



Analytical Report

Analysis of ETFE Net and Sediment Samples

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Hartley Anderson Limited

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Analysis*	Units	Matrix	Sample ID									
			Fox-1	Fox-2	Golf-1	Golf-2	Golf-3	Lima	Mike-2	Mike-3	November	
2,4,6-trinitrotoluene	µg/net	ETFE net	6	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
2,4-dinitrotoluene	µg/net	ETFE net	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
2,6-dinitrotoluene	µg/net	ETFE net	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
2-amino-4,6-dinitrotoluene	µg/net	ETFE net	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
4-amino-2,6-dinitrotoluene	µg/net	ETFE net	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1

Notes
* = Dinitrotoluene and trinitrotoluene content determined by solvent extraction with analysis by HPLC-UV
† = The data presented within this report relate only to the samples as received at the laboratory

Analysis*	Units	Matrix	Sample ID									
			G before	G after	F before	F after	N after	L before	L after	J before	M before	M after
2,4,6-trinitrotoluene	mg/kg	Sediment	358	6.00	2.16	0.79	10400	9.69	39900 [†]	2450	2.45	11.5
2,4-dinitrotoluene	mg/kg	Sediment	< 1.00	< 1.00	< 1.00	< 1.00	< 10.00	< 1.00	99.5	< 1.00	< 1.00	< 1.00
2,6-dinitrotoluene	mg/kg	Sediment	< 1.00	< 1.00	< 1.00	< 1.00	31.5	< 1.00	152	< 10.00	< 1.00	< 1.00
2-amino-4,6-dinitrotoluene	mg/kg	Sediment	< 5.00	< 0.50	< 0.50	< 0.50	< 0.50	< 0.50	174	< 5.00	< 0.50	< 0.50
4-amino-2,6-dinitrotoluene	mg/kg	Sediment	< 5.00	< 0.50	< 0.50	< 0.50	< 5.00	< 0.50	24.9	< 0.50	< 0.50	< 0.50

Notes
* = Dinitrotoluene and trinitrotoluene content determined by solvent extraction with analysis by HPLC-UV
† = Sample over calibration range after a 1:100 dilution
‡ = The data presented within this report relate only to the samples as received at the laboratory

Appendix A

Guidelines on Use of Report

Guidelines on Use of Report

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Assessment of the Levels of TNT and Degradation Products in Marine Sediments and Waters

Analysis of ETFE Net and Sediment Samples

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Abbreviations

AQC	Analytical Quality Control
BiP	Blast in place
CCS	Calibration check standards
ETFE	Ethylene tetrafluoroethylene
HPLC	High performance liquid chromatography
LOD	Limit of detection
MLUR	Ministerium für Landwirtschaft, Umwelt und ländliche Räume
RIB	Rigid inflatable boat
TNT	Trinitrotoluene
UXO	Unexploded ordnance

1. Introduction

1.1 Sample Details

Nine ethylene tetrafluoroethylene (ETFE) net samples and ten sediment samples were collected from the North Sea prior to, and after, the clearance of unexploded ordnance (UXO). The samples were analysed for dinitrotoluene and trinitrotoluene content. Table 1.1 provides details on the sample locations, type of UXO including initial charge size and the samples acquired at each location.

Table 1.1: Sample location details

Sample Identification	Depth [m]	Type	Initial charge size	Clearance	Samples Acquired
Foxtrot (F)	18.0	Mk 1-4 (Possibly a Mk-6 mine, 430 kg)	~200 kg Amatol (estimated 60 % of the original 340 kg explosive charge remained)	High Order	2 x ETFE net, 1 x pre sediment sample, 1 x post sediment sample
Golf (G)	19.5	British possible Mk-6 mine	~344 kg Amatol (estimated at 80% of original 430 kg)	Low Order followed by High Order	3 x ETFE net, 1 x pre sediment sample, 1 x post sediment sample
Juliet (J)	19.5	British Mk 1-4 mine	340 kg Amatol	Low Order	1 x pre sediment sample
Lima (L)	17.5	British Mk 6 mine	430 kg Amatol	Low Order	1 x ETFE net, 1 x pre sediment sample, 1 x post sediment sample
Mike (M)	18.5	British Mk 1-4 mine	170 kg Amatol (estimated 50-60% original 340 kg)	High Order	2 x ETFE net, 1 x pre sediment sample, 1 x post sediment sample
November (N)	17.5	British Mk 1-4 mine	340 kg Amatol explosive main charge intact.	Low Order followed by High Order	1 x ETFE net, 1 x post sediment sample
Notes Pre sediment sample refers to sediment collected prior to high order, or low order clearance methods Post sediment sample refers to sediment collected after high order, or low order clearance methods ETFE = Ethylene tetrafluoroethylene					

Appendix A outlines the guidelines for use of this report.

2. Methodologies

2.1 Sample Collection

ETFE net samples were acquired from the water surface at the clearance site from a rigid inflatable boat (RIB). Sediment samples were taken by a diver within 5 m of the UXO and the remains or crater following clearance.

2.2 Sample Preparation

Sediment samples were air dried at < 30 °C and ground to pass through a 710 µm sieve for homogeneity.

2.3 Sample Extraction and Analysis

2.3.1 ETFE nets

A solid:liquid extraction with acetonitrile was performed on the nets and an aliquot was run on a high-performance liquid chromatography (HPLC) system coupled to a diode array detector. The system is calibrated with a 5-point calibration of explosives and any positive results are automatically calculated by the software against this calibration. Method and system blanks are run alongside analytical quality control (AQC) samples and calibration check standards (CCS).

2.3.2 Sediment

A solid:liquid extraction with acetonitrile was performed on the sediment and an aliquot was run on an HPLC system coupled to a diode array detector. The system is calibrated with a 5-point calibration of explosives and any positive results are automatically calculated by the software against this calibration. Method and system blanks are run alongside AQC and CCS samples.

3. Results

3.1 ETFE Net Samples

Concentrations of 2,4,6-trinitrotoluene (2,4,6-TNT) in the ETFE net samples were < 1 µg/net in all samples, except sample Fox-1, which had a concentration of 6 µg/net.

Concentrations of 2,4-dinitrotoluene, 2,6-dinitrotoluene, 2-amino-4,6-dinitrotoluene and 4-amino-2,6-dinitrotoluene were < 1 µg/net in all samples.

3.2 Sediment Samples

2,4,6-TNT concentrations ranged from 0.79 mg/kg for sample F after to 39900 mg/kg for sample L after, with a mean of 5310 mg/kg and high variability (RSD 237 %).

2,4-dinitrotoluene concentrations ranged from < 1.00 mg/kg for seven samples to 99.5 mg/kg for sample L after.

2,6-dinitrotoluene concentrations ranged from < 1.00 mg/kg for six samples to 152 mg/kg for sample L after.

2-amino-4,6-dinitrotoluene concentrations ranged from < 0.50 mg/kg for eight samples to 174 mg/kg for sample L after.

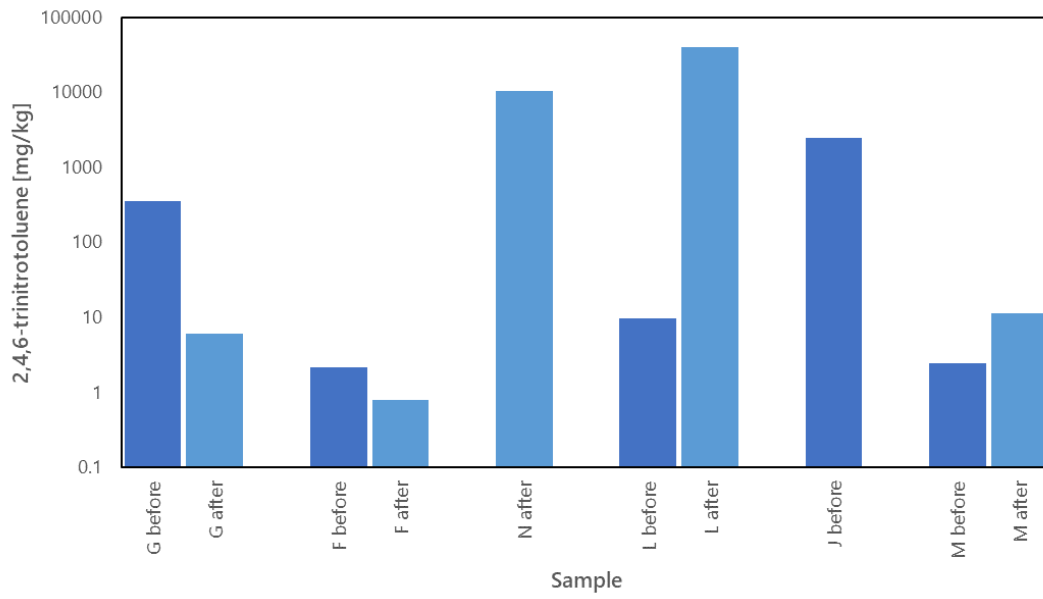
4-amino-2,6-dinitrotoluene concentrations ranged from < 0.50 mg/kg for seven samples to 24.9 mg/kg for sample L after.

Table 3.1: Summary of Analysis of ETFE Net Samples

Sample ID	Analysis				
	2,4,6-trinitrotoluene	2,4-dinitrotoluene	2,6-dinitrotoluene	2-amino-4,6-dinitrotoluene	4-amino-2,6-dinitrotoluene
Fox-1	6	< 1	< 1	< 1	< 1
Fox-2	< 1	< 1	< 1	< 1	< 1
Golf-1	< 1	< 1	< 1	< 1	< 1
Golf-2	< 1	< 1	< 1	< 1	< 1
Golf-3	< 1	< 1	< 1	< 1	< 1
Lima	< 1	< 1	< 1	< 1	< 1
Mike-2	< 1	< 1	< 1	< 1	< 1
Mike-3	< 1	< 1	< 1	< 1	< 1
November	< 1	< 1	< 1	< 1	< 1
Minimum	< 1	< 1	< 1	< 1	< 1
Maximum	6	< 1	< 1	< 1	< 1
Mean	-	-	-	-	-
Standard Deviation	-	-	-	-	-
RSD [%]	-	-	-	-	-
Notes Data presented as µg/net					

Table 3.2: Summary of Analysis of Sediment Samples

Sample ID	Analysis				
	2,4,6-trinitrotoluene	2,4-dinitrotoluene	2,6-dinitrotoluene	2-amino-4,6-dinitrotoluene	4-amino-2,6-dinitrotoluene
G before	358	< 1.00	< 1.00	< 5.00	< 5.00
G after	6.00	< 1.00	< 1.00	< 0.50	< 0.50
F before	2.16	< 1.00	< 1.00	< 0.50	< 0.50
F after	0.79	< 1.00	< 1.00	< 0.50	< 0.50
N after	10400	< 10.00	31.5	< 0.50	< 5.00
L before	9.69	< 1.00	< 1.00	< 0.50	< 0.50
L after	39900*	99.5	152	174	24.9
J before	2450	< 1.00	< 10.00	< 5.00	< 0.50
M before	2.45	< 1.00	< 1.00	< 0.50	< 0.50
M after	11.5	< 1.00	< 1.00	< 0.50	< 0.50
Minimum	0.79	< 1.00	< 1.00	< 5.00	< 5.00
Maximum	39900	99.5	152	174	24.9
Mean	5310	-	-	-	-
Standard Deviation	12600	-	-	-	-
RSD [%]	237	-	-	-	-
Notes					
Data presented in mg/kg dry weight					
* = Sample over calibration range after a 1:100 dilution					



Notes

Log₁₀ scale used to present the data

Figure 3.1: Sediment 2,4,6-trinitrotoluene concentrations

4. Discussion

Munitions were dumped in the North and Baltic Sea after the First and Second World Wars, resulting in millions of tonnes of conventional explosives and chemical weapons remaining on the seabed (Böttcher et al., 2012). UXO can present a hazard to vessels and infrastructure through self-detonations, as well as environmental concern through the leakage of toxic compounds present within the metal shells. Prolonged time on the seabed has resulted in the corrosion of casings and subsequent release of explosive chemicals such as 2,4,6-TNT to the surrounding environment, with corrosion rates influenced by pressure, temperature and salinity (Beck et al., 2018; Beck et al., 2019). Munitions lying on the seabed surface are likely to undergo faster rates of corrosion compared to those buried in sediment, which are subjected to anoxic conditions (George et al., 2015).

UXO disposal can be undertaken by the commonly used high order detonation, or by low order deflagration. High order detonation, or blast in place (BiP), is a common method used to reduce the risk to shipping and infrastructure through controlled explosion of munitions. However, this is often not 100 % effective, resulting in significant quantities of explosive compounds remaining on the seabed (Maser & Strehse, 2020). High order detonation involves the use of a counter charge in close proximity to the UXO in order to detonate any remaining explosive material present. However, studies have demonstrated that the levels of noise generated during explosions can have negative impacts on marine mammals due to their sensitivity to noise trauma (von Benda-Beckmann et al., 2015). Sound waves travel faster in water when compared to air; therefore, the noise from large blasts may impact marine mammals out with the 1 km mitigation zone.

Low order deflagration is an alternative technique used for UXO clearance and involves the use of a small charge to ignite the explosive contents to ignite and burn out, without detonation. This results in lower noise levels that are related to the charge used, rather than the size of the device detonated (Robinson et al., 2020).

Sediment concentrations of 2,4,6-TNT did not show any consistent patterns between pre- and post-clearance. Some sample locations, such as location Golf, demonstrated a reduction in sediment 2,4,6-TNT concentrations after device clearance, whereas location Lima demonstrated an increase of 4 orders of magnitude after clearance.

The temporal changes observed in sediment 2,4,6-TNT concentrations did not show any consistent patterns with the detonation method used. However, location Lima, which had the higher post detonation concentration, was subjected to low order deflagration only. Low order deflagration involves the use of charges to crack the ammunition casing to allow the contents to burn out, which may lead to a release of explosive contents into the surrounding environment if any unconsumed munitions compounds remain (Novik, 2023), resulting in the increase in 2,4,6-TNT observed.

A reduction in 2,4,6-TNT concentrations was observed at sample locations Fox and Golf, which were subjected to high order detonation. A post detonation reduction in 2,4,6-TNT concentrations could be a result of sediment redistribution, particularly with 'clean' sediment from blast crater formation.

Concentrations of munitions compounds recorded in marine sediment at UXO dumping sites are variable (Beck et al., 2018). For example, 2,4,6-TNT concentrations ranged from < limit of detection (LOD) to 1.2 mg/kg in the Dutch sector of the North Sea (den Otter et al., 2023) whilst a maximum value of 7.1 mg/kg was recorded at Kolberger Heide (MLUR, 2007). Maser et al. (2023) investigated 2,4,6-TNT levels around a wreck containing munitions and found a maximum concentration of 12.6 mg/kg, along with increased concentrations of nitroaromatic compounds found in fish and mussels around the wreck. Concentrations several orders of magnitude higher (19300 mg/kg) were measured next to UXO, with a reduction in concentrations observed at increasing distance (Barton & Porter, 2004). The pre- and post-detonation samples in this study also demonstrate highly variable concentrations, with concentrations of 2,4,6-TNT ranging from 0.79 mg/kg to 39900 mg/kg. The concentrations observed in study were considerably higher at some of the pre-detonation sample locations, suggesting the release of explosive compounds through corrosion and deterioration of munitions casings.

Concentrations of munitions compounds on the ETFE nets were below their respective limit of detection in all samples except Fox-1, where a concentration of 6 µg/net was recorded. The low concentrations recorded may be due to dilution of munition compounds within the water column, or a lack of sorption to the ETFE net surface. The low levels of TNT and degradation products observed in the ETFE net samples correlates with modelled TNT release from submerged munitions (Wang et al., 2013) which predicted munitions concentrations in the order of ng/L in water near munitions. Similarly, trace levels (< 1 µg/L) of munitions compounds (dinitrobenzene, dinitrotoluene and amino-dinitrotoluenes) were recorded in seawater near submerged munitions in Nova Scotia (Rodacy et al., 2001). The reported levels are consistent with mesocosm experiments which show slow dissolution and rapid dilution of dissolved munitions in water overlying exposed munition fragments under low flow conditions (Rosen & Lotufo, 2010).

5. Conclusions

The aim of this report has been to evaluate the concentrations of 2,4,6-TNT and other munitions compounds in seawater and sediment prior to, and after, the clearance of UXO by high order detonation and low order deflagration.

Sediment concentrations of 2,4,6-TNT did not show any consistent patterns between pre- and post-clearance, or with the clearance method used. Sample location Lima, cleared by low order deflagration only, reported a significant increase in sediment 2,4,6-TNT concentrations, suggesting a release of munitions compounds from the casing. The level recorded was comparable to 2,4,6-TNT levels in close proximity to munitions.

A reduction in 2,4,6-TNT concentrations was observed at sample locations Fox and Golf, which were subjected to high order detonation. The decrease observed is likely to be due to sediment redistribution from blast crater formation.

Sample location Mike was subjected to high order detonation and reported an increase in 2,4,6-TNT concentrations.

The remaining sample locations Juliet and November did not have a complete set of pre and post sediment samples and therefore changes in 2,4,6-TNT concentrations after UXO clearance could not be assessed.

The low concentrations recorded in the ETFE net samples may be due to dilution of munition compounds within the water column, or a lack of sorption to the ETFE net surface.

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Appendix A

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