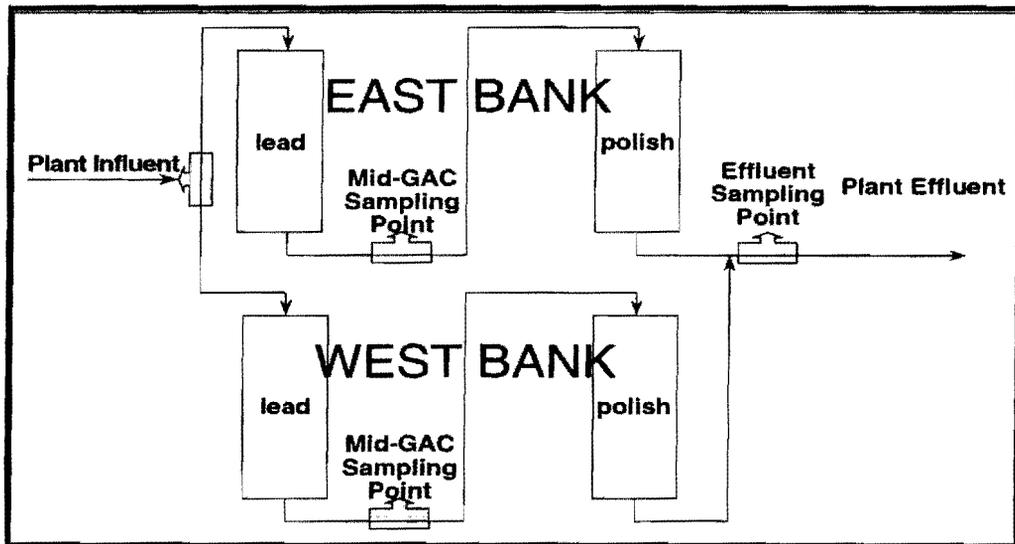




Dynamic Field Activity Case Study: Treatment System Optimization, Umatilla Chemical Depot



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Notice

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Abbreviations

1,3-DNB	1,3-dinitrobenzene
1,3,5-TNB	1,3,5-trinitrobenzene
2,4-DNT	2,4-dinitrotoluene
2,6-DNT	2,6-dinitrotoluene
C _e	concentration of effluent
C _i	concentration of influent
DEQ	Department of Environmental Quality
EPA	U.S. Environmental Protection Agency
FAM	field-based analytical methods
GAC	granular activated carbon
GPM	gallons per minute
HMX	high melting explosive
HPLC	high performance liquid chromatography
mg/L	micrograms per liter
NB	nitrobenzene
O&M	operation & maintenance
OU	operable unit
QC	quality control
RDX	Royal Demolition Explosive
RI	remedial investigation
ROD	record of decision
TNT	trinitrotoluene
UMCD	Umatilla Chemical Depot

Operation and Maintenance Umatilla Chemical Depot, Hermiston, Oregon

Abstract

The Army used a dynamic field activity (i.e., a project that combines on-site data generation with on-site decision making) to optimize the treatment system at the Umatilla Chemical Depot in 1999. The use of field-based analytical methods (FAMs) allowed them to maximize the usefulness of granular activated carbon and minimize the number of samples sent to a fixed laboratory for confirmation. The data provided by the FAMs met project requirements and improved the overall project quality control by providing rapid feedback on treatment problems as they occurred. Since its implementation, the optimized treatment system has been providing the Army with an annual savings of at least 45 percent.

Background

The Umatilla Chemical Depot (UMCD) was established as an Army ordnance depot in 1941 for the purpose of storing and handling munitions. It covers nearly 20,000 acres in northeastern Oregon in Morrow and Umatilla Counties, approximately five miles west of Hermiston, Oregon, and six miles south of the Columbia River. In 1988, UMCD was included in the Department of Defense's Base Realignment and Closure (BRAC) Program, which required its conventional ordnance storage mission to be transferred to another installation.

Beginning in the 1950s, UMCD operated an explosives washout plant on site. Munitions were opened and washed with hot water to remove and recover explosives. The plant was cleaned weekly, and the wash water, which contained high concentrations of explosives, was disposed of in two nearby unlined lagoons. The lagoons received a total of about 85 million gallons of wash water during plant operations. Although lagoon sludges were removed regularly during operation, explosives contained in the wash water percolated through the soil and into the groundwater below the lagoons.

A CERCLA remedial investigation (RI) of the explosives washout lagoons was initiated in 1988 to determine the nature and extent of contamination. Investigators discovered a 330-acre groundwater plume in an unconfined sandy aquifer made up primarily of Royal Demolition Explosive (RDX) with concentrations ranging up to 6,816 $\mu\text{g/L}$. Trinitrotoluene (TNT) was also in the groundwater at elevated concentrations (3,900 $\mu\text{g/L}$), but the TNT was generally confined to the area under and near the lagoons. In 1994, the Record of Decision (ROD) for the groundwater operable unit (OU) selected groundwater extraction and granular activated carbon (GAC) treatment as the remedy. Exhibit 1 lists the chemicals of concern and their associated cleanup levels.

**Exhibit 1
Groundwater Remediation Requirements**

Chemicals of Concern	Cleanup Criteria	Highest Concentration
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	2.1 µg/L	6,816 µg/L
2,4,6-trinitrotoluene (TNT)	2.8 µg/L	3,900 µg/L
1,3,5-trinitrobenzene (1,3,5-TNB)	1.8 µg/L	441 µg/L
1,3-dinitrobenzene (1,3,-DNB)	4.0 µg/L	24.4 µg/L
2,4-dinitrotoluene (2,4-DNT)	0.6 µg/L	497 µg/L
2,6-dinitrotoluene (2,6-DNT)	1.2 µg/L	5.3 µg/L
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazine (HMX)	350 µg/L	1,448 µg/L

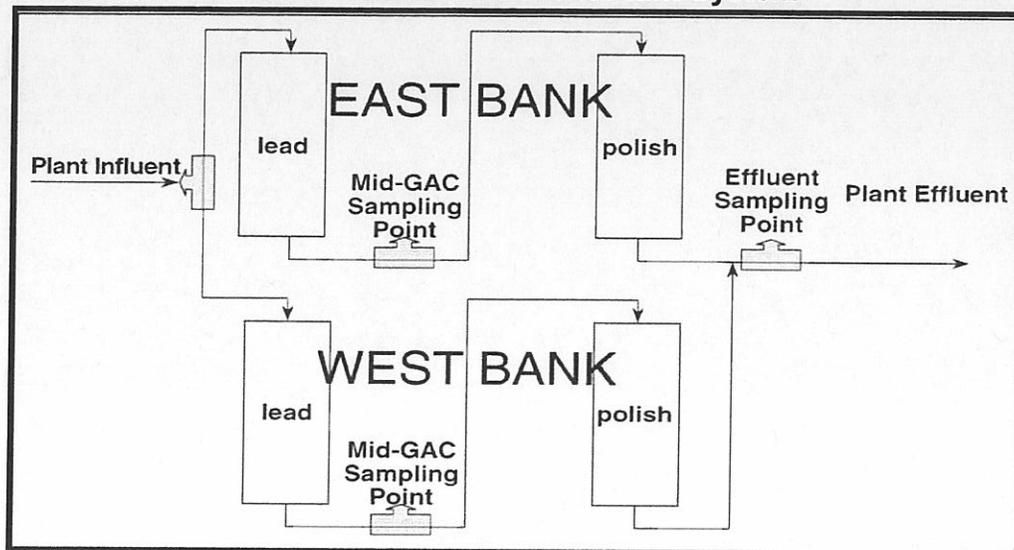
The Army Corps of Engineers (the Corps), in coordination with EPA, took responsibility for the design and operation of the treatment system. By using FAMs in the operation of the treatment plant and by undertaking a dynamic optimization process, the BRAC Cleanup Team, which included the Corps, the Corps' contractor, Oregon Department of Environmental Quality (DEQ), and EPA, demonstrated a quantifiable savings in the plant's annual operational expenses of greater than 45 percent, which represents a cost reduction of approximately \$180,000 per year. In addition, unquantifiable savings were achieved through better treatment plant quality control (QC).

Original Treatment System Design

Startup of the treatment system occurred in January 1997. Three extraction wells pumped approximately 1,300 gallons per minute (gpm) of contaminated groundwater to two parallel treatment lines, each containing two tanks with 20,000 pounds of GAC. Exhibit 2 presents a schematic drawing of the treatment system. Water entered the treatment area in a single pipe that split to feed two parallel systems that each contained a lead tank and a polishing tank. Water exiting the polishing tanks of each system was recombined before it was piped into three aquifer recharge areas. Twenty-seven groundwater monitoring wells and the three extraction wells were used to evaluate the progress and effectiveness of the cleanup. These wells were sampled on a regular schedule. The sample analysis was performed by an off-site laboratory to get a complete evaluation of all the contaminant levels.

The original operating procedure for each of the tank systems included sampling the water at a port in the piping between the lead and polishing tank (i.e., mid-GAC) on a weekly basis and analyzing the samples using an on-site colorimetric method. Based on FAM results, when concentrations of RDX exceeded 5 µg/L at a mid-GAC port, the system was shut down. Water

Exhibit 2
Schematic Drawing of Umatilla Chemical Depot
Groundwater Treatment Plant System



samples were then collected for off-site analysis from each mid-GAC port and from the effluent sampling point to confirm that breakthrough had occurred in at least one of the lead tanks and that no breakthrough had occurred in the polishing tanks. Because the reliability of the FAM data had already been demonstrated prior to designing the procedure, the contents of both lead tanks were changed out for off-site regeneration before confirmatory data were received. The minimally contaminated polishing tank then became the lead tank. RDX was chosen as the primary chemical to monitor because it was the contaminant with the highest concentration and the lowest affinity for the GAC, therefore, it would be the first chemical exhibiting breakthrough.

Analytical Method Selection

Before designing the treatment system, the Corps and EPA conducted a study of all the available commercial and emerging FAMs to determine if any could be used for RDX and TNT. Their search uncovered three methods for RDX and five methods for TNT, all of which were classified as either immunoassay, colorimetric, continuous flow immunosensor, or fiber optic biosensor. All were tested and compared with SW-846 Method 8330, which utilizes high performance liquid chromatography (HPLC). Method 8330 has documented quantitation limits of 0.84 $\mu\text{g/L}$ for RDX and 0.11 $\mu\text{g/L}$ for TNT.

Based on the results of these tests, the colorimetric method was selected. It demonstrated detection limits of 3.8 $\mu\text{g/L}$ for RDX and 0.9 $\mu\text{g/L}$ for TNT (Craig et al., 1996), which was

acceptable because the action level set at the mid-GAC sampling port was 5 µg/L for RDX (the team has since shown that the site specific detection limit for RDX is 2.0 µg/L). The Standard Operating Procedures for FAMs used at UMCD can be viewed at: <http://www.epa.gov/superfund/programs/dfa/casestudies>.

Initial Year of Operation

During the initial year of operation, the BRAC Cleanup Team encountered two incidents that demonstrated the FAM was providing an added layer of QC protection. The first occurred after one routine change out. The FAM results indicated that the treatment system effluent contained 16 µg/L RDX so the system was immediately shut down. A subsequent investigation indicated that not all of the spent GAC had been removed from the tank when the GAC was replenished. If off-site analysis with the normal turnaround time of three weeks had been used to minimize analytical costs, the contaminated discharge would have continued during that period. In the second incident, FAM data allowed the team to correct a problem with a corroded butterfly valve that was allowing contaminated influent water to bypass the lead carbon unit. Again, the use of off-site analysis as the sole source of analytical data would have resulted in contaminated groundwater bypassing the lead GAC unit for several weeks before the problem was caught and corrected.

Although the startup period indicated that the FAM was providing significant benefits for the project by providing a high level of QC and by meeting the project requirements, other aspects of the treatment system seemed to be inadequate. The primary problem that the system operator noticed was that breakthrough was occurring on the lead tanks much sooner than expected, resulting in a very high expenditure on GAC. After some discussion, the BRAC Cleanup Team decided to perform an optimization study that would seek to reduce the systems operating costs.

Dynamic Optimization Study

To determine areas where efficiencies could be gained, the Corps reviewed the entire treatment system design. Initially, the study examined the system design parameters (e.g., size of the unit, flow rate, type of charcoal, and regeneration and charcoal make-up process). One problem they found and fixed immediately was that the type of GAC they were using did not contain an adequate pore size to effectively remove RDX from groundwater. By changing the GAC specification they were able to significantly improve the treatment system.

Another problem they discovered involved the system flow rate, which was providing too little contact time for the RDX. Because changing the system to provide an adequate dwell time involved expensive redesign and construction, the BRAC Cleanup Team set out to improve the system's efficiency and lower operating costs with the existing design. The team decided that the

best method of optimizing the system was to determine the most cost effective use of GAC and treatment monitoring analysis for the project as a whole, rather than as individual components. As a result, they developed and tested five sampling and analysis scenarios over four test cycles of carbon change-out starting in December 1997. After the first two test cycles, the team added a sixth scenario that would accommodate changes in contaminant concentrations over time. A summary and comparison of all six scenarios is presented in Exhibits 3 and 4.

The exhibits show the conditions for sampling and analysis for each scenario. The start-up conditions are the same for each, with RDX and TNT being tested with FAMs at the mid-GAC and effluent points. Each of the subsequent rows describes the sampling protocol that was used for each phase of the different scenarios. They describe where samples are collected (e.g., mid-GAC), the conditions for their collection (e.g., on a weekly basis), the analytical method used (e.g., FAM for RDX), and the conditions for ending or changing the sampling and analysis strategy (e.g., RDX values exceed 5 µg/L at mid-GAC).

Scenario 2 represents the initial, or baseline, treatment design in which the system was shut down and the GAC of the first tank was replaced as soon as concentrations at the point between the two tanks (i.e., mid-GAC) exceeded 5 µg/L RDX. The new scenarios allowed RDX concentrations at the mid-GAC to substantially exceed 5 µg/L because the GAC in the lead tank had not been optimally loaded, and the polishing tank, with careful observation, prevented the plant's effluent from exceeding the cleanup criteria.

The BRAC Cleanup Team took a dynamic approach to improve efficiency based on evaluations of the data being generated. For instance, the sixth scenario involved sampling decisions based upon an innovative approach of using the ratio of the RDX concentrations between the effluent at the mid-GAC point (C_e) and the influent to the treatment system (C_i). When the ratio was less than 0.25, they would collect samples once every two weeks. As concentrations at the mid-GAC increased, and the ratio of C_e/C_i was between 0.25 and 0.50, samples would be collected once a week. Once the ratio of concentrations rose above 0.50, samples would be collected daily until break through in the lead tank was detected. These sampling protocols would allow the lead tank GAC to be fully utilized.

The results of the first three test cycles were used to select the two most cost effective scenarios for comparison during the fourth test cycle. These were scenarios 4 and 6. Based on the findings of the fourth test cycle, scenario 6 was selected as the most cost effective option. Exhibit 5 presents a summary of treatment cost data for each scenario through the four test cycles.

Cost Savings Analysis

There are two useful ways of calculating cost savings for this case study: to compare the cost of the optimized treatment design with the cost of the original treatment design; and to compare the cost of using the FAM as part of the system design with using the off-site analysis

Exhibit 3
Comparison of Dynamic Optimization Study Scenarios 2, 4, and 6¹

Activity		Scenario 2 (original design)	Scenario 4	Scenario 6
Start-up	Sample Location	Mid-GAC and Effluent	Mid-GAC and Effluent	Mid-GAC and Effluent
	FAM	RDX and TNT	RDX and TNT	RDX and TNT
	8330	None	None	None
1 st Sampling Protocol	Sample Location	Mid-GAC	Mid-GAC	Mid-GAC and Effluent
	FAM Rate	RDX Weekly	RDX Weekly	RDX Not Applicable
	Condition	< 5 ppb	< 150 ppb	Begin on week 5
	8330	None	None	None
2 nd Sampling Protocol	Sample Location	Mid-GAC and Effluent	Effluent	Mid-GAC and Effluent
	FAM Rate	RDX Not Applicable	RDX Every other day	RDX Every other week
	Condition	> 5 ppb = shutdown (Mid-GAC)	> 150 ppb, until breakthrough at effluent	$0 \leq (C_e/C_i) \leq 0.25$
	8330	Mid-GAC and effluent confirmation @ shutdown	Mid-GAC and effluent confirmation @ shutdown	None
3 rd Sampling Protocol	Sample Location			Mid-GAC and Effluent
	FAM Rate			RDX Every week
	Condition			$0.25 \leq (C_e/C_i) \leq 0.50$
	8330			None
4 th Sampling Protocol	Sample Location			Mid-GAC and Effluent
	FAM Rate			RDX 3 per week
	Condition			$(C_e/C_i) > 0.50$, until RDX break through
	8330			Mid-GAC and effluent confirmation @ shutdown

alone. The following discussion provides an estimate of the quantifiable benefits of using FAMs in the optimized treatment system.

¹ Each of the six scenarios used different sampling protocols that dictated the type and frequency of analyses. For scenario 2, the original sampling design, there were only two protocols used after startup. As long as RDX concentrations at the mid-GAC were below 5 ppb, samples were collected weekly. Once mid-GAC concentrations exceeded 5 ppb, the system would be shutdown and samples would be collected at the mid-GAC and effluent for analysis with SW-864 Method 8330.

Exhibit 4
Comparison of Dynamic Optimization Study Scenarios 1, 3, and 5

Activity		Scenario 1		Scenario 3		Scenario 5	
Start-up	Sample Location	Mid-GAC and Effluent		Mid-GAC and Effluent		Mid-GAC and Effluent	
	FAM	RDX and TNT		RDX and TNT		RDX and TNT	
	8330	None		None		None	
1 st Sampling Protocol	Sample Location	Mid-GAC		Mid-GAC		Mid-GAC	
	FAM Rate	RDX	Weekly	RDX	Weekly	RDX	Weekly
	Condition	< 50 ppb		< 100 ppb		< 150 ppb	
	8330	None		None		None	
2 nd Sampling Protocol	Sample Location	Mid-GAC		Mid-GAC		Mid-GAC and Effluent	
	FAM Rate	RDX	3 per week	RDX	Bi-weekly	RDX	Not Applicable
	Condition	100 ppb		< 200 ppb		>150 ppb =shutdown (Mid-GAC)	
	8330	Mid-GAC during sampling scheme		None		Mid-GAC and effluent confirmation @ shutdown	
3 rd Sampling Protocol	Sample Location	Mid-GAC and Effluent		Mid-GAC and Effluent			
	FAM Rate	RDX	Every other day	RDX	Every other day		
	Condition	>100 ppb mid-GAC, until breakthrough @ effluent		>200 ppb mid-GAC, until breakthrough @ effluent			
	8330	Mid-GAC and effluent confirmation @shutdown		Mid-GAC and effluent confirmation @ shutdown			

Optimized Treatment System Savings

Although the optimization study cannot be used to directly compare scenario 2 (the original treatment design) with scenario 6 (the optimized treatment design),² a minimum cost savings can be extrapolated from the study by first comparing the cost difference between scenarios 2 and 4 in cycle I, then by comparing the cost difference between scenarios 4 and 6 in cycles III and IV. In cycle I, scenario 4 (\$0.344/1,000 gallons) is about 30 percent less expensive than scenario 2 (\$0.486/1,000 gallons). In cycles III and IV, scenario 6 (\$0.344 and \$0.371/1,000 gallons) is 5 to 18 percent less expensive than scenario 4 (\$0.360 and \$0.449/1,000

² Treatment costs for scenario 2 in cycles II, III, and IV were not representative because the treatment system protocols for scenario 2 called for a completely new lead tank to be installed as soon as breakthrough occurred at the mid-GAC, whereas during the optimization study the lead tank was partially loaded with RDX to accommodate the other five scenarios. Direct comparison is further complicated by the fact that RDX influent concentrations fell from 700 µg/L in cycle I to 300 µg/L in cycle IV.

Exhibit 5
Comparison of Water Treatment Cost for Each Scenario Tested

Test Cycle Number	Scenario	Carbon Cost³	Analytical Cost³	Other O&M Cost³	Total Cost³
I	1	\$0.162	\$0.143	\$0.085	\$0.390
	2	\$0.280	\$0.117	\$0.089	\$0.486
	3	\$0.163	\$0.097	\$0.085	\$0.345
	4	\$0.162	\$0.097	\$0.085	\$0.344
	5	\$0.169	\$0.114	\$0.085	\$0.368
	6	N/A	N/A	N/A	N/A
II	1	\$0.275	\$0.196	\$0.089	\$0.560
	2	\$0.856	\$0.198	\$0.109	\$1.16
	3	\$0.275	\$0.157	\$0.089	\$0.521
	4	\$0.280	\$0.160	\$0.089	\$0.529
	5	\$0.350	\$0.200	\$0.091	\$0.641
	6	N/A	N/A	N/A	N/A
III	1	\$0.123	\$0.199	\$0.083	\$0.405
	2	\$0.343	\$0.128	\$0.091	\$0.562
	3	\$0.123	\$0.158	\$0.083	\$0.364
	4	\$0.123	\$0.154	\$0.083	\$0.360
	5	\$0.183	\$0.106	\$0.085	\$0.374
	6	\$0.123	\$0.138	\$0.083	\$0.344
IV	1	N/A	N/A	N/A	N/A
	2	N/A	N/A	N/A	N/A
	3	N/A	N/A	N/A	N/A
	4	\$0.152	\$0.213	\$0.084	\$0.449
	5	N/A	N/A	N/A	N/A
	6	\$0.152	\$0.135	\$0.084	\$0.371

³ Costs are calculated per 1,000 gallons.

gallons). Therefore, scenario 6 should be at least 30 percent less expensive than scenario 2. The treatment system currently treats approximately 600,000,000 gallons per year. An extrapolation of this estimate indicates that the optimized system is saving approximately \$95,000 in operating expenses per year.⁴

⁴ The yearly operating cost for scenario 2 based on cycle I would have been \$291,600; the yearly operating cost for scenario 4 based on cycle I would have been \$206,400; since scenario 6 was 5 to 18 percent less expensive than scenario 4 in cycles III and IV, a conservative extrapolation results in a yearly operating cost of \$196,080 (\$206,400 minus 5 percent) for scenario 6 in cycle I. The estimated savings are therefore \$95,520.

Another check on this estimate is to examine the number of days the GAC lasted before and after the optimization system was initiated. Previously, the GAC required removal after 40 to 60 days, even after the more appropriate GAC was used. The new system, however, allowed the GAC to last between 125 and 170 days over the first four cycles of its use. While this comparison provides only a rough indication of the impact of the new treatment design and does not attempt to estimate the affect of decreasing concentrations over time, it does provide corroborating evidence that the new protocols are more efficient in their use of GAC than the original system.

Cost Savings of Using Field-Based Analytical Method

Although the previous calculation provides ample evidence for the financial benefits of optimizing a groundwater treatment system using FAMs, it does not measure the full impact of this approach because many project managers use only off-site analysis for treatment system monitoring. Consequently, it is useful to determine the cost savings of this project compared with what it might have cost if FAMs were not considered at all.

Cost of RDX/TNT Colorimetric Method

The cost of the FAM from the manufacturer was \$24 per analysis. Since RDX and TNT require two separate analyses, the two together cost \$48. However, when the cost of labor, expendables, data validation, and data management are also considered, the FAM cost the project \$237.70 for analysis of RDX and \$289.99 for both RDX and TNT. A detailed breakdown of how these costs were derived is provided in Appendix A.

Cost of SW-846 Method 8330

The off-site laboratory contracted to run Method 8330 charged \$275 for regular turnaround (3 weeks, providing results on all nitroaromatics and nitroamines). The fully loaded cost (labor, shipping, data validation, and data management) of an analysis for the project was \$466.26 (see Appendix A for detailed costing information). However, if Method 8330, conducted at an off-site laboratory, were the only source of analytical data, 36-hour turnaround would be required at an additional cost of \$400. Because a 36-hour turnaround was very rarely used by the project, the fully loaded cost is not available, however, it would likely be approximately \$600 per sample based on the fully loaded cost of the regular turnaround off-site samples.

Total Savings

The first step in calculating the additional project savings from using FAMs is to estimate the cost increase of using quick turnaround off-site analysis in place of the analytical procedures used in the original treatment system design. Although the exact figure is difficult to calculate because both off- and on-site analyses were used in these protocols (i.e., scenario 2), this calculation can be simplified by observing that at each sampling event, the analytical cost would have, at a minimum, doubled if off-site analysis were the only method (see Exhibits 3 and 4). For example, during startup, RDX and TNT were analyzed with FAMs at an analytical cost of \$290. Had off-site analysis been used, the cost would have been \$600. Therefore, the analytical cost of the original treatment design would have been at least twice the amount found in the optimization study's scenario 2 of cycle I. These analytical protocols would result in a total project cost of \$0.603 per 1,000 gallons⁵. The same logic used to estimate the savings from scenario 6 may be used to derive the total project savings. Consequently, because scenario 4 is 45 percent less expensive than scenario 2 (in cycle I) would have been if it had used only off-site analysis, scenario 6 must be at least 45 percent less expensive as well. Therefore, total project savings are approximately \$180,000 per year (i.e., approximately twice the savings of \$95,000 calculated for the optimized system versus the original system).

Moreover, additional, unquantifiable, savings have been demonstrated by having the data available in the field to make site operating decisions. As mentioned earlier, rapid sample analysis enabled a timely resolution to a leaking valve problem and a carbon change-out mistake. Consequently, these data have resulted in better project QC and more effective site remediation. Likewise, it was this increased confidence in the project's QC procedures that provided regulators with the security to allow a plant operating strategy that completely utilizes the GAC until breakthrough is documented at the final effluent point.

Lessons Learned

Although the capabilities and limitations of the FAM for this groundwater pump-and-treat system were thoroughly researched before it was selected, a number of problems were discovered during the initial stages of its integration into the project. The lessons learned from this experience include:

- Method requirements must be clearly provided to the contractor;
- Site-specific matrices may require method modifications;
- FAM data were essential for the optimization process; and
- Undocumented analytical issues may exist for well researched methods.

⁵ From Exhibit 5: analytical cost of $\$0.117 \times 2 = \$0.234 + \$0.369$ (GAC and O&M) = \$0.603

Method Requirements Must Be Clearly Provided to the Contractor

During the project start-up period, the on-site laboratory was unable to produce the detection limits that a Corps researcher had documented. By investigating the problem, the BRAC Cleanup Team discovered a number of problems that had been caused by the contractor's misunderstanding of the method requirements. First, the FAM operator was neither a chemist nor had she been properly trained because the contractor did not understand the level of expertise that was needed to perform the work. Second, the laboratory facilities were inadequate because there was no running water within the trailer where the analyses were taking place. Third, cross-contamination was a problem because both the trailer used for analysis and the path for accessing water was dirty. Finally, some of the equipment selected was inadequate, such as pumps that did not provide enough suction for filtration. Consequently, it is critical that the project manager write a statement of work in such a way that contracting firms understand the level of training and qualifications necessary to perform and interpret the analysis and the conditions required for setting up the field laboratory.

Site-Specific Matrices May Require Method Modifications

The BRAC Cleanup Team initially was unable to obtain the required level of performance from the FAM due to the high levels of nitrates in the groundwater. By working with the method developers, the team was able to make modifications to improve performance for the site-specific matrix. To ensure that the modifications were properly documented, the site-specific SOPs for RDX and TNT analyses were also modified.

FAM Data Were Essential for the Optimization Process

The BRAC Cleanup Team found that their ability to analyze RDX and TNT within hours enabled them to proceed with the treatment plant optimization process. If the team had not been able to assess and report contaminant concentrations in the effluent water quickly, regulators would not have allowed the treatment plant to completely use the GAC units before they were replaced.

Undocumented Analytical Issues May Exist for a Well-Researched Method

The BRAC Cleanup Team discovered a number of significant issues affecting the results during the project start-up period as well as once the project was fully implemented even though the reliability of the FAM had been thoroughly tested in a laboratory. Their findings included:

- Method blanks should be run with each batch to identify contamination problems.

- Analyst-specific response curves should be developed because several steps in the method are very sensitive to analyst technique.
- Blank spikes developed with a second source standard and run through the extraction and analysis procedure provide valuable information on data quality.
- It is critical that the analyst note the color of the colorimetric response because an elevated absorbance reading, due to particulate matter, can be misinterpreted.
- Dinitroaromatic compounds will cause a false positive response.
- The method is very sensitive to the brand of acetone, deionized water, and reagents used.
- The method is heat and possibly light sensitive, consequently, the work station requires a constant temperature.

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Appendix A Treatment System Analytical Costs

Field Sampling and Analysis Costs (RDX and TNT)

Description	Quantity	Units	Unit Rate (\$)	Cost/Sample (\$)
Labor				
Sampling Labor	0.25	Lh*	31.70	7.93
Extraction and Analysis	2.00	Lh	16.83	33.66
QA Review/Reporting	1.00	Lh	27.79	27.79
Database and Statistics	0.50	Lh	27.79	13.90
Administration and Other	1.00	Lh	27.79	27.79
Direct Labor Subtotal				111.06
Fringe @ 33.75%				37.48
Labor and Fringe Subtotal				148.55
Test Kits				
EnSys Kit	1	each	24.00	24.00
Subtotal				24.00
Supplies				
Empore Filters w/10% loss	2	each	9.35	18.70
Glass fiber filter	1	each	0.30	0.30
Test tubes	2	each	0.80	1.60
Alumina-A Cartridge	1	each	1.75	1.75
Acetone	100	ml	0.01	1.00
Syringe and 0.45 filter	1	each	3.00	3.00
Sample Bottles	2	each	4.00	8.00
Misc. foil, DI water, vials	2	each	2.50	5.00
Subtotal				39.35
Equipment				
Vacuum Flasks	1	each	100.00	2.00
Vacuum Pump	1	each	448.00	1.00
Filter Apparatus	1	each	173.00	4.00
Photometer	1	each	1700.00	3.00
Misc. Bottles, Tubing, etc.	1	each	100.00	1.00
Subtotal				11.00
ODCs and Labor/Fringe Subtotal				222.90
G&A @ 18.27%				40.72
Total Estimated Cost				263.62
Fixed Fee @ 10%				26.36
Grand Total				289.98

* Labor hours

Field Sampling and Analysis Cost (RDX only) per Sample

Description	Quantity	Units	Unit Rate (\$)	Cost/Sample (\$)
Labor				
Sampling Labor	0.25	Lh*	31.70	7.93
Extraction and Analysis	1.50	Lh	16.83	25.25
QA Review/Reporting	0.50	Lh	27.79	13.90
Database and Statistics	0.25	Lh	27.79	6.95
Administration and Other	1.00	Lh	27.79	27.79
Direct Labor Subtotal				81.80
Fringe @ 33.75%				27.61
Labor and Fringe Subtotal				109.41
Test Kits				
EnSys Kit	1	each	24.00	24.00
Subtotal				24.00
Supplies				
Empore Filters w/10% loss	2	each	9.35	18.70
Glass fiber filter	1	each	0.30	0.30
Test tubes	1	each	0.80	0.80
Alumina-A Cartridge	1	each	1.75	1.75
Acetone	75	ml	0.01	0.75
Syringe and 0.45 filter	1	each	3.00	3.00
Sample Bottles	2	each	4.00	8.00
Misc. foil, DI water, vials	2	each	2.50	5.00
Subtotal				38.30
Equipment				
Vacuum Flasks	1	each	100.00	2.00
Vacuum Pump	1	each	448.00	1.00
Filter Apparatus	1	each	173.00	4.00
Photometer	1	each	1700.00	3.00
Misc. Bottles, Tubing, etc.	1	each	100.00	1.00
Subtotal				11.00
ODCs and Labor/Fringe Subtotal				182.71
G&A @ 18.27%				33.38
Total Estimated Cost				216.09
Fixed Fee @ 10%				21.61
Grand Total				237.70

* Labor hours

Fixed Laboratory 8330 Sampling and Analysis Cost per Sample

Description	Quantity	Units	Unit Rate (\$)	Cost/Sample (\$)
Labor				
Sampling Labor	0.25	Lh*	31.70	7.93
Packaging and Shipping	0.50	Lh	31.70	15.85
QA Review/Reporting	2.00	Lh	27.79	55.58
Database and Statistics	0.50	Lh	27.79	13.90
Administration and Other	1.00	Lh	27.79	27.79
Direct Labor Subtotal				121.04
Fringe @ 33.75%				40.85
Labor and Fringe Subtotal				161.89
Other Direct Costs				
Shipping Costs	1	each	15.00	15.00
Sample Bottles	1	each	4.00	4.00
Bubble Wrap, Ice, PPE, etc.	1	each	2.00	2.00
Cooler Replacement	1	shipment	0.50	0.50
Subtotal				21.50
Laboratory Costs				
8330 Analysis	1	each	150.00	150.00
EDF	0.25	package	100	25.00
Subtotal				175.00
ODCs and Labor/Fringe Subtotal				358.39
G&A @ 18.27%				65.48
Total Estimated Cost				423.87
Fixed Fee @ 10%				42.39
Grand Total				466.26

* Labor hours

Appendix B

Standard Operating Procedures for Analysis of TNT and RDX in Groundwater Using Colorimetric Method

The following standard operating procedure (SOP) was developed by the Army Corps of Engineers for use at the Umatilla Chemical Depot. It is repeated below as it appeared in their site specific Sampling and Analysis Plan. The procedure includes a modification to account for high nitrate levels in the water.

Description

This SOP describes a field analytical method for determining TNT and RDX concentrations in water. The method uses solid phase extraction to remove and pre-concentrate the analytes from water. In the method, a 2 L water sample is passed through a stack of two membranes to pre-concentrate TNT on the top disk and RDX on the bottom disk. Acetone is used to elute RDX from the bottom disk, and a chemical reaction is induced that causes a color change indicative of RDX in the solution. The RDX concentration is estimated from the absorbance at 510 nm on a Hach DR2000 spectrophotometer. Next, the top disk is eluted with acetone and a different chemical reaction is induced causing a color change indicative of TNT. The TNT concentration is estimated from the absorbance at 540 nm on the Hach DR2000. The contract required detection limit for TNT is 1.0 µg/L and for RDX is 5 µg/L. Sample extraction and analysis may take between 1.5 and 5 hours per sample depending on the number of parallel extraction apparatus.

Safety Precautions

- Extraction and analysis should be performed in a well ventilated area.
- Laboratory technicians should wear chemical resistant gloves and safety glasses.

Extraction Procedure

Materials Needed (per sample)

2 Empore extraction membranes
aluminum foil
2 25x200 mm glass test tubes
filter flask apparatus
vacuum pump
tweezers
timer (minutes/seconds)

Reagents Needed (per sample)

2 L of sample
acetone, technical grade
DI water
tap water/DI water/acetone for cleaning

Pitfalls

- Never let the disks go dry. Throw the disks out and start over if they do. Keep the disks covered with at least $\frac{1}{4}$ inch of fluid during the extraction phase.
- Apply the vacuum gradually so as not to damage the membranes. If you see particles in the acetone extracts at this point, vacuum was applied too suddenly.
- Do not shake the sample prior to filtering.

Procedure

1. Use gloves during the entire procedure.
2. Use tweezers to place two Empore extraction membranes centered on the lower portion of the filter apparatus; cover squarely with the upper portion of the filter apparatus and clamp securely. Do not touch the membranes with your hands. A glass fiber filter may also be used to remove particulate.
3. Slowly add 30 ml acetone to the stack and allow it to soak for 10 minutes.
4. Slowly apply vacuum to the filter flask apparatus until there is minimum dripping of acetone (evidence that both filters are completely saturated). Shut off vacuum; add 10 ml of D. I. water. Let set for 10 minutes or until about $\frac{1}{4}$ inch of liquid remains, whichever occurs first. The next two steps go quickly, so have materials measured and in place before starting.
5. Fill the reservoir with sample before the fluid level is reduced to $\frac{1}{4}$ inch. Reapply vacuum ever so slightly. The sample may be filtered through at a rate of 10 to 100 ml/min.
6. Continue filling the reservoir until 2 L of sample has penetrated the membrane. Do not allow the fluid level to fall below $\frac{1}{4}$ inch until the entire sample has been passed through.

7. Add 10 ml of DI water to the reservoir just before the last of the sample penetrates into the membrane. This will aid in washing out the nitrate interference.
8. Continue to apply vacuum for about 2 minutes after the last of the sample has been extracted. This is to remove excess water.
9. Remove the upper portion of the filter apparatus from the filter stack and discard the glass fiber filter, if used.
10. Remove both the disks and set them face up on a clean piece of aluminum foil marked "T" for top disk and "B" for bottom disk, these will be used later for your TNT and RDX extracts.
11. Reassemble the filter apparatus and rinse first with DI water and second with acetone.
12. Disassemble the filter apparatus and pour the water from the 2,000 ml Pyrex flask into a waste container.
13. Wash a 25 x 200 mm tube with DI water, rinse with acetone, label the tube (RDX or TNT, sample number, date), place it in the flask, and replace the funnel.
14. Place the RDX disk membrane (bottom) on top of the lower portion of the filter apparatus. Reassemble the filter stack.
15. Add 7 ml of acetone to the reservoir and soak for exactly 3 minutes.
16. Apply vacuum and aspirate acetone into the 25x200 mm tube until dripping stops.
17. Remove the membrane and discard. Cap the 25 x 200 mm test tube. If possible, samples should be analyzed on the day of extraction. Otherwise, the meniscus should be marked on the test tube and the tube refrigerated. If the fluid level falls below the meniscus line, the tube should be refilled with acetone to its original level.
18. Reassemble the vacuum apparatus with the TNT (top) disk, which was set aside in Step 10 and a fresh 25 x 200 mm test tube (washed as described in Step 13).
19. Add 25 ml acetone to the reservoir and allow soaking for exactly 3 minutes.
20. Apply vacuum and aspirate into the 25 x 200 mm tube. Cap the 25 x 200 mm test tube. If possible, samples should be analyzed on the day of extraction. Otherwise, the meniscus should be marked on the test tube and the tube refrigerated. If the fluid level falls below the meniscus line, the tube should be refilled with acetone to its original level.

21. Decontaminate the reservoir and filter holder by washing with tap water, rinsing with DI water, and final rinsing with acetone.
22. Collect all liquids generated during the decontamination process for incorporation into the treatment plant process.

RDX Analysis

Materials Needed (per sample)

10 ml syringe with 0.45 μm filter
2 13 ml holding vials
30 ml syringe with 0.45 μm filter
desiccator and desiccants
Alumina-A filter
2 matched Hach cuvettes/stoppers
Hach DR2000 set to 510 nm
5 ml syringe with 0.45 μm filter
50 ml reaction vial
analytical balance
Kimwipes™
spatula
Miscellaneous glass volumetric pipettes, flasks, and graduated cylinders

Reagents Needed (per sample)

5 ml acetone, technical grade
Hach NitriVer 3 powder pillow
20 ml DI water
0.2 g of zinc dust, 100 to 325 mesh
RDX Standards for laboratory control
Miscellaneous amounts of tap water/DI water/acetone for cleaning
0.75 ml of acetic acid solution (77 percent glacial acetic acid and 23 percent DI water)

Pitfalls

- The reaction of the acidified extract with zinc is the most crucial step in obtaining consistent and correct results. The step should be done as quickly as possible (10 seconds at the longest). The reaction is also temperature dependent and should be performed in a

cool setting. If the extract was refrigerated, make sure the extract is between 60-80 °F before beginning the analysis.

- The zinc syringe should be tapped gently so that the zinc is at the bottom of the syringe before removing the plunger.
- Check the filters at the bottom of the syringes to make sure that they are securely fastened before adding extract.
- Some samples may display a milky or cloudy appearance even after being filtered into the sample cuvette. These samples should be re-filtered and the cuvette cleaned. If the extract is still cloudy, read and record the absorbance, make a note of the cloudiness in the laboratory log, and indicate that this is a false positive. If a pink color also is present, this should be taken as a positive reaction for RDX; however, the associated result will be biased high.
- Make sure that the NiriVer pillow is completely dissolved in the reaction vessel containing 20 ml water. Do not let this solution sit for more than 10 minutes before using.
- Be sure to record the volumes used for all dilutions, not just the dilution factor. This will aid in checking for any mathematical errors.
- Let the bubbles dissolve before reading the absorption.
- Store the zinc dust and prepared zinc syringes in the desiccator.
- The test also will show a positive reaction for HMX.

Preparation Before Analysis

Using the spatula, place approximately 0.2 g of zinc dust into the barrel of a 5 ml syringe with a 0.45 μm filter attached. Replace the plunger. Store all zinc syringes in a desiccator with desiccant for at least 24 hours before they are used.

Procedure

1. Condition the alumina-A filter with 5 ml of acetone. Pour 5 ml of acetone into the 10 ml syringe with the alumina-A filter. Let the acetone filter through at a rate of one ml per minute.

2. Shake the 10 ml syringe dry and reuse for the next step.
3. Pour 5 ml of extract into the 10 ml syringe with the alumina-A filter. Filter the extract into a labeled 13 ml holding vial. Filter at a rate of 1 ml per minute.
4. Pour 5 ml of extract into the 10 ml syringe with attached filter. Filter the extract into a labeled 13 ml holding vial. Reserve the remaining 1 ml extract for possible dilutions.
5. Add 0.75 ml of the acetic acid solution to each 13 ml holding vial. Shake to mix and set aside for several minutes.
6. Add 20 ml DI water to a 50 ml reaction vessel. If the reaction vessel came supplied with DI water, remove the supplied water before adding fresh DI water. Add the NiriVer pillow to the 50 ml reaction vessel. Shake until completely dissolved. If batching samples, be sure to label the reaction vessel. Let set for at least 5 minutes but no longer than 10 minutes.
7. Slowly remove the plunger of the 5 ml zinc syringe, shaking the powder down. Holding the syringe over the reaction vessel, pour the extract into the 5 ml zinc syringe. Replace plunger, invert once and filter rapidly into the 50 ml reaction vessel containing 20 ml DI water. This step must be done as quickly as possible, approximately within 10 seconds.
8. Shake the reaction vessel to mix and wait at least 10 minutes, but no longer than 15 minutes, for color to develop.
9. Filter the sample into a clean DR2000 cuvette. Note in the laboratory logbook any obvious color.
10. Zero the instrument and obtain a background absorbance. (see Operation of Hach DR2000)
11. Read the absorbance of the sample and record along with any color changes.
12. Between samples, clean the cuvettes with DI water and acetone (in that order) using a stopper and shaking vigorously.
13. Periodically check that the instrument is correctly reading zero with the reference cuvette.
14. Calculate the concentration of the extract using the following equation:

$$RDX (\mu\text{g/L}) = A_i \times DF \times VCF \times RF$$

where

A_i = (absorbance of sample - absorbance of blank)

DF = dilution factor

VCF = volume correction factor is equal to 1.4 when the extraction volume is 7 ml

RF = response factor is listed in the laboratory

For sample concentrations where the absorbance is greater than 0.800, the reserved sample extract should be diluted with acetone, taken through the reaction steps, and the absorbance read and recorded.

TNT Analysis

Materials Needed (per sample)

30 ml syringe with 0.45 μ m filter attached

Hach DR2000 set at 540 nm

2 matched Hach cuvettes/stoppers

Miscellaneous glass volumetric pipettes, flasks, and graduated cylinders

Reagents Needed (per sample)

Developer solution

DI water/acetone for rinsing

TNT standard for laboratory control

Pitfalls

- The test will also react for TNB and DNT.
- If the extract was refrigerated make sure the extract is between 60-80 °F before beginning the analysis.

Procedure

1. Zero the instrument and obtain a background absorbance. (see Operation of Hach DR2000)
2. Pour 25 ml of extract into a 30 ml syringe with attached filter. Filter the sample into the sample cuvette.

3. Read and record the initial absorbence.
4. Add one drop of EnSys TNT developer solution.
5. Shake tube continuously for 3 seconds.
6. Read the final absorbence and record. Also note any color present in the extract and how the color developed.
7. Periodically check the instrument is correctly reading zero with the reference cuvette.
8. Calculate the concentration using the following equation:

$$\text{TNT } (\mu\text{g/L}) = [\text{Af} - (2 \times \text{Ai})] \times \text{DF} \times \text{VCF} \times \text{RF}$$

where

Ai = initial absorbence

Af = final absorbence

DF = Dilution factor

VCF = volume correction factor equal to 1.25 for 25 ml extraction volume

RF = response factor listed in the laboratory

Samples with TNT final absorbencies grater than 0.800 require dilutions. Use the reserved sample extract, perform the analysis, and record the results.

Quality Control/Quality Assurance

A laboratory control standard should be analyzed each day that an analysis is performed, and is used to verify that the analysis portion of the procedure is performed acceptably. The absorbence must be within 0.307 to 0.373 for RDX and 0.174 to 0.272 for TNT for the test to be in control. If the standard is not in control, try again, paying particular attention to the zinc step.

A blank must be extracted each day that samples are extracted. The method blank and its associated samples should all be analyzed at the same time. The blank must be clean and colorless. If any contamination is noted, review the glassware cleaning procedures or possible sources of cross contamination. Note problem and resolution in the logbook.

A blank spike must be extracted each week that samples are extracted. This blank spike is used to verify that the extraction portion of the procedure is being performed in an acceptable manner. A 2 L portion of DI water should be spiked with RDX and/or TNT and carried through the extraction procedure. Spike in 80 μl of a standard solution in acetone containing RDX and/or TNT at 500 mg/L each. The concentration of standard in the final extracts will be 20 $\mu\text{g/L}$. The

blank spike and its associated samples should be analyzed at the same time. The acceptable range for spike recovery is 60 to 140 percent.

Field duplicates must be extracted and analyzed at a rate of 10 percent. The precision goal is ± 50 percent RPD. Select duplicates that represent various concentration levels.

The reliability of the method is operator dependent. Each operator needs to do five qualifying spike samples through the extraction and analysis procedures to produce their own response factors for TNT and RDX analysis. The response factors need to be reevaluated periodically or when a major change in the procedure occurs.

All results and comments should be recorded in ink in a laboratory notebook with the name of the analysis and date clearly entered.

Operation of Hach DR2000

1. Turn on the Hach. The instrument will read "Selftest" followed by "Method?" Select "0" and press "read/enter".
2. Rotate the wavelength dial to the desired setting: 510 for RDX and 540 for TNT. Approach the wavelength from the high side when adjusting.
3. Fill both cuvettes to the line with acetone.
4. Insert the "reference" cuvette into the cell holder with the side marked "25 ml" on the right.
5. Close the light shield and press "Clear/Zero" to establish the reference.
6. Remove the reference and place the "sample" cuvette in the holder with the side marked "25 ml" on the right.
7. Press "Read/Enter" and record the absorbance in the laboratory logbook as ABS background.
8. If the reading is greater than ± 0.002 , clean the cuvettes and repeat the procedure.
9. Proceed with sample analysis.

Cleaning Cuvettes

1. Fill the matched cuvettes with 5 ml of water.
2. Cap each cuvette and shake vigorously for 3 seconds.
3. Empty into a waste container.
4. Fill the cuvettes with 5 ml of acetone.
5. Cap and shake for 3 seconds.
6. Empty into waste container.
7. Repeat the acetone wash.
8. Wipe the outside of the cuvettes with Kimwipes™. Take care especially to clean the side labeled "25 ml" and the side opposite.

General Interferences

1. Do not use the reagents beyond the expiration date.
2. TNT samples must be analyzed immediately after adding the Developer Solution. RDX samples must be analyzed within 60 minutes of the color incubation step.
3. Operate test kits at less than 39°C (100°F). Store at less than 80°F and out of direct sunlight.
4. Store all standards in the refrigerator.

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