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Chemical Sensing of Explosive Targets in the Bedford Basin, Halifax Nova Scotia

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Chemical Sensing of Explosive Targets in the Bedford Basin, Halifax, Nova Scotia

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ABSTRACT

Sandia National Laboratories has conducted research in chemical sensing and analysis of explosives for many years. Recently, our focus has been on the classification of unexploded ordnance (UXO) in shallow water, unearthed mortar rounds and shells, and anti-personnel/anti tank mines on land by sensing the low-level explosive signatures associated with these objects. The objective of this work is to develop a field portable chemical sensing system that can be used to examine mine-like objects (MLO) and UXO to determine whether there are traces of explosives associated with these objects. A sampling system that can extract explosives from water has been designed and demonstrated previously. This sampler utilizes a flow-through chamber that contains a solid phase microextraction (SPME) fiber to extract and concentrate the explosive molecules. Explosive molecules are then thermally desorbed from the concentrator for rapid desorption into an ion-mobility spectrometer (IMS) for identification. Three variations of this sampling system were evaluated during the Halifax field tests. This chemical sensing system is capable of sub-part-per-billion detection of TNT and related explosive compounds. This paper will describe a demonstration of this system performed in Bedford Basin, Halifax, Nova Scotia.

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INTRODUCTION

The most common UXO detection methods used today are anomaly detectors such as magnetometers and other metal detectors. Unfortunately, these techniques are often unable to discriminate between UXO or MLOs and metallic litter, such as shell fragments or other detritus. As a result, the false alarm rate associated with these techniques is quite high. Other systems, including sonar systems, ground penetrating radar, and other technologies, are capable of detecting anomalies in the environment that indicate the presence of UXO and MLOs. Software algorithms that attempt to reduce the false alarm rate by distinguishing between UXO and naturally occurring items such as rocks, are often incorporated into these systems. The successes of these systems varies, and have not been widely deployed in the field, often due to cost or difficulties of moving these large and rather complex systems into the field.

We are developing a small, portable detection system that can be used to determine whether there are explosive molecules associated with objects submerged in shallow water. Often, these objects will have been detected using other techniques, but by combining two different detection technologies, the false alarm rate can potentially be reduced to near zero.

Our approach is to incorporate off-the-shelf technology to the greatest extent possible. Only a few basic detection instruments are available to be used in a chemical sensor system. We evaluated the available technology for application to this sensing system and determined that the most practical instrument currently available for field detection is an ion mobility spectrometer (IMS). The IMS has a good balance of sensitivity and specificity for this application. By specificity we mean that the IMS is capable of determining the identities of several different explosive molecules in the same sample and isolating their signals. Using an IMS, it is possible to estimate the proportions of individual explosive compounds within the sample and to identify related degradation products as well. The IMS is sufficiently simple to operate that the analyses may be reduced by microprocessor to simple yes/no results. It is also adaptable to miniaturization and portable operation. For these tests, a gas chromatograph (GC) with an electron capture detector was used to confirm the results of the IMS analysis. The GC also provided quantitative results.

The use of a concentrator system in conjunction with an IMS for explosive detection in the marine environment has been demonstrated previously in “staged” tests [1]. This report describes the analysis of targets that have been submerged in the marine environment. The targets that were sampled have been submerged for a minimum of 60 years; some targets were believed to have been in place since World War I, while others were from World War II. The targets were not classified by age.

Halifax Explosion, Bedford Harbor, December 6, 1917

Nova Scotia is one of the ten provinces of Canada. It is bounded on the North by the Gulf of St. Lawrence and Northumberland Strait, across which lies Prince Edward Island; on the East and South by the Atlantic Ocean; and on the West by New Brunswick (Figure 1).



Figure 1. Map showing location of Halifax, Nova Scotia, Canada.

World War I demanded and consumed large amounts of materials, munitions and personnel. In early 1917 Halifax Harbor was officially introduced as a convoy system. Halifax harbor is a deep natural harbor. The inner harbor, known as Bedford Basin, was ideal for assembling convoys of warships which escorted the transport ships to protect them from German U-boats. Figure 2 shows the vast number of ships the Bedford Basin could accommodate.



Figure 2. Convoy Assembly in Bedford Basin circa 1943. As many as 600 ships would gather to form these convoys. M.M.A., Charles A. Vaughan Collection, Maritime Museum, Halifax, Nova, Scotia.

On Thursday, December 6, 1917, the port city was busy with the movement of war ships. Around eight o'clock that morning, the Norwegian relief ship SS IMO left its mooring in Bedford Basin and headed down the harbor for open sea. At the same time, a French ship, the Mont Blanc, that was used for transporting munitions was heading into the harbor to await a convoy escort. Stored in the holds of the Mont Blanc and stacked on her deck were 35 tons of benzol, 300 rounds of ammunition, 10 tons of gun cotton, 2,300 tons of picric acid and 400,000 pounds of TNT. The two ships collided, causing the picric acid on the Mont Blanc's deck to explode. The impact of the collision forced the Mont Blanc to drift towards Halifax.

At 9:06 a.m. debris from the munitions ship blew skyward a mile high. There was approximately 20 minutes between the collision of the boats and the explosion. It was enough time for spectators, including many children to run to the waterfront. Out of a population of less than 50,000 over 1900 people died and 9000 were injured, including 200 blinded by flying glass. This was the world's largest man-made explosion before Hiroshima (Figure 3).

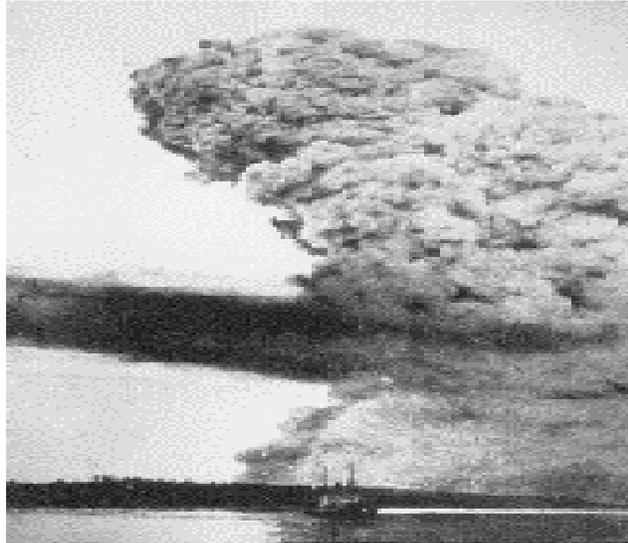


Figure 3. Mile high cloud from Halifax explosion. Photograph of Explosion Cloud courtesy of the Nova Scotia Provincial Archives

Next came shock waves, which created a man-made tsunami that sank many other vessels in the harbor. Sixteen hundred buildings were destroyed and 2.5 km² of the industrial section was leveled.

Although the explosion was extensive, much of the ordnance had not detonated. Much of it was thrown into the sea. For the most part, much of it was never recovered.

During World War II, Halifax was again the primary port for ships heading to Europe. Ordnance was sometimes lost overboard, several ships collided and sunk with their cargo, and there were several explosions that once again scattered ordnance.

Today, a considerable amount of unexploded ordnance remains in Bedford Basin. As a part of a continuing clean-up effort, Explosive Ordnance Disposal (EOD) divers are removing the most hazardous items. The ability to determine which UXO is still live is an important aspect, and the ability to “sniff” UXO is likely to assist with this determination.

Experimental

Projected Sample Concentration

A major part of our previous research, which was required before applying any detection technology to this task, was the determination of source concentrations, which we define as the signal strength. We have an ongoing research project directed at estimating this signal strength through the development of a mathematical model. The model is being validated by laboratory and field experiments. The calculated concentrations for buried UXO ranges from part-per-billion (ppb or $1:10^9$), to $1:10^{18}$ by mass. Similar estimates have been given by Spangler and Hogan, et.al.^{1,2} Field analyses have verified these estimates. Water phase concentrations depend on the permeability of the explosive fill through polymeric casings or seals, or around threaded joints on the UXO. This can range from relatively high rates for polymeric materials to very low (nearly negligible) for metallic cased UXOs. A reasonable target concentration for a chemical sensor, therefore, seems to be about $1:10^{12}$ to $1:10^{15}$. This concentration, whether in vapor or water, is several orders of magnitude less than the sensitivity of any currently available instruments that may be readily adapted to portable use. It is necessary to enhance the signal by using concentration techniques before submitting the explosive analyte to the detection instrument.

Sampling Locations

When sampling a water column for the presence of explosives, the sampling location is critical. Prior tests [1] have shown that as water flows past a target item, the signature being generated by that item is entrained in a narrow column; it does not exhibit significant lateral diffusion. Samples must be taken directly downcurrent from the target. Tests have been done where samples have been collected directly downcurrent from a target and have yielded strong signatures. A sample taken with an off-axis displacement of as little as one foot will often yield no signature at all. Therefore, sampling direction was determined by the current flow. Water samples were collected at distances of 0.3 meters, 1.0 meters, 2.0 meters, and 3.0 meters downcurrent from the target at a vertical distance of approximately 0.3 meters above the sea bottom. The targets were located at depths ranging from 10 meters to 30 meters. Sediment samples were collected at the same distances downcurrent and in-line with the water samples.

Samples were collected in the Bedford Basin off the ammo pier at Rent Point, at the location where the Claire Lily, a transport, sank, in the Trongate depression, and at Black Rock point (Figure 4), where a large amount of cordite can be found. These sites were chosen because of their accessibility and the variety of UXO present at these sites.



Figure 4. Sampling at Black Rock Point. Sediment and water samples were collected.

During the sampling process, the divers used a video camera that both provided real-time visual data to the surface crew, and recorded the sampling process. However, because it was not possible to communicate with the divers in real-time during the sampling process, the divers selected the targets to be sampled. Prior to deployment, a briefing was held that explained the purpose of the test, defined the sampling distances from the target, outlined videotaping requirements, and explained the sampling procedures. Since the targets had not been located or identified prior to the sampling dives, nor could previously located targets always be re-acquired, the divers located a target, then followed the procedures outlined during the pre-dive briefing to collect the appropriate samples. Previous work by other researchers has shown that shells that had been breached or broken open typically did not produce a signature.^[2] Therefore, only intact shells were sampled during the current exercise. Video taping of diver activities assisted with post-test identification of the targets, but identification was often difficult because of turbidity and marine growth on the targets. All target identifications in this report should therefore be considered to be tentative. The analytes being released by these targets should be considered to be positive identifications.

Sample collection and concentration methods

The process of detecting explosive signatures in water includes three basic steps. The first step involves sampling water or sediment near a suspected target. Sampling location has been shown to be critical in obtaining accurate analytical results and will be discussed later. The second step involves separating and concentrating the explosive molecules from the water and finally, the third step involves transferring the explosive analyte to a detector for processing.

Tests in our laboratory have shown that solid phase microextraction (SPME) fibers exhibit sufficient selectivity and concentration ability to enable one to detect the signature being released by submerged UXOs. A portion of the current testing program involved the evaluation of different water collection methods. Hence, three variations of the sample collector were evaluated. The first was a submersible hand-held device that could be taken to the target by a diver; the second was a surface sampler that sampled water collected by a diver-held hose and a surface-mounted pump; the third was simply grab sample collection using high density polyethylene

bottles. Each will be described in turn, and the relative effectiveness of each described in the data analysis section.

The submersible hand-held sampler was built by modifying a Mityvac® vacuum pump (Prism Enterprises, Inc., San Antonio, TX) to draw water past a SPME fiber. The Mityvac® pump as received from the manufacturer is shown in Figure 5.

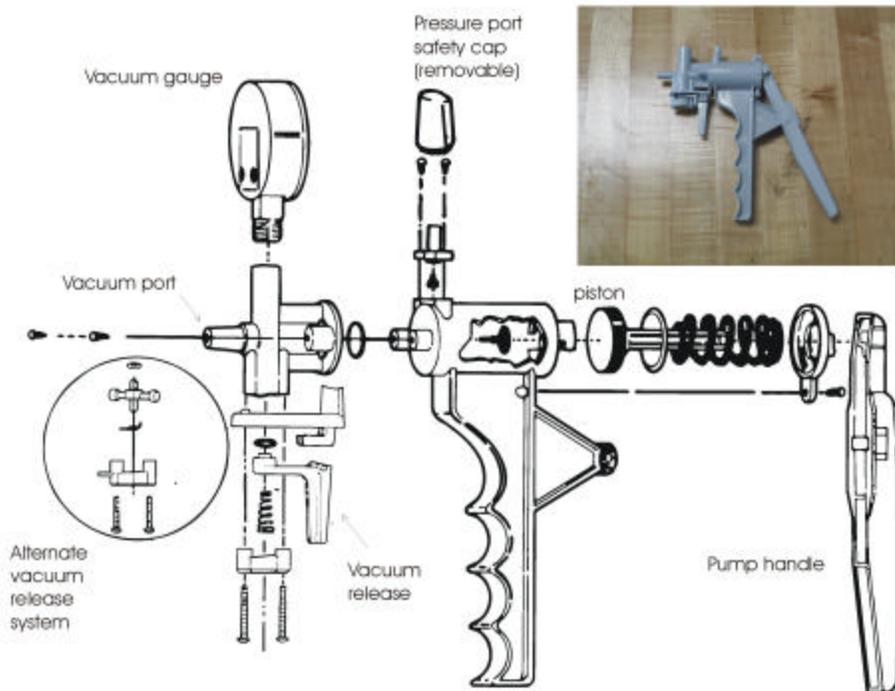


Figure 5 Mityvac® vacuum pump as received from manufacturer

The Mityvac® was modified (Figure 6) by removing the vacuum release assembly and sealing the resulting orifice. The front vacuum port was also plugged, and the vacuum gauge was removed. The pressure port safety cap was then removed. These modifications resulted in water being drawn into the port that previously held the vacuum gauge, and being vented through the pressure port as the pump was actuated. A plastic adapter that holds a standard SPME fiber holder and fiber was built to interface with the vacuum gauge port. This adapter had a series of small holes around the tip to channel water into the adapter and across the SPME fiber. A small plastic support was glued to the pump body to help support the adapter / sampling chamber. Approximately 7 milliliters of water was cycled with each pump actuation.

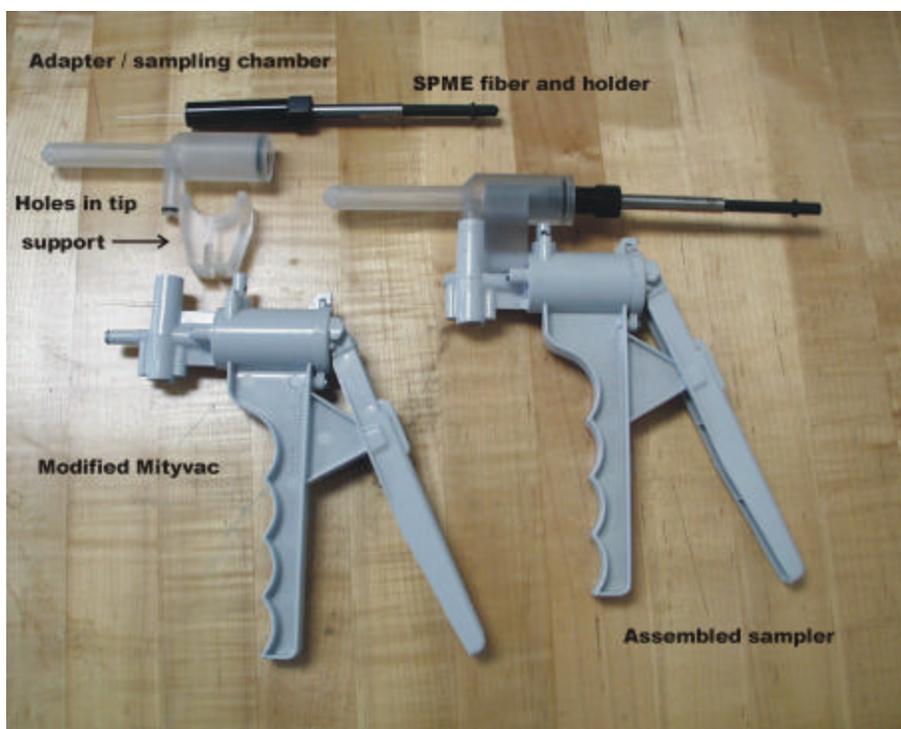


Figure 6. Modified Mityvac

Tests in our laboratory had shown that this SPME / Mityvac® sampling system exhibited sufficient sensitivity to detect TNT and 2,4-DNT at a concentration of 400 parts-per-trillion. For these tests, the pump was actuated a total of 10 times (total volume of water sampled was therefore 70 mL). The water sampled was discharged into a beaker for disposal. Analysis was done by thermally desorbing the SPME fiber into a PCP (West Palm Beach, FL) Model 111 Ion Mobility Spectrometer. Figure 7 shows this sampling system in use.

Although the MityVac® based sampler worked well in the laboratory, the divers identified one problem when using this sampler. The water discharge port on the pump was found to be too small. During the laboratory testing, the pump rate was adequate to obtain a sample in approximately 20 seconds. However, when deployed in 10 meters of water, the sampler was very sluggish and required several minutes to collect the sample. Enlarging the discharge port and/or increasing the strength of the internal return spring could possibly solve this problem. Because of this problem, the MityVac® sampler was not used for all tests.

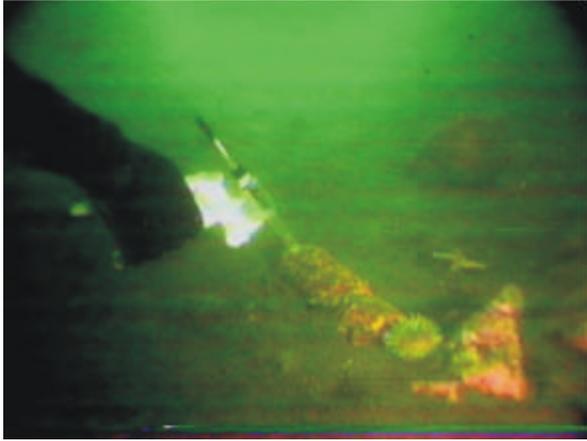


Figure 7. Mityvac based sampling system in use.

The second sampling method that was evaluated was a high density polyethylene block (Figure 8) that allowed water to flow through a collection chamber into which a SPME fiber could be inserted. A small, 12 volt water pump (PAR-MAX 3, model 30600-0012, ITT Jabsco, Costa Mesa, CA) was used to draw water through the sampling block and a length of ½ inch diameter polyethylene tubing that was carried by the diver. This pump is capable of providing 3.4 gallons per minute at a pressure of 40 psi with a 10 foot head. Tubing lengths sufficient to sample the bottom of the Trongate depression (ca. 30 meters) were used. It should be noted that the length of the input section of tubing can be quite long, even if a small pump is used, because the limiting factor for the pump's capacity is the head height, i.e. the distance the pump has to lift the water column above the surface of the water. The sampling chamber, Figure 8, is equipped with a septum inlet to obtain uninterrupted water flow while changing out SPME fibers. SPME fibers were left in the water stream for one minute. Grab samples were also collected using this system by collecting water as it exited the pump discharge port.

When using this flow-through system, we delayed sample collection until sufficient time passed to allow the tubing to be flushed with the desired water sample. Typically, it required about 50 seconds for our pump to clear the sampling line. Therefore, samples were not collected until at least 2 volumes had passed through the line (2 minutes).

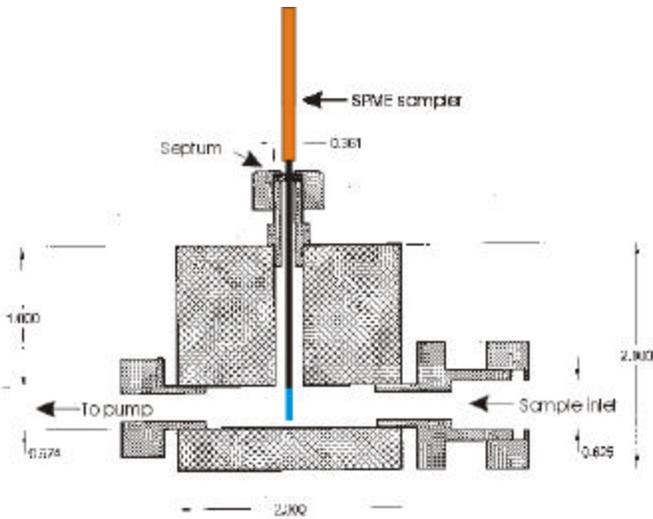


Figure 8. Flow through sampling system

Figure 9 shows this system in use by a diver.



Figure 9. Flow through sampling system in use by diver.

Figure 10 shows this sampling system being used topside to collect SPME samples, and Figure 11 shows this system being used to collect grab samples.



Figure 10. Flow through SPME sampling system being used to collect samples.



Figure 11. Flow through sampling system being used to collect grab samples.

When using either the Mityvac® system or the flow-through system, a pump is used to pass a sample of water through the concentrator, which contains a solid phase microextraction fiber. The SPME fiber removes the explosive molecules from the water stream and concentrates them for subsequent desorption into the IMS.

The third sampling method was simply diver-collected grab samples. Empty 237mL (8 oz.) amber high-density polyethylene bottles (Fisher Scientific, P/N 02-925-3D) were taken to the sampling location by divers. Laboratory tests have shown that, at a concentration of 10 part-per-billion, these bottles adsorb less than 1% of the analyte after a period of 7 days at room temperature. At the locations specified in the sampling plan, the lids

were opened to fill the bottles and then recapped. The bottles, which were pre-labeled with the distance from the target, were placed into a cooler upon return to the surface.

Sample storage and shipment

Most SPME samples were analyzed on-site using an Ion Mobility Spectrometer. The grab samples, along with some duplicate SPME samples, were returned to Sandia for extraction and analysis by gas chromatography using an electron capture detector. These samples were packed in ice upon collection and shipped, via overnight express, to Sandia for analysis. The samples were frozen upon receipt at Sandia and maintained at -20°C until analyzed.

Sediment samples

Sediment samples were collected by manually filling 237mL (8 oz.) amber high-density polyethylene bottles (Fisher Scientific, P/N 02-925-3D) with seabed material collected 0.3, 1.0, 2.0, and 3.0 meters from the target. The seabed was often rocky, so sediment samples were not always available. All sediment samples were returned to Sandia via overnight shipment. The samples were frozen until extraction and analysis procedures were begun.

Sediment Extraction procedures

- 1) Approximately 1gm (to 0.1mg) aliquots of sediment were placed in a 40mL scintillation vial. Larger pebbles (> ca. 4mm diameter) were removed prior to weighing the sample. Excess water was drained from the sediment, but the samples were not dried prior to analysis.
- 2) 15 mL of HPLC grade acetonitrile (Fisher Scientific) was added to the scintillation vial.
- 3) The sample was subjected to ultrasonication for 30 minutes in a cooled ultrasonic bath.
- 4) At the end of the allotted ultrasonication time, the supernatant acetonitrile liquid was clarified by filtration using a Cameo 0.45 µm nylon syringe filter screwed onto the Luer-lock fitting of a Becton Dickson 20ml disposable syringe.

The sediment extracts and many of the water grab samples were analyzed on a HP 6890 GC/µECD using method SW846 8095, “Explosives by Gas Chromatography.” Other samples were analyzed using a SPME extraction followed by desorption into an ion mobility spectrometer. The GC parameters are listed below. If necessary, additional sample dilutions were performed to keep the instrument response in the proper range.

GC/ECD Parameters for the Analysis of Halifax Samples

- 1) The SPME sample was introduced to a HP 6890 GC equipped with a split/splitless injector, a 6 m long x 0.53mm i.d. x 0.1 μ m film thickness RTX-225 capillary column and a μ -ECD (electron capture detector).
- 2) The instrument conditions for each analysis was as follows:
 - a) The injection port temperature was 225°C and a single tapered silanized 4mm i.d. glass liner without glass wool resides in the inlet.
 - b) The split vent of the injection port which, when open, has a split flow of 50 mL/min is closed just prior to injection. This permits the majority of the analyte to be swept onto the column much like the direct injection technique. After .75 minutes the split vent is opened to permit the remainder of the acetonitrile solvent to be vented to atmosphere, thereby narrowing the solvent peak. The vent stays open for the remainder of the analytical run.
 - c) The Helium carrier flow through the column at the initial oven temperature of 100°C is 7.4 mL/min.; the column head pressure at this point is 1.5psig. The oven is held at 100°C for 2 minutes, and then is programmed at 10 ° C/min to 200°C and held at this final temperature for 7 minutes. The analytical run is performed in Constant Flow mode which means the column head pressure rises from 1.5 psig at 100°C to 2.2 psig at 200°C to maintain the constant flow rate of 7.4 mL/min as the helium gas viscosity increases with temperature. The total analysis time is 19 minutes.
 - d) Finally, detection of the analytes takes place in the μ -ECD which has an additional make up nitrogen flow of 60 mL/min along with the 7.4 mL/min of helium carrier flow. The dinitro-aromatics are strongly electrophilic, and respond at the picogram level in the μ -ECD.

A 1000 pg/ μ L solution, containing a suite of nitroaromatic compounds, was analyzed to verify the 100 to 10000 picogram calibration curve. The 112 % recovery for the 1000pg standard was within the 85 to 115 % recovery limits specified for a Continuing Calibration Verification (CCV) by the SW-846 8095 method for Nitroaromatics by GC/ECD. A typical chromatogram is shown in Figure 12.

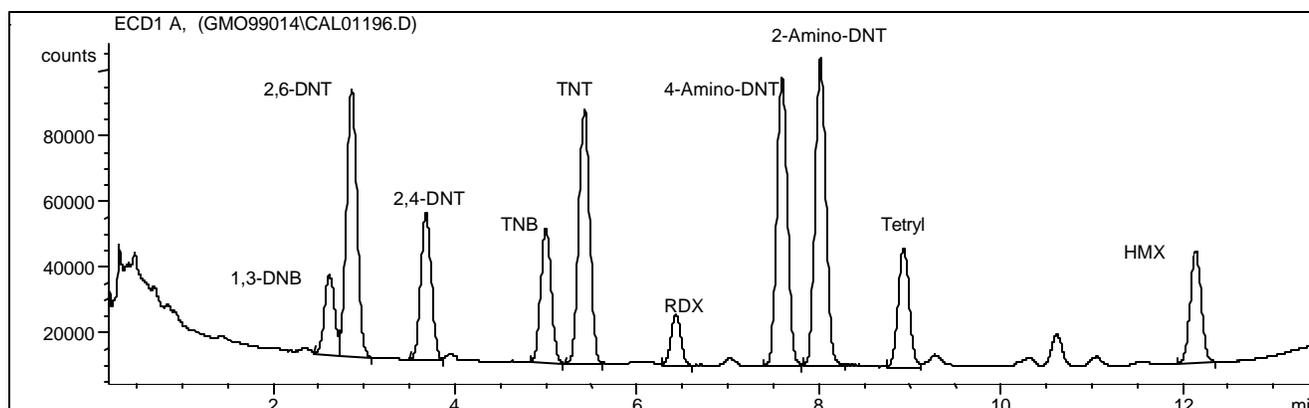


Figure 12 Typical chromatogram of an EPA 8330 calibration mixture.

GC Quality Control samples

Quality control samples were run on the HP 6890 GC/ μ ECD using method SW846 8095, “Explosives by Gas Chromatography” to verify the validity of the GC data.

1. The following quality control samples were run for each set of samples analyzed:
 - a. A laboratory method blank (sediment known to be free of explosive residue) was subjected to the same extraction and analysis procedures as all the samples.
 - b. A laboratory control sample (an extract of soil from (a)) that had been spiked with the analytes of interest. The spike was prepared using water from the basin that had been spiked with 1 part-per-billion (ppb) 2,6-dinitrotoluene (2,6-DNT), 1 ppb dinitrobenzene, 1 ppb 2,4-dinitrotoluene (2,4-DNT), 1 ppb 2,4,6-trinitrotoluene (TNT), 1 ppb trinitrobenzene (TNB), 1 ppb 2-amino-dinitrotoluene (2-Am-DNT), 1 ppb 4-amino-dinitrotoluene (4-Am-DNT), and 1 ppb cyclo-1,3,5-trimethylene-2,4,6-trinitramine (RDX) and run through the same analysis procedures as all the samples. This control sample was used to verify the accuracy of recovery of the analytes in the presence of possible matrix effects.
 - c. Finally, samples were bracketed by a continuing calibration verification to ensure that the original calibration is valid for those samples.

Ion Mobility Spectrometer

A PCP (West Palm Beach, FL) model 111 Ion Mobility Spectrometer was used to desorb SPME fibers that had been collected using the MityVac[®] sampler and the flow-through sampler topside. The IMS analysis is qualitative only. No attempt to quantify the amount of analyte was attempted. For these analyses, the inlet was maintained at 225°C for desorption of the SPME fibers. Purified air was used as the carrier and drift gases (200 mL / min and 100 mL / min, respectively), with approximately 5 ppb methylene chloride added to the drift gas as a dopant. SPME desorption time was approximately 30 seconds.

Data Analysis

The data shown on the following pages provides both identification and quantitation of the analytes collected near each target. Photos are provided for most targets from which a signature was obtained. In other cases, photographs were not obtained due to poor visibility in a particular location. Data obtained from Rent Point, Claire Lily, the Trongate, Black Rock Point, and the water and sediment blanks are presented.

SUMMARY

We have demonstrated the ability to collect, concentrate, and detect explosive molecules in water and seabed sediment being released by buried or submerged explosive ordnance. In many cases, there appears to be a correlation between the signature detected in the water column and the signature detected in the corresponding sediment. Detection levels ranged from 0.05 to >100 parts per billion. The variability of detections as a function of distance from the target is likely due to the filamentous nature of the plumes emanating from the target. Different sampling methods, i.e. underwater or surface grab samples and underwater or surface SPME extraction sample provided similar results. The technology used for these demonstrations is commercially

available hardware, adapted and modified for this purpose. Work remains to improve the design and integration of the various components to produce a field portable chemical detection system, but it has been demonstrated that unexploded ordnance can be detected using a chemical sensor even after many years of submersion.

Appendix 1 Detailed Data Summary

The tables shown in this appendix provide both summary results and detailed analytical results for each target sampled. The data tables should be read as follows:

The summary table lists the method of sample collection, the distance from the shell, and whether or not explosives were detected. If a detection occurred, the tables following the summary table provide detailed lists of the analytes found in that sample. Each sample that shows a positive detection in the summary table will have a corresponding column in one of the adjoining tables. “nd” indicates that no explosives were detected in the sample. An example is illustrated here:

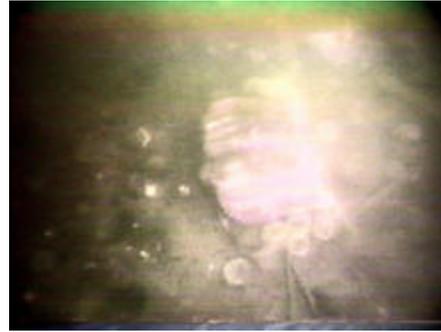
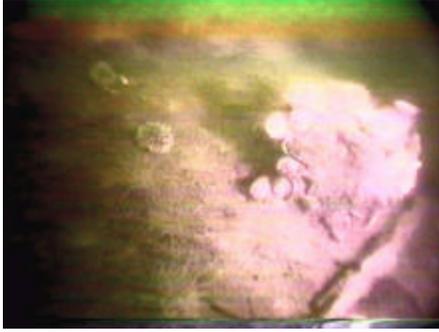
Summary of method and detection				
Method	0.3 meters from shell	1 meter from shell	2 meters from shell	3 meters from shell
Underwater grab, GC analysis. See Table A	positive detection	positive detection	nd	no sample collected
Surface grab, GC analysis. See Table A	positive detection	positive detection	nd	no sample collected
Underwater SPME, MityVac. See Table B	positive detection	positive detection	nd	no sample collected
Surface SPME, flow-through. See Table B	positive detection	positive detection	nd	nd
Sediment samples, GC analysis. See Table C	nd	nd	positive detection	positive detection

Water samples, GC Analysis with ECD detection	0.3 meters from shell	0.3 meters from shell	1 meter from shell	1 meter from shell
Sampling method	underwater grab	surface grab	underwater grab	surface grab
2,6-DNT	nd	nd	nd	nd
Dinitrobenzene	nd	nd	nd	nd
2,4-DNT	0.05	nd	nd	nd
TNT	nd	nd	14.19	nd
TNB	nd	nd	nd	nd
4-AM-DNT	0.14	trace	123.47	trace
2-AM-DNT	0.06	nd	107.86	nd

Table A. Sample Water analysis by GC

Water samples, SPME / MS analysis	0.3 meters from shell	0.3 meters from shell	1 meter from shell	1 meter from shell
Sampling Method	Surface SPME	MityVac SPME	MityVac SPME	Surface SPME
2,6-DNT	nd	nd	nd	nd
Dinitrobenzene	nd	nd	nd	nd
2,4-DNT	nd	nd	nd	nd
TNT	trace	trace	detect	detect
TNB	nd	nd	nd	nd
2- or 4-AM-DNT	nd	nd	nd	trace

Table A. Sample Water analysis by IMS



Sample Site: Rent Point

Sample Identifier: H

Likely ID – 6 inch shell

All results are in parts-per-billion

Summary of method and detection				
Method	0.3 meters from shell	1 meter from shell	2 meters from shell	3 meters from shell
Underwater grab, GC analysis. See Table A	positive detection	positive detection	nd	no sample collected
Surface grab, GC analysis. See Table A	positive detection	positive detection	nd	no sample collected
Underwater SPME, MityVac. See Table B	positive detection	positive detection	nd	no sample collected
Surface SPME, flow-through. See Table B	positive detection	positive detection	nd	nd
Sediment samples, GC analysis. See Table C	nd	nd	positive detection	positive detection

Water samples, GC Analysis with ECD detection	0.3 meters from shell	0.3 meters from shell	1 meter from shell	1 meter from shell
Sampling method	underwater grab	surface grab	underwater grab	surface grab
2,6 DNT	nd	nd	nd	nd
Dinitrobenzene	nd	nd	nd	nd
2,4 DNT	0.05	n	nd	nd
TNT	nd	nd	14.19	nd
TNB	nd	nd	nd	nd
4-AM-DNT	0.14	trace	123.47	trace
2-AM-DNT	0.06	nd	107.86	nd

Table A . Sample H water analysis by GC

Data table continuation

Sample Site: Rent Point

Sample Identifier: H

Likely ID – 6 inch shell

All results are in parts-per-billion

Water samples, SPME / IMS analysis	0.3 meters from shell	0.3 meters from shell	1 meter from shell	1 meter from shell
Sampling Method	Surface SPME	MityVac SPME	MityVac SPME	Surface SPME
2,6 DNT	nd	nd	nd	nd
Dinitrobenzene	nd	nd	nd	nd
2,4 DNT	nd	nd	nd	nd
TNT	trace	trace	detect	detect
TNB	nd	nd	nd	nd
2- or 4-AM-DNT	nd	nd	nd	trace

Table B. Sample H water analysis by IMS

Sediment samples, GC Analysis with ECD detection	2 meters from shell, sample 1	2 meters from shell, sample 2	3 meters from shell
2,6 DNT	117.1	509.6	48.66
Dinitrobenzene	79.81	nd	nd
2,4 DNT	557.0	39.95	199.8
TNT	0.005	13.9	1.57
TNB	11.39	23.06	6.49
4-AM-DNT	5.58	47.44	nd
2-AM-DNT	7.22	91.3	nd

Table C Sample H sediment analysis by GC



Sample Site: Rent Point

Sample Identifier: B3

Likely ID – 6 inch shell

All results are in parts-per-billion

Summary of method and detection				
Method	0.3 meters from shell	1 meter from shell	2 meters from shell	3 meters from shell
Underwater grab, GC analysis. See Table D	positive detection	positive detection	nd	Positive detection
Surface grab, GC analysis. See Table D	positive detection	nd	nd	nd
Underwater SPME, MityVac See Table E	positive detection	positive detection	nd	No sample collected
Surface SPME, flow-through. See Table E	positive detection	positive detection	nd	nd
Sediment samples, GC analysis. See Table F	positive detection	no sediment available	positive detection	nd

Data table continuation

Sample Site: Rent Point

Sample Identifier: B3

Likely ID – 6 inch shell

All results are in parts-per-billion

Water samples, GC Analysis with ECD detection	0.3 meters from shell	0.3 meters from shell	1 meter from shell	3 meters from shell
Sampling method	Underwater grab	Surface grab	Underwater grab	Underwater grab
2,6 DNT	nd	nd	nd	nd
Dinitrobenzene	nd	nd	nd	5.92
2,4 DNT	nd	0.04	nd	nd
TNT	nd	nd	nd	nd
TNB	nd	nd	nd	nd
4-AM-DNT	0.56	nd	1.0	nd
2-AM-DNT	0.22	nd	0.04	nd

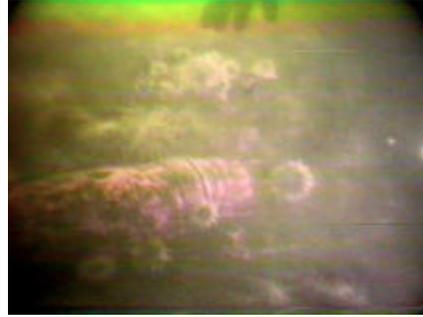
Table D. Sample B3 water analysis by GC

Water samples, SPME / IMS analysis	0.3 meters from shell	0.3 meters from shell	1 meter from shell	1 meter from shell
Sampling Method	Surface SPME	MityVac SPME	MityVac SPME	Surface SPME
2,6 DNT	nd	nd	nd	nd
Dinitrobenzene	nd	nd	nd	nd
2,4 DNT	nd	nd	nd	nd
TNT	nd	nd	nd	nd
TNB	nd	nd	nd	nd
2- or 4-AM-DNT	trace	trace	trace	trace

Table E. Sample B3 water analysis by SPME

Sediment samples, GC Analysis with ECD detection	0.3 meters from shell	2 meters from shell
2,6 DNT	nd	nd
Dinitrobenzene	nd	nd
2,4 DNT	nd	222.34
TNT	167.39	1.12
TNB	89.28	11.42
4-AM-DNT	552.6	5.22
2-AM-DNT	nd	nd

Table F. Sample B3 sediment analysis by GC



Sample Site: Rent Point

Sample Identifier: C2

Likely ID – 250 lb bomb

All results are in parts-per-billion

Summary of method and detection				
Method	0.3 meters from shell	1 meter from shell	2 meters from shell	3 meters from shell
Underwater grab, GC analysis. See Table G	Positive detection	Positive detection	No sample collected	No sample collected
Surface grab, GC analysis	nd	nd	nd	nd
Underwater SPME, MityVac	nd	nd	No sample collected	nd
Surface SPME, flow-through	nd	nd	nd	nd
Sediment samples, GC analysis. See Table H	Positive detection	Positive detection	Positive detection	No sediment available

Data continuation

Sample Site: Rent Point

Sample Identifier: C2

Likely ID – bomb

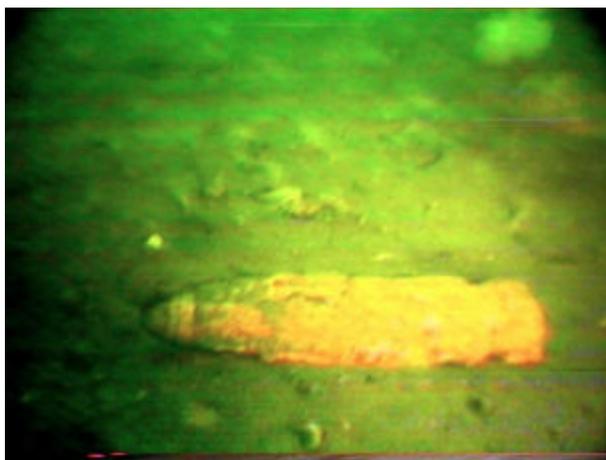
All results are in parts-per-billion

Water samples, GC Analysis with ECD detection	0.3 meters from shell	1 meter from shell
Sampling method	Underwater grab	Underwater grab
2,6 DNT	nd	nd
Dinitrobenzene	nd	nd
2,4 DNT	0.08	0.08
TNT	nd	nd
TNB	nd	nd
4-AM-DNT	nd	nd
2-AM-DNT	nd	nd

Table G. Sample C2 water analysis by GC

Sediment samples, GC Analysis with ECD detection	0.3 meters from shell	1 meter from shell	2 meters from shell
2,6 DNT	415.9	nd	nd
Dinitrobenzene	nd	4.82	nd
2,4 DNT	250.7	nd	2.06
TNT	0.99	2.32	0.13
TNB	0.29	0.19	1.72
4-AM-DNT	nd	1.83	nd
2-AM-DNT	0.49	nd	nd

Table H. Sample C2 sediment analysis by GC



Sample Site: Rent Point

Sample Identifier: D3

Likely ID – 155 mm shell

All results are in parts-per-billion

Summary of method and detection				
Method	0.3 meters from shell	1 meter from shell	2 meters from shell	3 meters from shell
Underwater grab, GC analysis. See Table I	Positive detection	Positive detection	Positive detection	No sample collected
Surface grab, GC analysis. See Table I	Positive detection	Positive detection	nd	nd
Underwater SPME, MityVac. See Table J	Sampler not used	Sampler not used	Sampler not used	Sampler not used
Surface SPME, flow-through. See Table J	Positive detection	Positive detection	Broken SPME fiber	No sample collected
Sediment samples, GC analysis. See Table K	No sediment available	No sediment available	Positive detection	Positive detection

Data table continuation

Sample Site: Rent Point

Sample Identifier: D3

Likely ID – 155 mm shell

All results are in parts-per-billion

Water samples, GC Analysis with ECD detection	0.3 meters from shell	0.3 meters from shell	1 meter from shell	1 meter from shell	2 meters from shell
Sampling method	Underwater grab	Surface grab	Underwater grab	Surface grab	Underwater grab
2,6 DNT	nd	nd	nd	nd	nd
Dinitrobenzene	nd	nd	nd	nd	nd
2,4 DNT	0.04	nd	0.9	nd	nd
TNT	nd	nd	nd	nd	nd
TNB	0.10	trace	nd	nd	nd
4-AM-DNT	0.06	nd	0.13	trace	0.21
2-AM-DNT	nd	nd	0.07	nd	0.07

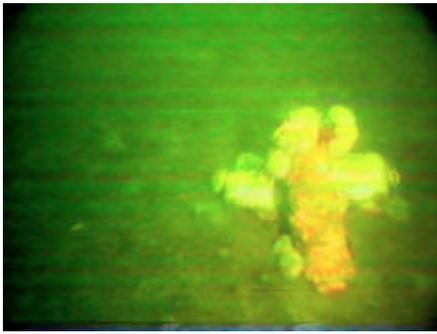
Table I. Sample D3 water analysis by GC

Water samples, SPME / IMS analysis	0.3 meters from shell	1 meter from shell
Sampling Method	Surface SPME	surface SPME
2,6 DNT	nd	nd
Dinitrobenzene	nd	nd
2,4 DNT	nd	nd
TNT	trace	trace
TNB	nd	nd
2- or 4-AM-DNT	nd	nd

Table J. Sample D3 water analysis by SPME

Sediment samples, GC Analysis with ECD detection	2.0 meters from shell	3 meters from shell
2,6 DNT	nd	nd
Dinitrobenzene	nd	nd
2,4 DNT	nd	nd
TNT	0.36	0.6
TNB	0.61	1.3
4-AM-DNT	nd	1.46
2-AM-DNT	nd	nd

Table K. Sample D3 sediment analysis by GC



Sample Site: Rent Point

Sample Identifier: E2

Likely ID – unknown

All results are in parts-per-billion

Summary of method and detection				
Method	0.3 meters from shell	1 meter from shell	2 meters from shell	3 meters from shell
Underwater grab, GC analysis. See Table L	Positive detection	nd	nd	No sample collected
Surface grab, GC analysis. See Table L	Positive detection	nd	nd	No sample collected
Underwater SPME, MityVac. See Table M	Positive detection	Positive detection	nd	No sample collected
Surface SPME, flow-through. See Table M	Positive detection	nd	nd	No sample collected
Sediment samples, GC analysis. See Table N	Positive detection	Positive detection	Positive detection	No sample collected

Data table continuation

Sample Site: Rent Point

Sample Identifier: E2

Likely ID – unknown

All results are in parts-per-billion

Water samples, GC Analysis with ECD detection	0.3 meters from shell	0.3 meters from shell	0.3 meters from shell
Sampling method	Underwater grab, sample 1	Underwater grab, sample 2	Surface grab
2,6 DNT	nd	nd	nd
Dinitrobenzene	nd	nd	nd
2,4 DNT	nd	0.9	nd
TNT	nd	0.02	nd
TNB	0.76	nd	0.03
4-AM-DNT	1.50	0.09	0.04
2-AM-DNT	0.50	nd	nd

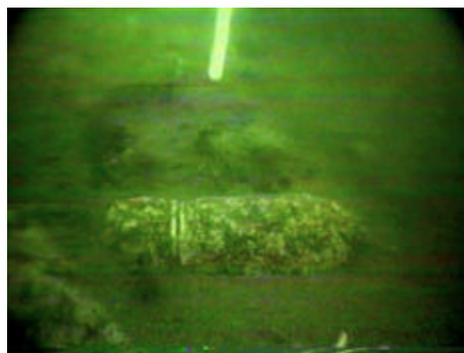
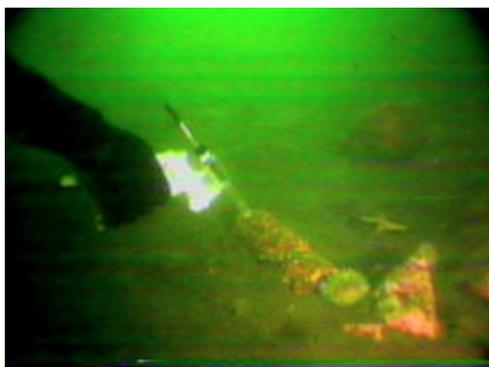
Table L. Sample E2 water analysis by GC

Water samples, SPME / IMS analysis	0.3 meters from shell	0.3 meters from shell	1 meter from shell
Sampling Method	Surface SPME	MityVac SPME	MityVac SPME
2,6 DNT	nd	nd	nd
Dinitrobenzene	nd	nd	nd
2,4 DNT	nd	nd	nd
TNT	nd	nd	nd
TNB	nd	nd	nd
2- or 4-AM-DNT	trace	trace	trace

Table M. Sample E2 water analysis by SPME

Sediment samples, GC Analysis with ECD detection	0.3 meters from shell	1 meter from shell	2 meters from shell
2,6 DNT	nd	nd	nd
Dinitrobenzene	nd	nd	nd
2,4 DNT	nd	nd	nd
TNT	8.87	0.38	0.53
TNB	1.97	0.29	0.58
4-AM-DNT	12.83	nd	nd
2-AM-DNT	3.94	nd	nd

Table N. Sample E2 sediment analysis by GC



Sample Site: Rent Point

Sample Identifier: F2

Likely ID – 4 inch shell

All results are in parts-per-billion

Summary of method and detection				
Method	0.3 meters from shell	1 meter from shell	2 meters from shell	3 meters from shell
Underwater grab, GC analysis. See Table O	Positive detection	Positive detection	nd	No sample collected
Surface grab, GC analysis. See Table O	Positive detection	nd	nd	No sample collected
Underwater SPME, MityVac. See Table P	Positive detection	nd	No sample collected	No sample collected
Surface SPME, flow-through. See Table P	Positive detection	Positive detection	nd	No sample collected
Sediment samples, GC analysis. See Table Q	No sample available	Positive detection	No sediment available	Positive detection

Data table continuation

Sample Site: Rent Point

Sample Identifier: F2

Likely ID – 4 inch shell

All results are in parts-per-billion

Water samples, GC Analysis with ECD detection	0.3 meters from shell	0.3 meters from shell	1 meter from shell
Sampling method	Underwater grab, sample 1	Surface grab	Underwater grab
2,6 DNT	nd	nd	nd
Dinitrobenzene	nd	nd	nd
2,4 DNT	0.05	3.14	trace
TNT	0.02	nd	nd
TNB	nd	nd	nd
4-AM-DNT	0.03	nd	nd
2-AM-DNT	nd	nd	nd

Table O. Sample F2 water analysis by GC

Water samples, SPME / IMS analysis	0.3 meters from shell	0.3 meters from shell	1 meter from shell
Sampling Method	Surface SPME	MityVac SPME	Surface SPME
2,6 DNT	nd	nd	nd
Dinitrobenzene	nd	nd	nd
2,4 DNT	nd	nd	nd
TNT	small peak	small peak	trace
TNB	nd	nd	nd
2- or 4-AM-DNT	small peak	trace	trace

Table P. Sample F2 water analysis by SPME

Sediment samples, GC Analysis with ECD detection	1 meter from shell	3 meters from shell
2,6 DNT	851.9	nd
Dinitrobenzene	nd	nd
2,4 DNT	245.4	0.04
TNT	1.32	1.03
TNB	0.81	0.6
4-AM-DNT	nd	nd
2-AM-DNT	nd	nd

Table Q. Sample F2 sediment analysis by GC



Sample Site: Rent Point

Sample Identifier: G1

Likely ID – 5 inch shell, fuse missing

All results are in parts-per-billion

Summary of method and detection				
Method	0.3 meters from shell	1 meter from shell	2 meters from shell	3 meters from shell
Underwater grab, GC analysis. See Table R	Positive detection	Positive detection	Positive detection	Positive detection
Surface grab, GC analysis. See Table R	Broken SPME fiber	Positive detection	Positive detection	Positive detection
Underwater SPME, MityVac. See Table S	Positive detection	Positive detection	System not used	System not used
Surface SPME, flow-through. See Table S	Positive detection	Positive detection	Positive detection	Positive detection
Sediment samples, GC analysis. See Table T	Positive detection	Positive detection	trace	No sediment available

Data table continuation

Sample Site: Rent Point

Sample Identifier: G1

Likely ID – 5 inch shell, fuse missing

All results are in parts-per-billion

Water samples, GC Analysis with ECD detection	0.3 meters from shell	1 meter from shell	1 meter from shell	2 meters from shell	2 meters from shell	3 meters from shell	3 meters from shell
Sampling method	Underwater grab, sample 1	Underwater grab	Surface grab	Underwater grab	Surface grab	Underwater grab	Surface grab
2,6 DNT	nd	nd	nd	nd	nd	nd	nd
Dinitrobenzene	nd	nd	nd	nd	nd	4.11	1.02
2,4 DNT	0.02	1.97	0.06	nd	0.73	trace	nd
TNT	nd	0.07	trace	0.07	0.04	nd	nd
TNB	nd	nd	nd	nd	nd	nd	nd
4-AM-DNT	nd	1.27	nd	nd	trace	nd	nd
2-AM-DNT	nd	0.79	nd	nd	trace	nd	nd

Table R. Sample G1 water analysis by GC

Water samples, SPME / IMS analysis	0.3 meters from shell	0.3 meters from shell	1 meter from shell	1 meter from shell	2 meters from shell	3 meters from shell
Sampling Method	Surface SPME	MityVac SPME	Surface SPME	MityVac SPME	Surface SPME	Surface SPME
2,6 DNT	nd	nd				
Dinitrobenzene	nd	nd	trace	nd	nd	detect
2,4 DNT	trace	trace	detect	detect	detect	detect
TNT	trace	nd	detect	detect	nd	nd
TNB	nd	nd	nd	nd	nd	nd
2- or 4-AM-DNT	nd	nd	detect	nd	trace	nd

Table S. Sample G1 water analysis by SPME

Sediment samples, GC Analysis with ECD detection	0.3 meters from shell	1 meter from shell	2 meters from shell
2,6 DNT	2.45	nd	nd
Dinitrobenzene	4.16	nd	nd
2,4 DNT	115.26	nd	nd
TNT	0.68	0.95	trace
TNB	2.62	0.67	nd
4-AM-DNT	0.51	nd	nd
2-AM-DNT	1.5	nd	nd

Table T. Sample G1 sediment analysis by SPME



Sample Site: Rent Point

Sample Identifier: A1

Likely ID – 6 inch shell

All results are in parts-per-billion

Summary of method and detection				
Method	0.3 meters from shell	1 meter from shell	2 meters from shell	3 meters from shell
Underwater grab, GC analysis. See Table U	Positive detection	Positive detection	Positive detection	System not used
Surface grab, GC analysis. See Table U	Positive detection	nd	System not used	System not used
Underwater SPME, MityVac. See Table V	No sample collected	Positive detection	Positive detection	System not used
Surface SPME, flow-through. See Table V	Positive detection	nd	System not used	System not used
Sediment samples, GC analysis. See Table W	No sediment available	Positive detection	Positive detection	No sediment available

Data table continuation

Sample Site: Rent Point

Sample Identifier: A1

Likely ID – 6 inch shell

All results are in parts-per-billion

Water samples, GC Analysis with ECD detection	0.3 meters from shell	0.3 meters from shell	1 meter from shell	2 meters from shell
Sampling method	Underwater grab, sample 1	Surface grab	Underwater grab	Underwater grab
2,6 DNT	nd	nd	nd	nd
Dinitrobenzene	nd	nd	nd	nd
2,4 DNT	0.12	0.10	nd	0.43
TNT	nd	nd	nd	nd
TNB	nd	nd	nd	nd
4-AM-DNT	nd	nd	0.37	nd
2-AM-DNT	nd	nd	0.18	nd

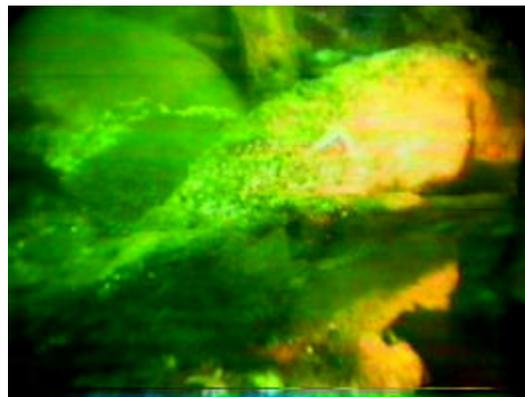
Table U. Sample A1 water analysis by GC

Water samples, SPME / IMS analysis	0.3 meters from shell	1 meter from shell	1 meter from shell	2 meters from shell	2 meters from shell
Sampling Method	Surface SPME	Surface SPME	MityVac SPME	Surface SPME	MityVac SPME
2,6 DNT	nd	nd	nd	nd	nd
Dinitrobenzene	nd	nd	nd	nd	nd
2,4 DNT	Detect	Detect	Trace	Trace	nd
TNT	Trace	nd	nd	nd	nd
TNB	nd	nd	nd	nd	nd
2- or 4-AM-DNT	nd	nd	nd	trace	trace

Table V. Sample A1 water analysis by SPME

Sediment samples, GC Analysis with ECD detection	1 meter from shell	2 meters from shell
2,6 DNT	nd	nd
Dinitrobenzene	nd	88.45
2,4 DNT	nd	30.26
TNT	1.28	0.81
TNB	2.07	1.81
4-AM-DNT	8.09	nd
2-AM-DNT	nd	nd

Table W. Sample A1 sediment analysis by GC



Sample Site: Claire Lily

Sample Identifier: K1

Likely ID – small bomb

All results are in parts-per-billion

Summary of method and detection				
Method	0.3 meters from shell	1 meter from shell	2 meters from shell	3 meters from shell
Underwater grab, GC analysis. See Table X	Positive detection	Positive detection	Positive detection	nd
Surface grab, GC analysis. See Table X	System not used	System not used	System not used	System not used
Underwater SPME, MityVac. See Table Y	System not used	System not used	System not used	System not used
Surface SPME, flow-through. See Table Y	Broken SPME fiber	Positive detection	Positive detection	nd
Sediment samples, GC analysis. See Table Z	Positive detection	No sediment available	No sediment available	No sediment available

Data table continuation

Sample Site: Claire Lily

Sample Identifier: K1

Likely ID – small bomb

All results are in parts-per-billion

Water samples, GC Analysis with ECD detection	0.3 meters from shell	1 meter from shell	2 meters from shell
Sampling method	Underwater grab, sample 1	Underwater grab	Underwater grab
2,6 DNT	nd	nd	nd
Dinitrobenzene	nd	nd	nd
2,4 DNT	nd	0.4	0.3
TNT	nd	0.58	0.01
TNB	nd	1.24	nd
4-AM-DNT	0.33	3.97	0.12
2-AM-DNT	0.11	3.92	0.11

Table X. Sample K1 water analysis by GC

Water samples, SPME / IMS analysis	1 meter from shell	2 meters from shell
Sampling Method	Surface SPME	Surface SPME
2,6 DNT	nd	nd
Dinitrobenzene	nd	nd
2,4 DNT	detect	detect
TNT	detect	detect
TNB	detect	detect
2- or 4-AM-DNT	detect	detect

Table Y. Sample K1 water analysis by SPME

Sediment samples, GC Analysis with ECD detection	0.3 meters from shell
2,6 DNT	nd
Dinitrobenzene	nd
2,4 DNT	nd
TNT	0.07
TNB	1.64
4-AM-DNT	nd
2-AM-DNT	0.39

Table Z. Sample K1 sediment analysis by GC



Sample Site: Claire Lily

Sample Identifier: D3

Likely ID – 250 lb bombs

All results are in parts-per-billion

Summary of method and detection				
Method	0.3 meters from shell	1 meter from shell	2 meters from shell	3 meters from shell
Underwater grab, GC analysis. See Table AA	Positive detection	nd	Positive detection	No sample collected
Surface grab, GC analysis. See Table AA	No sample collected	No sample collected	No sample collected	No sample collected
Underwater SPME, MityVac. See Table BB	System not used	System not used	System not used	System not used
Surface SPME, flow-through. See Table BB	Positive detection	nd	nd	nd
Sediment samples, GC analysis. See Table CC	No sediment available	No sediment available	No sediment available	No sediment available

Data table continuation

Sample Site: Claire Lily

Sample Identifier: D3

Likely ID – 250 lb bombs

All results are in parts-per-billion

Water samples, GC Analysis with ECD detection	0.3 meters from shell	0.3 meters from shell	2 meters from shell
Sampling method	Underwater grab, sample 1	Underwater grab, sample 2	Underwater grab
2,6 DNT	nd	nd	nd
Dinitrobenzene	nd	nd	nd
2,4 DNT	nd	nd	0.05
TNT	0.07	nd	nd
TNB	nd	nd	nd
4-AM-DNT	nd	0.55	nd
2-AM-DNT	nd	0.4	nd

Table AA. Sample D3 water analysis by GC

Water samples, SPME / IMS analysis	1 meter from shell
Sampling Method	Surface SPME
2,6 DNT	nd
Dinitrobenzene	nd
2,4 DNT	nd
TNT	nd
TNB	nd
2- or 4-AM-DNT	trace

Table BB. Sample D3 water analysis by SPME

Sediment samples, GC Analysis with ECD detection	0.3 meters from shell
2,6 DNT	121.3
Dinitrobenzene	nd
2,4 DNT	nd
TNT	0.37
TNB	1.73
4-AM-DNT	nd
2-AM-DNT	nd

Table CC. Sample D3 sediment analysis by SPME



Sample Site: Claire Lily

Sample Identifier: M1

Likely ID – 8 or 9 inch shell

All results are in parts-per-billion

Summary of method and detection				
Method	0.3 meters from shell	1 meter from shell	2 meters from shell	3 meters from shell
Underwater grab, GC analysis. See Table DD	Positive detection	nd	Positive detection	nd
Surface grab, GC analysis.	nd	nd	nd	nd
Underwater SPME, MityVac	System not used	System not used	System not used	System not used
Surface SPME, flow-through. See Table EE	nd	nd	nd	nd
Sediment samples, GC analysis. See Table EE	Positive detection	nd	nd	nd

Data table continuation

Sample Site: Claire Lily

Sample Identifier: M1

Likely ID – 8 or 9 inch shell

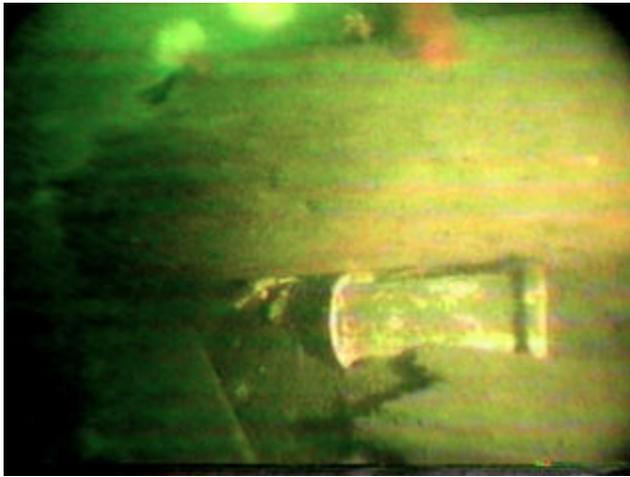
All results are in parts-per-billion

Water samples, GC Analysis with ECD detection	0.3 meters from shell	2 meters from shell
Sampling method	Underwater grab, sample 1	Underwater grab
2,6 DNT	2.0	1.72
Dinitrobenzene	nd	nd
2,4 DNT	0.16	nd
TNT	nd	nd
TNB	nd	nd
4-AM-DNT	nd	0.97
2-AM-DNT	nd	0.64

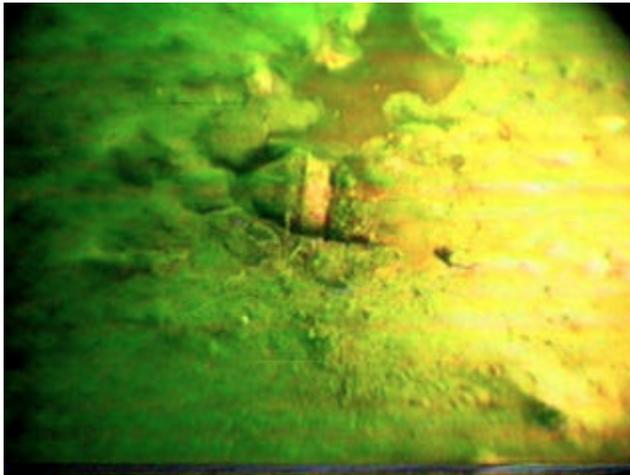
Table DD. Sample M1 water analysis by GC

Sediment samples, GC Analysis with ECD detection	0.3 meters from shell
2,6 DNT	11.45
Dinitrobenzene	14.25
2,4 DNT	12.57
TNT	0.13
TNB	2.42
4-AM-DNT	nd
2-AM-DNT	0.39

Table EE. Sample M1 sediment analysis by GC



Sample Site: Rent Point
Sample Identifier: B1
Likely ID – unknown
No detections



Sample Site: Rent Point
Sample Identifier: D2
Likely ID – 4 inch shell
No detections



Sample Site: Rent Point
Sample Identifier: D2
Likely ID – 250 lb bomb
No detections

Trongate depression:

No photos are available due to the cloudiness of the water. Two targets were sampled. Their condition was unknown (possibly broken open). No signatures were detected. The ability to pump water from a depth of 30 meters for sampling was demonstrated.

Black Rock Point

A large ship carrying cordite (nitrocellulose pellets containing nitroglycerine and petroleum jelly) pellets went aground at black rock point and spilled its cargo. The cordite (approx. 1/8-inch diameter by 1/2 to 3/4 inch long) pellets are washing onto the beach and are present in the water. We were asked to sample the beach area and the water near the beach to see if any nitroglycerine was leaching into the water. Ten samples were taken and analyzed. Nitroglycerine was not detected. Laboratory tests using cordite pellets from this beach showed that they did not leach nitroglycerine. Dissolution and extraction indicated that essentially all of the nitroglycerine had been depleted. The cordite pellets would burn vigorously when lit with a match. This behavior is presumably due to the remaining nitrocellulose.

Blanks

During the course of the sampling period, 8 sediment blanks were taken as were 15 water blanks. No explosives were detected in any of the blanks.

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