

# Proceedings of the Annual International Conference on Soils, Sediments, Water and Energy

---

Volume 12

Article 17

---

January 2010

## Lessons Learned For Future Designs From Thermal Treatment Of Perchlorate In Soil

Shouvik Gangopadhyay  
*ECC*

Paul Nixon  
*Impact Area Groundwater Study Program*

Scott C. Michalak  
*United States Army Corps of Engineers New England District*

Follow this and additional works at: <https://scholarworks.umass.edu/soilsproceedings>

---

### Recommended Citation

Gangopadhyay, Shouvik; Nixon, Paul; and Michalak, Scott C. (2010) "Lessons Learned For Future Designs From Thermal Treatment Of Perchlorate In Soil," *Proceedings of the Annual International Conference on Soils, Sediments, Water and Energy*: Vol. 12 , Article 17. Available at: <https://scholarworks.umass.edu/soilsproceedings/vol12/iss1/17>

This Conference Proceeding is brought to you for free and open access by ScholarWorks@UMass Amherst. It has been accepted for inclusion in Proceedings of the Annual International Conference on Soils, Sediments, Water and Energy by an authorized editor of ScholarWorks@UMass Amherst. For more information, please contact [scholarworks@library.umass.edu](mailto:scholarworks@library.umass.edu).

## Chapter 16

### LESSONS LEARNED FOR FUTURE DESIGNS FROM THERMAL TREATMENT OF PERCHLORATE IN SOIL

Shouvik Gangopadhyay<sup>1</sup>, Paul Nixon<sup>2</sup>, and Scott C. Michalak<sup>3</sup>

<sup>1</sup>ECC, PB 519 Gaffney Road, Camp Edwards, MA 02542; <sup>2</sup>Impact Area Groundwater Study Program, 1803 West Outer Road, Camp Edwards, MA 02542; <sup>3</sup>United States Army Corps of Engineers New England District, 696 Virginia Road, Concord, MA 01742-2751

**Abstract:** A soil remediation project using *ex-situ* thermal desorption to remove explosive compounds was modified at the latter stages of planning to include treatment of perchlorate. The design-build approach consisted of a bench scale study, which verified the treatability of perchlorate at baseline treatment conditions, and pilot scale studies, which helped refine the process parameters for full-scale treatment. During initial full-scale operations, a high number of treated soil batches failed to meet the project treatment criteria. Analytical results of samples collected from various stages along the treatment train indicated a potential for perchlorate contamination to by-pass the primary treatment process. Slower feed rates and higher operating temperatures did not show any conclusive positive impact on treatment efficiency. Recycling the particulates from the air pollution control equipment back into the feed soil was initially considered, but was deemed infeasible with the current equipment design configuration. A cost-effective solution was achieved by reducing the size of the treated soil sample batch volume, thereby reducing the amount of soil requiring re-treatment when occasional failures occurred. Future plant designs intended for treatment of perchlorate could potentially improve destruction efficiency by recycling particulates to the beginning of the process instead of the treated soil discharge.

**Key words:** soil remediation, treatment, pilot-scale, process improvement, explosive compounds, RDX, HMX, perchlorate.

## 1. INTRODUCTION

Several sites at a military installation were found to contain surface and sub-surface contamination consisting of perchlorate and the explosive compounds hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) and octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrocine (HMX) in a wide range of concentrations. Remediation of these sites was deemed necessary as the groundwater beneath the sites is classified as a sole source drinking water aquifer.

Once the soils were excavated, several alternatives were available for managing the excavated soils. These included landfill disposal, soil washing, and thermal desorption. Taking into consideration the expected volume of soil requiring treatment, the thermal desorption alternative became the more economically attractive alternative. Additionally, soil washing was expected to be ineffective for chunks of explosive materials found in some of the source area sites. Furthermore, on-site treatment eliminated the potential liabilities typically associated with landfill disposal.

The volume of soil excavated from the sites and treated by a thermal desorption process was approximately 40,000 cubic yards (cy). The remediation goal was agreed on with the regulatory community and set based on the reporting limit of the available laboratory analyses— 4 ppb for perchlorate based on EPA Method 314.0 and 120 ppb for the explosive compounds RDX and HMX based on EPA SW846/Method 8330. Since that time, the remediation soil standards proposed by the

state regulatory agency for perchlorate and explosives compounds (RDX and HMX) are orders of magnitude higher than the agreed on remediation goals for this project.

Environmental Chemical Corporation (ECC) was contracted by the United States Army Corps of Engineers (USACE) to mobilize and operate a Thermal Treatment Unit (TTU) at the site. This paper provides a general description of the system, the studies conducted to determine feasibility of the system, and lessons learned from the TTU operations. This project was the first known full-scale application of thermal destruction of perchlorate in contaminated soil using a TTU.

## 2. TREATMENT PROCESS

The TTU consists of a solids treatment system and an air pollution control (APC) system. The solids treatment system contains a soil feed system, direct-fired rotary drum (dryer), and product discharge system. The APC system contains a cyclone, thermal oxidizer, evaporative cooling chamber, bag house, induced draft blower, and a stack. The fuel source for the burners is vaporized liquid propane. The process flow diagram is shown in Figure 1.

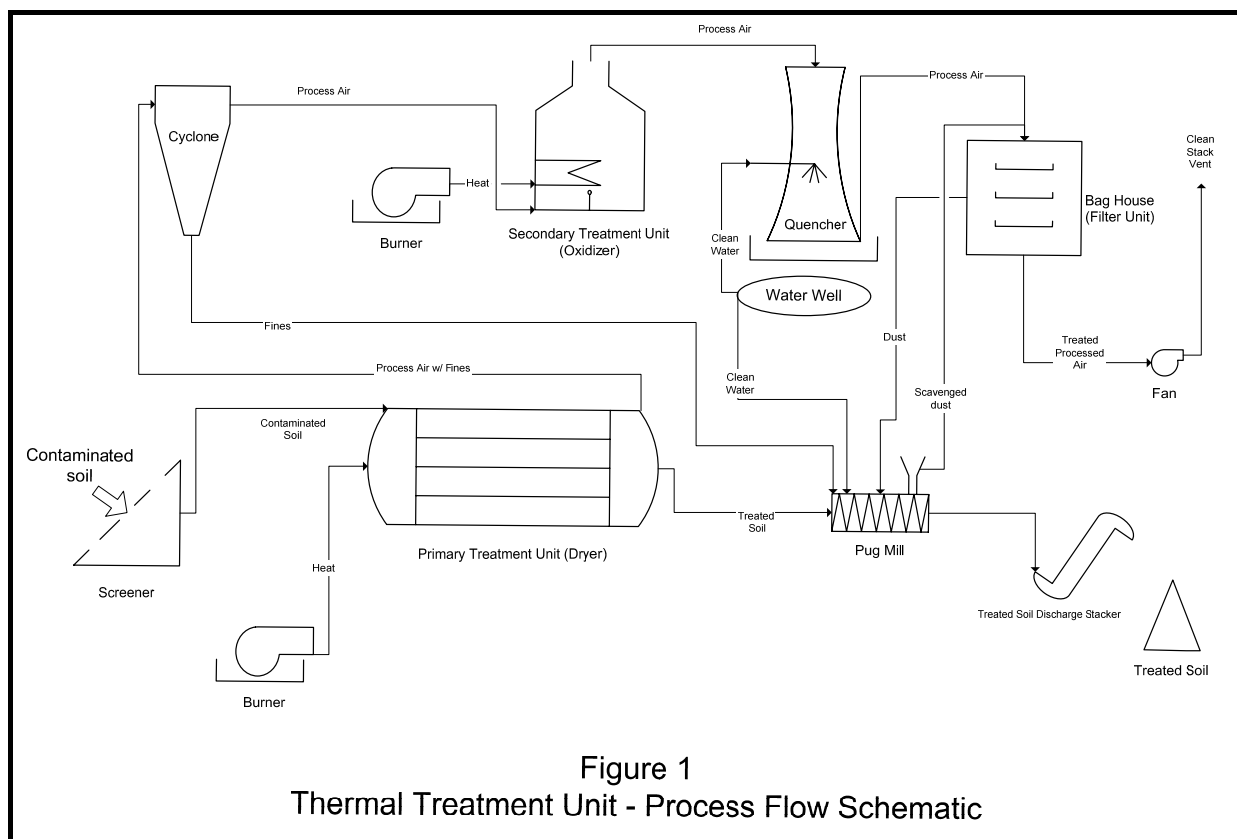


Figure 1  
Thermal Treatment Unit - Process Flow Schematic

Figure 1.

Soil is prepared for treatment by initially screening out items greater than 1 inch in diameter using mechanical means. Contaminants are removed from the soil in the parallel flow, direct-fired rotary drum. In the parallel flow design configuration, soil particulates travel the entire length of the drum during which time they are heated to, and maintained at, the target temperature until they are discharged to the pugmill.

The pugmill mixes the hot treated soil with cooling water. This re-humidification process controls dust emissions and prepares the soil for future handling. A negative draft is induced on the headspace of the pugmill and any steam generated within the pugmill is vented to the bag house.

Exhaust gases leaving the rotary drum pass through a cyclone, which is the primary control for air entrained particulates. Particulates removed in the cyclone are gravity fed to the pugmill where they are blended into the treated soil before exiting the system.

The exhaust gas leaving the cyclone continues to a thermal oxidizer which heats the gas to a specific temperature, determined on a contaminant and site specific basis, in order to destroy the remaining contaminants. Before entering the baghouse, the hot gases exiting the thermal oxidizer are cooled with air-atomized water spray nozzles in the evaporative cooling chamber to a temperature below 450°F.

The bag house is designed to remove fine particulates entrained in the exhaust gases. The fines form a cake on the bag surface, which is periodically cleaned by a pulse of air. A screw auger transfers these fines to the soil discharge pugmill and blends the fines back into the treated soil. An induced draft (ID) blower located after the bag house is used to maintain a negative pressure on the air pollution control system. Clean exhaust gases are emitted to the atmosphere through a vertical stack.

### 3. TREATABILITY STUDY

Full-scale operations at previous project sites had established that an operating temperature of approximately 650°F was sufficient to remove explosive contaminants (predominantly RDX and HMX). However, perchlorate had previously never been treated in a thermal desorption process. A literature search revealed that ammonium perchlorate sublimates and undergoes thermal destruction at about 725°F.

A treatability study was designed to determine the target operating parameters for full-scale treatment conditions. The test program was carried out at Hazen Research, Inc., Golden, Colorado. The apparatus used consisted of a cylindrical quartz rotary kiln in a muffle furnace, with all off-gases collected in a 2-stage condensing system that separated the condensed water vapor and particulates from the non-condensable gases. The test soils were carefully weighed and spiked with a known quantity of perchlorate and homogenized before adding the soil to the reactor. The rotary kiln reactor was rotated during the test to simulate full-scale operation, and heated up at a controlled rate to the desired operating temperature. When the rotary kiln reactor and soils reached the desired temperature, a timed run was initiated. An outline of the tests is shown in Table 1. All products (soils, particulates, water vapor and non-condensable gases) were analyzed for residual perchlorate. Two sets of tests were carried out with the soils spiked to 100 ppb (low level) and 100,000 ppb (high level).

Table 1. Operating Conditions, Treatability Study

Operating Condition Number	Concentration of Perchlorate in Feed Soil (ppb)	Treated Soil Temperature (OF)	Residence Time at Temperature	Oxygen Content
1	100,000	650	10 minutes	> 7%
2	100,000	900	10 minutes	> 7%
3	100,000	1150	10 minutes	> 7%
4	100,000	650	30 minutes	> 7%
5	100,000	1150	30 minutes	> 7%
6	100,000	900	10 minutes	<1%
7	100	650	10 minutes	> 7%
8	100	900	10 minutes	> 7%
9	100	1150	10 minutes	> 7%
10	100,000	725	10 minutes	> 7%
11	100,000	775	10 minutes	> 7%

°F = degrees Fahrenheit

ppb = parts per billion

Table 2 shows the results obtained from analysis of the soil constituents and Table 3 shows the analytical results from the condensates collected during each run. The analytical data indicate that explosive compounds were destroyed under all conditions tested. Perchlorate was removed in six of the initial nine conditions evaluated. The tests which failed were at the lowest treatment temperature,

650°F. Since perchlorate was removed successfully at the two higher operating temperatures of 900°F and 1150°F, two additional tests were run at intermediate temperatures of 725°F and 775°F. Traces of perchlorate were present in the 725°F treated soils but none at 775°F.

Table 2. Soil Analytical Results, Treatability Study

Description	Pre-spike and Pre-Test	Run 1, 100 mg/kg, 650 °F, 10 min., >7% O2	Run 2, 100 mg/kg, 900 °F, 10 min., >7% O2	Run 3, 100 mg/kg, 1150 °F, 10 min., >7 % O2	Run 4, 100 mg/kg, 650 °F, 30 min., >7% O2	Run 5, 100 mg/kg, 1150 °F, 30 min., >7% O2	Run 6, 100 mg/kg, 900 °F, 10 min., <1% O2	Run 7, 0.1 mg/kg, 650 °F, 10 min., >7% O2	Run 8, 0.1 mg/kg, 900 °F, 10 min., >7% O2	Run 9, 0.1 mg/kg, 1150 °F, 10 min., >7% O2	Run 10, 100 mg/kg, 725 °F, 10 min., >7% O2	Run 11, 100 mg/kg, 775 °F, 10 min., >7% O2
Units	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
Perchlorate	3.5J	51	1.3U	1.3U	25	1.3U	1.3U	41	1.3U	1.3U	74	1.3U
HMX	120U	120U	120U	120U	120U	120U	120U	120U	120U	120U	120U	120U
RDX	120U	120U	120U	120U	120U	120U	120U	120U	120U	120U	120U	120U

J = estimated result                      U = non-detect result  
 °F=degrees Fahrenheit      mg/kg = milligrams per kilogram

Table 3. Condensate Analytical Results, Treatability Study

Description	Detection Limit	Run 1, 100 mg/kg, 650 °F, 10 min, >7 % O2	Run 2, 100 mg/kg, 900 °F, 10 min, >7 % O2	Run 3, 100 mg/kg, 1150 °F, 10 min, >7 % O2	Run 4, 100 mg/kg, 650 °F, 30 min, >7 % O2	Run 5, 100 mg/kg, 1150 °F, 30 min, >7 % O2	Run 6, 100 mg/kg, 900 °F, 10 min, <1 % O2
Units	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L
Perchlorate	0.35	160	110	140	140	150	220

µg/L = micrograms per liter                      °F = degrees Fahrenheit

The condensed streams collected from the rotary kiln reactor were analyzed and showed that less than 0.05 percent of the perchlorate anions in the feed soils were present in the condensate. The treatability study thus successfully demonstrated that perchlorate contaminated soils can be thermally treated and that > 99.9% of perchlorate was decomposed in the rotary kiln operating at 775°F or higher. The remaining 0.01% balance would be destroyed in the full scale system in the thermal oxidizer operated at 1400°F.

#### 4. PILOT AND PERFORMANCE TESTING

During shakedown operations, the initial set point for the soil discharge temperature was 775°F, based on the treatability study results for perchlorate. TTU performance is often system specific and each unit must be optimized to ensure adequate performance. Furthermore, the site soils were generally wet (up to 18% moisture by weight). Drying the soil requires approximately half of the residence time, leaving the remaining half for thermal destruction of the contaminants.

Twenty spike tests were conducted to determine the minimum temperature and other operating parameters required to ensure the treatment of all contaminants. The feed rates for the testing period varied between 20 to 40 tons per hour, at an approximate residence time of 13 minutes. Measured amounts of the spiking agents were added based on feed rate. Results from the tests are included in Table 4.

Table 4. Spike Test Results For Perchlorate

Test #	Feed Perchlorate Concentration (part per billion)	Treated Soil Perchlorate Concentration (part per billion)	Minimum Temp Goal (F)	Average Temp (F)	Average Feed Rate (tons per hour)	Treatment Efficiency
1	100,000	45	775	807	25	99.955%
2	82,000	4500	725	756	30	94.543%
3	121,000	3500	675	705	21	97.102%
4	76,000	280	825	832	35	99.633%
5	83,000	20	875	886	33	99.976%
6	77,000	88	825	833	35	99.885%
7	82,000	13	875	886	33	99.984%
11	1,000	<0.8	925	958	30	100.000%
12	1,100	<0.9	900	911	26	100.000%
13	900	<1.0	875	877	25	100.000%
14	1,200	<1.1	850	853	26	100.000%
15	1,100	3.5	800	804	27	99.683%
16	110,100	3.3	925	936	29	99.997%
17	109,000	13	900	912	33	99.988%
18	102,700	65	875	884	35	99.937%
19	111,300	140	850	857	35	99.874%
20	105,500	340	800	807	37	99.678%

The first ten tests involved spiking feed soil with high concentrations of reagents (approximately 100 parts per million). HMX was successfully treated in all tests at temperatures ranging from 600°F to 700°F. Results from the first three tests (1 through 3), run at minimum operating condition of 775°F, 725°F and 675°F respectively, indicated that all perchlorate was not adequately removed from the feed soil. Subsequently, the remaining four tests (4 through 7) were run at minimum operating temperatures of 825°F and 875°F. Analytical results showed that the treated soils still contained perchlorate at levels above the 4 ppb project remediation goal. It should be noted that the control of treatment temperature during this early stage of shakedown testing was inconsistent and may have contributed to inefficiencies in treatment. The treatment temperature control was improved in subsequent operations after the burner control components were further tuned.

Spiking events eleven through twenty were designed to determine both the treatment temperature for perchlorate and the effect that feed concentrations had on the treatment of perchlorate. These tests were run with minimum temperatures of 925, 900, 875, 850, and 800°F, respectively, repeated for two different types of feed soils. Five of these events (16 through 20) were run with native soils spiked with a high concentration (approximately 100 ppm) of perchlorate and the other five (11 through 15) were run with native soils spiked with a low concentration (approximately 1 ppm of perchlorate). Results (Table 4) showed that the high concentration spiked soil was treated to below the project remediation goal only at the highest temperature (Test 16 at 925 °F). The low concentration spiked soils were successfully treated to below the project remediation goal in all the tests.

Figures 2 and 3 illustrate the variation of removal efficiency of perchlorate with respect to average operating temperatures. Removal efficiency of the soils containing low concentrations of perchlorate rose sharply between 800 and 850°F. Beyond 850°F, perchlorate was not detected in any of the treated soils samples. Removal efficiency of the soils containing a higher concentration of perchlorate rose sharply in the beginning, and continued to rise gradually after 800°F.

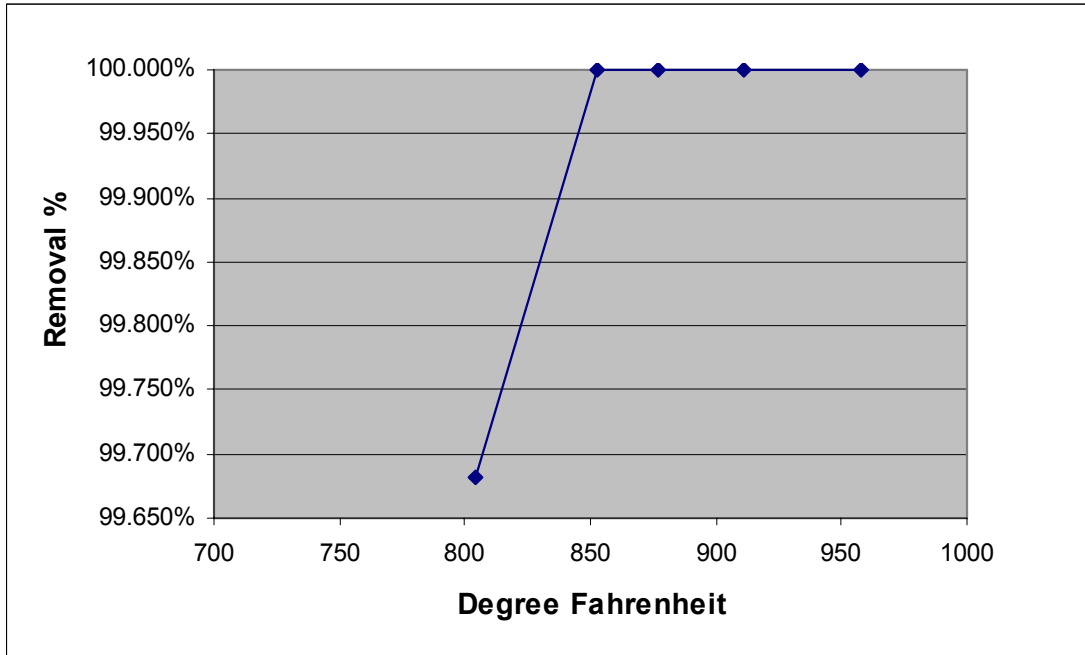


Figure 2. Removal Efficiency vs Temperature (Low Spike Concentration)

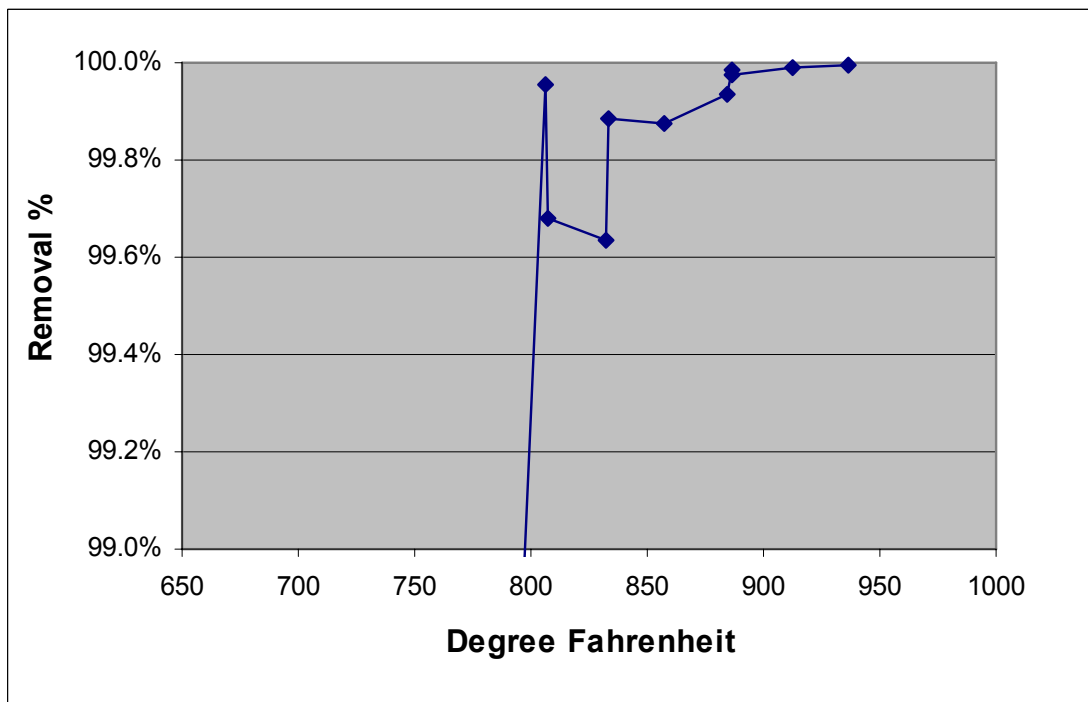


Figure 3. Removal Efficiency vs Temperature (High Spike Concentration)

Once operating conditions were sufficiently field tested during this period, the treatment unit underwent a proof of performance (POP) test. The primary objectives of the performance test, conducted over a three day period, were to demonstrate that a) the air emissions would meet the criteria established by the state Air Permit, b) the TTU would meet the project specified treatment criteria for soil, and c) the equipment could be safely operated in a controlled manner.

Feed material for the test included approximately 2,500 tons of soil from the contaminated sites and treated soil from spiking events that had failed the treatment criteria. A portion of the soil was also spiked with target compounds in order to demonstrate the effectiveness of the thermal treatment system's air pollution control system. Composite samples of the feed material were collected and analyzed at a rate consistent with treated soil verification testing to verify the levels of the contaminants of concern before treatment and to establish a baseline for air permit emissions calculations. Samples were also collected from the cyclone discharge and from the baghouse dust stream prior to being mixed with the treated soil.

Analytical results of samples from the feed soil, treated soil, and baghouse and cyclone dust are presented in Tables 5, 6 and 7, respectively. The analytical results indicated that the pre-spiked feed soil for the POP had varying concentrations of perchlorate ranging from 46 to 100 ppb. The major contributing source of perchlorate in the feed soil was from recycled stockpiles that had previously failed to meet the targeted cleanup standard for perchlorate during the spike tests.

Table 5. POP Test Feed Soil Summary Data

Sample ID	RDX (ppb)	HMX (ppb)	Perchlorate (ppb)	Moisture %
<b>Day 1</b>				
FS033104-019	120U	120U	100	14
FS033104-020	120U	120U	91	14
<b>Day 2</b>				
FS040104-024	120U	120U	66	18
FS040104-027	120U	120U	57	16
FS0401044-030	120U	120U	46	17
<b>Day 3</b>				
FS040204-033	120U	140	50	14
FS040204-036	330	120U	63	14

Table 6. POP Test Treated Soil Summary Data

Sample ID	RDX (ppb)	HMX (ppb)	Perchlorate (ppb)	Moisture %
<b>Day 1</b>				
TS033104-019	120U	120U	4.3J	12
TS033104-020	120U	120U	5.8U	14
<b>Day 2</b>				
TS040104-024	120U	120U	3J	15
TS040104-025	120U	120U	3.5J	13
TS040104-026	120U	120U	3.3J	14
TS040104-027	120U	120U	1.9J	12
TS040104-028	120U	120U	4.3J	11
TS040104-029	120U	120U	10	14
TS040104-030	120U	120U	2.8	18
TS040104-031	120U	120U	2.8J	14
TS040104-032	120U	120U	5.9U	15
<b>Day 3</b>				
TS040204-033	120U	120U	5.6U	11
TS040204-034	120U	120U	5.6U	11
TS040204-035	120U	120U	5.7U	13
TS040204-036	120U	120U	5.7U	12
TS040204-037	120U	120U	5.6U	10
TS040204-038	120U	120U	5.7U	12
TS040204-039	120U	120U	5.6U	11
TS040204-040	120U	120U	5.7U	12
TS040204-041	120U	120U	5.7U	12

Table 7. POP Test Baghouse/Cyclone Summary Data

Sample ID	RDX (ppb)	HMX (ppb)	Perchlorate (ppb)	Moisture %
<b>Day 1</b>				
BD033104	120U	120U	22	2
CD033104	120U	120U	46	0
<b>Day 2</b>				



Sample ID	RDX (ppb)	HMX (ppb)	Perchlorate (ppb)	Moisture %
BD040104	120U	120U	9.5J	1
CD040104	120U	120U	5U	0
<b>Day 3</b>				
BD04020	120U	120U	5U	0
CD040204	120U	120U	5U	0
HMX= octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine      RDX = hexahydro-1,3,5-trinitro-1,3,5-triazine				
U = non-detect result      J = estimated result				

A total of 20 post treatment samples were collected during the POP test. Nineteen of the twenty treated sample results indicate that perchlorate was treated to below the Method 314.1 detection level of 4 ppb. This level of success (95%) indicated that the treatment system and operational parameters were effective in meeting the soil remediation goal.

The baghouse and cyclone samples were below the treatment goals of 120 ppb for explosives on all three days of the POP. As listed in the tables, the perchlorate results were inconsistent for the soil samples from the cyclone and baghouse streams.

During the shakedown and spike testing period, interferences in the analytical method used for soil samples were discovered. Based on the fact that interferences were persistent during the analysis of the feed and treated soils using EPA Method 314.0, the project team proposed using an alternate Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) method (EPA Method SW846/8321), which is more applicable to a soil matrix as opposed to the Method 314.0 which is more applicable to water samples. A series of samples were analyzed using both methods; the results indicated that false positives were sometimes detected using Method 314.0. The analytical change from EPA Method 314.0 to EPA Method SW846/8321 was approved by the regulatory agencies prior to implementation.

## 5. FULL SCALE OPERATIONS

Based on the results from the POP test, the following treatment parameters were established to meet the performance goals:

- Soil treatment temperature: 839°F, minimum
- Soil feed rate: approximately 40 tph, maximum
- STU discharge temperature: 1489°F, minimum.

At the start of full-scale operations, an unforeseen problem was encountered with the treatment process. Of the initial twenty 500 cy piles that were treated, only fourteen met the treatment standards (30% failure rate). The treatment failures were all for residual perchlorate contamination with concentrations ranging from 7 to 22 ppb. A review of the TTU process was conducted using data from the performance tests, as shown in Figures 4 and 5. Attempts were made to correlate the soil temperature and the feed rate to the failure of the treatment system. However, as can be seen in two representative charts from two spiking rounds, no direct correlation could be observed between the failure rate and the operating parameters viewed at the time of sample collection. A review of the sampling and laboratory analysis was also conducted to determine the cause and to reduce the frequency of treatment failures. No apparent causes for the treatment failures were identified in the analytical process, particularly since switching over to EPA Method 8321 seemed to have reduced interference that were previously observed.

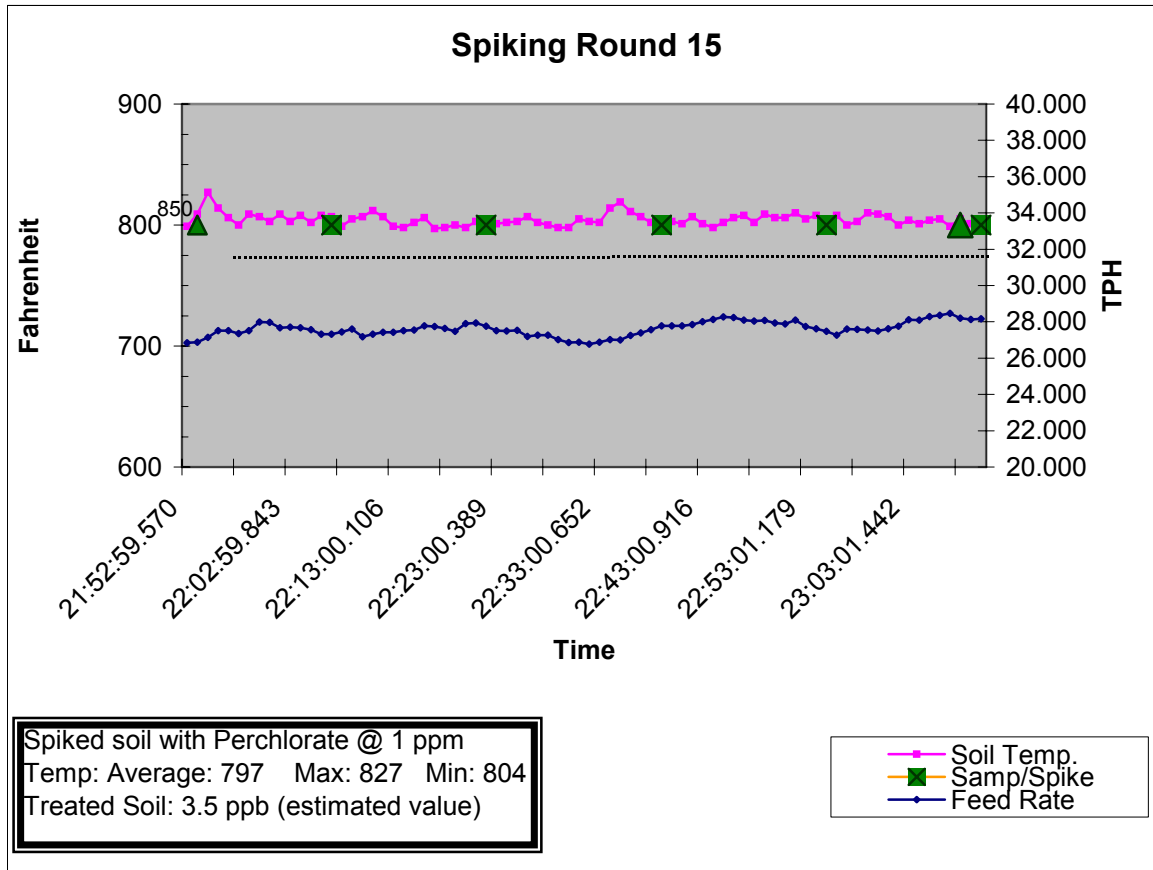


Figure 4. Operating Parameters during performance testing – Spike Test 15

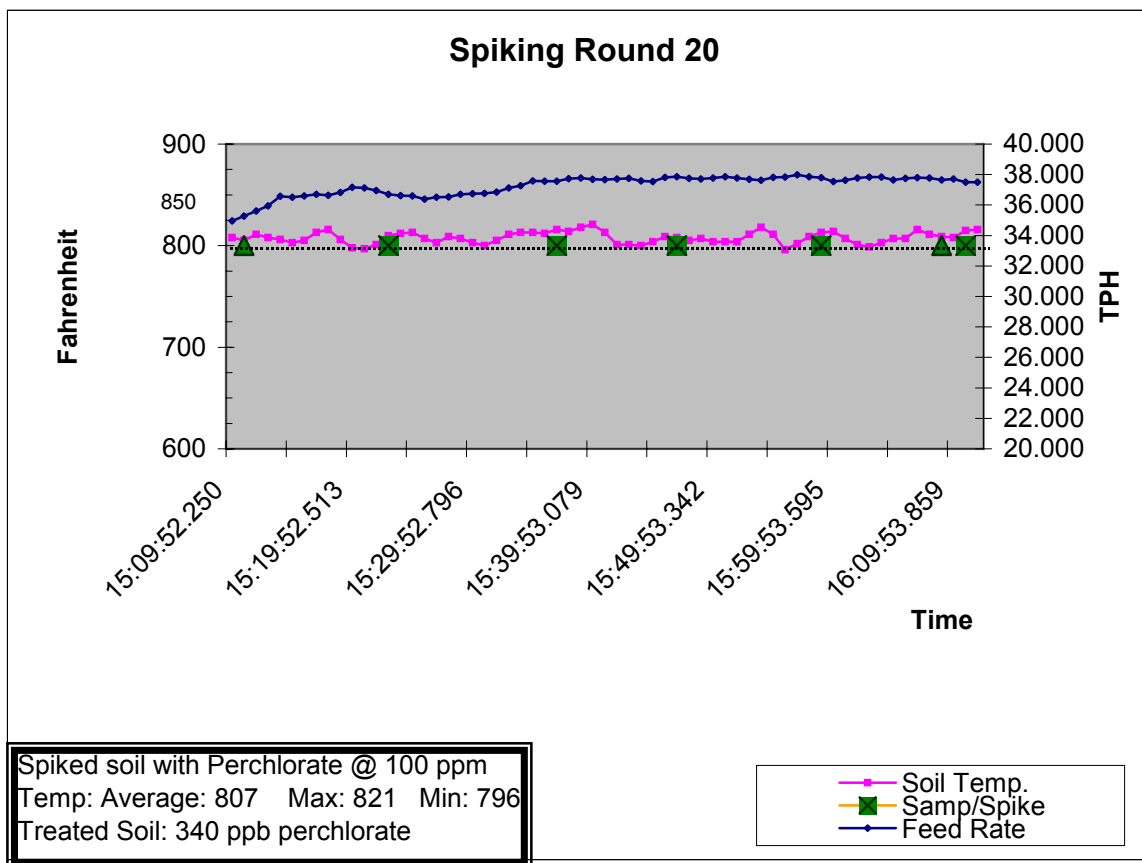


Figure 5. Operating Parameters during performance testing – Spike Test 20

Other factors that were examined were methods used for keeping the soil material dry. Typically, the soil was mixed with lime and this had been suspected to be interfering in the analytical method earlier; however, levels of lime mixed during initial full-scale operations were low and its usage did not correlate with the treatment failures.

The levels of perchlorate in the treated stockpiles seemed to be attributable to fines that were collected in the cyclone and mixed in the pugmill with the treated soil. It was noted that analytical results from the baghouse and cyclone samples steadily showed detects of perchlorate throughout the treatment process. Although these detects were less than the treatment goals of 4 ppb, it was feasible that a fraction of feed soil, in the form of fine particles suspended in the exhaust gas, was traveling through the system at a much higher rate than the average residence time for treated soil. These fines are captured in the cyclone; a portion is gravity fed into the pugmill while the remainder is carried over to the rest of the APC. These fine particulates could potentially have been part of any sample collected to measure the effectiveness of the treatment process.

Based on literature and previous testing results, the fine particles that were carried over to the thermal oxidizer would have been effectively treated based on the operating temperature. However, trace levels of particulates were also seen in the samples from the baghouse. These were surmised to be originating from either the relic concentration of previous high concentration spiking tests, or from particulates, which were carried over with the steam that is vented from the pugmill to the baghouse.

The following steps were taken to eliminate the failures.

- The treatment temperature was increased to 950 °F.
- The baghouse was thoroughly decontaminated and the recovered fines were blended into the feed stockpile

- The feed rate was temporarily lowered to 30 tph then later adjusted upwards to the maximum permissible 40 tph
- An attempt was made to operate the TTU by closing the discharge valve from the cyclone to the pug mill. However, the amount of particulate loading was beyond the design capacity of the baghouse making this procedure not feasible for continuous operation of the TTU.

The above steps did not consistently produce the desired results. Within the project constraints, it was not feasible to stop operations in order to re-design the system. Thus, a practical operational change was made in order to overcome this issue with the treatment process.

The treated soil stockpile area was initially configured to manage soils in batches of 500 cy piles, which corresponded to the sampling frequency for explosives. The sampling frequency of treated soils for perchlorate was increased through regulatory concerns to one every 100 cy. If any of the five samples representing the treated soil stockpile was above the treatment standard, the entire 500 cy pile was retreated through the TTU. The treated soil stockpile area was reconfigured so that the treated soil could be managed in 100 cy stockpiles. This is illustrated in Figures 6 and 7. Reconfiguring the stockpile area drastically reduced the percentage of soil stockpiles that showed an occasional failure due to particulate carryover. This also mitigated the consequence of a treated soil failure by greatly reducing the quantity of soil requiring re-treatment. During subsequent full-scale operation, two daily composite samples were collected from the material transferred from the baghouse to the pugmill and from the cyclone dust effluent, respectively. These samples were analyzed for perchlorate. Only one out of 85 bag house samples was above the remediation goal of 4.0 ppb and 12 out of 85 cyclone samples were above 4.0 ppb. These typically were on occasions were the treated soil samples also failed to meet the treatment criteria, further validating the source of the failure.



Figure 6. Site Infra-structure designed for 500 cy piles



Figure 7. Site Infra-structure re-designed for 100 cy piles

In summary, a total of 60,036 tons were processed and 55,123 tons were successfully treated during full-scale operations. The failure rate for perchlorate during full-scale operations was 8.9%. As described previously, only fourteen of the initial twenty 500 cubic yard piles met the treatment standards (30% failure rate). After the treated storage area was reconfigured to accommodate the separation of 100 cy piles and the process was optimized, the failure rate dropped to 1.33%. The majority of soil on the project was processed at this lower failure rate.

The TTU successfully treated 100% of explosives throughout the project with no detectable concentrations in the treated soil.

As previously discussed, operating data imply that low levels of perchlorate in the baghouse and cyclone streams are likely due to short-circuiting within the process so that small portions of contaminated soil are not exposed to the treatment temperature for the necessary amount of time. Recycling these fines back through the process was discussed but determined to be impractical under the existing systems configuration.

## 6. OBSERVATIONS FOR FUTURE DESIGN

This project was the first documented project in the country to thermally treat perchlorate contaminated soils, utilizing thermal destruction of an inorganic non-volatile contaminant as opposed to vaporization of organics for subsequent treatment by additional process units. Previous projects had demonstrated the successful applications of thermal desorption to treat explosive contaminated soil and this project had the same success. This project demonstrated that the same technology can be adapted to remove perchlorate from contaminated soil. It should be noted that factors such as feed characteristics, drying practices, pre-heating durations and residence time are important site specific

criteria that influence successful treatment, and should be re-assessed in the design phase for future projects.

Perchlorate-contaminated soils seemed to occasionally bypassed the primary treatment process and entered the air pollution control equipment. This resulted in occasional low-level detections of perchlorate in the treated soil. Future TTU designs intended for treatment of perchlorate to low treatment goals, as used during this project, could try to account for this by recycling dust to the beginning of the treatment train instead of the treated soil discharge. Conceptually, recycling the dust to the soil feed may help to reduce the frequency of failures in future systems, however that was not feasible with the equipment that was on-site for this project. Furthermore, based on the low failure rate, it may be more efficient to re-treat the failed soil as was done for this project.

Observations from analytical results indicated that LC/MS/MS method (Method 8321) is a performance-based method that provides definitive, reliable results confirmed by MS, especially at low levels. The method number has been subsequently changed to EPA SW846/Method 331. This method is less susceptible than method 314.0 to matrix interferences, such as those experienced in the treated soils during and prior to the performance test. Use of this method will assure that the treatment goal of 4 µg/kg can be confidently assessed for all samples. It is recommended that future project designs incorporate this particular method for analysis of solid matrices.

Lime, used as a soil amendment for drying, also seemed to cause interference in the analytical methods. Thus, alternate approaches to keeping the feed soil dry should be considered in future projects.

## REFERENCES

- Bircumshaw, L.L., and Newman, B.H., 1954, "The Thermal Decomposition of Ammonium Perchlorate I. Introduction, experimental, analysis of gaseous products, and thermal decomposition experiments", *Proceedings of the Royal Society of London*, A227, 115-132.
- Guillory, W. A., and King, M., 1970, "Thermal Decomposition of Ammonium Perchlorate", *AIAA Journal*, vol. 8, no. 6, 1134-1136.
- Keenan, Arthur G., and Siegmund, Robert F., 1969, "Thermal Decomposition of Ammonium Perchlorate", *Quarterly Reviews, Chemical Society*, vol. 23(3), 430-452.
- P.W.M. Jacobs; H.M. Whitehead, 1969, "Decomposition and Combustion of Ammonium Perchlorate", *Chemical Reviews*, vol. 69, 551-590.
- Manelis, G. B., et al, 2003, Thermal Decomposition and Combustion of Explosives and Propellant, Chapter 13, *Ammonium Perchlorate*, 189-220, Taylor and Frances, ISBN# 0415299845.
- ECC, 2004. Thermal Treatability Study Report for Impact Area Groundwater Study Program, Massachusetts Military Reservation, Camp Edwards, MA. January 27, 2004.
- ECC 2004. Proof of Performance Test Report for Impact Area Groundwater Study Program, Massachusetts Military Reservation, Camp Edwards, MA. August 9, 2004.
- ECC 2006. TTU Completion Report for Impact Area Groundwater Study Program, Massachusetts Military Reservation, Camp Edwards, MA. February 10, 2006.
- MADEP (Massachusetts Department of Environmental Protection) 2004 "Revised Conditional Approval" of Comprehensive Plan Application No. 4P03015.