Comparative sampling methodologies for detecting and quantifying 2,4,6 trinitrotoluene post-blast traces in water

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ABSTRACT

This study addresses the analytical challenges associated with recovering explosive residues, focusing on the identification of 2,4,6-trinitrotoluene (TNT) in water samples. It evaluates the practicality, efficiency, and representativeness of three sampling methodologies: traditional grab sampling (GS), composite sampling (CS), and 3-D multi-increment sampling (3D-MIS). High-Performance Liquid Chromatography (HPLC) was employed for explosive identification. Post-blast sampling of TNT residues from high-order and low-order deflagrations was conducted to assess each method's efficacy and limitations in detecting trace and bulk contaminations. The experiments were conducted at the Alford Technologies Group range in Broadmead, UK, with analysis performed at the Defence Academy in Shrivenham, UK. Key findings highlight the varying effectiveness of each sampling method, with implications for enhancing detection sensitivity and accuracy in post-blast scenarios. This study underscores the importance of selecting appropriate sampling strategies tailored to different contamination scenarios, thereby informing more effective response protocols in CBRNE incidents involving water environments.

1.0 INTRODUCTION

Sampling and analysis play a crucial role across industries to monitor workplace pollutants and ensure compliance with health and safety procedures. In the context of Chemical, Biological, Radiological, Nuclear, and high-yield Explosives (CBRN(e)) incidents, effective chemical analysis becomes even more important as it provides conclusive evidence regarding the presence or absence of hazardous agents (Andrewes, 2023; OPCW, 2024; Petersen et al., 2005). Despite international prohibitions, such as the Chemical Weapons Convention (CWC), the use of toxic nerve agents and explosives remains a significant threat, as demonstrated in recent conflicts and domestic incidents, such as the Salisbury poisoning (de Koning et al., 2023; OPCW Technical Secretariat, 2018). Thus, effective identification, recovery, and remediation of hazardous chemicals are essential for mitigating exposure risks to both civilians and military personnel.

However, one of the key challenges in CBRN(e) incidents is ensuring that sampling methodologies are accurate, representative, and cost-effective. Contaminants, especially in explosive residues, are often distributed unevenly across the environment, making it difficult to achieve consistent results with traditional sampling methods (Walsh et al., 2014). Ideally a sample should represent the actual distribution and composition of contaminants, but heterogeneity due to distribution and composition, can introduce significant errors (Walsh et al., 2014). Accuracy in sampling is emphasised by the North Atlantic Treaty Organisation (NATO), which recommends standard operating procedures (SOP), rules of procedure (ROP), and quality control (QC) measures such as field blanks (NATO, 2015). However, the sampling method itself plays a crucial role in ensuring accurate results.

Grab sampling, commonly used for its simplicity and low cost, is often the least representative method due to its failure to address the heterogeneous nature of contamination (Minkkinen & Esbensen, 2009). While grab sampling remains the preferred approach for water collection according to the NATO's Sampling and Identification of Biological, Chemical, and Radiological Agents (SIBCRA) guidance (NATO, 2015), its

limitations highlight the need for alternative strategies. Composite sampling, which combines multiple samples into a single bulk sample, improves efficiency but can reduce detection sensitivity, especially in liquid samples. Nonetheless, this drawback can be mitigated by using filtration techniques (France et al., 2015). Composite sampling has proven effective in producing representative results in CBRN(e) scenarios without compromising data integrity (France et al., 2015; Patil, 2011). The most representative method, multi-increment sampling (MIS), systematically collects samples across a grid, forming a pooled sample that captures spatial heterogeneity. (Walsh et al., 2014). When conducted in three dimensions, MIS provides a comprehensive assessment of contamination, making it the most effective method for representing explosive residues in water.

The aim of the study is to evaluate the effectiveness, reliability, and representativeness of these three sampling methodologies for detecting both trace and bulk contamination in water following explosive detonations and deflagrations. The specific objectives are (i) to evaluate the performance of these sampling methods in capturing the distribution and composition of hazardous residues in heterogeneous environments; (ii) to assess the effectiveness of both high-order detonations (which produce underwater blasts) and low-order deflagrations (which minimise the environmental impact but have limited available data to date); and (iii) to provide recommendation on the most suitable sampling approach for accurate and comprehensive contamination assessments in CBRN(e) scenarios.

2.0 MATERIALS AND METHODS

2.1 Experimental set up

Six pipe bombs were manufactured using seamless mild steel S235 J tubes supplied by Precision Profiles, each filled with 100 g TNT and 20 g PENO (80% Pentaerythritol tetranitrate (PETN) and 20% marker) donor fill. Three devices were detonated as high order explosions and three as low order deflagrations. Each was placed in 1000 L water filled intermediate bulk containers (IBC). Following detonation or deflagration, and after the area was declared safe, samples were collected in order of ease of sampling. Therefore, grab samples were collected and sample split in situ to form the composite sample first, then the multi-increment samples were taken. The experiment was carried out at Alford Technology's explosive range in Broadmead, UK. To prevent pressure, build up, the IBC roofs were removed using a handsaw. To enhance water collection post-detonation/deflagration in the IBC, four commercially available paddling pools were placed around the elevated IBC (Figure 1). The 1000 mm x 1160 mm x 1200 mm IBCs were constructed from a galvanized steel cage, with a high-density polyethylene body.

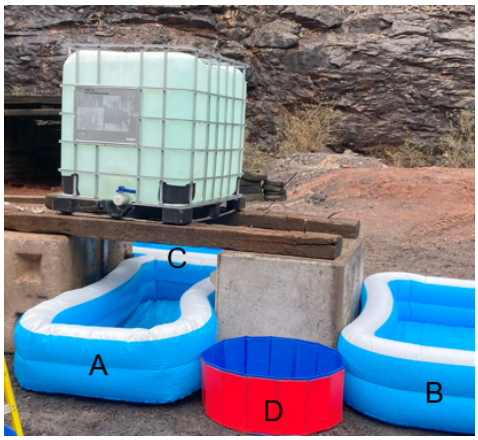

Figure 1: Experimental set up at Alfords range showing all four paddling pools A, B, C and D as well as the IBC filled with 1000 L of water suspended on top of two solid wood beams.

To achieve low order deflagration, the Vulcan method, used by the British military, was used (Parliament, 2020). The method utilises a Magnesium Jet Forming Cone disc activated by 20 g PENO donor charge and a Davey Bickford Electric Detonator. The Vulcan device was taped to the pipe bomb using steel leg attachments and electrical tape. The assembly, including the pipe bomb with attached Vulcan, was suspended centrally within the IBC using wire, aiming to evenly distribute the blast force and minimise damage to the IBC with equal amounts of water on each side (Figure 2).

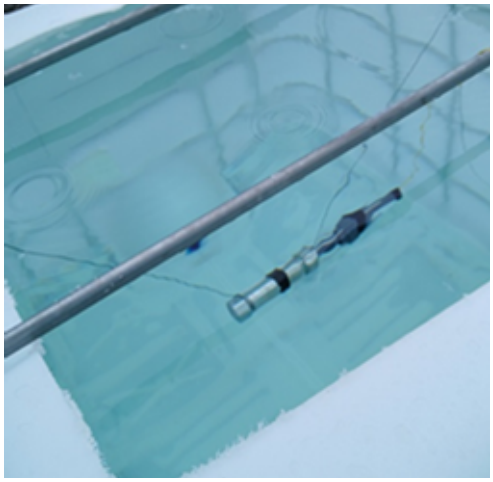

Figure 2: Pipe bomb suspended in 1000 L IBC tank for low order deflagration.

To initiate the high order detonations, one cap of each pipe bomb was removed and 20 g of PENO, a PETN based plastic explosive, was placed directly in contact with the TNT (Figure 5, left image). The detonator was then directly placed into the PENO. Like the LO disposals, the pipe bomb was placed as centrally within the IBC as possible (Figure 3, right image).

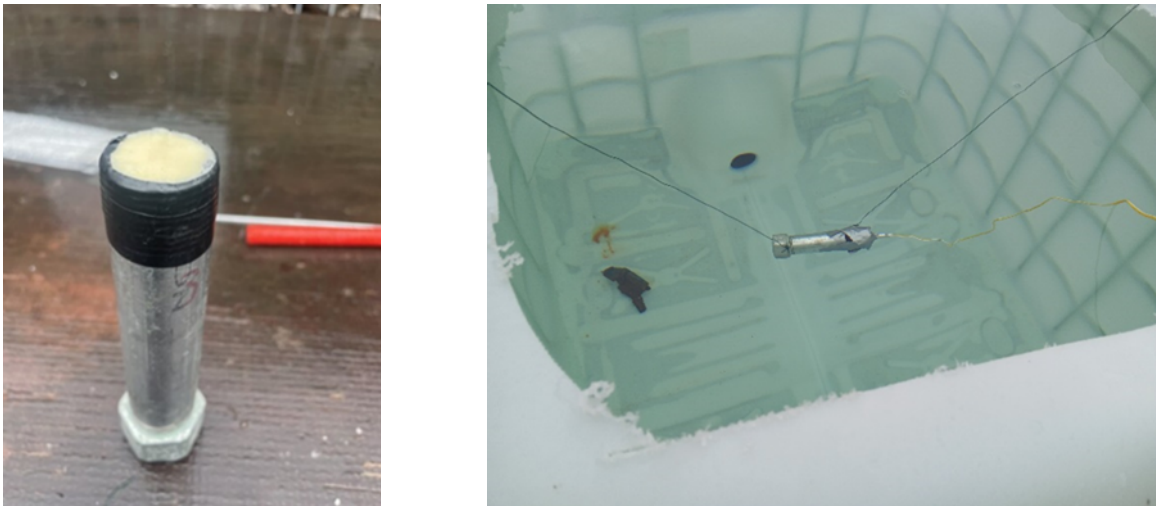

Figure 3: Pipe bomb rigged for high order detonation with PENO donor charge (left panel) and suspended pipe bomb in IBC tank for high order detonation with detonator attached (right panel)

2.2 Composite and grab sampling

Sampling involved composite and grab techniques using sample splitting which are endorsed by institutions such as the OPCW and NATO. This method allows the reduction of sampling materials required. Essentially, grab samples were split into two portions: one retained as an individual grab sample and the

other used to create a composite sample. To execute this, 100-ml grab samples were split immediately after collection (Figure 4). One 50-ml aliquot was stored in a 50-ml amber vial as an individual grab sample for hazard mapping. The remaining 50-ml aliquot was placed in a 500-ml amber bottle to form the composite sample. Sampling occurred promptly after detonation once safety was assured.

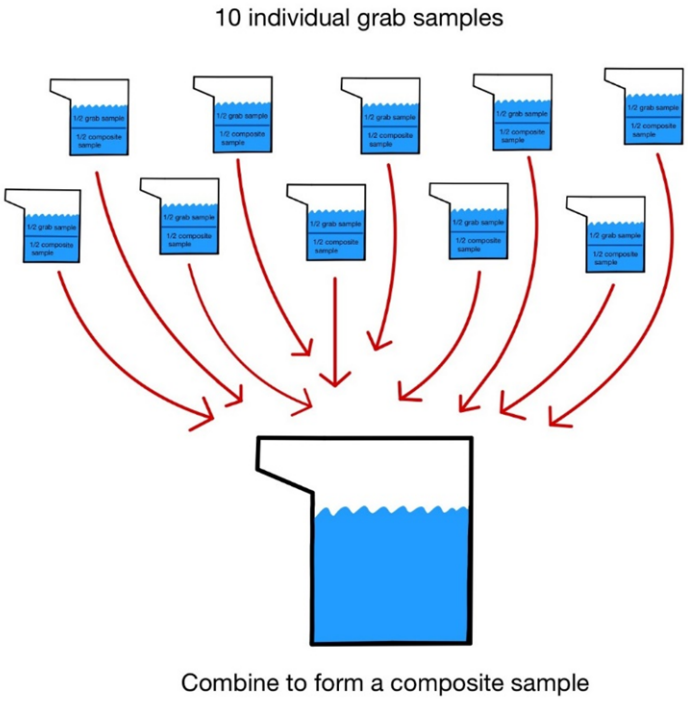

Figure 4: Overview of the composite and grab sampling

Random selection of the grab samples was determined by the sampler rather than utilising an algorithm or software, reflecting common procedures in CBRN (e) protocols. Duplicates were taken resulting in 20 grab samples and two composite samples, each composed of 10 subsamples, collected for each of the three detonations and three deflagrations. Six of these samples were collected from the surface of the IBC and one sample was taken each from the four paddling pools to make up the 10 grab samples and composite sample (Figure 1).

2.3 3-D multi-increment sampling (3-D MIS)

3-D multi-increment sampling (3-D MIS) was employed by sectioning the lot within the metal cage surrounding the IBC into 12 sub-lots across two layers. Each sub-lot was sampled from approximately the same positions (Figure 5). In total, 12 subsamples, six from layer one and six from layer two, were collected. All samples were taken in duplicates.

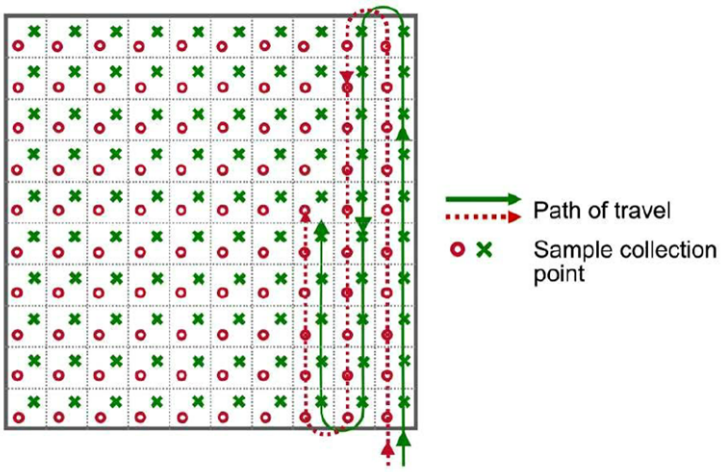

Figure 5: Illustration of multi-increment sampling (Walsh,2014).

2.4 Quality Control measures

Procedural blank controls, also known as field blanks, were collected at the range. Additionally, background samples were collected before detonations and duplicate samples post detonations to prevent contamination during sampling, transportation (NATO, 2015). For quality control, NATO guidelines recommend including a field duplicate, a field blank, and a background sample for every ten samples collected (NATO, 2015).

2.6 HPLC analysis

The HPLC used is Agilent 1260 with a limit of detection (LoD) > 1ppm. To ensure quick, efficient analysis a HPLC method was set up to allow fast analysis at 3 minutes with a flow rate of 1.50 ml and an injection volume of 10µl. The 3-minute runs allowed for the TNT (RT ≈ 2.4 min) to be detected. A C18 column from Agilent was used in reverse phase with a mobile phase made up of 60:40 ACN: Water. To facilitate external calibration for qualitative analysis, internal reference standards of TNT concentrations 5, 10, 20, 30, 40 and 50 ppm were freshly manufactured and filtered before analysis. To ensure the proportion of variance was acceptable, standard curves were only accepted if the R-squared was above 0.99. Each HPLC was set up to run the set of standards first, then followed by the samples. The collected QC samples, duplicate, blank and background samples, were included and analysed alongside. Following each complete run, two washing steps were completed taking approximately 20 minutes. One with water and one with acetonitrile.

3.0 RESULTS AND DISCUSSION

3.1 Optimisation of sample preparation for HPLC analysis

To determine the optimal sample preparation method for HPLC analysis, an initial experiment was carried out to compare two mobile phase compositions as follows: 100% water and 50:50 Acetonitrile (ACN): Water. Each comparison utilised a single grab sample from each IBC. The results demonstrated that filtering the water samples through a 0.2 µm nylon filter substantially enhanced the detection of low concentrations of TNT (Table 1). Further to this, LO deflagrations result in significantly higher residual concentrations compared to HO detonations (Table 1). Specifically, the mean concentrations for LO deflagrations ranged from 6.12 to 22.9 ppm, whereas the mean concentrations for HO detonations ranged between 1.4 and 2.16 ppm (Table 1). Notably, the concentrations in high order 'E' samples were below the limit of detection (LoD) of the HPLC and could not be quantified through external calibration. However, a TNT peak was detected in the chromatogram, confirming the presence of the analyte. These findings indicate that as the

concentration of the analyte is close to the LoD of the analytical equipment, a greater number of samples are necessary to ensure reliable detection.

Table 1: Comparison of the two sample preparation methods

Detonation type	Samples	Sample prep methods	Concentration (ppm)	Retention time (RT)
LO	A	Filtration	5.067	2.317
LO	B	Filtration	10.725	2.319
LO	D	Filtration	22.439	2.321
HO	C	Filtration	2.139	2.499
HO	E	Filtration	2.137	2.241
HO	F	Filtration	2.136	2.243
LO	A.1	Dilution	6.569	2.318
LO	B.1	Dilution	14.817	2.319
LO	D.1	Dilution	23.137	2.315
HO	C.1	Dilution	0.000	/
HO	E.1	Dilution	0.000	/
HO	F.1	Dilution	0.000	/

For samples C.1, D.1, E.1 no residual concentration of TNT was detected, as increasing the volume of samples with low concentrations, lowers the sensitivity of detection and produced false negatives. The results further indicated that significantly higher residues persisted following LO deflagrations compared to HO detonations. Specifically, TNT concentration ≥ 5 ppm remained after LO detonations. For subsequent analyses, all samples were subjected to filtration using a 0.2 µm nylon filter.

3.2 Grab samples

Twelve grab samples were collected in two batches of six each. The samples were stored at 4°C until analysis. Before extracting triplicate subsamples, sampling vials were inverted three times to mitigate any distributional heterogeneity. Subsamples were taken using single use plastic syringes and filtered using 0.2µm Nylon filters before storing at 4°C until HPLC analysis. Out of 36 samples analysed for all three LO deflagrations (A, B and D), all samples were found to contain TNT concentration above 5 ppm (Figure 7). A large disparity between residual concentrations was detected between the three firings with average concentrations of 4.55 ppm, 8.92 ppm and 22.05 ppm for A, B and D respectively. Firing A was induced using a Vulcan, and intended to fire as a HO. Consequently, firing B and D were fired without a Vulcan device. The three firings show a high variability between the degree of low order firings. Reproducibility of LO deflagrations are difficult to achieve (Pennington et al., 2008).

Comparatively, for the post HO detonations (C, E and F), only 44% of the grab samples from HO 'C', 72% from HO 'E' and 53% from HO 'F' could be integrated using the HPLC software. However, concentrations could only be calculated for HO 'C' and 'E' (Figure 7). Average concentrations were 1.28 ppm, 1.59 ppm and below the LoD for firings C,E and F respectively, indicating that more than 98% of the TNT was consumed in the detonation.

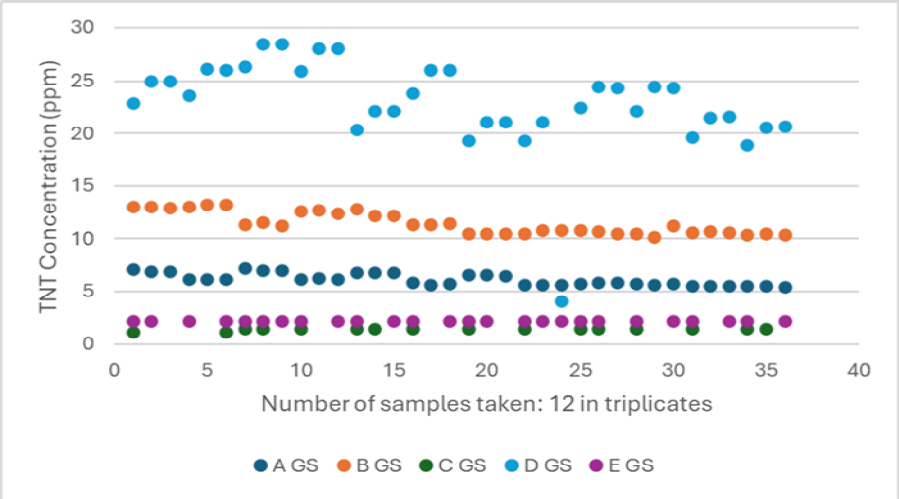

Figure 7: Comparison of all grab samples (GS) taken across all six firings within the intermediate bulk container (IBC) and paddling pools

The results indicated that grab samples collected from the paddling pools exhibited a higher variance in TNT concentrations compared to those obtained from the IBC (Figure 8). This variability suggests that environmental factors, such as water movement and sedimentation processes, may influence the distribution of contaminants in the paddling pools. Additionally, the overall concentration of TNT in the paddling pool samples was significantly lower than that found in samples taken from the IBCs.

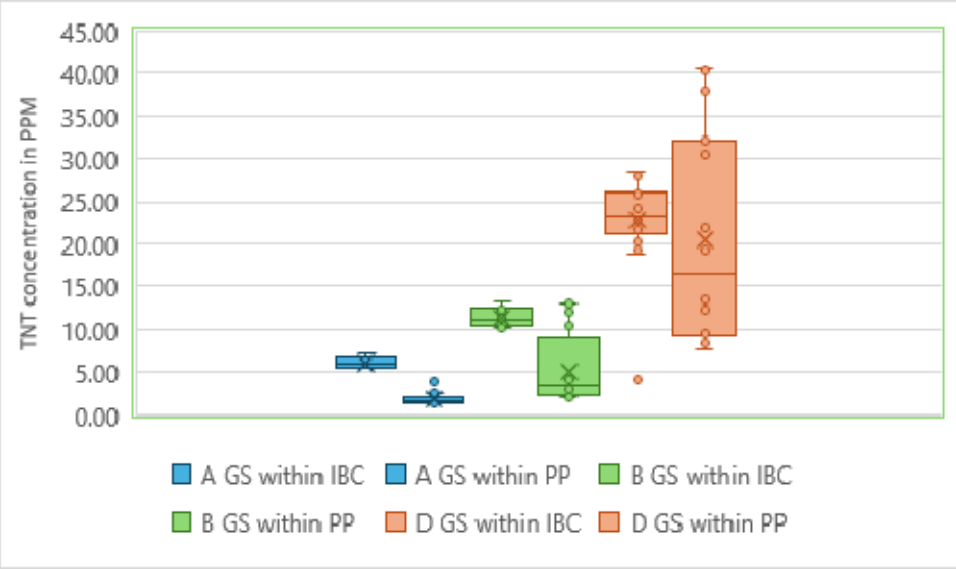

Figure 8: Visual comparison of grab samples (GS) taken within the intermediate bulk container (IBC) and within paddling pools (PP) for all low orders (LO).

Furthermore, it was observed that the higher the residual concentration post-clearance operation correlated with increased variance in the grab samples both inside the IBC and in the paddling pools (Table 2).

Table 2: Concentration of grab samples (GS) taken within the IBC and paddling pools

Conditions	Intermediate bulk container		Paddling pools	
	Mean (ppm)	SD	Mean (ppm)	SD
LO-A	6.12	0.57	4.55	2.16
LO-B	11.41	1.02	8.92	4.08
LO-D	22.9	4.25	22.05	7.38
HO-C	1.4	0.11	1.28	3.84
HO-E	2.16	0.01	1.59	1.04
HO-F	-0.22	0.06	-0.024	0.05

This discrepancy in concentration may be attributed to several factors, including dilution effects from surrounding water and potential degradation of TNT over time. The increased variance in TNT levels could also indicate the heterogeneous nature of contamination in the paddling pools, where localised hotspots may exist due to the uneven deposition of residues (Figure 9).

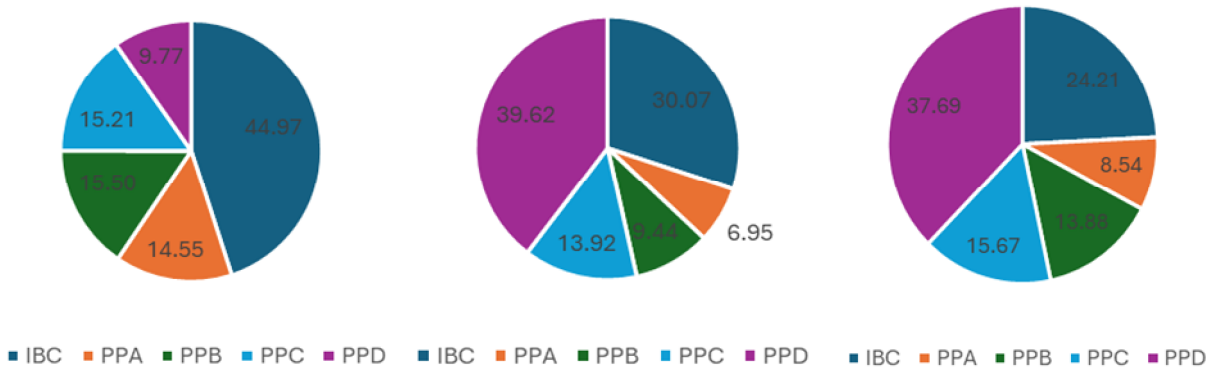

Figure 9: % concentration recovery of TNT from all low order firings from paddling pools A, B, C, D and IBC A, B and D

The distribution of TNT concentrations in the four paddling pools and IBCs A, B, and D, respectively is shown in Figure 9. For example, samples from paddling pool A had concentrations of 9.77%, 39.62%, and 37.69%, indicating that the distribution of TNT varies across the three firings. This variability is evident throughout the results. No definitive conclusions can be drawn regarding the heterogeneous distribution of the post-blast samples, as other contributing factors, such as interactions with the plastic of the paddling pools, cannot be ruled out. This suggests that there is not a single optimal location for sample collection; rather, a series of sampling locations is recommended to obtain a comprehensive overview of the contamination.

3.3 Composite samples

Given that 40% of the composite sample originated from the paddling pools, the results exhibited a notable bias that may have influenced overall concentration assessments. To address this potential bias, the samples underwent a triplicate subsampling process after the composite bottle was inverted three times to ensure uniform distribution. Following this, the samples were subjected to the same preparation protocol, including filtration and refrigeration, until HPLC analysis could be performed. Concentration calculations were only feasible for the LO samples (Table 3), aligning with existing literature that suggests such findings are common in similar studies (France et al., 2015). This observation emphasises the importance of careful sample handling and preparation, as well as the need to consider the impact of source variability on

analytical outcomes. The bias introduced by the paddling pool samples warrants further investigation to fully understand its implications on contamination assessments.

Table 3: Mean and standard deviation of composite samples.

IBC	Low/High order	Sampling method	Mean (ppm)	SD
A	LO	CS	3.67	0.9
B	LO	CS	9.11	0.82
D	LO	CS	20.31	2.16
C	HO	CS	-0.28	0
E	HO	CS	-0.26	0.02
F	HO	CS	/	/

Thus, a significant limiting factor is the additional dilution of trace contaminants following HO detonations. Nevertheless, composite sampling is a viable method for estimating overall contamination levels, particularly when dealing with LO deflagration (Figure 10). This approach effectively captures the distribution of contaminants, thereby providing a more accurate assessment of contamination levels in environments affected by explosive incidents.

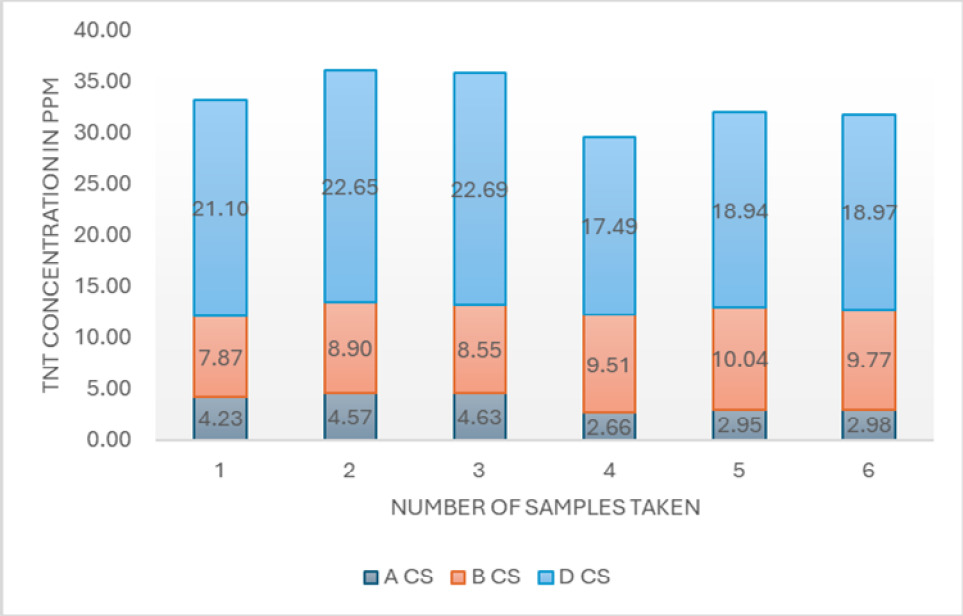

Figure 10: Comparison of duplicate composite sampling of triplicates (CS) for all low order (LO) deflagrations.

A clear trend was observed, indicating that as the concentration of contaminants increased in post-deflagration samples, there was a corresponding rise in the standard deviation (Figure 11). This suggests that higher contaminant levels are associated with greater variability in sample measurements, potentially due to the heterogeneous distribution of residues in the environment.

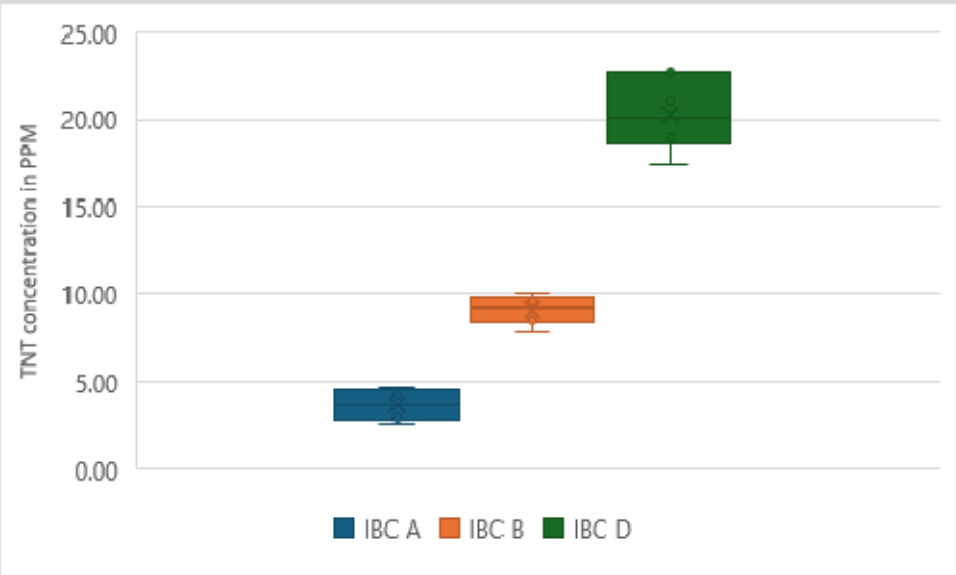

Figure 11: Distribution of composite sample concentrations of the low order deflagrations on IBC tank.

3.4 3-D multi-increment sampling

3-D MIS was conducted exclusively for the LO deflagrations, as the HO detonation left only a quarter of the water in the split IBC, rendering it an inadequate representation of the total water body (Figure 12).

Figure 12: Destruction of IBC post high order detonation

MIS, being the most labour-intensive method assessed in this study, demands extensive planning, specialised equipment, and significant time investment. This contrasts with the urgency of typical CBRN(e) incidents, highlighting a key limitation of MIS for rapid deployment in such scenario. Despite these challenges, the results suggest that spatial sampling methods, like 3-D MIS, warrant further investigation for accurately representing contamination in large bodies of water. Interestingly, one of the three deflagration experiments exhibited substantial variance between samples. Specifically, deflagration 'D' showed a high SD ± 10.7 ppm, indicating significant variability and questioning the reliability of the calculated concentration in comparison to the other two deflagrations and their SDs of ±1.7 ppm and ±0.7 ppm for deflagration ‘A’ and ‘B’ respectively. This large variance notably skewed the mean values, emphasising the need for careful consideration of sampling methods when dealing with heterogeneous contamination (Table 4, Figure 11).

Table 4: Mean and standard deviation of all multi-increment samples taken in the low order deflagration from IBC tanks A, B, D.

IBC	Sampling method	Mean (ppm)	SD
A	MIS	10.76	1.7
B	MIS	14.83	0.7
D	MIS	32.03	10.7

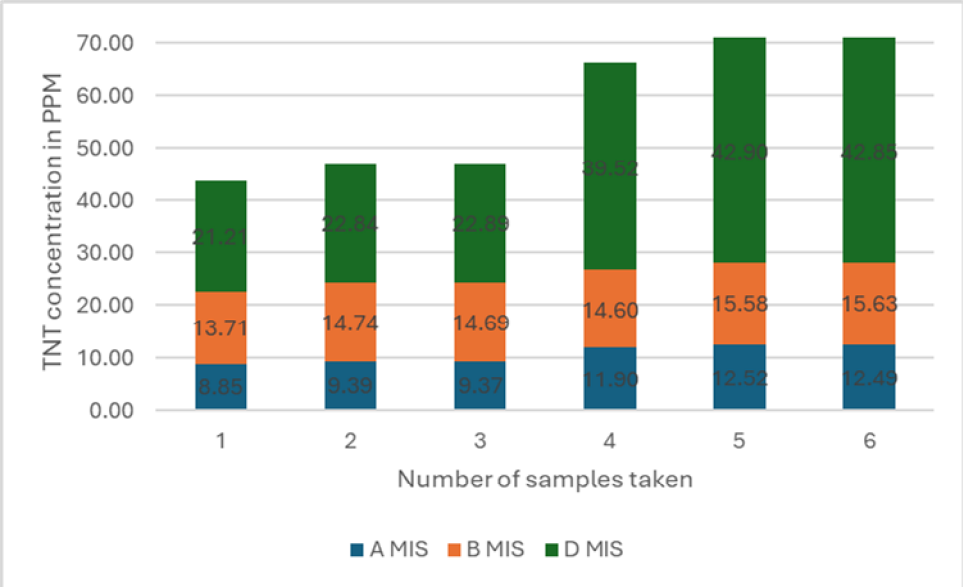

Figure 11: TNT concentration across the duplicate multi-increment samples (MIS) taken for all low order (LO) deflagration (average value from triplicates).

3.5 Overall comparison

Generally, the CS showed the lowest concentration, while the GS was found to be in the middle and the MIS showed the highest detected concentrations of TNT (Table 5). This disparity can be attributed to the three-dimensional sampling approach across two layers. The absence of spatial sampling in CBRN (e) incidents could therefore be a potential concern. However, it is crucial to acknowledge that this comparison involves 36 samples versus 6 samples and 6 samples, introducing a potential source of error that should be noted for clarity to the reader.

order (LO) deflagration 'A, B and D'.Table 5: Comparison of grab (GS), composite (CS) and multi-increment sampling (MIS) mean and standard deviation for high-order (HO) detonation and low-order (LO) deflagration.

Type of detonation	IBC	Sampling method	Mean (ppm)	SD
LO	A	GS	6.12	0.57
LO	A	CS	3.67	0.9
LO	A	MIS	10.76	1.72
LO	B	GS	11.41	1.02
LO	B	CS	9.11	0.82
LO	B	MIS	14.83	0.71

LO	D	GS	22.9	4.25
LO	D	CS	20.31	2.16
LO	D	MIS	32.03	10.74
HO	C	GS	1.4	0.11
HO	C	CS	-0.28	0
HO	C	MIS	/	/
HO	E	GS	2.16	0.01
HO	E	CS	-0.26	0.02
HO	E	MIS	/	/
HO	F	GS	-0.22	0.06
HO	F	CS	/	/
HO	F	MIS	/	/

4.0 CONCLUSION

This study highlights the importance of selecting appropriate sampling methodologies for CBRN(e) recovery efforts, emphasizing the need to align the sampling strategy with the specific objectives and environmental context. While grab sampling remains essential for rapid hazard mapping, its inherent variability makes it prone to significant errors, necessitating a minimum of triplicate analysis to mitigate false negatives. The study highlights that as analyte concentrations approach the LoD of the HPLC, an increased number of samples is required for accurate representation, making GS less reliable for post-blast contamination assessment in aqueous environments. CS offers a more efficient alternative by consolidating multiple samples into a single analysis, reducing time and resources. However, this method can be limited by its inability to accurately recover low concentrations of residues without additional pre-concentration steps, as demonstrated in cases where samples from the paddling pools diluted the true concentration. Thus, while composite sampling is useful for broad contamination assessments, it may not be suitable for detecting trace amounts of hazardous materials. 3D-MIS showed the highest recovery of TNT indicating that it is the most effective method for capturing the heterogeneous dispersion of contaminants in bodies of water. Despite its higher complexity and resource requirements, MIS offers superior accuracy in representing true contamination levels, particularly in spatially heterogeneous environments. However, its logistical demands and the need for extensive planning limit its practicality in time-sensitive CBRN(e) incidents. The main distinction between CS and 3D-MIS lies in the systematic versus random collection approach. While GS and CS rely on randomness, MIS adopts a systematic approach, proving more effective in providing comprehensive post-blast water concentration profile. The study also highlights the risk of false negatives in grab and composite sampling, especially at low concentrations, and stresses the critical need for comprehensive training for first responders to improve sampling strategies in heterogeneous environments. Finally, this research points to the limitations of current assumptions of homogeneity in environmental sampling and advocates for further exploration of multi-increment techniques. Future studies should ensure triplicate sampling at all experimental stages to enhance the reliability and confidence of results, especially in complex CBRN(e) recovery scenarios.

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