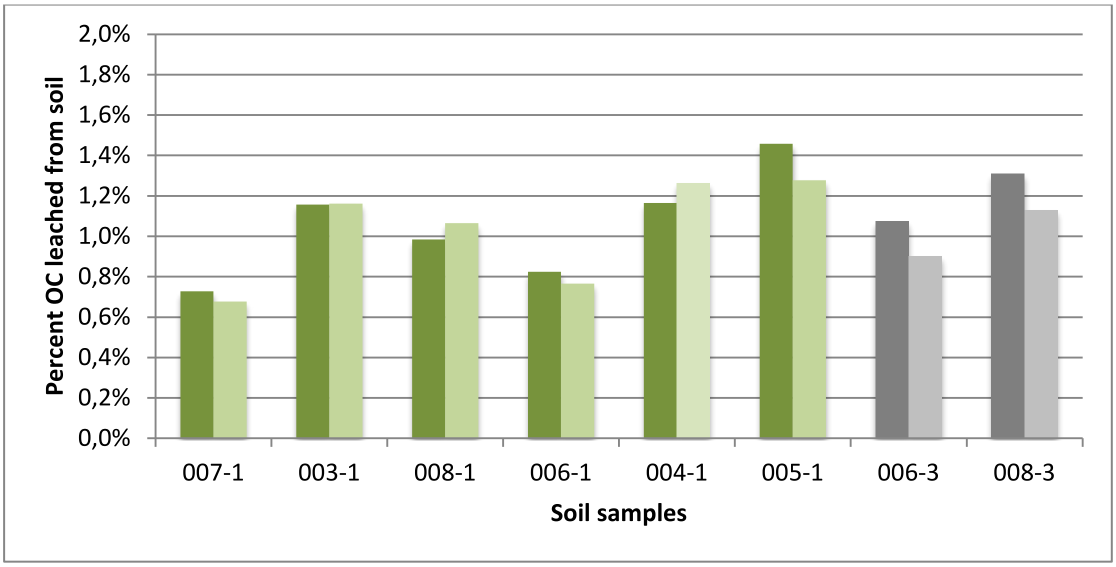

Figure 9: Percentage of organic carbon (OC) leached from the soil, based on DOC values in Figure 8, mass of soil in flasks and calculated fraction of organic carbon in the soil (Table 2). Dark colors represent flasks with MEA-NO_2, while light colors indicate flasks with DMA-NO_2. Samples are sorted according to decreasing organic content. Soils rich in organic material are shown in green, while mineral soil are shown in grey.

This does nevertheless raise the issue of the role of DNOM enhancing the transport of nitramines from the terrestrial to the aquatic environment. Since the nitamines are sorbed to organic matter it is likely that the nitramines also be sorbed to dissolved humic macromolecules and thus transported with the DNOM in the water. The soils in the region around Mongstad are in this study found to be very acid. Such soils exhibit inherently also a large flux of DNOM. It is therefore reason to believe that the leaching of nitramines from soils to surface waters may be significantly enhanced due to high flux of DNOM in the watercourses around Mongstad.

6 Conclusion

The rather hydrophilic properties of the studied nitramines, indicated by the low log K_{OC} values, means that a significant fraction of the compounds may be found in the water phase. Pilot tests show also that the nitramines appear not to be hydrolyzed nor biodegraded rapidly in the environment. This implies that the studied nitramines may eventually be leached out of the soils and into surface waters. The transport of nitramines in the water phase may be further enhanced by sorption to DNOM.

Both DMA-NO_2 and MEA-NO_2 show a stronger sorption to soil rich in organic matter (i.e. >75% LoI; Table 2) than to mineral soils. This is especially the case for DMA-NO_2, which has an 2-3 times greater sorption to organic rich soil than MEA-NO_2. This implies that the soils O- and A-horizons play the most important role in the retention of nitramines in soils. This is especially important within the 30 km proximity near Mongstad, where about half of the land area has thin soils with generally less than 2 cm O- and A horizons (Appendix A).

The slow sorption rate of the DMA-NO_2 indicates further that sorption equilibrium may not be achieved within 96 - 192h. This could result in much of the DMA-NO_2 bypassing the organic horizon and thereby reaching the mineral horizons where the sorption is fairly low.

The sorption test ISO/TS 21268:2007 is an experimental test and cannot be directly related to the conditions in the environment as the test is conducted with levels of nitramines and at a liquid to soil ratio which will not be encountered under natural conditions. Uncertainties in the final results are aggregated by all steps in the experiment and the analytical determination of nitramines. No direct assessments of uncertainties were made within the scope of this study, nevertheless clear preliminary conclusions may be drawn based on the limited variability achieved for K_{oc} values.

This pilot study shows clearly that the nitramines are not completely sorbed by the soils. Further studies are therefore required in order to determine the fate and transport of the nitramines in the environment.

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Appendix A

Distribution of land characteristics within the 30km from Mongstad (Skog og Landskap, 2011, NGU, 2011).

Table A.1: Land type within the 30 km radius from Mongstad.

Land type	Area (%)
Forest	39
Open solid ground terrain	47
Marsh	5
Cultivated soil	8
Other	1

Table A.2: Forest tree type within the 30 km radius from Mongstad.

Tree types in forest	Area (%)
Coniferous	55
Deciduous	39
Mix forest	6

Table A.3: Soil quality for tree growth in forest areas within the 30 km radius from Mongstad.

Soil quality for tree growth in forest	Area (%)
Very high	22
High	35
Medium	9
Low	7
Impediment	27

Table A.4: Land cover within the 30 km radius from Mongstad.

Land cover	Area (%)
Thick organic soil cover	5
Thick soil cover	20
Thin soil cover	42
Rock outcrop	9
Boulders or scree	0.2
Not registered	25

Table A.5: Distribution of general mineral soil types within the 30 km radius from Mongstad.

Unconsolidated deposits (General mineral soil type)	Area (%)
Moraine	8.9
Glaciofluvial deposits	0.4
Marine and fjord deposits	0.4
Fluvial deposits	0.2
Erosion material	8.7
Landslide material	2.8
Peats and bogs	2.4
Rock outcrop/thin soils	76.2