ESTCP
Cost and Performance Report

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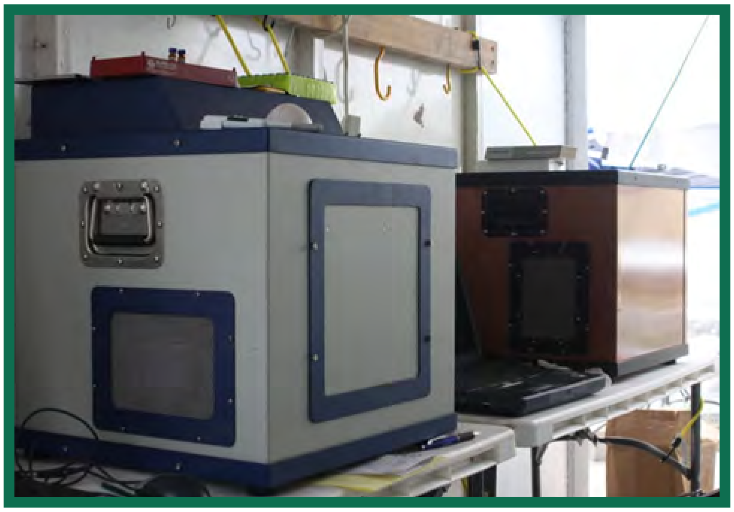

Field Portable GC-MS Unit for Semi-Volatile Compound Analysis in Groundwater

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ESTCP

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ACRONYMS AND ABBREVIATIONS

1,3-DNB	1,3- dinitrobenzene
2,4-DNT	2,4-dinitrotoluene
3,4-DNT	3,4-dinitrotoluene
bgs	below ground surface
CIT	cylindrical ion trap
CPS	counts per second
DI	deionized water
DoD	U.S. Department of Defense
ECB	Environmental Chemistry Branch
ERDC	Engineer Research and Development Center
ESTCP	Environmental Security Technology Certification Program
GC	gas chromatograph
GC-ECD	gas chromatograph-electron capture detector
GC-MS	gas chromatograph-mass spectrometer
HPLC	high performance liquid chromatography
LAAP	Louisiana Army Ammunition Plant
LAP	loading, assembling, and packing
LCC	life-cycle costs
LCS	laboratory control sample
LC-S	laboratory control-spike
LTM	low thermal mass
MAAP	Milan Army Ammunition Plant
MC	munitions constituents
MDL	method detection limit
MS	mass spectrometry
MS/MS	tandem mass spectrometry
MSD	mass selective detector
MSL	mean sea level
NB	nitrobenzene
NPV	net present value
NS	net savings
PAH	polyaromatic hydrocarbon
PCB	polychlorinated biphenyl
QA	quality assurance

ACRONYMS AND ABBREVIATIONS (continued)

QC	quality control
QSM	Quality Systems Manual
RDX	hexahydro-1,3,5-trinitro-1,3,5-triazine
RPD	relative percent difference
SIM	selected ion monitoring
SPE	solid phase extraction
SPME	solid phase micro extraction
SUV	sport utility vehicle
TNB	1,3,5-trinitrobenzene
TNT	2,4,6-trinitrotoluene
USEPA	U.S. Environmental Protection Agency
UV	ultraviolet

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1.0 EXECUTIVE SUMMARY

1.1 BACKGROUND

Periodic groundwater sampling is often required as part of a long-term monitoring program. Traditional sampling and analytical techniques require shipping multiple liters of water to fixed laboratories that perform regulatory-approved analytical methods. The typical analysis and data reporting time at most analytical laboratories can be up to 45 days, which delays vital information on contaminant concentrations being reported to the customer. Additionally, most sample holding times have been tested for a small set of environmental matrices where the assumption has been made that analyte concentrations will not change significantly if analyzed within this window, typically 7 to 40 days (Jenkins et al., 1995a; Jenkins et al., 1995b; Jenkins and Grant, 1987). The use of a field portable gas chromatograph-mass spectrometer (GC-MS) alleviates these concerns.

1.2 OBJECTIVES OF THE DEMONSTRATION

The objective of this ESTCP demonstration was to 1) demonstrate the suitability of field analysis for a suite of contaminants of concern (semivolatile munitions constituents) and 2) demonstrate the utility, comparability, and cost savings of groundwater analysis using the Griffin 450 GC-MS.

This effort was designed to demonstrate the advantages and limitations of field-portable analytical instrumentation for the detection and quantification of munitions constituents in groundwater, which eliminates the need to ship water samples overnight, under chain of custody, to a fixed analytical laboratory. Specifically, we tested the Griffin 450 GC-MS and compared the in-field results to traditional munitions constituents (MC) analysis using laboratory-based high performance liquid chromatography (HPLC) with ultraviolet (UV) absorbance detection following U.S. Environmental Protection Agency (USEPA) Method 8330.

1.3 TECHNOLOGY DESCRIPTION

Mass spectrometry (MS) analysis systems can provide valuable chemical information on almost any type of sample. Traditionally, MS has been confined to fixed-site laboratory analysis due to the size and fragility of the instruments typically employed for this application. Griffin has made efforts toward miniaturization, enabling this technology to be brought to the field to perform analysis. The Griffin instruments use a cylindrical ion trap (CIT) as the mass analyzer; this device is a simplified geometry of the classic hyperbolic ion trap and therefore more easily miniaturized. The Griffin instruments also use a low thermal mass (LTM) gas chromatograph (GC) as the GC. With a smaller ion trap, the vacuum manifold becomes smaller, and the resulting pumping and power requirements are reduced. The LTM GC column eliminates the need for a convective oven, greatly reducing the size and power consumption compared to standard GC systems. These modifications to the instrument design all serve to decrease the size and weight of the instrument. Griffin has also worked to ruggedize the instrument, enabling transport into the field for on-site analysis. The improved electronic stability and sensitivity of the Griffin 450 provided higher quality data, especially in humid environments, compared to the previous Griffin 400 model GC-MS.

1.4 DEMONSTRATION RESULTS

The instrumentation was tested on 28 groundwater samples from two distinct field sites for a variety of analytes with concentrations ranging over three orders of magnitude. The compounds evaluated were: nitrobenzene (NB), 1,3-dinitrobenzene (1,3-DNB), 2,4-dinitrotoluene (2,4-DNT), 1,3,5-trinitrobenzene (TNB), 2,4,6-trinitrotoluene (TNT) and Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX). Split groundwater samples were collected and analyzed for these compounds to compare the results from a field portable GC/MS method to the results from a conventional fixed laboratory method. Detection limits for the field portable instrumentation are sufficient to meet regulatory threshold levels, generally around 0.002 mg/L. Linear regression comparison of the in-field results to traditional laboratory-based analysis suggests comparability between the techniques, with the slope of the regression for all analytes being between 0.8 and 1.2, except for TNB and RDX. However, the slope of the regression for RDX is between 0.8 and 1.2 for all concentrations below 10 mg/L.

As all of the paired results for NB were non-detects, only a limited evaluation was possible. However, the NB results were consistent in that both the field and laboratory methods reported non-detects for NB for all split sample analyses. The field method for RDX possessed a negative bias relative to the fixed laboratory method and exhibited relatively large variability across all concentration ranges evaluated. The field results were about 70% of the laboratory results on the average. Therefore, it is recommended that the field method be used to obtain only screening-level data for RDX. The field and laboratory results were essentially equivalent for concentrations less than or equal to 0.3 and 0.2 mg/L for 1,3-DNB and 2,4-DNT, respectively. The comparison was limited by the relatively small data set owing to several non-detects and the relatively small concentration range evaluated (about 0.01 – 0.1 mg/L). Results for TNT were reliable for screening only below a concentration of 0.05 mg/L; however, between 0.05 and 10 mg/L, results from the field and laboratory were equivalent. The field method consistently exhibits a significant positive bias for TNB (F=1.5 L). There was a very strong correlation between the laboratory and field methods for concentrations greater than about 0.05 mg/L to the highest reported concentration, but the performance of the field method was relatively poor at smaller concentrations. The TNB field results >0.05 mg/L would need to be adjusted for bias prior to being reported.

The results indicate that similar reporting limits can be obtained using the field-portable instrument when coupled to solid phase extraction (SPE) sample preparation, although instrument stability at the low concentration range can be an issue. Furthermore, the linear dynamic range is somewhat limited, as compared to HPLC analysis, for samples with high analyte concentrations.

The cost savings of the field method were found to be $29,600 a year, based on 12 week-long field trips per year, with a breakeven point of 3.54 years.

1.5 IMPLEMENTATION ISSUES

Field portable GC-MS appears at this point to be suitable only for screening RDX, due to significant scatter in the comparison to laboratory results across the concentration range tested. The regression line data demonstrate that the slope is within our 0.8 to 1.2 limit except for TNB and RDX. The TNB data is skewed somewhat by two samples with high concentrations. A similar effect is observed for RDX with one high concentration sample skewing the results. These samples reflect the linear dynamic range limitations of the current instrument when large sample preconcentration factors result from the SPE procedure. Additionally, deployment of the technology requires skilled labor at this point. Deployment of the technology to field sites is feasible for any site that has sufficient space and access for deployment traditional groundwater collection activities.

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2.0 INTRODUCTION

This document describes the field deployment and operation of the Griffin 450™ GC-MS for the detection and quantification of MC in groundwater.

2.1 BACKGROUND

The long-term monitoring requirement for facilities often involves periodic sampling of groundwater for several years, even after activities have ceased. Traditional sampling and analytical techniques require shipping multiple liters of water to fixed laboratories that perform regulatory-approved analytical methods. The typical analysis and data reporting time at most analytical laboratories can be up to 45 days, which delays vital information on contaminant concentrations being reported to the customer. Additionally, most sample holding times have been tested for only a small set of environmental matrices where the assumption has been made that analyte concentrations will not change significantly if analyzed within this window, typically 7 to 40 days (Jenkins et al., 1995a; Jenkins et al., 1995b; Jenkins and Grant, 1987). The use of a field-portable GC-MS alleviates these concerns. While the ability to screen groundwater by direct sampling or solid phase micro extraction (SPME) has been tested, additional sample preparation and analysis options are desirable to ensure regulatory-acceptable in-field quantitation. Although, field-portable instrumentation has been successfully used previously in the analysis of volatile compounds (Jenkins et al., 1995a) it has not been extended to the analysis of semi-volatile analytes, such as explosives.

Gas chromatography with a mass selective detector (MSD) is an approved method for analysis of organic contaminants (USEPA Method 8270). The Griffin 450 GC-MS instrument tested and produced by Griffin is capable of air and liquid sampling, directly or via an SPME fiber. The CIT technology used in this system allows for miniaturization of the mass analyzer, while still maintaining the high caliber of analysis associated with traditional quadrupole mass spectrometry. In addition, a shock mount platform is used to protect the pump and electronic components, allowing for transport to remote sites. This mass spectrometer allows for analysis and follow-up investigations, including the use of tandem mass spectrometry (MS/MS) capabilities to confirm the identity of contaminants and unknown compounds present in the sample matrix. Unknown compounds have the potential to produce false positives when using non-selective detectors such as UV absorbance.

2.2 OBJECTIVES OF THE DEMONSTRATION

The objective of this ESTCP demonstration was to 1) demonstrate the suitability of field analysis for a suite of contaminants of concern (semivolatile MC), and 2) demonstrate the utility, comparability, and cost savings of groundwater analysis using the Griffin 450 GC-MS.

The research plan for this demonstration was to collect groundwater samples at the Louisiana Army Ammunition Plant (LAAP), in Minden, LA, and the Milan Army Ammunition Plant (MAAP) in Milan, TN, using standard well purging and sampling methods (USEPA Region 1, 1996), and analyze the samples 1) in field utilizing the Griffin 450 GC-MS instrument and 2) in the laboratory using HPLC (USEPA Method 8330B). The previous generation Griffin 400 GC-

MS was used as a comparison to the Griffin 450. The following analytes were evaluated: NB, 1,3-DNB, 2,4-DNT, TNB, TNT, and RDX.

The cost savings of the field method were found to be $29,600 a year, based on 12 week-long field trips a year, with a breakeven point of 3.54 years.

2.3 REGULATORY DRIVERS

USEPA methods 8330 and 8095 are two standard analytical methods for explosives. These methods involve collecting up to 1 L of water, shipping the sample to a laboratory, and using solid phase extraction and concentration for sample preparation prior to analysis. Both of these techniques use non-specific detectors, UV absorbance in the case of 8330, and election capture detection in the case of 8095. Because these methods have non-specific detectors, dual chromatography column confirmation is required for absolute analyte confirmation and quantitation. The use of GC-MS, which is also an accepted methodology (USEPA Methods 8270 and 529), allows for the analytes to be detected and confirmed with only a single chromatographic separation due to the selectivity of the mass spectrometer. Additionally, by using a field-portable instrument, samples can be collected and analyzed in an expedited manner, removing the cost and delay associated with sample transport to a fixed laboratory.

3.0 TECHNOLOGY

This document compares the Griffin 450 GC-MS to the more traditional HPLC-UV explosives analysis following USEPA Method 8330B. The improved electronic stability and sensitivity of the Griffin 450 provided higher quality data, especially in humid environments, compared to the previous Griffin 400 model GC-MS.

3.1 TECHNOLOGY DESCRIPTION

Typical MS analysis systems can provide valuable chemical information on almost any type of sample. Traditionally, MS has been confined to fixed-site laboratory analysis due to the size and fragility of the instruments typically employed for this application. Efforts have been made toward miniaturization enabling this technology to be brought to the field to perform analysis. Griffin uses a CIT as the mass analyzer; this device is a simplified geometry of the classic hyperbolic ion trap and therefore more easily miniaturized. The Griffin GC-MS also uses an LTM GC as the GC. With a smaller ion trap, the vacuum manifold becomes smaller, and the resulting pumping and power requirements are reduced. With the LTM GC column, the GC oven is removed and replaced by heat tape, decreasing the power requirements further. These modifications to the instrument design all serve to decrease the size and weight of the instrument relative to a traditional bench-top GC-MS. Griffin has also worked to ruggedize the instrument, enabling transport into the field for on-site analysis.

3.1.1 Schematic Diagram of the Technology

The GC-MS system consists of a heated inlet, guard columns, LTM GC, a vacuum chamber, a CIT, a turbo molecular pump, diaphragm pump, and system electronics. The inlet, guard columns, LTM GC, and the vacuum chamber are shown in Figure 1. Additionally a typical field setup for both the Griffin 400 and 450 is shown in Figure 2. The field extraction is preformed on the setup in the front left corner of the field work area shown.

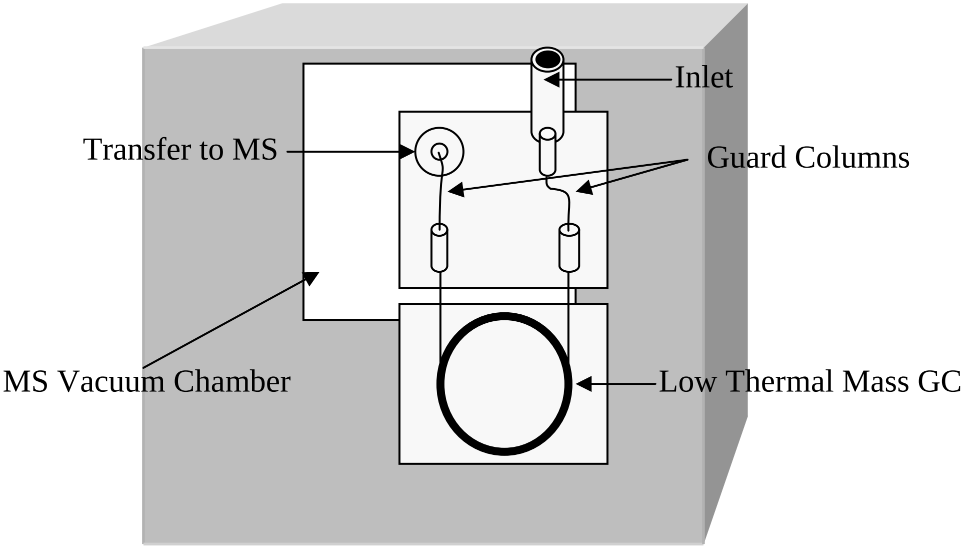


Figure 1. Components of the ICx Griffin 450 GC-MS.

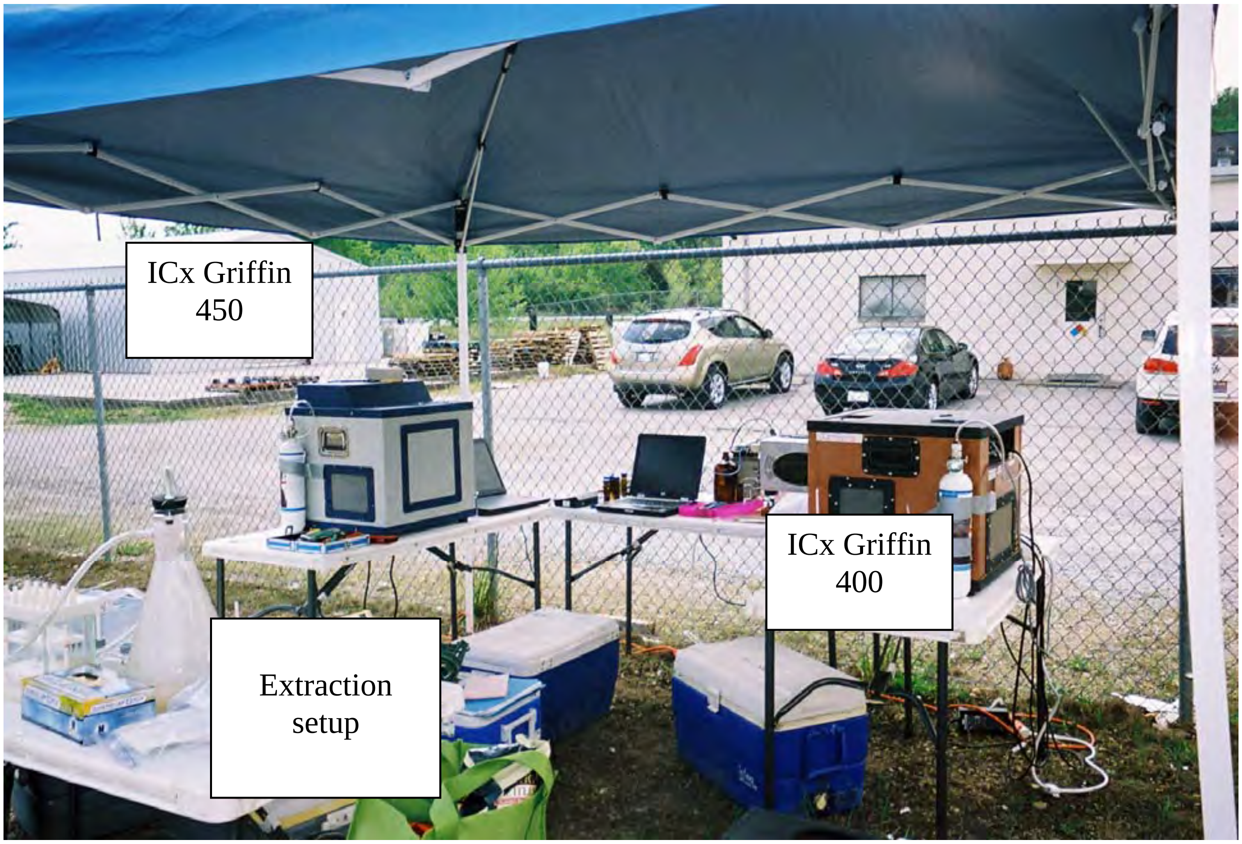


Figure 2. Field setup for the ICx Griffin 400, the ICx Griffin 450, and the extraction setup.

3.1.2 Development of the Technology

Existing research by the U.S. Army Engineer Research and Development Center (ERDC) has demonstrated the applicability of ICx Griffin’s Minotaur 400 field portable GC-MS to analyze MC in groundwater (Russel et al., 2007; Macmillan and Splichal, 2005; NASA, 2007; Bednar et al., 2009; Kirgan et al., 2008), specifically NB, 1,3-DNB, 2,4-DNT, TNB, TNT, and RDX. Figure 3 provides structural representations of the compounds. The current demonstration leveraged this work serving as a base of comparison.

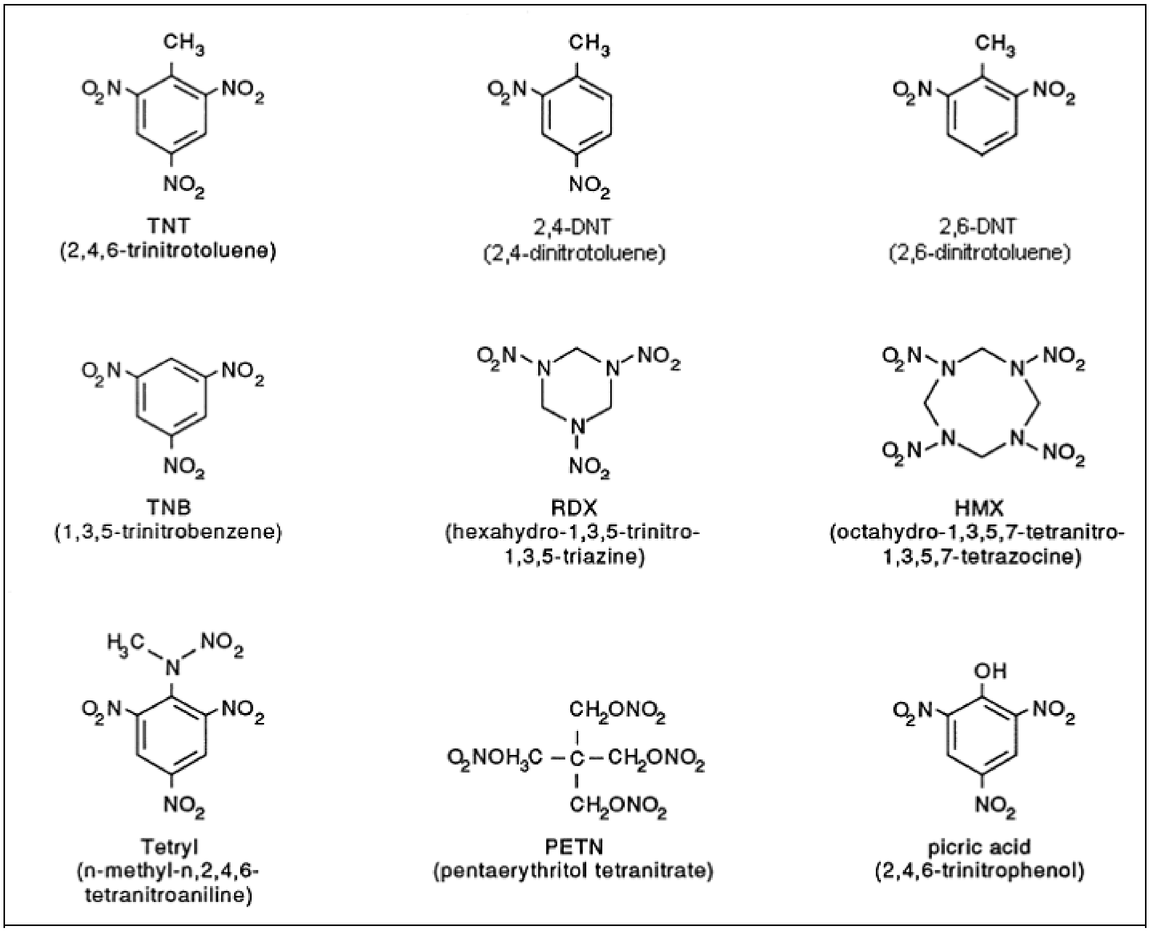


Figure 3. Structural representation of known munitions constituents present at LAAP.

Results from several studies conducted under the sponsorship of the Long-Term Monitoring Program were reported in the original proposal. These studies showed analyte degradation or loss during storage and shipment to fixed laboratories (Jenkins, et al., 1995a; Kirgan et al., 2008; USEPA, 1998) and identification of unknown contaminants utilizing the ICx Griffin 400.

Field trials have included two classes of analytes, munitions constituents, and polyaromatic hydrocarbons (PAHs). The PAH study indicated favorable comparisons (Bednar et al., 2009) between the ICx Griffin 400 GC-MS field analysis and traditional laboratory GC-MS analysis following USEPA Method 8270C. Previous field studies utilizing the field GC-MS instruments at LAAP have shown agreement of field data and HPLC data for TNT and 1,3-DNB. Figure 4 shows the chromatogram obtained in the field from the ICx Griffin 400 GC-MS for Well 104. Figure 4 shows the comparability to laboratory determined numbers for TNT. The agreement between the sets of data is acceptable with a bias of less than 20%. An additional contaminant was detected in many of the field samples at LAAP; it was identified as the plasticizer N-(n-butyl) benzene sulfonamide using the capabilities of the ICx Griffin 400 GC-MS.

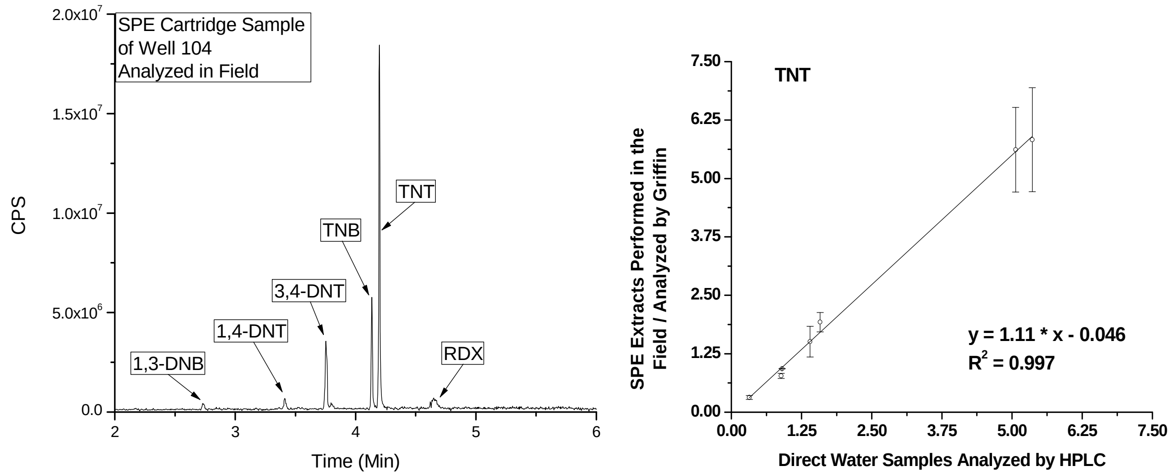


Figure 4. Typical chromatogram obtained by the Griffin 400 GC-MS (left) and Griffin 400 GC-MS results from the field work compared to the HPLC Laboratory results for TNT (right).
(CPS [counts per second])

3.1.3 Development Conducted under ESTCP

The new Griffin 450 instrument has also undergone extensive laboratory testing, including method development and detection limit determination, as shown in Table 1. The verification standard was analyzed after the detection limit study. The detection limit study demonstrated that the instrument can, for most analytes, reach detection limits of less than 0.001 mg/L. The regulatory limits for most of the analytes are 0.001 mg/L or higher (e.g., 0.002 mg/L for TNT and RDX); these values are also in the range for HPLC method detection limits (MDL), which are generally on the order of 0.0005 mg/L. The values reported for the 0.001 mg/L low-level laboratory control sample are the results of triplicate analyses collected on non-consecutive days. This low-level laboratory control sample is below the calculated MDL for NB and RDX yet used a larger SPE concentration factor to achieve acceptable recovery. The Department of Defense (DoD) Quality Systems Manual (QSM) limits for a mid-range laboratory control sample (LCS) are used to show acceptable recovery, with all analytes meeting these limits except TNB, which is slightly lower (as described in Section 3). However, application of these mid-calibration range recovery limits to the lower end of an instrument's detection range represents an extremely conservative situation, and therefore the TNB recovery is deemed acceptable.

Extensive field testing on the Griffin 450 instrument has consisted of the evaluation of 28 groundwater samples collected from monitoring wells at two demonstrations sites. LAAP and MAAP were used as demonstration sites.

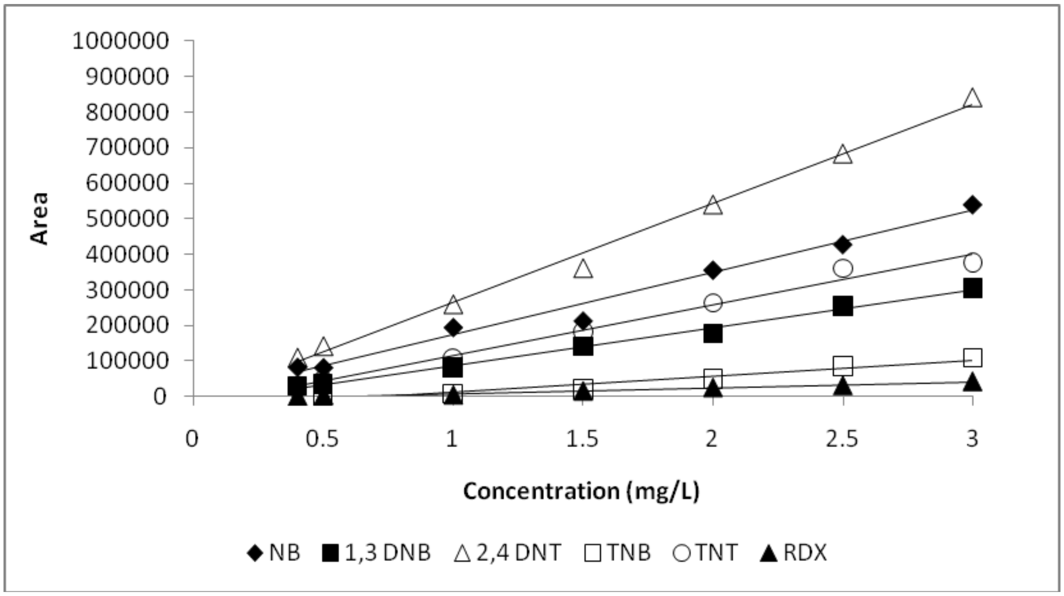
Table 1. Detection limits for munitions constituents using SPE extraction and detection on the ICx 450 GC-MS.

Analyte	Regulatory/Decision Levels (mg/L)	MDL (mg/L)*	0.001 mg/L low-level laboratory control sample*	% REC
NB	0.005**	0.0014	0.0012	120
1,3-DNB	0.001	0.0005	0.0011	110.9
2,4-DNT	0.005	0.0006	0.0008	75.8
TNB	0.01***	0.0002	0.0005	52.2
TNT	0.002	0.0003	0.0013	133.2
RDX	0.002	0.0005	0.0012	117.0

*mean recoveries
**KS regulatory limit, USEPA limit 17 mg/L
***Chronic water quality criteria

3.1.4 Calibration of the Technology

The calibration for the Griffin GC-MS used mixed analyte standards with concentrations of 0.4, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, and 3.5 mg/L, each containing 5 mg/L of 3,4-dinitrotoluene (3,4-DNT) as an internal standard to correct for instrumental drift. Linear response functions were obtained for each analyte (Figure 5, Table 2), and typically had correlation coefficients greater than 0.95. SPE of groundwater samples between 1.56 and 10.9 µg/L fall within the GC-MS calibration range when a concentration factor of 320 is used.



Calibration curve data was collected in the field at MAAP.

Figure 5. Calibration curves for NB, 1,3-DNB, 2,4-DNT, TNB, TNT, and RDX.

Table 2. Calibration curve data parameters.

Analyte	Quantitation Mass Monitored (*m/z*)	Retention Time (minutes)	Calibration Curve*	R^2
NB	123	1.43	C=(A-90.48)/174516.95	0.98
1,3-DNB	167	3.65	C=(A+19849.76)/106797.08	0.99
2,4-DNT	165	4.03	C=(A+12325.51)/278102.7	0.99
TNB	213	4.55	C=(A+32385.71)/44806.29	0.95
TNT	210	4.59	C=(A+29116.76)/143635.9	0.99
RDX	128	5.00	C=(A+7259.44)/15834.53	0.98

*C is the concentration of the analyte and A the area of the quantitation masses monitored.

3.1.5 Expected Applications

GC-MS is an accepted analytical methodology for a wide range of organic compounds (USEPA Methods 8270 and 529), with MC being one such class of analytes. This technology has been tested by the ERDC Long-Term Monitoring research program for detection and quantitation of MC in groundwater. Furthermore, it has been used for near-real-time quantitation of PAHs in dredged material during active dredging operations on the lower Mississippi River (Bednar et al., 2009). Finally, the technology is currently under evaluation for use at an Alabama Superfund site for field detection and quantitation of polychlorinated biphenyls (PCBs) in support of a site contamination delineation investigation. The utility of this technology is outlined by the wide range of potential applications for field portable GC-MS instrumentation.

3.2 ADVANTAGES AND LIMITATIONS OF THE TECHNOLOGY

The instrumentation provides in-field, near-real-time, confirmatory GC-MS analysis of MC in groundwater. These data improve the quantitiation of contamination found in the field and prevent errors due to potential degradation processes occurring during transport to a fixed laboratory.

The cost savings for analyzing samples in the field versus shipping to a fixed-site laboratory is based on the shipping costs as well as fiscally intangible cost related to delays in data reporting from fixed-site laboratories. Shipping costs can be more than $40 per sample, depending on distance transported and the amount of ice required to maintain regulatory temperatures, whereas field analysis has no such cost.

Operating costs for the field instrumentation are lower due to less solvent and helium gas usage, and single chromatographic analysis versus the laboratory techniques that require dual column confirmation for both HPLC and gas chromatograph-electron capture detector (GC-ECD) analyses. Both field analysis and traditional analysis have charges incurred for field mobilization to collect samples. The field analysis has the added benefit of near-real time data reporting, rather than traditional laboratory turnaround times of 30 days or more. Additionally, the mass spectrometer allows for analysis and confirmation of analytes in one chromatographic analysis, rather than two when using non-selective detectors, such as UV absorbance.

The limitations with this technology are environmental concerns, such as heat and humidity, which have been shown before to be detrimental to the quality of data obtained from the Griffin 400 (Russel et al., 2007; Kirgan et al., 2008). High humidity has been shown to cause the baseline to drift (increase noise in the base line), thereby increasing the limit of detection (Figure 6). The temperature and humidity ranged from 10-35°C and 25-90% relative humidity during the course of the current demonstrations. No dependence on temperature or humidity was observed for the Griffin 450 GC-MS during this study. The Griffin 450 has updated system electronics and the addition of the inlet control board, which may explain the improved stability.

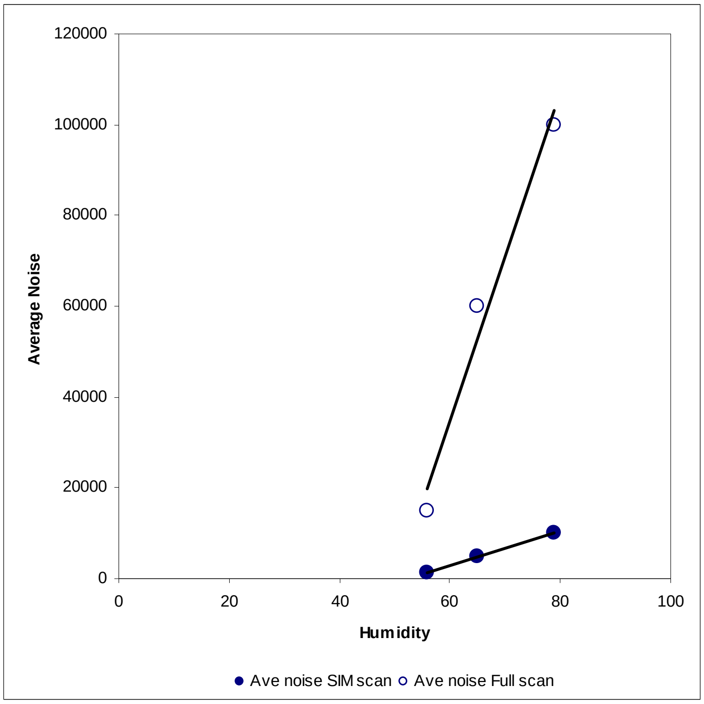


Figure 6. Average baseline noise observed on the Griffin 400 for both selected ion monitoring (SIM) and full scan monitoring with varying humidity conditions. Below 55% humidity, the average baseline noise is constant.

Differences between the Griffin 400 GC-MS and the Griffin 450 GC-MS that result in improved field operation include:

- Updated vacuum system (including a new turbomolecular pump) provides lower trap pressures, thus better sensitivity
- New detector with onboard preamp board, also increases sensitivity by reducing noise
- More robust injector assembly
- New inlet control board on the Griffin 450 GC-MS provides software control of heated zones.

Additionally, the current field instrumentation, Griffin 400 and 450, required highly trained and experience analysts, limiting the deployment by field personnel.

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4.0 PERFORMANCE OBJECTIVES

Both qualitative and quantitative performance objectives were indentified for the GC-MS technology. Qualitative measures included the day-to-day operational performance parameters, i.e., operation by portable generator-produced electricity and response to humidity. The quantitative measures included a statistical comparison of field-generated data to the laboratory-based data produced by the benchmark method, USEPA Method 8330B. The performance objectives are summarized in Table 3.

Table 3. Performance objectives.

Performance Objective	Data Requirements	Success Criteria	Results
Quantitative Performance Objectives			
Agreement between analytical methods for analytes of interest. A lack of bias with GC-MS method	Data sets each consisting of at least 20 total paired points (measurements from split samples)	Lack of statistically significant differences at the 95% level of confidence using statistical tests for paired data sets or a bias that is less than 20%. A linear relationship through the origin with a slope of nearly one (0.80 - 1.20).	Yes for 1,3-DNB, 2,4-DNT and TNT No RDX and TNB
The ability to provide accurate results in clean matrices	LCS recoveries from in-field analyses	LCS recoveries that all fall within the acceptance ranges in the DoD QSM.	Yes for NB, 1,3-DNB, 2,4-DNT and TNT Limitations with RDX and TNB
Ability to quantify analyte concentrations at the levels of interest in aqueous matrices	Detection limits that meet commonly used decision limits for explosives in groundwater (regulatory or risk-based thresholds)	Detection limits less than the decision limits. (e.g., RDX<0.002 mg/L)	Yes for all compounds at the 0.001 mg/L spike level. Agreement between field and laboratory methods was only obtained for concentrations >0.05 mg/L for TNT and TNB
Ability to recover analytes in environmental matrices	Matrix spike and matrix spike duplicate recoveries consistent with fixed laboratory analyses	Matrix spike and Matrix spike duplicate recoveries that fall within the acceptance ranges in the DoD QSM for spike recoveries and 20% for the relative percent differences (RPDs)	Yes for NB, 1,3-DNB, 2,4-DNT and TNT Limitations with TNB. No for RDX

Table 3. Performance objectives (continued).

Performance Objective	Data Requirements	Success Criteria	Results
Qualitative Performance Objectives			
Ease of use and GC-MS operates as expected	Feedback from field technician on usability of technology and time required	A single field technician able to take measurements and troubleshoot any problems that arise	Problems encountered were solved with replacement of consumables
Ease of deployment	Deployment with standard equipment, e.g., 5kW generator, single trailer needed to transport equipment	Standard field deployment	No problems encountered with deployment
Technology robustness	Signal-to-noise does not change relative to humidity; different matrices do not adversely affect data quality	Data quality not affected by humidity or sample matrix composition (e.g., RPD <20%)	Data quality was not affected by humidity

The primary objective was to obtain quantitative results for MC in groundwater that are statistically comparable to traditional techniques by the benchmark laboratory method, USEPA Method 8330B. Method 8330B sets the criteria for sample duplicates to ±20%; this was the success metric utilized for the field instrument.

In particular, acceptance criteria (Table 4) are presented for LCSs, which are prepared by spiking reagent water and processed on a batch basis (at a frequency of at least 5%). One liter samples were used for the LCS analysis. A blank and a laboratory control spike (LC-S) were analyzed daily, as each day is considered an analytical batch, which resulted in these quality control (QC) samples being analyzed at a rate higher than 5%.

Table 4. DoD QSM % LCS recovery limits and regulatory/decision levels.

Analyte	DoD QSM % Recovery Limits	Regulatory/ Decision Levels (mg/L)
NB	50-140	0.005*
DNB	45-160	0.001
DNT	60-135	0.005
TNB	65-140	0.01**
TNT	50-145	0.002
RDX	50-160	0.002

*KS regulatory limit, USEPA limit 17 mg/L
**Chronic water quality criteria

Paired groundwater samples (at least n=20) were used to compare the field method to the lab method. The number of data points was adequate to do linear regressions and Kendall Thiel line analysis (e.g., plots of the lab results versus the field results). The slope and intercept provided information about bias, as the slope should be 1 and the intercept 0 when there is no bias. The lab and the field method data sets were evaluated using linear regression fits and tests for paired data sets. Additionally, the sign test, t-test, Wilcoxon, or one-sample test for proportions were performed to determine using the paired data sets to determine if there is significant bias.

The qualitative objectives were designed to assess the overall instrument performance in the field. Serious degradation in the signal-to-noise level has been observed during operation of the previous generation Griffin 400 GC-MS in high humidity conditions. This resulted in data that was unusable and in extreme cases unobtainable, as the baseline noise overwhelmed the detector.

Additionally, the ease of deployment and operation of the instrument in the field were subjective measures of demonstration success. Demonstration operations were carried out from a central location, using a portable generator, with no uncorrectable instrument failures encountered, such as pump or electronic failure. However, the older Griffin 400 instrument had reached the end of its expected lifetime and produced results that were not quantitatively comparable to either the laboratory analysis or the newer Griffin 450 instrument. One analyst was able to maintain and operate the Griffin 450 GC-MS system for the duration of both demonstrations. However, there were a few instrument problems mainly resulting from transport of the instrument to the site. All of the issues were overcome by replacing instrument consumables. This does highlight a limitation of the technology, that currently it requires highly trained operators for successful deployment.

5.0 SITE DESCRIPTION

LAAP and MAAP were chosen as test sites for the ICx Griffin field portable GC-MS. These sites were chosen based on having several munitions constituents present at various concentrations over several orders of magnitude to test the versatility of the instrument. Both sites have a humid climate, which allowed for the observation of instrument behavior and response as a function of humidity. Groundwater wells less than 30 m deep are located on both sites, which aided in sample collection, as deeper wells require more tubing and different sampling pumps than were available to our research project.

5.1 SITE LOCATIONS

5.1.1 LAAP

The following excerpts were taken from Pennington et al. (1999).

> LAAP is a government-owned, contractor-operated facility located 35.4 km (22 miles) east of Shreveport, LA. The primary mission of the 6062-ha (14,974-acre) plant was to load, assemble, and package ammunition items, manufacture ammunition metal parts, and provide associated support functions for ammunition production.
>
> LAAP was placed on the National Priorities List in March 1989 due to contamination caused by past disposal of explosives-laden wastewater in 16 unlined surface impoundments located in Area P (Figure 7).

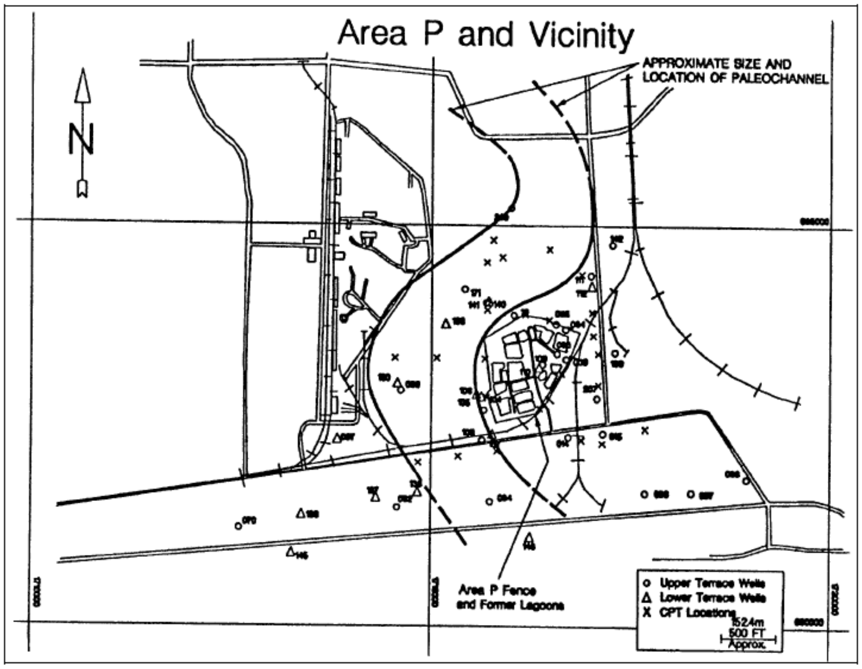

Figure 7. LAAP – Area P and vicinity.
(Taken from Pennington et al. [1999])

5.1.2 MAAP

MAAP is a government-owned, contractor-operated facility located 161 km (100 miles) east/northeast of Memphis in the central section of west Tennessee, east of Milan, TN. Constructed in 1941, the primary mission of the 9122-ha (22,540-acre) facility was to produce and store fuses, boosters, and small- and large-caliber ammunition.

The O-line at the MAAP (Figure 8) is a conventional munitions demobilization facility. Effluent from the removal of munitions was discharged into 11 unlined settling ponds with an estimated capacity of 5.5 million gallons. Sediments were routinely dredged from the ponds and stored on the ground. The ponds were lined in 1981 and the accumulated sediments placed into the ponds.

Currently, MAAP does loading assembling and packing (LAP) for fuzes and other ammunition items, such as, demolition charges, mortar rounds, and 155-mm projectiles. MAAP also stores and tests ammunition (USEPA, 1998).

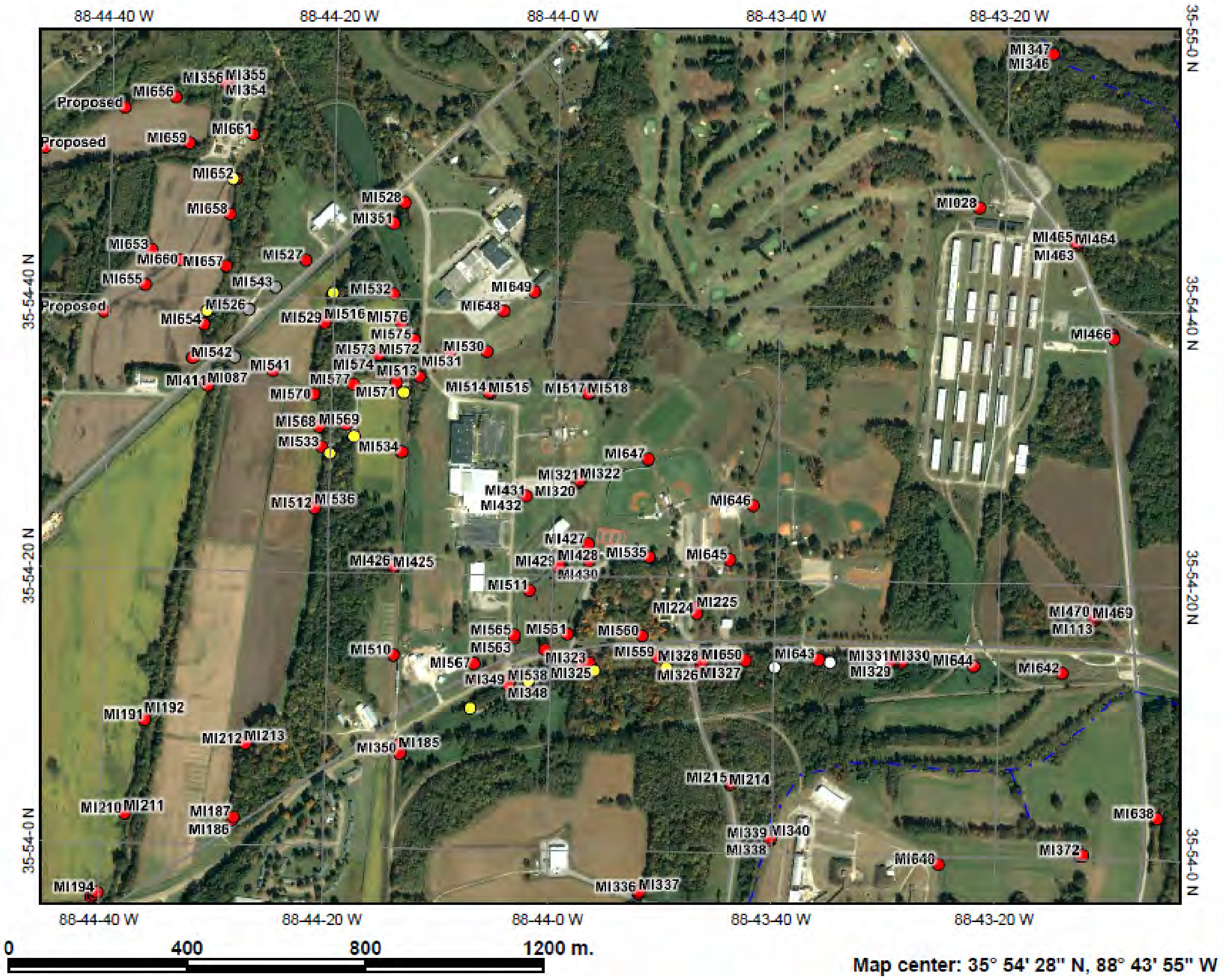

Figure 8. MAAP – O-line.

5.2 SITE GEOLOGY/HYDROGEOLOGY

5.2.1 LAAP Geology

Area P of LAAP consists of an upper and lower terrace separated by intermittent clay. The upper terrace consists of very fine silt, clays, and silty clays, while the lower terrace consists of fine sands and a trace of gravel. The terrace deposits date from the Pleistocene Age and overlay the Eocene Age Cane River Formation.

5.2.2 LAAP Hydrogeology

There are only slight seasonal variations in the groundwater level at LAAP. The low permeability of the soil precludes rapid movement and recharge. The groundwater levels reach their highest levels in winter and lowest in the fall. The movement of groundwater in the lower terrace is to the southeast, while the upper terrace the movement is to the east.

5.2.3 LAAP Geomorphology

LAAP is located within the Western Gulf Coastal Plain physiographic province. Two major landforms are found within LAAP, dissected uplands and rolling prairie. Regionally, LAAP lies within the North Louisiana Syncline, a subsurface structural feature located east of the Sabine Uplift and west of the Monroe-Sharkey Platform. The groundwater flow regime is significantly modified by small uplifts in the area, which modify the local structural geology. Ground surface elevations range from about 40 m (130 ft) above mean sea level (MSL) to the east near Dorcheat Bayou and 24 m (80 ft) above MSL to the west near Clarke Bayou.

5.2.4 MAAP Geology

MAAP lies on the eastern flank of the Upper Mississippi River Embayment. Sediments consisting of gravel, sand, clay, lignite chalk, and limestone have been deposited in the embayment. The fluvial deposits date from the Tertiary and Quaternary age.

5.2.5 MAAP Hydrogeology

The principal sources of groundwater in western Tennessee are the Claiborne and Wilcox sands. This unconfined aquifer yields groundwater to private, municipal, and industrial wells in the area. Groundwater flow in this aquifer generally is about 20 ft/mi to the northwest, following the direction of the regional dip of this sand. The Memphis Sand aquifer is thick, laterally continuous, and highly transmissive.

5.2.6 MAAP Geomorphology

MAAP is located on the Memphis Sand of the Claiborne Group of Tertiary age in the Gulf Coastal Plain of western Tennessee. Regionally, MAAP lies within the Upper Mississippi River Embayment. The Memphis Sand ranges to 900 ft thick and is covered in most places by Tertiary and Quaternary age fluvial deposits and Quaternary age loess and alluvium deposits.

5.3 CONTAMINANT DISTRIBUTION

Site maps of the monitoring wells at LAAP and MAAP are shown in Figures 9 and 8, respectively. Information on the contaminant distribution at both sites is given below. Tables 5 and 6 list the monitoring wells at LAAP and MAAP, the dates they were sampled, and the presence (denoted by X) or the absence (denoted n.d.) of the contaminant.

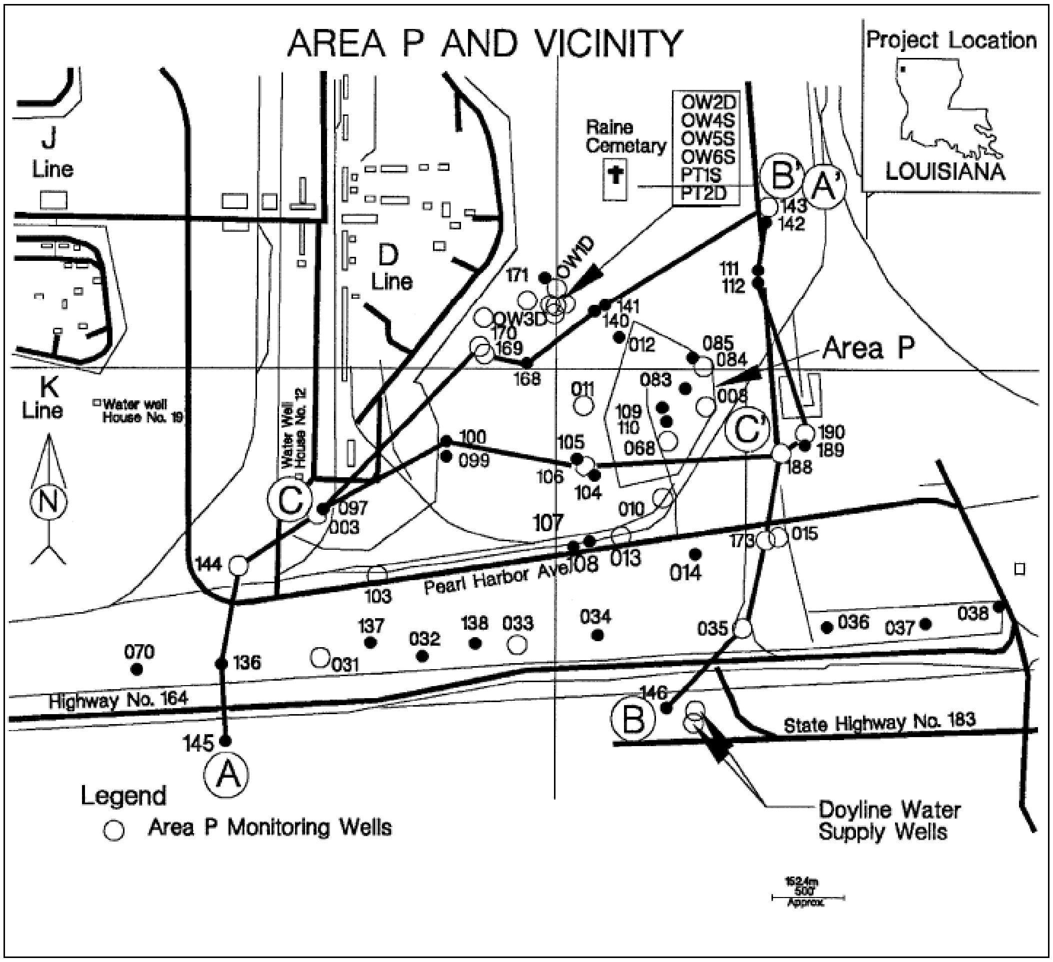

Figure 9. LAAP – Area P and vicinity showing where monitoring wells are located.
(taken from Pennington et al. [1999])

5.3.1 LAAP CONTAMINANT DISTRIBUTION

The contamination at LAAP lies in the area surrounding the former wastewater lagoons at Area P. Monitoring wells were installed during the remediation of Area P, which included the incineration of soil and the treatment of wastewater and rainwater collected within the 16 lagoons. The overall water quality of Area P was shown to be improving in 1994 (Pennington et al., 1999); however RDX concentrations of 16 ppm have been detected in Well 104 as recently as 2008.

Table 5. Information on wells at LAAP.

Well #	Date Sampled	1,3-DNB	2,4-DNT	TNB	TNT	RDX	Terrace	Top of screen (m bgs*)	Bottom of screen (m bgs)	Screen length (m)
85	3/24/10	X**	X	X	X	X	Upper	6.86	9.91	3.05
104	3/23/10	X	X	X	X	X	Upper	6.86	9.91	3.05
105	3/23/10	X	X	X	X	X	Lower	15.09	16.61	1.52
108	3/22/10	X	X	X	X	X	Lower	21.95	24.38	2.44
110	3/24/10	X	X	X	X	X	Lower	22.86	25.91	3.05
111	3/22/10	n.d.***	n.d.	n.d.	n.d.	X	Upper	10.06	13.11	3.05
112	3/22/10	X	X	X	X	X	Lower	21.28	24.32	3.05
140	3/23/10	X	X	X	X	X	Upper	4.57	7.62	3.05
141	3/23/10	X	X	X	X	X	Lower	18.9	21.95	3.05
142	3/24/10	n.d.	n.d.	X	X	X	?	?	?	?

*bgs = below ground surface
**X = presence of contaminant
***n.d. = absence of contaminant

Table 6. Information on wells at MAAP.

Well #	Date Sampled	NB	1,3-DNB	2,4-DNT	TNB	TNT	RDX
M1355	4/22/10	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
M1514	4/22/10	n.d.	n.d.	X	X	X	X
M1516	4/22/10	n.d.	n.d.	X	X	X	X
M1531	4/20/10	n.d.	n.d.	n.d.	n.d.	X	X
M1533	4/21/10	n.d.	n.d.	X	X	X	X
M1534	4/23/10	n.d.	n.d.	n.d.	X	X	X
M1536	4/21/10	n.d.	n.d.	X	n.d.	X	X
M1537	4/21/10	n.d.	n.d.	X	X	X	X
M1538	4/21/10	n.d.	n.d.	n.d.	X	X	X
M1569	4/23/10	n.d.	n.d.	n.d.	n.d.	X	X
M1570	4/21/10	n.d.	n.d.	X	X	X	X
M1571	4/23/10	n.d.	n.d.	n.d.	n.d.	X	X
M1573	4/23/10	n.d.	n.d.	n.d.	X	X	X
M1645	4/20/10	n.d.	n.d.	X	n.d.	X	X
M1653	4/20/10	n.d.	n.d.	n.d.	n.d.	X	X
M1654	4/22/10	n.d.	n.d.	X	X	X	X
M1658	4/20/10	n.d.	n.d.	X	n.d.	X	X
M1660	4/20/10	n.d.	n.d.	n.d.	X	X	X

5.3.2 MAAP CONTAMINANT DISTRIBUTION

The contamination at MAAP lies in the area surrounding the former settling ponds. Monitoring wells were installed in 1979 and indicated the presence of explosives and heavy metals. MAAP is shown in Figure 8. The monitoring well locations (Figure 8) for the M-Line are located in the northwest quadrant of MAAP between Highway 79 and state road 104.

5.3.3 MAAP

See Figure 8 for locations of monitoring wells.

6.0 TEST DESIGN

The Griffin 450 GC-MS was used to analyze semivolatile MC in the field. MC that have been found on the sites include NB, 1,3-DNB, 2,4-DNT, TNB, TNT, and RDX. Samples were collected in 4-liter amber bottles after purging of the wells. Analyte concentrations in these samples were compared between the Griffin 450 to HPLC analyses. HPLC analyses were conducted at the ERDC-Environmental Chemistry Branch (ECB) by USEPA Method 8330B.

The initial hypothesis was that there are no statistical differences between the analysis conducted in the field on the ICx Griffin instruments and the analysis conducted at the ERDC-ECB, that is, concentrations of the analytes in samples analyzed by GC-MS in the field are comparable to those analyzed in the laboratory by HPLC, USEPA Method 8330B. Analyte degradation due to transportation of the water samples back to the laboratory is possible, which would result in a higher concentration determined with the field method. However, all analyses were conducted within traditional analyte holding times, and the samples were stored at 4°C and shielded from light.

Data analyses were on an analyte-by-analyte basis. All data sets were first analyzed to determine if the data are normally distributed and if the variances are homogeneous. Concentrations for each analyte were compared using standard statistical analyses to determine if significant differences exist between the treatments (i.e., the Griffin analysis and the HPLC analysis). Wells with analyte concentrations above and at the detection limit (0.0016 mg/L for a concentration factor of 320) were targeted for this study. However, there were analytes where many of the wells have concentrations that are below the detection limit. In those cases, comparisons were made for analytes that have detectable concentrations.

6.1 CONCEPTUAL EXPERIMENTAL DESIGN

The field demonstration was a comparison of the field-deployable GC-MS and traditional laboratory HPLC analysis, based on USEPA Method 8330B. Specifically, 4-L groundwater samples will be collected using traditional sampling methodology. The 4-L water sample was split; one portion was analyzed in the field, and the second was shipped back to the ERDC-ECB for HPLC analysis. Standard method QC sample analyses were employed, including sample duplicates, matrix spikes and matrix spike duplicates, and laboratory control samples. These analyses were used to confirm the quality of the field and laboratory data and verify that analyte recoveries were within DoD QSM limits.

The primary objective was to obtain quantitative results for MC in groundwater that are statistically comparable to traditional techniques by the benchmark laboratory method, USEPA Method 8330B. Method 8330B sets the criteria for sample duplicates to ±20%. In particular, acceptance criteria (Table 4) are presented for LCSs, which are prepared by spiking reagent water, and processed on a batch basis (at a frequency of at least 5%).

One liter samples were used for the LCS analysis. A blank and an LC-S were analyzed daily, as each day is considered an analytical batch, which resulted in these quality control samples being analyzed at a rate higher than 5%.

Paired groundwater samples (n=28) were used to compare the field method to the lab method. The number of data points was adequate to do linear regressions and Kendall Thiel line analysis (e.g., plots of the lab results versus the field results). The slope and intercept provided information about bias, as the slope should be 1 and the intercept 0 when there is no bias.

The lab and the field method data sets were evaluated using linear regression fits and tests for paired data sets. Additionally the sign test, t-test, Wilcoxon or one-sample test for proportions were performed to determine if using the paired data sets would determine if there is significant bias.

6.2 BASELINE CHARACTERIZATION

Water samples were collected from 10 monitoring wells at Area P of LAAP and from 18 monitoring wells at MAAP for the analysis of NB, 1,3-DNB, 2,4-DNT, TNB, TNT, and RDX. The pre-demonstration sampling was also performed at LAAP utilizing nine monitoring wells. Wells at LAAP were sounded to determine the groundwater level before the sampling pump was deployed. Samples were collected once the pH, conductivity, dissolved oxygen, temperature, and turbidity stabilized as monitored with a field meter (YSI 556 MPS Multi probe system, YSI Incorporated, Yellow Springs, OH). The pump was placed at the midpoint of the screened interval and pumped through a YSI monitoring unit. The ERDC team personnel collected the groundwater samples at LAAP using dedicated Teflon tubing. Well water samples at MAAP were collected by ARCADIS U.S., Inc. (2849 Paces Ferry Road, Suite 400, Atlanta, GA 30339) as part of normal monitoring activities at the site. Groundwater samples were collected in 4 liter amber jugs and split for field and traditional laboratory analysis.

Samples for traditional laboratory analysis were collected, stored, and shipped in a manner that prevented the degradation of the munitions constituents present, including packing on ice and storage in the dark. Each sample was labeled to identify the site, well number, and time and date of collection.

Contaminant concentrations were determined in the field with the Griffin 450 GC-MS; in the laboratory they were determined by HPLC using USEPA Method 8330B. The wells selected at LAAP have a range of munitions constituents from ~0.001 to 8 mg/L. The wells selected at MAAP have a range of munitions constituents from ~0.001 to 0.4 mg/L. Therefore, these wells represented ideal cases to test the versatility of the instrument over a range of analyte concentrations.

The only wastes were the purge water and water from decontamination activities. The wastewater from LAAP activities were containerized and transported back to ERDC for disposal.

ARCADIS was responsible for disposal of the wastewater generated from well sampling at MAAP as the field demonstration event was conducted simultaneously with the site's scheduled long-term monitoring sampling. Wastes generated from the field extraction and analysis were containerized and transported back to ERDC for disposal.

6.3 LABORATORY STUDY RESULTS

Table 7 lists the concentrations determined for well water samples for both LAAP and MAAP analyzed by the Environmental Chemistry Branch laboratory in Vicksburg, MS. The analyte extraction efficiency has been shown to be the same for both the fixed laboratory method and the field method, (Kirgan, et al., 2008). The concentration factors for the field and the laboratory analysis were also the same, as the same sample and final volumes were used for both extraction methods.

Table 7. HPLC Laboratory results for groundwater samples at LAAP and MAAP.

Well #	NB	1,3-DNB	2,4-DNT	TNB	TNT	RDX
108	<0.0005	0.0082	0.0738	0.7259	0.6142	2.0165
111	<0.00005	<0.00005	<0.00005	<0.00005	<0.00005	<0.00005
112	<0.00004	0.0003	0.0011	0.0003	0.0004	0.0248
105	<0.0010	0.0340	0.0093	0.7398	0.2231	0.2231
104	<0.0010	0.3286	0.1901	8.2453	6.5697	13.6107
140	<0.00025	0.0834	0.0372	0.0234	0.7790	2.9515
141	<0.00025	0.0311	0.1009	1.1211	1.2344	0.7841
142	<0.00004	<0.00004	<0.00004	<0.00004	<0.00004	<0.00004
85	<0.0010	0.0029	0.0247	6.7785	1.7333	4.0635
110	<0.0005	0.0461	0.0710	0.3817	0.6814	4.2326
MI660	<0.0001	<0.0001	0.0004	0.0007	0.0398	0.0681
MI658	<0.00008	0.0001	0.0009	0.0009	0.0958	0.1426
MI653	<0.00004	<0.00004	0.0001	0.0001	0.0011	0.0045
MI645	<0.00004	<0.00004	0.0002	0.0001	0.0004	0.2103
MI531	<0.00003	<0.00003	<0.00003	0.0001	0.0009	0.0011
MI570	<0.0001	<0.0001	<0.0001	0.0004	0.0047	0.0076
MI533	<0.00003	0.0001	0.0003	0.0008	0.0225	0.0711
MI536	<0.00005	<0.00005	0.0002	0.0002	0.0034	0.0348
MI537	<0.00004	<0.00004	0.0001	0.0035	0.0349	0.0341
MI538	<0.00004	<0.00004	0.0001	0.0018	0.0321	0.0700
MI654	<0.00005	<0.00005	0.0004	0.0006	0.0103	0.0755
MI355	<0.00003	<0.00003	<0.00003	0.0001	<0.00003	<0.00003
MI514	<0.00005	<0.00005	0.0003	0.0068	0.0857	0.0097
MI516	<0.00005	<0.00005	0.0001	0.0004	0.0160	0.0206
MI534	<0.00003	<0.00003	<0.00003	0.0004	0.0032	0.0026
MI569	<0.00003	<0.00003	<0.00003	0.0001	0.0001	0.0003
MI571	<0.00003	<0.00003	<0.00003	<0.00003	0.0001	0.0001
MI573	<0.00003	<0.00003	0.0002	0.0003	0.0037	0.0048

Results shown are mg/L in groundwater.

6.4 FIELD TESTING

The field setup for both sites consisted of two workstations. The first workstation is used for sample extraction and preparation, and the second is used for GC-MS analysis. Electrical power was supplied by portable 5 kW generators.

All equipment was transported to the field sites in a 4 m covered trailer pulled by a government-owned sport utility vehicle (SUV). Deployment and setup with three field personnel took approximately 2 hours, including staging of generators, setup of field supplies, accessing wells, instrumentation/computer setup, and vacuum system pump-down. The first calibration standard was analyzed within 2 hours of arrival on site. After initial unloading of trailer and vehicles, the field sampling team members deployed to the first well to be sampled while the analytical team members continued instrument warm-up and workstation setup.

Three to six well samples along with the required spikes and duplicates were analyzed per day. Water samples were extracted using Porapak RDX SPE cartridges (Waters, 34 Maple Street, Milford, MA). The SPE cartridges were conditioned by eluting 15 mL of acetonitrile and then 15 mL of deionized water (DI) through the cartridge in the laboratory. They were stored on ice in a sealed Ziploc bag shielded from light until needed. Water samples of 0.05 to 1.6 L were used for extraction, depending on the expected concentrations of the munitions constituents, as overloading the SPE cartridge can lead to analyte breakthrough. The well water was drawn through the SPE cartridge at a rate of <20 mL per minute. The MC were eluted off the SPE cartridge with 5 mL of acetonitrile and collected in a 15 mL centrifuge tube. Extracts were brought to a final volume of 5 mL mixed thoroughly and then transferred to a 10 mL amber vial. A 1 mL aliquot was then transferred to a 1.5 mL amber vial, dried with sodium sulfate, and spiked with 5 μL of the internal standard, 3,4-DNT, for a final concentration of 5 mg/L.

The instrument calibration standards were analyzed and a calibration curve was determined concurrently with well water collection and extraction. Calibration verification standards were analyzed periodically to confirm instrument calibration. Analysis by GC-MS of the SPE extracts commenced once the calibration curve had been determined and a verification standard had been analyzed. The GC profile was such that the contaminants of interest were chromatographically resolved and ramped from 40°C to 280°C over approximately 10 minutes. Samples were analyzed on the GC-MS by injection of one microliter volumes onto the column, with the split flow adjusted such that there was a flow of greater than 20 mL/min out the split. The temperature profile and the MS conditions have been previously described (Russel et al., 2007; Bednar et al., 2009; Kirgan et al., 2008; USEPA, 1998).

Instrument shutdown at the end of the day consisted of performing a final check standard followed by the instrument's preprogrammed shutdown sequence. The final check standard ensured the calibration held after the last samples were analyzed. The shutdown sequence turned off all instrument electronics and shut down the vacuum system. The instrument was locked in the transport trailer overnight. At the end of the field demonstration, all sampling supplies and instruments were repacked and loaded for transport back to the ERDC-ECB for cleaning.

The Gantt chart provided in Table 8 outlines the schedule of field activities.

Table 8. Gantt chart for field activities.

Time	800	900	1000	1100	1200	1300	1400	1500	1600	1700
Setting up field laboratory	🟦	🟦								
Instrumentation setup	🟦									
Sampling		🟦	🟦	🟦	🟦	🟦				
Sample extraction			🟦	🟦	🟦	🟦	🟦	🟦		
Calibration standards			🟦	🟦	🟦					
Sample analyses					🟦	🟦	🟦	🟦	🟦	
Loading of field supplies								🟦	🟦	🟦
Final calibration check									🟦	
Shutdown of instruments										🟦

6.5 SAMPLING METHODS

Twenty-eight wells were sampled for MC analysis. For each well, a 4 L whole-water sample was collected. Additional water for quality assurance (QA)/QC samples was collected as needed. The QA/QC samples were also collected in 4 L amber bottles; they were then split into four 1 L amber bottles for analysis in the field and shipped back to ERDC-ECB for laboratory analysis. The Griffin GC-MS instrument was placed in a central location relative to the well locations. One of the wells was chosen each day for QA/QC because each day is an "analytical batch."

6.6 SAMPLING RESULTS

Split groundwater samples were collected and analyzed for the compounds NB, 1,3-DNB, 2,4-DNT, TNB, TNT, and RDX to compare the results from the Griffin 450, a field portable GC/MS, to the HPLC results from a conventional fixed laboratory.

The total number and types of samples collected for all sampling events are shown in Tables 9 and 10. Holding times for the groundwater samples were 7 days; however, the holding time once the water samples are extracted into acetonitrile is 30 days.

The results from the field and laboratory analysis of groundwater samples are shown in Tables 11 and 7. Results from control and matrix spike samples are given in Tables 12 and 13, respectively.

Table 9. Total number and types of samples collected.

Component	Matrix	Number of Samples	Analyte	Location
Pre-demonstration sampling	Groundwater	9	NB, 1,3-DNB, 2,4-DNT, TNB, TNT, RDX	Monitoring wells at LAAP
Technology performance sampling	Groundwater	10 LAAP 18 MAAP	NB, 1,3-DNB, 2,4-DNT, TNB, TNT, RDX	Monitoring wells at LAAP and MAAP

Table 10. Total number and types of samples collected.

Matrix	Analyte	Method	Container	Preservative[1]	Holding Time
Groundwater	NB	USEPA 8330B and modified USEPA 8270 and 529	1 L amber bottle	None	7 days
	1,3-DNB	USEPA 8330B and modified USEPA 8270 and 529	1 L amber bottle	None	7 days
	2,4-DNT	USEPA 8330B and modified USEPA 8270 and 529	1 L amber bottle	None	7 days
	TNB	USEPA 8330B and modified USEPA 8270 and 529	1 L amber bottle	None	7 days
	TNT	USEPA 8330B and modified USEPA 8270 and 529	1 L amber bottle	None	7 days
	RDX	USEPA 8330B and modified USEPA 8270 and 529	1 L amber bottle	None	7 days

Table 11. ICx Griffin 450 results for wells at LAAP and MAAP.

Well #	NB	1,3-DNB	2,4-DNT	TNB	TNT	RDX
108	<0.0178	0.0107	0.0643	1.1542	0.7663	3.1228
111	<0.0016	0.0009	<0.0007	0.0031	0.0015	<0.0006
112	<0.0015	0.0011	0.0007	0.0030	0.0027	0.0292
105	<0.0356	0.0407	0.0227	1.0887	0.1939	0.1939
104	<0.0356.	0.2980	0.1678	12.5725	6.7263	17.9812
140	<0.0089	0.0846	0.0355	0.0283	0.8421	1.9238
141	<0.0089	0.1059	0.1002	1.5073	1.1937	0.6502
142	<0.0015	<0.0006	<0.0007	0.0033	0.0008	0.0029
85	<0.0356	<0.0133	0.0256	10.2946	2.0208	2.8327
110	<0.0178	<0.0067	<0.0080	0.0594	0.0376	0.0442
MI660	<0.0036	<0.0013	<0.0016	<0.0006	0.0289	0.0285
MI658	<0.0030	0.0025	0.0017	0.0081	0.0977	0.0890
MI653	<0.0015	0.0010	<0.0007	<0.0002	0.0018	0.0040
MI645	<0.0015	<0.0006	<0.0007	<0.0002	0.0012	0.1384
MI531	<0.0011	<0.0004	<0.0005	<0.0002	0.0010	0.0030
MI570	<0.0045	<0.0017	<0.0020	<0.0007	0.0054	0.0091
MI533	<0.0011	<0.0004	<0.0005	<0.0002	0.0188	0.0680
MI536	<0.0018	<0.0007	<0.0008	0.0042	0.0028	0.0368
MI537	<0.0015	<0.0006	<0.0007	0.0037	0.0084	0.0146
MI538	<0.0015	<0.0006	<0.0007	0.0035	0.0127	0.0155
MI654	<0.0018	<0.0007	<0.0008	0.0282	0.0181	0.0367
MI355	<0.0011	<0.0004	<0.0005	0.0019	0.0012	0.0285
MI514	<0.0018	<0.0007	<0.0008	0.0052	0.0788	0.0042
MI516	<0.0018	<0.0007	<0.0008	0.0032	0.0094	0.0016
MI534	<0.0011	<0.0004	<0.0005	0.0020	0.0021	0.0133
MI569	<0.0011	<0.0004	0.0005	0.0022	0.0008	0.0015
MI571	<0.0011	<0.0004	<0.0005	<0.0002	0.0008	0.0014
MI573	<0.0011	<0.0004	0.0006	0.0023	0.0309	0.0708

Results shown are mg/L in groundwater.

Table 12. Griffin 450 LCS % recoveries.

	NB	1,3-DNB	2,4-DNT	TNB	TNT	RDX
DoD QSM Limits	50-140	45-160	60-135	65-140	50-145	50-160
LAAP Day 1	78	73	82	83	74	57
LAAP Day 2	58	47	60	73	59	**33**
LAAP Day 3	110	65	96	91	83	69
MAAP Day 1	100	98	91	81	82	55
MAAP Day 2	110	93	100	72	67	**41**
MAAP Day 3	99	100	110	**62**	70	57
MAAP Day 4	77	110	100	79	88	110

Reported to two significant figures. Values in **bold** are outside DoD QSM limits.

Table 13. Griffin 450 MS % recoveries.

	Sample ID	NB	1,3-DNB	2,4-DNT	TNB	TNT	RDX
DoD QSM Limits		50-140	45-160	60-135	65-140	50-145	50-160
LAAP Day 1	111MS	96	86	91	74	63	**45**
	11MSD	100	74	120	100	92	**38**
LAAP Day 2[*]	104MS	92	**27**	84	**750**	**490**	**-2200**
	104MSD	99	87	111	**-1000**	**-900**	**-3800**
LAAP Day 3	142MS	80	72	73	75	72	54
	142MSD	96	100	93	88	81	**49**
MAAP Day 1	MI531MS	120	110	110	66	55	**9.8**
	MI531 MSD	120	77	96	79	58	**26**
MAAP Day 2	MI536MS	110	68	100	**59**	54	**260**
	MI536 MSD	120	99	120	89	81	**200**
MAAP Day 3	MI355MS	**160**	110	110	**22**	61	**20**
	MI355MSD	140	93	110	**23**	66	**37**
MAAP Day 4	MI569MS	70	99	94	66	86	**33**
	MI569MSD	98	130	100	76	96	**34**

Values in **bold** are outside DoD QSM limits

[*] The well sample chosen was highly contaminated with the MCs of interest except for NB; therefore the spike was insignificant compared to the amount of analyte present, resulting in poor recoveries.

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7.0 PERFORMANCE ASSESSMENT

The analyte concentrations measured by the laboratory and field methods qualitatively agree for all MC studied. Graphical analysis of the plots of the Griffin field data versus the HPLC laboratory data for the individual MCs of interest show linear regression slope values between 0.80 and 1.20 for 1,3-DNB, 2,4-DNT and TNT (Table 14). Nitrobenzene was not detected from any of the well samples; therefore only comparisons of the non-detect and the control samples were possible. The linear regression comparison of the field results to the traditional laboratory results for RDX resulted in a slope of 1.2614; however, if only concentration below 5 mg/L are considered, the result is a slope of 0.8614. Trinitrobenzene was the only compound investigated that showed significant differences at the 95% level of confidence. The laboratory and field results for all the detected MC in groundwater are plotted in Figure 10, showing generally good agreement (slope=0.9834) between the two techniques below a concentration of 10 mg/L (see inset). Concentrations above 10 mg/L significantly bias the results, such that a slope of 0.7389 is observed.

Table 14. Slopes from linear regression analysis of Griffin 450 results versus traditional HPLC results for individual MC.

	NB	1,3-DNB	2,4-DNT	TNB	TNT	RDX
Slope complete data set	N.A.	.8635	0.881	1.5228	1.0271	1.2614
Slope of truncated data	N.A.	0.8635	0.881	0.9407	1.0377	0.8614

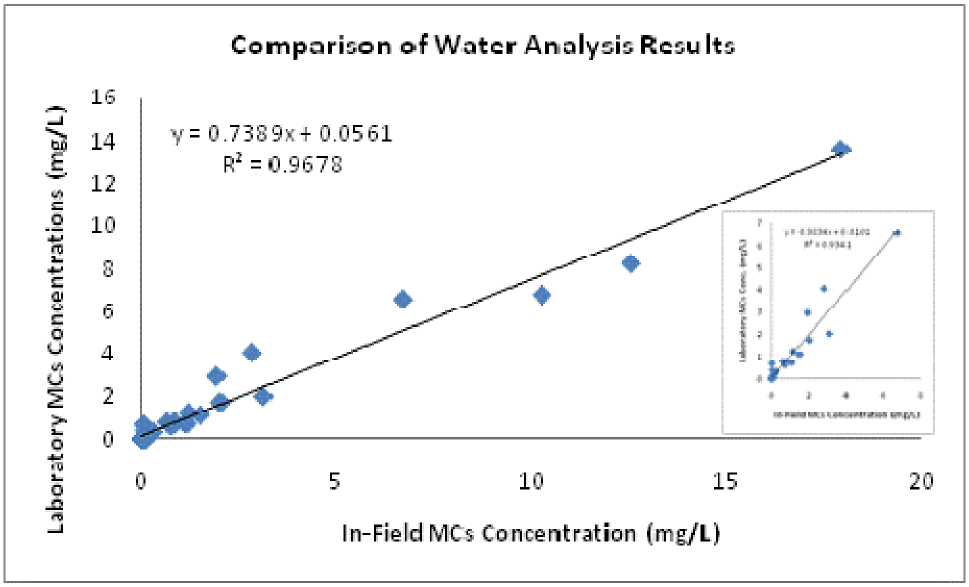

Figure 10. Comparison of field and laboratory MC concentration data for groundwater samples.

Inset shows the linear regression fit of data under 10 mg/L.

The compounds NB, 1,3-DNB, 2,4-DNT, TNB, TNT, and RDX were evaluated. Split groundwater samples were collected and analyzed for these compounds to compare the results from a field-portable GC/MS method (denoted by the variable *F*) to the results from a conventional fixed laboratory method (denoted by the variable *L*). NB was not detected in any of the groundwater samples; therefore, only a comparison of the spike recoveries was possible. Parametric and non-parametric linear fits were done for the remaining five compounds.

The regression line data in Table 14 demonstrates that the slopes are between 0.8 to 1.2 except for TNB and RDX. However, the TNB data was skewed somewhat by two samples with high concentrations. A similar effect was observed for RDX with one high concentration sample skewing the results. These samples reflect the linear dynamic range limitations of the field instrument. When large sample preconcentration factors result from the SPE procedure, the data can fall outside the linear dynamic range of the field instrument. When truncated data sets of samples below a given value (5 mg/L for instance) are considered, they show that there are ranges where the data is comparable to the laboratory results. See below, where F corresponds to Griffin field data and L corresponds to laboratory HPLC data.

Compound	**Relationship**	**Remarks**
1,3-DNB	$F \approx L$	*$F \leq 0.3$ ppm*
2,4-DNT	$F \approx L$	*$F \leq 0.2$ ppm*
TNT	$F = L$	*0.05 ppm $\leq F \leq 10$ ppm; $F < 0.05$ screening-level*
RDX	$F \approx 0.7 L$	*Use for screening-level purposes only*
TNB	$F = 1.5 L$	*0.05 ppm $\leq F \leq 10$ ppm; $F < 0.05$ screening-level*

The field method for RDX possessed a negative bias relative to the fixed laboratory method and exhibited relatively large variability across all concentration ranges evaluated. The field results were about 70% of the laboratory results on the average for concentrations below 1 mg/L. There was variable quantitative agreement for individual split samples. However, there was excellent qualitative agreement between the field and laboratory results. Therefore, it is suggested that the field method provides only screening-level data for RDX. The field method may possess positive biases for 1,3–DNB and 2,4-DNT. However, these biases are <0.001 mg/L on average and seem too small to be of any practical significance. The field method also consistently exhibits a significant positive bias for TNB. There was a very strong correlation between the laboratory and field methods for concentrations greater than about 0.05 mg/L to the highest reported concentration, but the performance of the field method was relatively poor at smaller concentrations. A positive bias was identified by both the sign test and Prentice-Wilcoxon test and via visual examinations of the box plots. The bias is relatively small (about 0.002 mg/L on average) but may be indicative of a lack of agreement between the field and laboratory method.

Control (Blank and LCS) and matrix spike (MS and MSD) samples were analyzed each day as part of the analytical batch of samples generally resulted in analyte recoveries within the DoD QSM limits (Table 4). However, not all results are within the acceptance limits, suggesting that poor recovery can be an issue if the samples are not thoroughly dry prior to injection into the instrument inlet. RDX, in particular, shows poor recovery of matrix spikes, indicating the difficulty with which is encountered with RDX analysis by GC methods.

Matrix spike recoveries were within DoD QSM acceptance limits for NB, 1,3-DNB, 2,4-DNT, and TNT for all days except the second day of sampling at LAAP. The well sample selected on Day 2 at LAAP was highly contaminated and the spike was too low to be detected in all cases except for NB, which was not present in the matrix water. RDX recoveries were within DoD

QSM limits in only 50% of the LCS control samples and were consistently low in the matrix spike samples.

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8.0 COST ASSESSMENT

One of the objectives of this demonstration was to document the cost savings associated with this field analysis technology compared with traditional laboratory analysis. The calculated cost savings of this technology were estimated to be 50% of the traditional analytical costs. However, the cost savings for a typical site were ~7-10%, based on actual accrued costs at MAAP.

8.1 COST MODEL

Documented costs include all equipment (capital) costs, disposal costs, shipping costs, sampling costs, labor costs, travel, and per diem. Table 15 documents and explains these costs. Table 16 documents the labor costs associated with field sampling and analysis. Table 17 details the cost per sample for both the traditional laboratory and field analysis. The cost for the field analysis is approximately 50% of the laboratory analysis per sample. However, analysis of actual accrued costs for MAAP, a typical site, showed that the field analysis was 90% of the laboratory analysis cost.

Table 15. Cost model for field analysis of groundwater.

Cost Element	Data to be Tracked	Estimated Costs	
Start-up	• Instrument purchase	Griffin 450	$105,000
Field deployment	• Personnel required and associated labor • Materials	Technician, 10 h	$900
		Materials	$100
Field analysis	• Personnel required and associated labor • Materials • Deployment costs	Field technician, 80 h Mass spectrometrist, 40 h	$7,200 $3,600
		Materials	$1011
		Per diem	$1740
		Truck	$750
		Mileage, $0.52 per mile	$507
Laboratory analysis	• Materials • Cost per sample analysis • Shipping	Materials	$ Included in cost per sample
		Analysis cost per sample	$250
		Shipping per sample	$40
Material cost	**Supplies for Well Water Sampling:** **Field sampling deployment:** Teflon tubing: • Depth of well and location of screened interval Sample containers: • Amber bottles (1 L) and vials (10 mL and 1.5 mL) Extraction consumables: • SPE columns • Acetonitrile • Centrifuge tubes (15 mL) • Disposable pipettes (10 mL)	Teflon tubing, per ft	$4.05
		Amber Bottles, per week	$545
		SPE cartridges, per week	$336
		Acetonitrile, 1 L	$130
		Centrifuge tubes, per week	$35
		Disposable pipets, per week	$19
	GC-MS Consumables • LTM column, guard columns and inlet liners • Helium	LTM column	$3000
		Consumables kit	$570
		Helium, per bottle	$317

Table 15. Cost model for field analysis of groundwater (continued).

Cost Element	Data to be Tracked	Estimated Costs	
Material cost (continued)	**HPLC Consumables** • Column • Solvent • Helium	Dual confirmation columns	$2000
		Acetonitrile, methanol	$500
		Helium	$ 0
Waste disposal	Purge water was brought back to the ERDC for disposal.	No cost tracking	
Operation and maintenance costs	No unique requirements anticipated, but issues that arise were noted.		

Table 16. Labor costs for field sampling and analysis.

Cost Element	Data Tracked During the Demonstration	Costs	
Mobilization costs[1]	• Instrument packing and unpacking	Lab technician, 8 h	$720
	• Materials for analysis	Supplies	$1850
Cost of analysis[2]	• Sample preparation	Lab technician, 8 h	$720
	• Sample analysis and data reporting	Lab technician, 8 h	$720
Waste disposal	• Disposal costs for water collected	NA[3]	NA[3]

[1] Cost for total trip

[2] Cost per 10 analysis

[3] Disposal costs and field sampling incurred for field- or laboratory-based analysis.

Table 17. Cost per sample.

	Laboratory Costs	Field GC-MS Costs
Overnight Shipping + Ice	$40/sample[1]	NA
Analysis	$250/sample	$60/sample[2]
Field Supplies	NA[3]	$105/sample[4]
Total	$290/sample	$165/sample

[1] $157.00 for overnight shipping of 30 lb cooler from Denver, CO, to Vicksburg, MS, which could contain four 1 L samples with ice

[2] Cost calculated as two field technicians for extraction and analysis at a rate of 2.5 samples/hour and includes instrument mobilization

[3] Costs included in the analysis

[4] Costs include ~120 analyses per chromatography column, helium, and other consumables

Data for the cost assessment was tracked in an Excel spreadsheet and included the following parameters: labor, materials, travel, and analysis. Labor was tracked according to the type of personnel required to collect samples (field technician, engineer, program manager, etc.) and their associated labor hours. Material purchases and analytical laboratory costs were tracked according to sample collection, analysis, and transport. Capital costs associated with the acquisition of the field- and laboratory-based analytical instrumentation were also included. The Griffin 450 GC-MS had an initial cost of $105,000, compared to a typical laboratory HPLC system that has an initial cost of approximately $70,000, depending on instrument manufacturer and specifications.

8.2 COST DRIVERS

Traditional fixed laboratory analytical cost will not decrease substantially with time; rather costs have remained relatively stable over the past 10 years for Method 8330 analysis. Furthermore, shipping costs will continue to increase with increasing fuel costs and transportation costs. Labor costs for field deployment will also increase; however, this increased expense is incurred for sampling regardless of field or laboratory analysis. The increased labor costs will, however, increase the field analysis cost. There are several intangible benefits to the field-portable instrumentation, which include near-real-time availability of the data and the identification of unknown compounds or new contaminants. The potential benefits of near-real-time analysis are more pronounced during a site investigation phase, where analyte concentrations could impact well installation locations. In such a scenario, groundwater monitoring wells could be installed as field instrumentation provided data on analyte concentration, effectively “plume mapping” the site in near-real-time. Additionally, the selectivity of the GC-MS for analyte confirmation allows the technology to be applied to other classes of contaminants, such as PAHs and PCBs, among others.

8.3 COST ANALYSIS

The MAAP site was selected as a representative site for the cost analysis. The site at MAAP was actively sampled and therefore more accurately represented adding the field analysis to a preexisting sampling regime. The life-cycle analysis comparing standard laboratory analysis and the Griffin 450 GC-MS field analysis are shown in Table 18. Total costs incurred for field and laboratory analysis through 10 years are shown; however, the field instrument has a life expectancy of approximately 7 years (as demonstrated by the current Griffin 400 instrument), compared to a laboratory instrument which might be expected to last 10 years. Figure 11 compares the total costs for the standard laboratory analysis and the Griffin 450 GC-MS field analysis. The field analysis total costs assumes 12 five-day sampling events yearly and 25 samples analyzed per sampling event for a total of 300 samples analyzed per year ($247,739/year). The total cost for Year 1 of the field analysis includes the purchase of the Griffin 450 GC-MS ($105,000). The laboratory analysis total cost assumes 300 samples are analyzed yearly (equivalent to 25 samples analyzed 12 times a year) for a yearly cost of $277,360. This cost also includes shipping at approximately $40/sample. It should be noted that this shipping cost is highly conservative, where it estimates that four 1 L samples could be shipped for $160 over a given distance (e.g., Denver, CO, to Vicksburg, MS). Due to the need to ship additional waters (e.g., for QC purposes), and the fact that distances could be greater, this shipping estimate should be considered a lower rather than upper bound.

The cost difference between the field and laboratory analysis is also shown in column 4 of Table 18. Figure 12 shows the cost difference between the field and laboratory analysis as a function of time. The breakeven point between the two analyses occurs in Year 3. The laboratory analysis costs assume that no startup costs were incurred. However, new HPLC instrumentation would more than likely need to be purchased during a 10-year cycle. The breakeven point occurs in Year 1 when the cost of a new HPLC ($80,000) is taken into account. There are, however, intangible benefits of the field instrumentation, including near-real-time availability of data, which may be important during well installation or plume delineation, as well as ability to identify unknown compounds with the mass spectrometer. While these capabilities may not be directly applicable to Long Term Monitoring activities (e.g., a set number of wells will already be installed), they are "value added."

Table 18. Life-cycle cost.

Year	Field Analysis Total Costs	Laboratory Analysis Total Costs	Laboratory - Field Analysis Costs
1	$352,739[1]	$277,360	$-75,379
2	$600,478	$554,719	$-45,759
3	$848,217	$832,079	$-16,138
4	$1,095,956	$1,109,438	$13,483
5	$1,343,695	$1,386,798	$43,103
6	$1,591,433	$1,664,158	$72,724
7	$1,839,172	$1,941,517	$102,345
8	$2,086,911	$2,218,877	$131,966
9	$2,334,650	$2,496,236	$161,586
10	$2,582,389	$2,773,596	$191,207

[1] Includes purchase of Griffin 450 at $105,000. Yearly field analysis costs are $247,739.

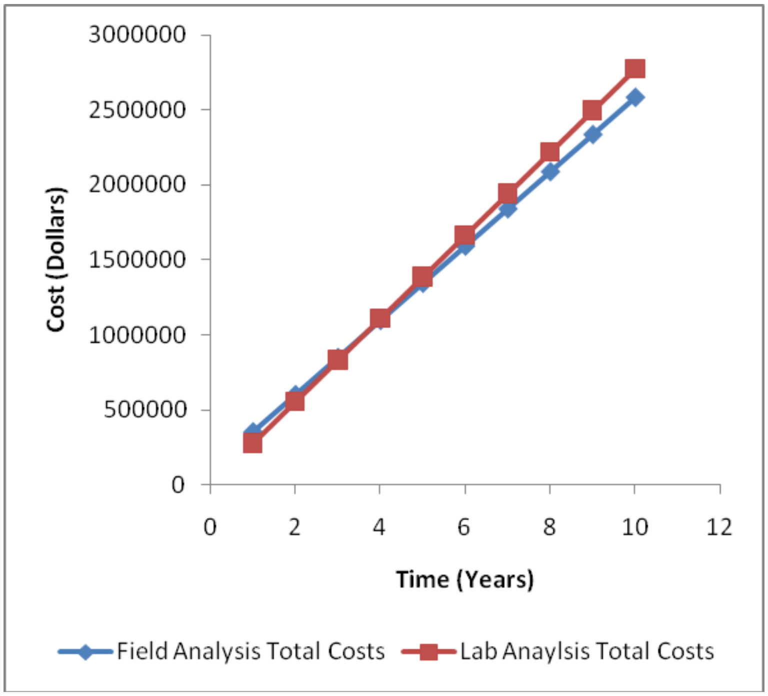

Figure 11. Total cost for traditional laboratory ($27,860/year) and field analysis ($247,739/year).

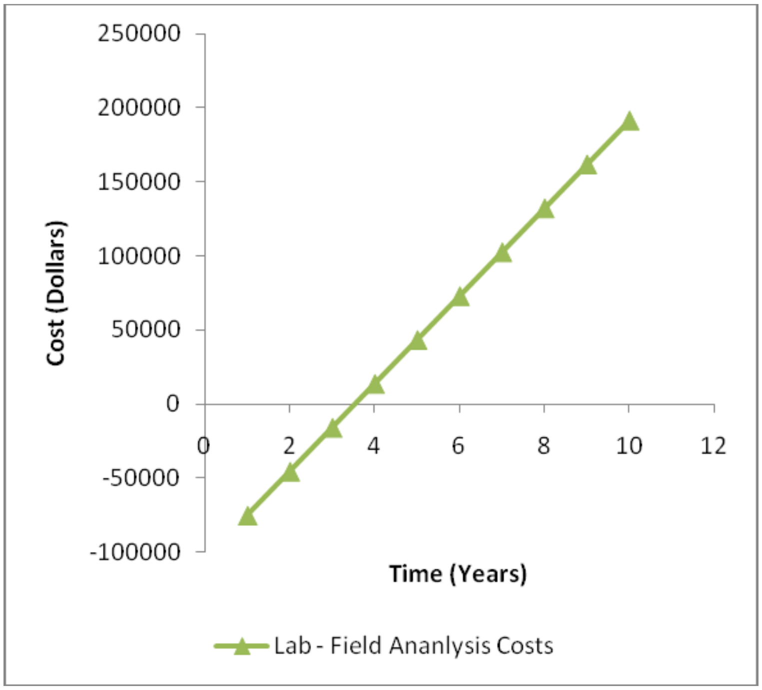

Figure 12. Difference in cost between the traditional laboratory analysis and the field analysis.
Breakeven point occurs at 3.54 years.

8.3.1 Net Present Value (NPV) Analysis

The life-cycle costs (LCC) of both the field and traditional laboratory analysis were calculated (Fuller and Petersen, 1996) for 7- and 10-year study periods. These study periods were selected based on life expectancy of the field and laboratory instruments. Future costs were discounted to NPVs using rates from Energy Price Indices and Discount Factors for LCC Analysis (Rushing et al., 2010). The 7-year LCC for the field and laboratory analysis were found to be $1,745,032 and 1,836,123, respectively (Table 19).

Benefits of the field analysis are primarily in the form of future operational savings; therefore, the net savings (NS) of the field analysis relative to the traditional laboratory analysis was calculated. The NS of the field analysis to the traditional laboratory analysis over 7 years is $91,091.02 in present-value dollars. The field analysis becomes cost effective at approximately 3.7 years when corrected for present-value.

Table 19. NPV life-cycle cost.

Study Period	LCC Field Analysis	LCC Laboratory Analysis	Net Savings (Laboratory - Field)
7	$1,745,032	$1,836,123	$91,091.02
10	$2,384,199	$2,551,712	$167,513.2

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9.0 IMPLEMENTATION ISSUES

A technical report is currently being drafted that will be useful for other organizations to lessen the learning curve required to successfully bring the demonstrated technology online. Furthermore, through discussions with researchers and innovative technology advocates at USEPA, we are pursuing regulatory acceptance of the technology for quantitation of MC in groundwater. Due to the fact that GC-MS is already a regulatory approved analytical methodology, acceptance of the current field application is based solely on the ability to generate laboratory-quality data with similar reporting limits and costs. Currently, a significant drawback to implementation of the technology is the requirement to have a trained and experienced analytical chemist on staff to operate the instrument, properly maintain it, and troubleshoot as needed. The instrument has been shown to have limitations in detection limits, stability, and linear dynamic range, when compared to traditional laboratory based analytical equipment. The field portable Griffin GC-MS appears to have quantitative capabilities for 1,3-DNB, 2,4-DNT, and TNT. However, at this point it appears to be only suitable for screening of TNB and RDX. The regression line data demonstrate that the slope is within our 0.8 to 1.2 limit except for TNB and RDX. The TNB data is skewed somewhat by two samples with high concentrations. A similar effect is observed for RDX with one sample skewing the results. These samples reflect the linear dynamic range limitations of the current instrument when large sample preconcentration factors result from the SPE procedure. Further testing would be required to make stronger conclusions owing gaps between the high and low concentrations and to relatively few data points in some data sets. The initial cost of the instrument, at approximately $100,000, also represents a formidable obstacle, which causes the breakeven cost point to be several years in the future, depending on the analytical workload of the user. However, the technology has applications far beyond MC in groundwater and therefore may be applicable to other environmental investigations that will add to the return on investment.

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APPENDIX A

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