

# **Final Report**

## **Operational Implementation of Ammonium Perchlorate Biodegradation**

**Air Force Research Laboratory  
Airbase & Environmental Technology Division**

**June 19, 1998**

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# **Operational Implementation of Ammonium Perchlorate Biodegradation**

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### **1. Introduction**

#### **1.1 Background Information**

Nearly every major weapon system that has solid propulsion, explosive devices, or pyrotechnic devices, contains perchlorate compounds. Ammonium perchlorate (AP) is the oxidizer and primary ingredient in solid propellant for most large rocket motors that typically contain 68-73 weight percent AP. For component and ingredient recovery, remanufacture, or demilitarization, high-pressure water washout is the currently accepted process of removal. In addition to this process, manufacturing and testing activities also produce large quantities of water contaminated with various concentrations of AP that must be treated as a hazardous waste. The United States Environmental Protection Agency (USEPA) has issued a Provisional RFD that could restrict discharge of AP to less than one part-per-million (ppm) and severely impact DoD propulsion contractors. The Minuteman III propulsion remanufacture program will remove over 35 million pounds of propellant from 1200 first and second stage motors in order to recover and reuse the cases. All of the major DoD propulsion contractors currently have either an AP disposal problem or a ground water contamination problem that could delay, add unnecessary costs, or otherwise jeopardize major production programs. This resulted in a "High" ranked Air Force need, # 405, "Disposal and Demilitarization of Solid Rocket Motors."

This technology is a low-cost biodegradation process that converts the perchlorate ion ( $\text{ClO}_4^-$ ) in process wastewater to chloride ( $\text{Cl}^-$ ). Perchlorate can be reduced from a concentration greater than 1.0% to a concentration below detection limits ( $< 0.5$  ppm). Effluents from this process can be discharged directly to conventional sewage treatment facilities. This process will:

- Reduce environmental liability to DoD and its contractors by reducing the generation of hazardous wastes
- Minimize adverse impact of environmental compliance to DoD support operations
- Reduce cost for solid rocket propellant and large rocket motor disposal

- Reduce cost for weapon system production - specifically the Minuteman III Propulsion Remanufacture Program
- Facilitate component, propellant, and propellant ingredient recovery and reuse
- Enable the continued use of AP, a critical defense material, in propulsion systems for the DoD

## **1.2 Official DoD Requirement Statements**

Increased regulatory constraints have curtailed the ability of the Air Force to dispose of rocket propellant by open-burning, open-detonation, or static firing. Treatment and conversion technologies are sought as environmentally acceptable alternatives to conventional disposal methods. This effort supports compliance with the Cooperative Threat Reduction (CTR) Program, START I, and START II Arms Control Treaties, AFMCR 136-5 "Demilitarization/Disposal Requirements Relating to the Design of New or Modification of Ammunition items," and AFLC SON 003-90 "Solid Propellant Rocket Motor Disposal."

This effort also meets requirements set forth in ESOH-902, "Destruction of Chemical Wastes Without High-Temperature Incineration," ESOH-405, "Disposal/Demilitarization of Existing Solid Rocket Motors from Large Rocket Propulsion Systems," 2.III.1.a, (Tri-Service Strategic-Plan) "Develop Technology for Rocket Motor Propellant Removal and Destruction," and 2.III.1.c, "Alternatives to Open Burning/Open Detonation (OB/OD) Destruction of Energetic Wastes." In addition, the recovery and reuse of Minuteman III Stage I & II cases is a requirement for the Minuteman Life Extension Program. Current methods of recovery generate significant quantities of secondary wastewater that contain perchlorate, asbestos, salts, corrosion inhibitors, metals, and other propellant ingredients. This biodegradation process will provide a safe, low-cost, environmentally acceptable method for disposal of process wastewater associated with production, remanufacturing, testing, demilitarization, and disposal.

**1.2.2 How Requirements Were Addressed.** The demonstrated ability to biologically reduce the perchlorate ion to chloride provides an efficient, inexpensive complete destruction process. Most perchlorate effluents contain numerous additional contaminants. Each perchlorate problem has its own unique set of contaminants and contaminant concentrations that must be accommodated by this process. The key to addressing these requirements is to determine how to adapt, design, and control this process to achieve maximum efficiency for each problem set. As this process is demonstrated on a wider range of effluents we have gained confidence that it can be adapted to almost any situation. In this program, the biodegradation process was adapted to reduce perchlorate in a brine effluent to chloride.

## **1.3 Objectives of the Demonstration**

The objective of this demonstration was to provide a production-scale, operational validation of the ammonium perchlorate (AP) biodegradation process that was developed by the Air Force Research Laboratory. Components of an existing pilot-scale demonstration unit were modified and integrated into existing waste treatment facilities at Thiokol's production plant near Brigham City, Utah. The demonstration occurred in two phases. First, the production-scale transportable system was assembled on an existing test site at Tyndall AFB, Florida to conduct functional and process demonstrations. Process control and operation was demonstrated with two bioreactors in

both series and parallel operational configurations. Upon successful completion of the Tyndall demonstration, the system was modified, disassembled, transported to Thiokol, and reassembled. The validation testing at Thiokol demonstrated both technical and cost performance in an integrated industrial waste treatment facility. Actual effluents from operational processes and perchlorate recovery units were treated and discharged to an existing sewage treatment plant.

#### **1.4 Regulatory Issues**

Alternative processes for disposal of propellants are required to achieve compliance with the Clean Air Act Amendments (CAAA) 1990, the Federal Water Pollution Prevention and Control Act (FWPPCA) 1987, the Federal Facility Compliance Act 1992 (P.L. 102-386), and Executive Order 12856 (Aug-93)/Federal Compliance with Right-to-Know Laws and Pollution Prevention Requirements. Each demonstration facility complied with its State Implementation Plan and NPDES permit requirements.

The original NPDES permit requirement at Thiokol for perchlorate discharge was 10 ppm. To achieve this requirement, perchlorate recovery and ion exchange processes were developed and implemented by Thiokol. However, these processes are creating much higher total dissolved solids (TDS) which would likely violate the NPDES permit requirement of 3800 ppm during any major remanufacture program, such as the Minuteman III, or a disposal effort such as the Titan solid rocket motor (SRM) disposal program. The Titan disposal program will removal solid propellant from 57 Titan SRM segments using water washout.

In 1992 the USEPA evaluated studies on medical patients who were given perchlorate to treat hyperactive thyroid glands (Graves' disease). This data was used to help determine the health risks of perchlorate exposure. The most sensitive indicators of perchlorate effects were the release of iodine from the thyroid and inhibition of iodine uptake. For these effects, the EPA identified a no observable adverse effects level (NOAEL) of 0.14 mg/kg/day and applied standard dose criteria with an uncertainty factor of 1000 to obtain a 4 parts-per-billion (ppb) drinking water criteria. In 1995, the EPA reevaluated the uncertainty factors. They included an uncertainty factor of 300 that made perchlorate concentration limits in drinking water range from 4-18 ppb. In 1997, improved analytical methods enabled perchlorate detection limits to decrease from approximately 1 mg/liter (or part-per-million, ppm) to 4 ppb.

In February 1997, the California Department of Health Services (DHS) began to analyze certain drinking water wells in Sacramento County and southern California suspected of containing perchlorate. As contaminated wells began to be identified, the California DHS reviewed the EPA data and established an 18 ppb action level for perchlorate in drinking water. On August 1, 1997, DHS informed drinking water utilities of its intention to develop a regulation that includes perchlorate as an unregulated chemical for which monitoring is required.

## 1.5 Previous Testing of the Technology

The results of laboratory studies were used to design, fabricate, and demonstrate at the pilot-scale, the operability of a complete AP biodegradation system using actual effluent from the washout of Minuteman stage 2 propellant washout by Aerojet Corporation's Propulsion Division. The design of the pilot-scale system was centered on a 350-gallon anaerobic reactor capable of treating up to 1000 gallons per day of dilute AP wastewater. A new facility was constructed to house the pilot-scale bioreactor system at the Tyndall AFB, Florida. Case Engineering of Lakeland, Florida constructed the pilot-scale system. This modular, skid-mounted system was delivered to Tyndall AFB in October 1994 on three trailers and completely assembled in 7 days. In May 1995, the pilot-scale system was operated continuously for over 600 hours using an extract prepared from dried brewer's yeast, reducing a 3000 ppm perchlorate feed stream to less than detectable limits. The pilot-scale system was also operated for over 900 hours at residence times as short as 12 hours using a commercially available, water-soluble, yeast extract called BYF-100. Although both nutrients performed well, the more expensive BYF-100 was more efficient as a nutrient source and aided in the reduction of biological oxygen demand (BOD) in effluent discharges.

## 2. Technology Description

### 2.1 Description

**2.1.1 Background.** In 1989 biodegradation was recognized as a viable process to treat dilute AP waste streams and remediate contaminated soil and ground water. Attaway and Smith<sup>1</sup> isolated an organism capable of reducing perchlorate and designated the bacterium HAP-1. Consequently, laboratory studies were conducted in batch mode and in continuously stirred tank reactors (CSTR's). Process variables that affect perchlorate reduction performance were addressed in bench-scale studies. These variables included temperature, pH, nutrient type, nutrient concentration, residence time, and perchlorate ion concentration. Nutrients had to be commercially available, relatively low cost, and demonstrate good performance with respect to perchlorate reduction. The most promising nutrients were dried brewer's yeast and yeast extracts. Typical treatment conditions identified were:

Temperature	37-42 °C
pH	6.5-7.6
Residence Time	8-24 hours
Perchlorate Concentration	< 6000 ppm
Degradation Rates	125 mg/l per hr

**2.1.2 Recent Discoveries.** Laboratory efforts culminated in the positive identification of the genus and species of the bacterium in the mixed culture responsible for perchlorate reduction as *Wolinella succinogenes*.<sup>2</sup> This discovery enabled the Air Force to take advantage of the scientific data and literature on the specific bacterium, leading to promising process enhancements. One discovery is the microaerophilic nature of *Wolinella succinogenes*. This means that this organism may prefer small concentrations of oxygen, or that oxygen could compete with perchlorate reduction as an alternate electron acceptor in certain metabolic pathways. Previously, it was assumed that oxygen did not play a critical part in perchlorate reduction because other microbes in the consortium would consume the oxygen to maintain anaerobic conditions. When strict anaerobic conditions were maintained, very stable, predictable perchlorate reduction was obtained at rates exceeding 0.5 g/l per hour. In addition, it was successfully demonstrated that the HAP-1 mixed culture could destroy AP in the presence of other energetic compounds including nitroglycerin, nitramines, stabilizers, and plasticizers. One patent has been granted on this process<sup>3</sup> and additional patents are pending.

**2.1.3 Optimization Studies.** Additional studies have demonstrated that this biodegradation process is much more durable, flexible, and predictable than originally perceived. Process optimization efforts have focused on reducing operating costs, tailoring process variables, and reconfiguring operations to treat representative industrial wastes. These efforts have resulted in an increased robustness of the process to effectively treat effluents containing over 1.0 percent (10,000 ppm) perchlorate. Perchlorate can also be reduced in effluents with a high salt content (> 2.3 % Na<sup>+</sup>, K<sup>+</sup>, Cl<sup>-</sup>) and other impurities (NO<sub>2</sub><sup>-</sup>, NO<sub>3</sub><sup>-</sup>, SO<sub>4</sub><sup>-2</sup>), and over a broad temperature range (20-42 °C). Lower cost nutrients were successfully demonstrated to significantly lower the primary operating expense. Dried brewer's yeast can be used directly, without extracting the critical nutrients. Although this increases BOD concentrations in the effluent, the total nutrient requirement and costs are reduced. Preliminary studies have shown that dried, sweet cheese whey may also be an effective nutrient by itself or in mixtures with brewer's yeast. Cheese whey is more soluble than brewer's yeast and only one fourth the cost. Using unprocessed yeast and cheese whey wastes may possibly reduce nutrient costs even further.

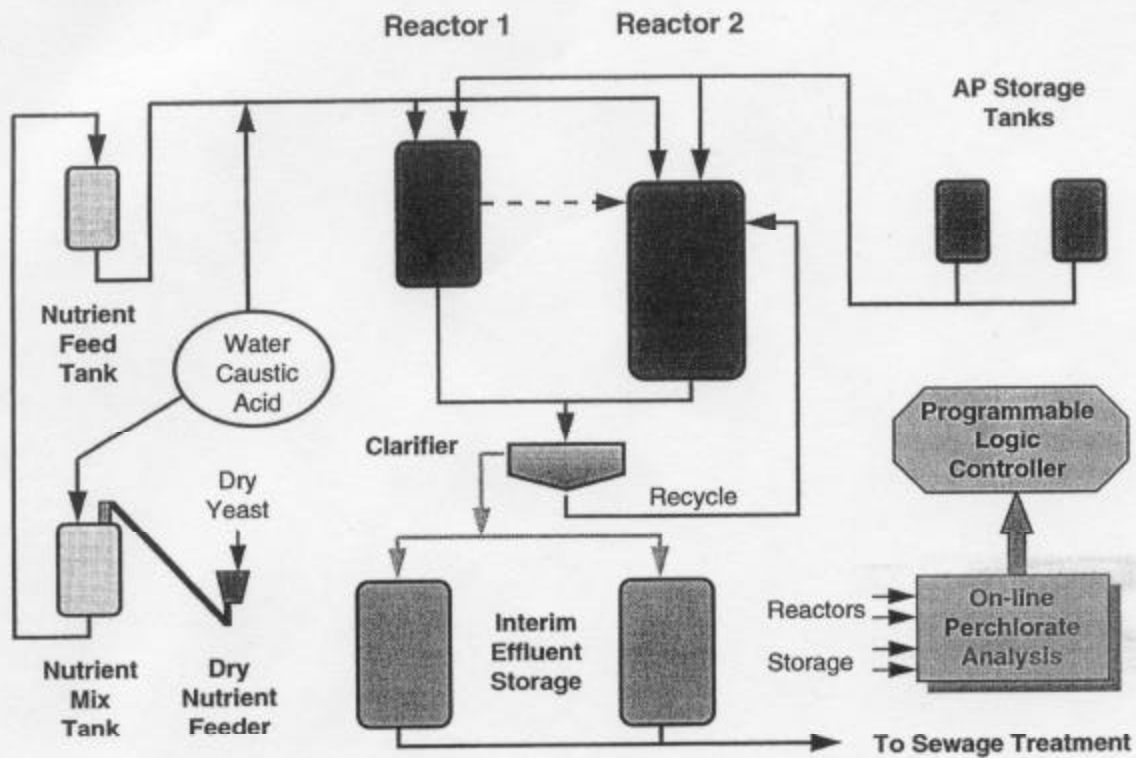
**2.1.4 Production-Scale Design.** The general requirements for developing production-scale demonstration system design were (See Figure 1 below):

- Robust Process Design - provide stable, dependable, repeatable operation
- Flexibility to handle different effluents - treat a broad range of feed rates and perchlorate and brine concentrations
- Low-cost operation - ability to use low-cost nutrients
- Maximum automation and flexibility of process control - Programmable Logic Control (PLC)



Figure 1. Proposed Ammonium Perchlorate Biodegradation Unit

### Ammonium Perchlorate Biodegradation Prototype at Thiokol



Specific design requirements include:

- Capacity - treat 450 gallons per day of saturated brine containing 3000-5000 ppm AP
- Perchlorate concentration - treat up to 1.0% perchlorate in effluents
- Reactor system - two anaerobic reactors that can be operated in parallel or series
- Contaminants - reduce perchlorate in the presence of nitrate and nitrite
- Nutrient - prepare and feed low-cost, high solids, dried brewer's yeast
- Materials of construction - able to handle corrosive environment of perchlorate, sulfuric acid, and sodium hydroxide
- Process variable control:
  - ✓ Temperature - control both reactors in the 30-40 °C range
  - ✓ pH - control reactors near pH 7.1 and nutrient slurry in the 2.0-4.0 pH range
  - ✓ Residence times - nominal 18-24 hours, capable of 12-30 hours
  - ✓ Reactor inerting - sparge with nitrogen generated by a membrane system
- Process control - on-line perchlorate analysis providing feedback for flow control

## 2.2 Advantages and Limitations of the Technology

This process is inherently reliable because of its near ambient operating conditions. However, loss of temperature or pH control could destroy microbes in the reactor and force re-inoculation of the system. This could interrupt operations for several days. This process, with dual anaerobic reactors, a clarifier, effluent storage tanks, and on-line perchlorate analysis providing feedback control, significantly mitigates against potential loss of biological activity. If perchlorate concentration begins to rise in a reactor indicating loss of perchlorate reduction activity, the control system will limit the perchlorate effluent feed, helping to preserve viable microbes in all process vessels. Once factors causing the upset have been identified, a reactor can quickly be re-inoculated by recycling effluent from the second reactor, clarifier, or effluent storage tanks.

### 2.2.1 Advantages:

- Process operates at near ambient conditions - inexpensive equipment & operation
- Process is fast for an anaerobic biodegradation process - residence time is typically less than 24 hours, rates can exceed 0.5 g/liter per hour, reduces the size & cost of equipment
- No post-treatment is necessary - reduces perchlorate to less than 0.5 ppm in a single stage and effluent can be discharged to sewer
- High concentration perchlorate (> 1.0%) can be fed to a continuously stirred tank reactor (CSTR) as long as reactor concentration is maintained below 6000-10,000 ppm
- Can treat up to 6000 ppm perchlorate effluents in a single step
- Can reduce perchlorate in the presence of >2% salts and other inorganic contaminants
- Relatively low operating cost - \$0.10 to \$0.20 per gallon for perchlorate concentrations up to 6000 ppm vs. conventional treatment costs in excess of \$1.00 per gallon
- Biodegradation of low concentration perchlorate is much less expensive than concentration/recovery processes
- The naturally occurring consortium of microbes appears very stable - the HAP-1 organism has not been lost or displaced after months of operation in unsterile environments, except when reactor was exposed to excess oxygen, high temperature, or extreme pH

### 2.2.2 Limitations:

- There is some practical limit to perchlorate equilibrium concentrations in a reactor concentrations above 6000-10,000 ppm appear to inhibit perchlorate reduction
- At perchlorate concentrations above 4000 ppm processing costs increase linearly with perchlorate concentration due to a proportional nutrient requirement
- BOD/COD concentrations of the effluent are proportional to nutrient addition - if local sewage treatment facilities cannot accept additional high BOD/COD effluents, then anaerobic and/or aerobic post-treatment would be required to lower the organic content

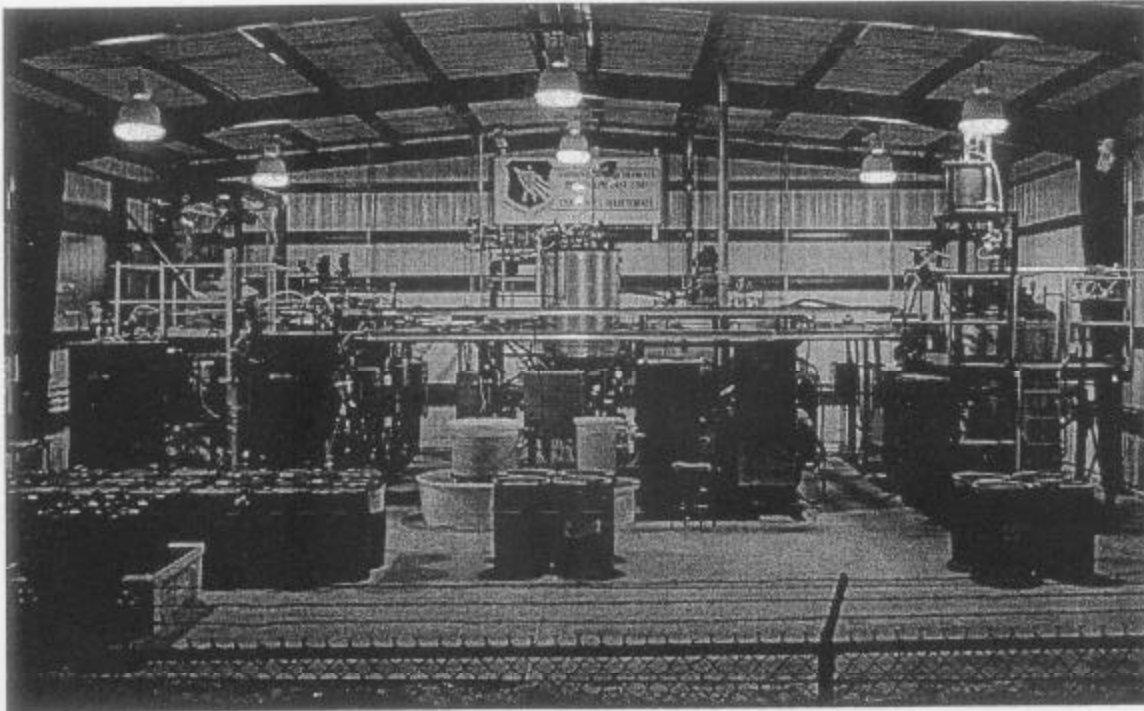
### 3. Site/Facility Descriptions

#### 3.1 Background

There are at least eight rocket motor production facilities, two perchlorate-manufacturing facilities, and many ordnance production facilities in the United States. Before current RCRA and other environmental laws were enacted and enforced, propellant, and propellant ingredient wastes were disposed of by open burning and open detonation (OB/OD), as stated earlier. Aqueous effluents were also collected in evaporation ponds, or, in some cases, discharged to the environment. In the early 1990s, Thiokol Corporation, constructed an industrial wastewater treatment plant at its production facility near Brigham City, Utah and deactivated their evaporation ponds. Current aqueous treatment costs at Thiokol are in excess of \$1.00 per gallon and these treatment processes generate sludges, spent activated carbon sorbents, spent ion exchange resins, salts, and brine solutions containing AP.

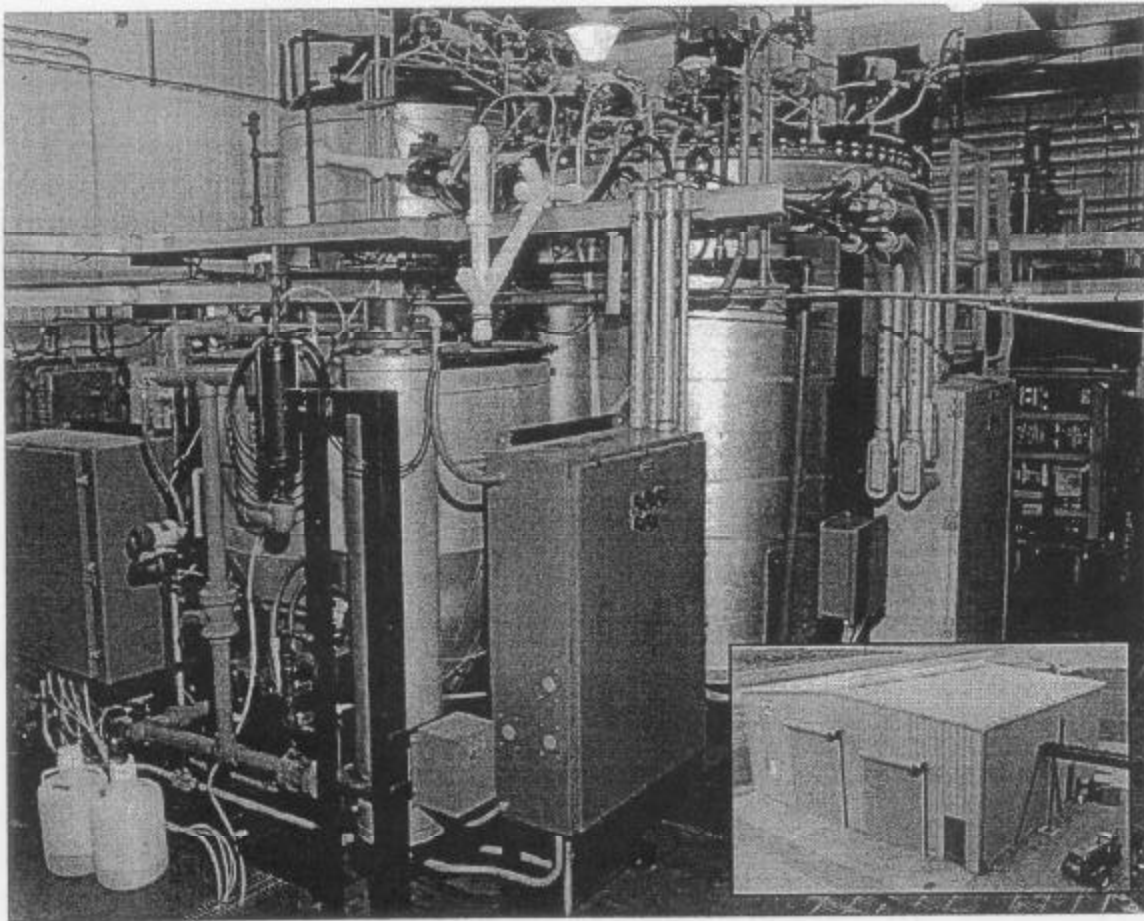
**3.1.1 Tyndall AFB Demonstration Site.** The first phase of the demonstration occurred at Tyndall AFB, Florida. The existing facility that housed the 1995 and 1996 demonstrations of the original pilot-scale system were used. Use of the existing infrastructure, laboratory facilities, and experienced operators minimized risks during system trouble-shooting and checkout. Operations at Tyndall AFB were concurrent with facility preparation and building construction at the Thiokol site.

**Figure 2. Building 1708 at Tyndall AFB, FL**



**3.1.2 Thiokol Demonstration Site.** Thiokol's Utah operations are located 25 miles northwest of Brigham City, Utah, near Promontory. Thiokol has been involved in propellant removal, component recovery, demilitarization, and large rocket motor production for over 35 years. In addition to production programs for tactical systems such as the Standard Missile, stages or components of major Navy and Air Force Strategic systems have been produced at this facility. These include Minuteman I, II, and III, Peacekeeper, and Trident I and II. Space Shuttle Solid Rocket Motors (SRMs) are also manufactured at this facility.

**Figure 3. Building M-705A at Thiokol Corporation**



The perchlorate biodegradation process was integrated into existing waste treatment processes at Thiokol's production facility. Several propulsion contractors and defense facility sites were investigated for potential application of AP biodegradation. Thiokol was selected for the following reasons:

- Perchlorate wastes are generated from production and demilitarization operations
- Perchlorate wastewater is segregated from wastewater containing other energetics

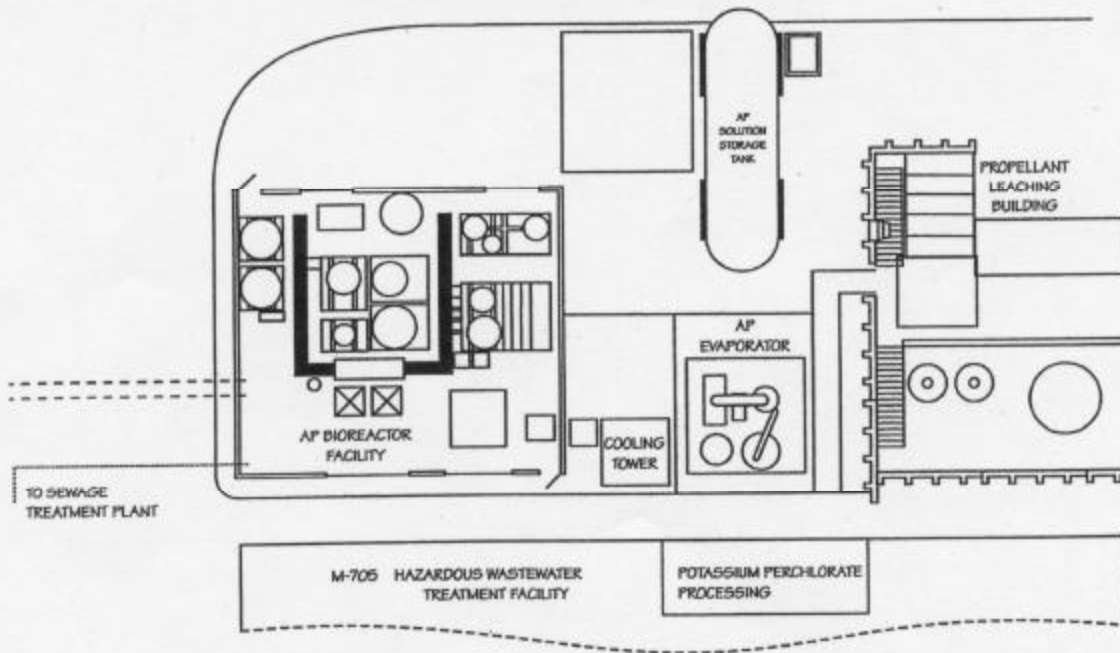
- A potassium perchlorate recovery process is in operation that produces a perchlorate containing brine effluent
- A second AP recovery process has recently begun operation. It may also produce an AP effluent that requires potassium precipitation and/or perchlorate biodegradation
- A new, state-of-the-art, industrial wastewater treatment plant is operational
- A new sewage treatment facility has been constructed and is in operation. It can easily treat high BOD/COD effluents generated by the AP biodegradation process.

### 3.2 Site/Facility Characteristics

The Demonstration site at Thiokol is located adjacent to building M-705, the existing IWTP. This facility contains the potassium perchlorate precipitation and recovery process that generates the perchlorate containing brine effluent. A schematic of the site plan with the location of 42 ft by 48 ft biodegradation facility is shown in Figure 4 below.

Figure 4. Thiokol Site Plan

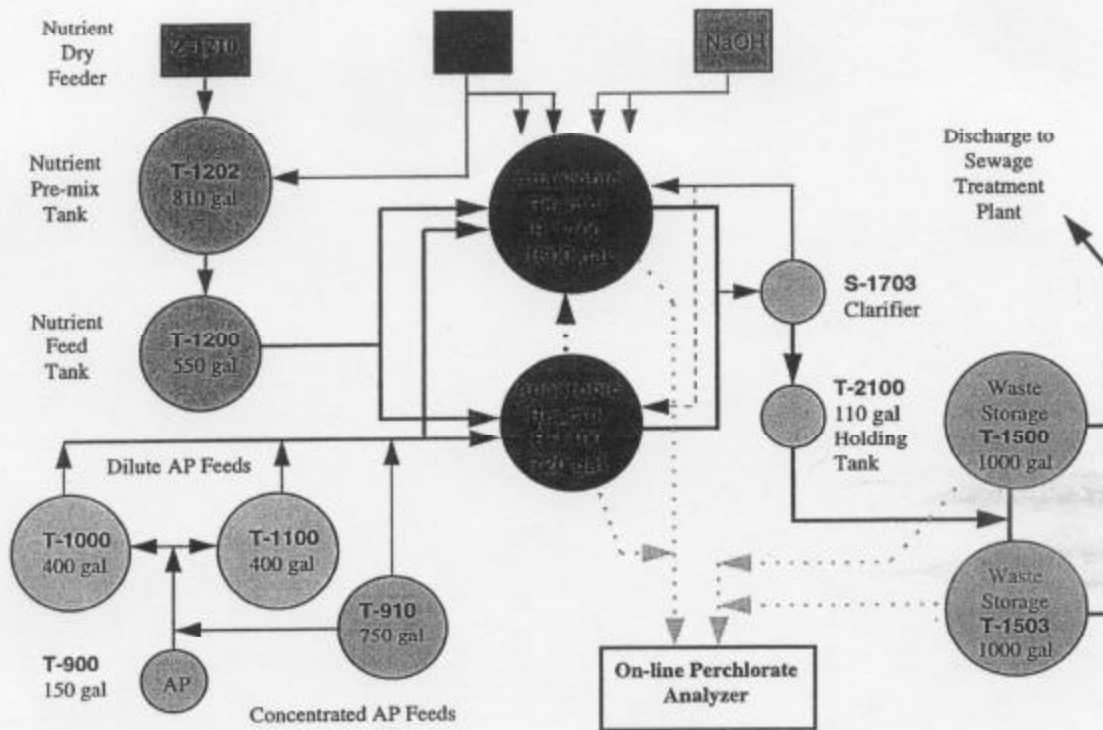
## THIOKOL SITE PLAN



The identification and general arrangement of the major equipment components of the modified process that will be demonstrated at Thiokol are shown in Figure 5 below.

Figure 5. Equipment Location and Arrangement

### Identification and Arrangement of Process Equipment



The process that produces the brine/perchlorate waste stream precipitates potassium perchlorate (KP) from concentrated ammonium perchlorate effluents. These effluents are concentrated by evaporation and ion exchange, followed by ammonia being stripped (and replaced by sodium) and excess potassium chloride (KCl) is added to effect the precipitation of KP. This results in a 10-30% salt ( $\text{Na}^+$ ,  $\text{K}^+$ , and  $\text{Cl}^-$ ) brine solution which contains 3000-5000 ppm perchlorate and additional nitrate and nitrite. Solubility curves were generated for both sodium and potassium perchlorate in order to minimize KCl additions, effect the optimal perchlorate precipitation, and determine appropriate compositions for surrogate waste streams used in laboratory and pilot-scale tests.



## 4. Demonstration Approach

### 4.1 Performance Objectives

**4.1.1 Contaminants.** The perchlorate anion ( $\text{ClO}_4^-$ ) is the primary regulated contaminant in the effluent and is readily reduced to chloride ( $\text{Cl}^-$ ). It has been demonstrated that this reduction is not inhibited by the presence of different cations ( $\text{NH}_4^+$ ,  $\text{Na}^+$ ,  $\text{K}^+$ ). In addition to perchlorate reduction, other contaminant anions including nitrate ( $\text{NO}_3^-$ ), nitrite ( $\text{NO}_2^-$ ), and chlorate ( $\text{ClO}_3^-$ ) are reduced. This is important since they represent degradation products or components of other energetic materials and corrosion inhibitors that may be present with perchlorate. Other products of reduction, chloride and nitrogen, are discharged.

**4.1.2 Objectives.** The treatment objective was to reduce all the perchlorate to chloride in a contaminated brine effluent. The lower detection limit for state-of-the-art HPLC perchlorate analytical methods for process wastewater is 0.5 ppm. The next goal was to purposely keep reduction rates for this process low when treating dilute perchlorate brines (20% brine) to minimize operating costs and nutrient consumption. On the other hand, when treating high perchlorate effluent, the goal was to optimize nutrient concentration to achieve reduction rates equal to or greater than 0.5 grams/liter per hour. Another objective was to demonstrate on-line perchlorate analysis for process control. By adjusting process control logic, residence time and nutrient consumption can be minimized and the process optimized towards cost and/or capacity.

Two operating conditions were evaluated during the Tyndall demonstration: 1) Parallel operations with a brine feed (5000 ppm AP brine fed at 10-20% rate) utilizing a mixed yeast and cheese whey nutrient solution, and 2) series operations with a high perchlorate feed (10% solution fed at a 1% rate to the first reactor) utilizing a dried brewer's yeast solution.

Typically, the process requires less than five or six residence times to stabilize after a condition change (i.e., five or six days when operated at a 24 hour residence time). At this time, a final analysis may be conducted prior to changing conditions. However, since this is a biological process, condition changes occasionally produce a lag-time while the organisms adapt to the new environment. Therefore, it is desirable to operate at some conditions for extended periods of time to ensure the culture has fully adapted (either positively or negatively) to the new condition. Funding support and program deadlines limited the length of the demonstrations. However, through the CRDA with Thiokol, operation will continue at least two years from the end of this program. Under the CRDA, performance and cost data will continue to be collected and reported.

**4.1.3 Process Waste.** Effluent from the anaerobic reduction has an elevated BOD/COD content directly proportional to the amount of nutrient added to the reactor. When treating 1000 ppm perchlorate, the effluent COD will be approximately 4,000 ppm and the BOD will be approximately 3,000 ppm. Typical domestic sewage has a BOD of approximately 200 ppm. If local sewage treatment facilities cannot accept new, high BOD/COD effluents, then aerobic post-treatment is required to lower the organic content. During the demonstrations at Tyndall AFB and Thiokol, the effluent from the anaerobic reactors were discharged directly to existing sewage treatment facilities. At Thiokol, a newly constructed, operational, sewage treatment facility has

excess capacity which can easily treat high BOD/COD effluents generated by the AP biodegradation process.

**4.1.4 Factors Affecting Technology Performance.** Process variables that affect perchlorate reduction were addressed in the laboratory studies discussed earlier. These variables included temperature, pH, oxygen concentration, nutrient type, nutrient concentration, residence time, other anions, and perchlorate ion concentration. These process variables can be controlled to optimize performance. Unknown factors that could impact performance are other possible contaminants present in industrial wastewater not previously identified in the laboratory.

## **4.2 Physical Setup and Operation**

**4.2.1 Facility Requirements.** The Tyndall site required minimal preparation for this demonstration. The most significant modification was converting the facility from 208 VAC, 3-phase electrical service to 480 VAC, 3-phase service. This enabled demonstrations at Tyndall AFB and Thiokol facilities to take place without an intervening modification to adapt the process to Thiokol's 480 VAC power requirement. This simplified transition time and resources.

A new facility was prepared for the Thiokol site. The entire system is housed in a building to protect it from the elements and low temperatures. Thiokol provided infrastructure and connection to all utilities, effluent streams, and the sewage treatment facility. After initial testing, Thiokol will operate the system for at least two years under the existing CRDA. While effluents are delivered to the process in batches and stored before treatment, the process itself is operated continuously, 24 hours per day, seven days per week.

**4.2.2 Inoculation and Startup.** An important step in startup is inoculating the reactor with the HAP-1 microbe consortium. To facilitate startup of field systems, or to recover from a total process upset, this program demonstrated reactor inoculation from lyophilized (freeze-dried) microbial samples. Lyophilized samples can be stored in sealed, evacuated glass vials for decades. When these vials are opened, the contents are added directly to a growth media containing nutrient (5-25 g/l of brewer's yeast, yeast extract and/or sweet cheese whey), pH buffers, and perchlorate (500-1000 ppm). The culture inoculum can be grown at temperatures of 25 to 40 °C in an anaerobic chamber or in sealed bottles. The lyophilized vials are prepared at Tyndall AFB from cultures in active laboratory reactors that are effectively reducing perchlorate. Typically, 0.5 ml of reactor contents and 0.5 ml of 24% sucrose are lyophilized in 10 milliliter (ml) vials. The contents of a lyophilized vial are then transferred to 100 ml bottles where perchlorate concentration is monitored and pH is adjusted daily to maintain pH 7.0-7.5. Sharp decreases in perchlorate concentration indicate that the culture is growing. At this time, the inoculum is transferred to 1 liter bottles with additional nutrient and perchlorate. One additional transfer to 20 liter carboys is necessary before inoculating the reactor. In the first reactor, 200 gallons of 12 g/l brewer's yeast containing 1000 ppm perchlorate are brought to pH ~7.0 and a temperature of 30-40 °C and subsequently sparged with 2.0 ft<sup>3</sup>/min of nitrogen. This reactor is inoculated by adding four 20 liter carboys to 200 gallons of media. When perchlorate concentration begins to drop rapidly, additional media and perchlorate effluent are metered into the reactor continuously at flow rates proportional to a 24 hour residence time until the reactor is at capacity. At this time the first set of operational conditions can begin.



### 4.3 Sampling Procedures

There are two types of sample media: 1) aqueous feed solutions consisting of perchlorate anions, various salts and corrosion inhibitors, and other soluble inorganic components and 2) biosludge samples from the reactors. These differing types of sample media had no effect on the sampling procedure and corresponding analyses for inorganics. Samples taken for plate count determinations were kept as anaerobic as possible, given the sampling environment, and the plating procedure executed within 30 minutes of sample collection. All samples were taken from various sample ports on the pilot scale facility. All valves and hoses used for sample collection were flushed prior to sample collection insuring fresh samples. Technicians and operators were trained in proper sampling procedures and QA/QC protocols used by AFRL at Tyndall AFB, Florida.

Standard Quality Assurance/Quality Control practices were employed with each set of sample collections. Field duplicates were collected and analyzed for the same parameters as the associated samples. They were preserved, transported, and documented in the same manner as the samples. Field duplicates were collected to measure the variability inherent in sampling processes. They were obtained by duplicating the entire sample acquisition process used to obtain all other samples. Frequency of field duplicate sampling is a minimum of one or 10% of the total number of samples taken. Split samples were also taken, preserved, transported, and documented using the same protocols as the related samples. Aqueous or dilute biosludge solutions were split due to the extreme variability of samples (i.e., soil, sediment). The samples were mixed in a large, appropriately cleaned, sample containers and aliquots were poured into the appropriate labeled sample containers. Split sampling is conducted once per sample batch.

Samples were taken daily and placed in polypropylene bottles to be frozen immediately, except for samples required for pH testing, plate counts, and solids tests which were performed upon receipt. Sample containers were labeled using sample location, date of collection, time of sample, name of person taking the sample, and the type of analysis required.

The effectiveness of the demonstration will be determined by the concentration of the perchlorate anion following treatment by the bioreactor. The demonstration will be successful if the effluent concentration of perchlorate is kept below Thiokol's permit limit of 10 ppm.

### 4.4 Analytical Procedures

**4.4.1 Types of Analyses.** Sampling and data collection consisted of monitoring the process variables and analyzing the influent and effluent streams. Analytical methods developed and used during bench scale studies and process development were used for the process demonstration. Analyses consisted of:

- 1) Anions -  $\text{ClO}_4^-$ ,  $\text{ClO}_3^-$ ,  $\text{Cl}^-$ ,  $\text{NO}_3^-$ ,  $\text{NO}_2^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{PO}_4^{2-}$ , & others if suspected
- 2) Cations -  $\text{NH}_4^+$
- 3) Chemical oxygen demand (COD)
- 4) Total suspended solids (TSS) and total dissolved solids (TDS)
- 5) HAP-1 and Other microbe cell counts

## 6) Biological oxygen demand (BOD)

Historical data indicates that metals present in low concentrations do not present a problem to this process. Therefore, metals were not analyzed. The sampling locations indicated in the Sample Plan Matrix (Appendix B), can be found on the general arrangement of the process equipment provided in Section 4.3.

**4.4.2 Selection of Analytical Methods.** The primary method to determine the concentration of the perchlorate anion used an ion ( $\text{ClO}_4^-$ ) specific electrode which measures electrical potential differences in conjunction with known additions of perchlorate containing standards. This is a standard method for perchlorate anion concentration. This method was used to determine perchlorate concentration for both discrete and continuous samples. The prototype unit was equipped with online perchlorate analysis capability. The specific ion electrode method can accurately detect perchlorate concentrations in reactor effluents to a lower limit of 10 ppm.

An ion chromatography method for perchlorate anion concentration determination was also employed. This method was developed in 1992 using a Dionex DX-300 Series Ion Chromatography Instrument employing a C-18 HPLC reverse-phase column and conductivity detection. The ion chromatography method can accurately detect perchlorate levels down to 0.5 ppm.

All other anion determinations ( $\text{ClO}_3^-$ ,  $\text{Cl}^-$ ,  $\text{NO}_3^-$ ,  $\text{SO}_4^{2-}$ , and  $\text{PO}_4^{2-}$ ), with the exception of nitrite ( $\text{NO}_2^-$ ), are completed with the Dionex DX-300 Series Ion Chromatography Instrument employing a AS-11 Ion Pac column from Dionex and conductivity detection. The Dionex system is computer controlled and integrated with AI-450 Chromatography Software.

Nitrite analyses are conducted using Standard Method 4500- $\text{NO}_2^-$  A<sup>4</sup>. The spectrophotometer used to measure absorbance upon preparation completion is a CARY 3E-UV, Visible Spectrophotometer. This is a standard method for nitrite anion determinations. Nitrite values are expressed in ppm units.

Chemical Oxygen Demand (COD) is determined by the HACH method described in detail in the HACH Water Analysis Handbook. This method is EPA approved. In the chemical oxygen demand test, the sample is heated for two hours at 150 °C with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, reducing the dichromate ion ( $\text{Cr}_2\text{O}_7^{2-}$ ) to green chromic ( $\text{Cr}^{3+}$ ) ion. The chloride ion can be a possible interference for this method. The COD catalyst powder contains mercuric sulfate to complex up to 1000 mg/L chloride. For higher chloride concentrations, the sample must be diluted appropriately so that the chloride concentration is less than 1000 mg/L. COD concentrations will be presented in parts per million (ppm).

Total Organic Carbon (TOC) is determined using the Shimadzu TOC-5000 equipped with the ASI-5000 autosampler. This analytical method is also EPA approved. To determine the quantity of organically bound carbon, the organic molecules must be broken down to single carbon units and converted to a single molecular form that can be measured quantitatively. TOC methods utilize heat and oxygen, ultraviolet irradiation, chemical oxidants, or combinations of these oxidants to convert organic carbon to carbon dioxide. The carbon dioxide is then measured directly by a non-dispersive infrared analyzer. TOC concentrations were reported in ppm units.

## 5. Performance Assessment

### 5.1 Performance Data: Tyndall AFB Demonstration

**5.1.1 Startup.** Both parallel and series operation were conducted successfully during the Tyndall AFB demonstration. The inoculum was grown from a lyophilized sample to validate the procedure presented in Section 4.2.2. The nitrogen generator was used to purge the reactor (R-1400) of oxygen to facilitate inoculation. The reactor was charged with approximately 200 gallons of nutrient (10 g/l) and 1000 ppm of ammonium perchlorate solution, the temperature and pH adjusted, and then the inoculum added. When the perchlorate concentration began to drop, additional perchlorate and nutrient solution were metered into the reactor. However, by the time R-1400 was full, perchlorate was be completely reduced at a residence time of 24 hours. The effluent from R-1400 was fed to R-1700 along with additional perchlorate and nutrient to inoculate the second reactor. With both reactors inoculated and accepting feed, parallel operation was initiated.

**5.1.2 Effluent Analysis.** The perchlorate used for inoculation and startup was prepared from a 10-12% ammonium perchlorate solution from a Minuteman stage 2 propellant washout so as to conserve the Thiokol brine solution. Table 1 describes the different feed materials used in the Tyndall AFB Demonstration.

**Table 1. Thiokol Effluent Characterization for Tyndall AFB Demonstration**

	ClO <sub>4</sub> <sup>-</sup> (mg/l)	Cl <sup>-</sup> (mg/l)	NO <sub>3</sub> <sup>-</sup> (mg/l)	NO <sub>2</sub> <sup>-</sup> (mg/l)	TDS (mg/l)
NaClO <sub>4</sub> Brine	4266	101,161	4662	299	271
NH <sub>4</sub> ClO <sub>4</sub> Concentrate	123,000	2286	346	128	125

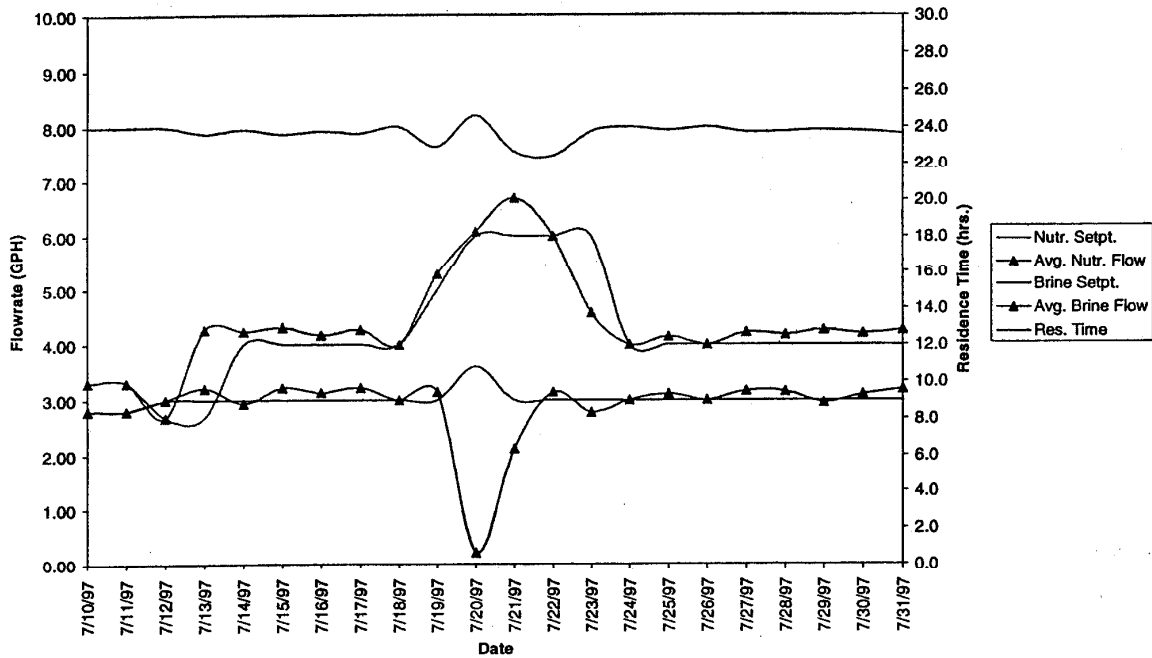
**5.1.3 Test Matrix.** Both parallel and series operational tests were conducted. Parallel operation on the brine effluent was conducted first. After successful inoculation and operation on 1000 ppm ammonium perchlorate, the feed was switched to a 10 % brine feed. The 10% concentration was necessary due to unexpectedly high total dissolved solids in this effluent. Even a 10% solution resulted in a 2.71% salt content in the reactor before nutrient, acid, and caustic additions. This was near the limit indicated by bench-scale studies before significant performance degradation was observed. A summary of the test conditions is provided in Table 2.

**Table 2. Test Condition Summary**

			Effluent	Nutrient Feed		Res. Time	Other
	Date Initiated	Reactor Config.	Feed Conc.	g/l	Type	hours	Events/Comments
1	7/1/97	Series	1000 ppm AP	5	100% yeast	24	Start-up

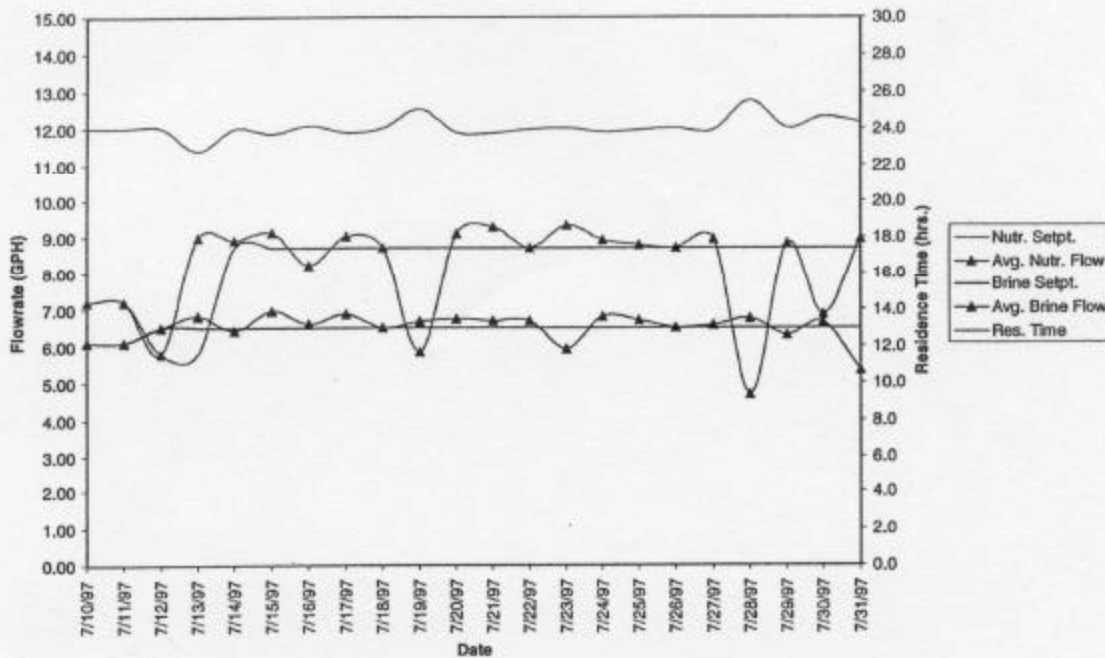
2	7/10/97	Parallel	1000 ppm AP		100% yeast	24	Start-up
3	7/14/97	Parallel	10% Brine	4	25:75 yeast:whey	24	Initiate brine feed Initiate whey nutrient
4	7/27/97	Parallel	10% Brine	4	50:50 yeast:whey	24	Increase yeast concentration
5	7/29/97	Parallel	10% Brine	4	100% yeast	24	Increase yeast concentration
6	8/1/97	Series	4000 ppm AP	16	100% yeast	24	Initiate series operation Initiate 10% AP feed
7	8/7/97	Series	6000 ppm AP	24	100% yeast	24	Increase yeast concentration
8	8/30/97	Series	8000 ppm AP	32	25:75 yeast:whey	24	Initiate whey nutrient

**5.1.4 System Functional Performance: Parallel Operation using Brine.** Overall the system performed as designed. Initially there was a concern that the flow control valves for the nutrient would easily plug from the yeast solids. Nutrient deficiencies could lead to perchlorate excursions in the reactor. Valve orifices had to be changed to larger  $C_v$  values than designed in order for the control valves to work as desired. Average flow rates were determined based on daily readings of system totalizers. Nutrient and perchlorate brine flow rates were maintained very close to set point values as seen in Figure 6. This resulted in a nearly constant residence time. The only deviation was due to a reduction in the brine flow to R-1400 due to elevated perchlorate levels in the reactor. This was caused by the culture adapting to the high salt concentrations. Water flow rates were assumed to be constant at the set point. Totalizer glitches sometimes caused inaccurate readings. However, when water flow was physically measured against the set point, flow was accurate to within measurement error (< 2%). The operational data is provided in Tables 3 and 4 (Appendix B).



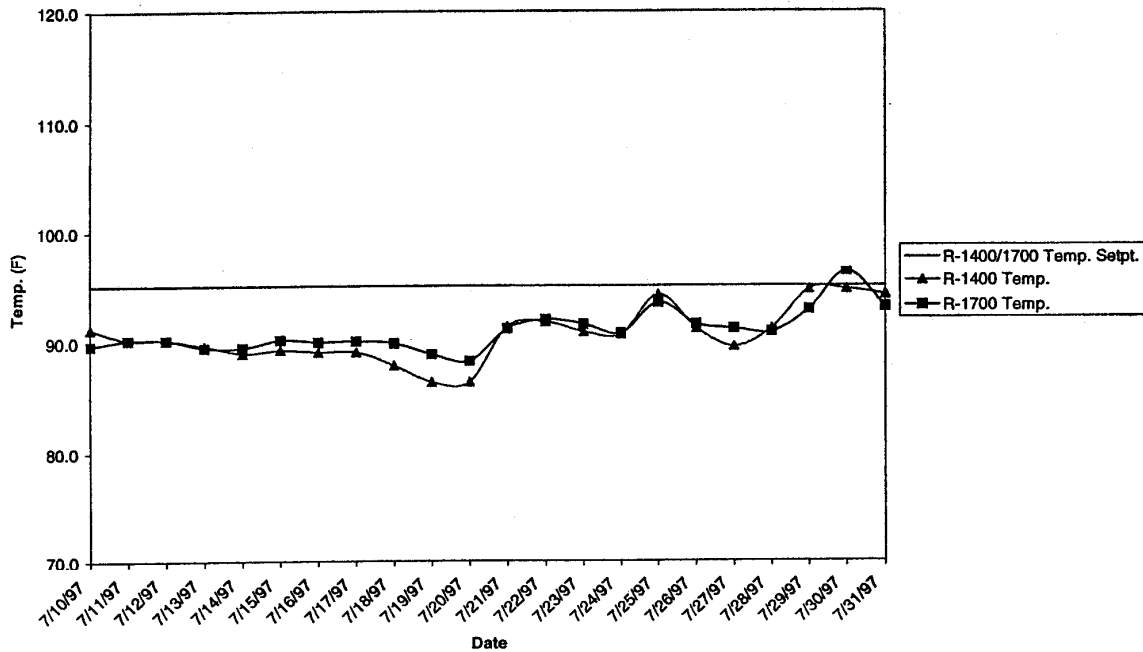
**Figure 6. R-1400 Setpoints and Average Flow Rates vs. Time**

Figure 7, shows the set-points, average flow rates, and residence time of R-1700 during parallel operation. Again, water flow was assumed to be constant at the set-point. Residence time and brine flow remained relatively constant through the demonstration. Undersized orifices in the control valve contributed to plugging the nutrient control valves. This was corrected with a larger  $C_v$  value and also by installation of a nutrient recirculation loop from the control valves on R-1400 and R-1700 back to the nutrient feed tank (T-1200).



**Figure 7. R-1700 Setpoints and Average Flow Rates vs. Time**

Temperature control for the Tyndall AFB demonstration was not optimal. The process was designed to be heated and cooled using glycol from an external process at the Thiokol facility. At Tyndall AFB, the auxiliary heater was designed to be used in a closed-loop configuration. In addition, the large glycol circulating pump in the loop caused significant heating of the process and reactors. Therefore the loop remained off during the demonstration. Regardless, reactor temperatures remained near the setpoint values as shown below in Figure 8.



**Figure 8. Reactor Temperature during Parallel Operation**

Nutrient preparation was performed manually. A hoist on the nutrient preparation skid platform hoisted 50 pound bags of the nutrient onto the platform. The contents of the bags were manually placed into the nutrient pre-mix tank (T-1202) where they were mixed with water and sulfuric acid (NOTE: The dry feeder was not utilized due to time constraints and setup requirements). Sulfuric acid was also manually added due to recurring pump seal problems (discussed later).

At Tyndall AFB, the use of cheese whey as a viable, alternative, reduced-cost nutrient source was also demonstrated. In the past, the laboratory used dried brewer's yeast or a commercial water soluble nutrient, BYF-100, as the primary sources of nutrient. Cheese whey is significantly cheaper than both dried brewer's yeast and BYF-100, but has been shown in the laboratory and field to perform identically. Cheese whey is also more water soluble than brewer's yeast and, when combined with the dried yeast, reduced plugging in the feed lines and control valves.

Sulfuric acid consumption was minimal. Sulfuric acid was used mostly for setting the pH of the nutrient below 2.0-- a method of "sterilizing" the nutrient solutions. This step in nutrient preparation was necessary to prevent fungal growth in the nutrient mix and feed vessels. In addition, the system was designed to use sulfuric acid for pH control in the reactors. Due to a problem with a pump seal, sulfuric acid was not utilized for pH control. The process by which the microbes destroy perchlorate is naturally acidic. Therefore, caustic was mainly used to keep reactor conditions neutral.

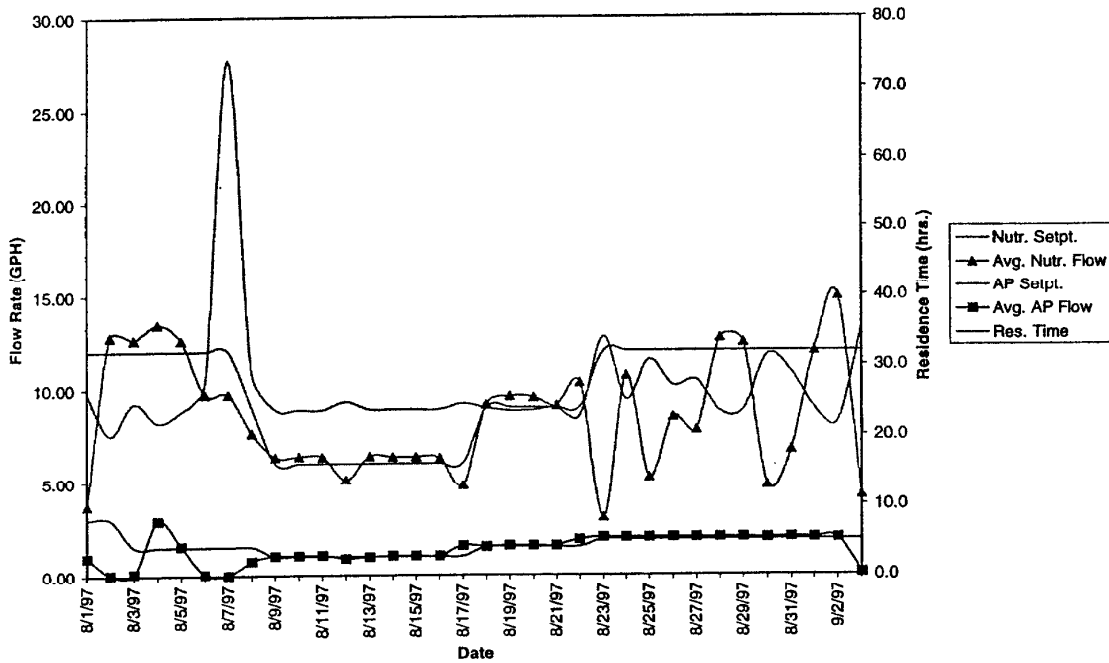
The reactor system contained an online perchlorate analyzer which used an ion specific electrode (ISE) identical to those used in the laboratory (discussed in Section 4.4.2). This analyzer is a dual channel unit designed to be used in both automatic and manual modes. The system PLC is configured to automatically trigger the sample loop pumps and, after a time delay, trigger the analyzer to sample the reactors and storage tanks in pairs, i.e., R-1400 and T-1500, then R-1700 and T-1503. When the PLC triggers the sample pumps, sample is circulated from the sample point to the clarifier. When the analyzer is triggered, a solenoid valve in the analyzer opens and draws sample from the sampling loop into an internal sample loop that sends the sample to the chamber. Perchlorate is measured in the chamber using the ISE. The measured sample is automatically flushed out of the loop to the clarifier. Originally, samples were pulled from the underside of the vessels, but the lines and pumps plugged frequently. Samples are now taken from dip tubes inserted into the top of each sampled tank. Analyzer readings were accurate (within 10%) for the brine even in the presence of large concentrations of chloride during parallel operation. During series operation, the readings were only reliable for the second reactor (R-1700) and the storage tanks due to perchlorate concentrations of the first reactor being outside the range of the standard used for low perchlorate concentration analysis. Currently there is an upgrade available for the existing system that enables the unit to perform sample dilutions and provide analysis at any concentration.

**5.1.5 System Functional Performance: Series operation using 10% AP.** The objective of this portion of the Tyndall AFB demonstration was to show that 10 -12% concentrated perchlorate solutions could be treated anaerobically using a two step process. A 10-12% AP solution was metered into the first reactor at a flow rate that would supply between 4000-6000 ppm perchlorate along with the necessary nutrient and dilution water. The first reactor, R-1400, would degrade part of the perchlorate, while the second reactor, R-1700, would degrade the remaining perchlorate.

During conversion from parallel to series operations, a problem with feed stream mixing occurred. Small heels of brine in the perchlorate feed tanks, pumps, and lines were not adequately flushed into the reactors. The high salt concentration brine reacted with the concentrated perchlorate precipitating KP in feed lines and control valves and dosed the system with ammonium. These two factors may have inadvertently raised ammonium and perchlorate concentrations fed to the reactors.

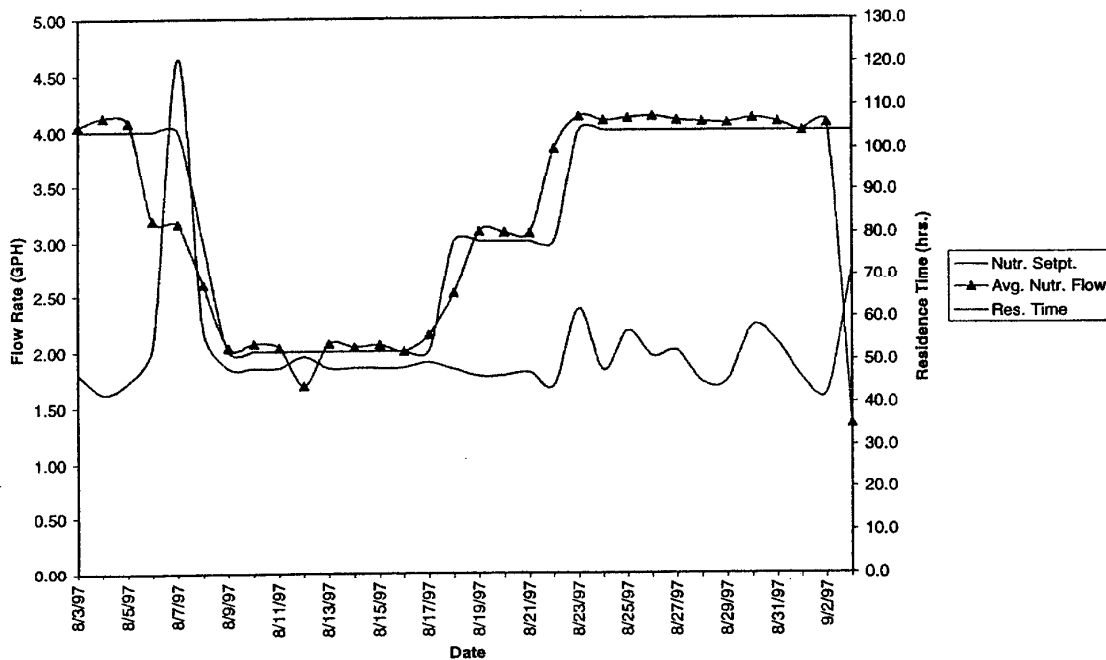
Overall, the system performed in series as designed. Nutrient plugging of the control valve with yeast solids was again a problem. The nutrient control valves were replaced with larger  $C_v$  values to achieve the functional performance needed. As shown in Figure 9, once excess KP was cleared from the feed lines and control valves, brine flow rates maintained near setpoints. Nutrient flow rates were maintained near setpoints until lines became plugged with yeast solids. Residence time remained constant during times when the control valves were not plugged. Again, water flow rates were assumed to be constant at the set point. Totalizer glitches sometimes caused inaccurate readings. However, when water flow was physically measured against the set point, flow was accurate to within measurement error (< 2%). The operational data is provided in Tables 5 and 6 (Appendix B).





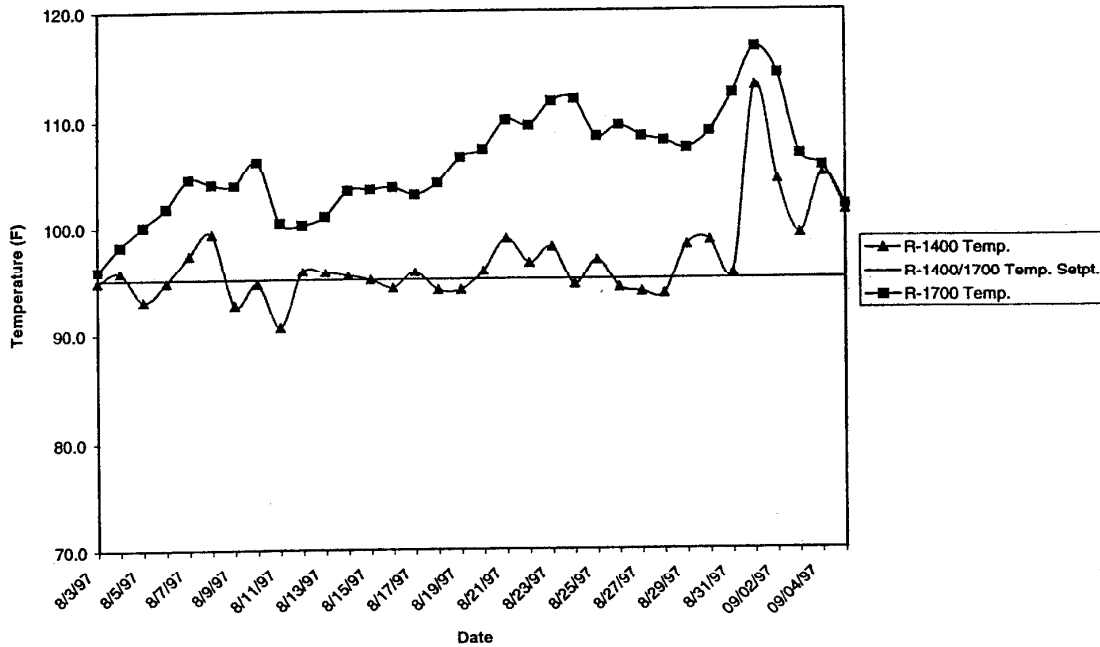
**Figure 9. R-1400 Set-points and Average Flow Rates vs. Time**

When configured in series, R-1700 is the second reactor. As shown in Figure 10, only a nutrient stream is fed to R-1700 to provide additional nutrients for the organisms reducing perchlorate. Flow rates were maintained within operational setpoints.



**Figure 10. R-1700 Set-points and Average Flow Rates vs. Time**

As in Section 5.1.4, temperature was not optimal during series operation. Figure 11 shows reactor temperatures and temperature setpoints. Temperature ran high and could not be controlled because, as stated in Section 5.1.4, the process was designed to be heated using an outside source of glycol at the Thiokol facility. As the temperature increased in the reactors, perchlorate reduction became erratic (See Figure 15). High temperatures were partially attributed to the high solids content and long solids retention time in the reactors. It has been previously demonstrated that when reactor temperatures exceed 42<sup>o</sup> C (108<sup>o</sup> F), perchlorate reduction is severely inhibited. Operating the clarifier at 100% recycle created a very high solids retention time and overwhelmed the reactors with suspended and dissolved solids. High recycle rates were acceptable for dilute effluent and nutrient feeds, but not for concentrated effluents. Therefore, the process was modified so that the recycle rate could be controlled to both reactors and to waste.



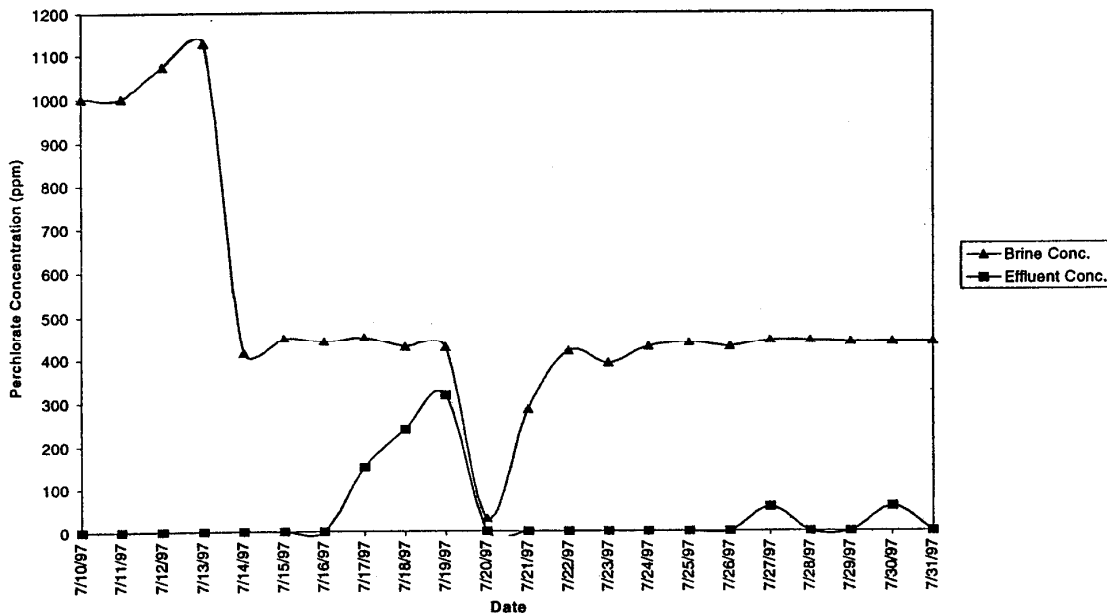
**Figure 11. Reactor Temperature during Series Operation**

Originally, the system was designed for solids recycle from the clarifier (S-1703) to R-1700. While at Tyndall, the solids recycle was modified to enable operators to manually switch solids recycling between R-1400 and R-1700. Solids recycle was intended for recycling HAP-1 and unused nutrient. The system may have been overwhelmed with solids (TSS and TDS) during series operation. Table 7 shows the increase in TSS and COD concentrations during the demonstration. During parallel operations in July, recycling of solids did not present a problem because of low nutrient concentration. During series operation, no means of solids wasting was available.

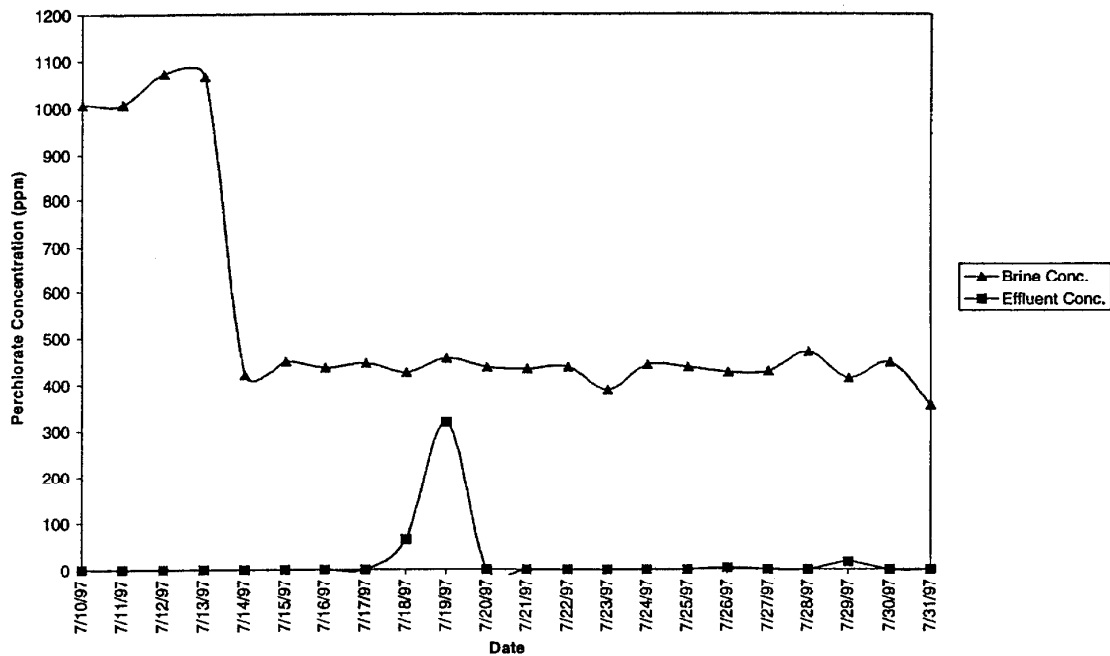
**Table 7. TSS and COD data for S-1703 (top/bottom)**

Date	S-1703 Top			S-1703 Bottom		
	TSS (g/l)	COD <sub>r</sub> (ppm)	COD <sub>t</sub> (ppm)	TSS (g/l)	COD <sub>r</sub> (ppm)	COD <sub>t</sub> (ppm)
7-17-97	1.01	2620	4700	30.20	4750	47325
7-24-97	1.04	5140	8925	22.97	6940	41025
7-31-97	1.01	2300	4550	37.37	4100	46875
8-7-97	1.90	8625	13490	7.4	9825	21800
8-11-97	14.40	14475	30740	44.0	14475	30740
8-13-97	8.8	12780	21175	40.1	14190	48650
8-14-97	8.6	12420	19850	35.2	12370	53900
8-18-97	6.6	11650	21550	31.5	12840	47725

**5.1.6 Biodegradation Performance in Parallel.** Figures 12 and 13 show brine and effluent concentrations for both R-1400 and R-1700 during parallel operations. The spike in perchlorate concentration in both reactors in Figures 12 and 13 was due to the microbial populations adjusting to high salt concentrations and a new nutrient source in the reactors. Figure 13 shows that the feed to R-1400 was temporarily interrupted for one day to aid in the adjustment. However, Figure 13 shows the feed was continued despite the upset. Both reactors recovered and performed well during the remainder of the parallel operation. Anion data for both reactors can be found in Tables 8 and 9 (Appendix B).

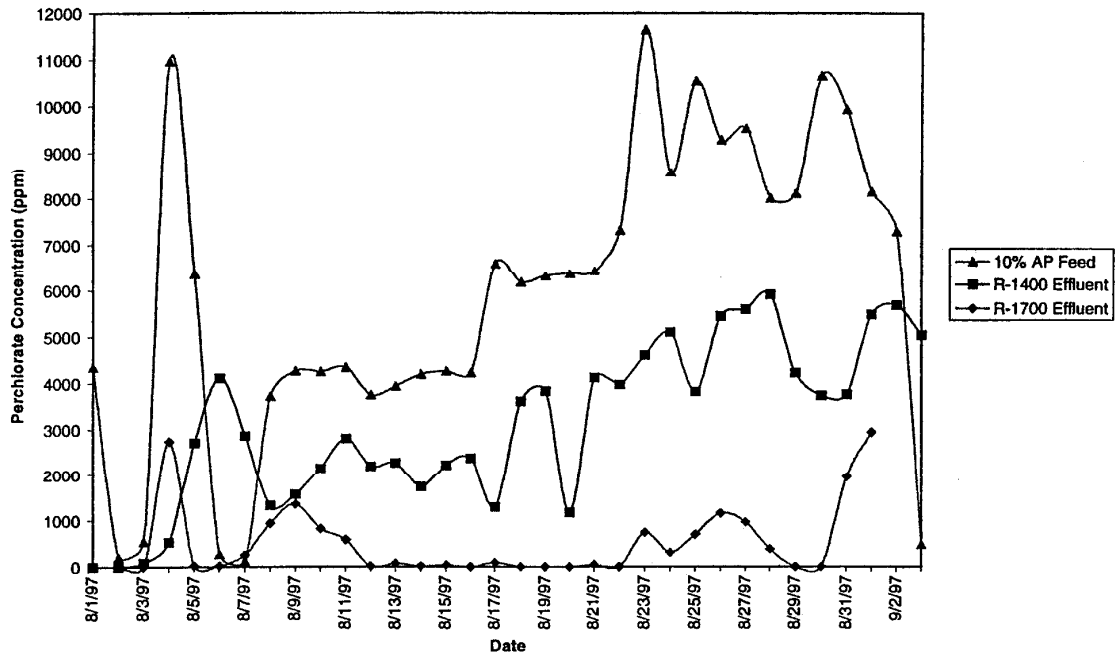


**Figure 12. R-1400 Brine and Effluent Concentrations during Parallel Operation**



**Figure 13. R-1700 Brine And Effluent Concentrations during Parallel Operation**

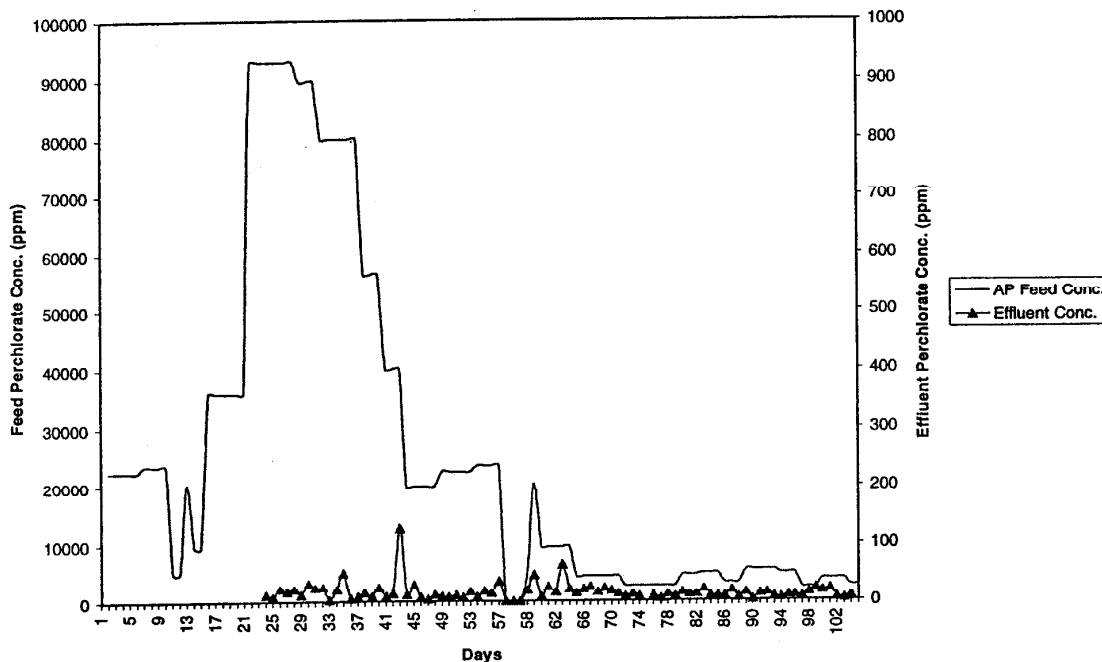
**5.1.7 Biodegradation Performance in Series.** During series operation, the optimal perchlorate feed concentrations were 4000, 6000, and 8000 ppm. Figure 14 shows that feed concentrations to R-1400 exceeded 10,000 ppm on several occasions. This was caused by flow control problems as a result of KP (potassium perchlorate) plugging and by the periodic washing out of solid KP into the reactor. These large perchlorate spikes in the feed cause comparable spikes in the reactors. The high perchlorate feed resulted in nutrient limited conditions. This, coupled with high temperatures and elevated TSS levels, caused perchlorate excursions in R-1700 after 8/22/97. However, during steady state performance with 4000 and 6000-ppm feeds, all the perchlorate was reduced in R-1700. Anion data for both reactors can be found in Tables 8 and 9 (Appendix B).



**Figure 14. Series Operation: Calculated Perchlorate Feed Conc. vs. R-1400/1700 Concentrations**

**5.2 Performance Data: Thiokol Validation**

**5.2.1 Summary.** Installation of the system was initiated 13 October, 1997. Installation was completed 30 October, 1997, and the initial software and functional tests were completed by 06 November, 1997. The system was operated and tested on water to train operators and repair items damaged during transit from Florida. Inoculation and startup from lyophilized vials brought from Tyndall AFB took place 08 December, 1997. Since startup, the system has performed nominally, and has processed a total of 9,155 gallons of effluent of varying concentrations from the perchlorate recovery production facility (see Figure 15). Thiokol will be issuing quarterly reports to the USAF in accordance with terms of the CRDA. All reports will be made available upon receipt.



**Figure 15. Thiokol AP Feed and Effluent Concentrations**

**5.2.2 Effluent Analysis.** Building M-705A is a pretreatment stage for discharge to the on-plant sewage treatment facility and is largely concerned with perchlorate concentration and TDS levels. Analyses are performed to identify nitrate, nitrite, and sulfate as required. These results may be found in Table 10, Appendix B.

**5.2.3 System Functional Performance.** As mentioned earlier, the system has performed nominally during the first quarter. Software modification and control improvements are being made to enhance system performance as necessary. Equipment modifications have been made as required. Centrifugal pumps on the AP feed skid and pumps used for pH control have been replaced with magnetic drive pumps. Process requirements have also been changed as necessary. Sodium hydroxide is used to pH the nutrient to pH>10 as a means of sterilizing the nutrient which minimizes acid usage. Also, to improve operability, sulfuric acid was replaced with citric acid for pH control.

**5.2.4 Biodegradation Performance.** Typical effluent from Thiokol's perchlorate production facility (ion exchange and potassium precipitation units) are very high in TDS (150-300 g/l) and relatively low in perchlorate (~5000 mg/l). However, Figure 15 shows during the first two months of operation, the perchlorate in the brine effluent was relatively high (20-90,000 mg/l). In addition, the effluent contained approximately 10,000 mg/l nitrite and 5000 mg/l nitrate. The effluent is produced in batch processes: therefore, a different batch was fed to the reactors every

2-8 days. Because of the high TDS, the effluent is diluted to 5-10% of its original concentration as it is fed to the reactors. Again, Figure 15 shows the actual perchlorate concentration in the undiluted feed and in the reactor effluent. Typical perchlorate concentrations in the reactors are less than 20 ppm using the ion-specific probe method. These results almost always translate to near non-detect levels using ion chromatography. Nitrate levels were nearly completely reduced, however, little nitrite reduction was observed (Table 10, Appendix B). During the first quarter, there have been no major upsets in operations and reinoculation has not been necessary.

### 5.3 Data Assessment

**5.3.1 Tyndall Demonstration.** Several design modifications were identified as a result of the validation testing:

- Caustic and perchlorate pumps were modified from single-seal to double-seal heads
- A circulation loop was installed in the nutrient delivery system to minimize plugging problems
- Nutrient valve port sizes were increased
- Dip-tubes were installed in the reactors to draw samples for the online perchlorate analyzer
- Multiple two-way valves were configured on the clarifier recycle to permit control of solids recycle to both reactors and to waste independently
- A vent line was installed on the clarifier
- Start-up screens were developed to determine and change set points based on specific, operator input parameters
- In the laboratory, caustic was determined to be a more viable means of nutrient sterilization vs. acid

**5.3.2 Thiokol Demonstration.** Several design modifications and new startup procedures have resulted from industrial operations at Thiokol:

- The system was started up entirely from lyophilized cultures produced at Tyndall AFB
- Magnetic drive pumps have replaced the double-seal head, centrifugal pumps for AP feed and pH control
- Caustic was used for nutrient sterilization instead of acid
- Citric acid replaced sulfuric acid for pH control—safer and more user-friendly

### 5.4 Technology Comparison

**5.4.1 Other Technologies.** There is no other perchlorate destruction technology (other than OB/OD) at this level of maturity. The recent problems with perchlorate in drinking water in California and Nevada have prompted both government and private studies in this area. The most promising competitive destruction technologies are catalytic and electrochemical processes.



While both have been demonstrated to work in the laboratory to some degree, there are many technical hurdles to overcome before they can be seriously considered for full-scale implementation. The major concerns are long-term performance, destruction efficiency, and cost.

Most waste streams are complex solutions of ions that compete with the desired reactions and foul the selective catalytic surfaces. Biodegradation has proven to be much more tolerant of the complex ionic solutions. Perchlorate biodegradation has demonstrated nearly complete destruction - to below detection limits. So far catalytic processes have not been able to demonstrate this high destruction efficiency. While biodegradation of perchlorate requires nutrient, catalytic processes require effluent pretreatment, electricity, expensive precious metal catalysts, and additional reactants such as reducing agents (hydrogen gas).

**5.4.2 Summary of Performance Data.** Biological processes are inherently stable, reliable, simple, cost-effective processes. This process is no different with respect to those characteristics. Costs may be greater than a simple BOD reduction process because nutrient has to be added to effect perchlorate reduction. The microbes used in this process, while enriched and specialized, are naturally occurring and very stable in the environment required to reduce perchlorate. Bench-scale bioreactors have been operated for several months at a time with no loss in performance and without the need for re-inoculation. As long as perchlorate-reducing conditions are maintained, the perchlorate-reducing consortia of microbes remains dominant.

The prototype demonstrated at Thiokol is much more complex in design than is necessary for typical wastewater treatment. Decentralized process control, gravity, positive displacement pump feeding, and inexpensive materials of construction can greatly simplify operation and reduce cost. Additional discussion is provided in Section 9, Lessons Learned.

In general, biological processes are slow. High perchlorate reduction rates ( $> 0.5$  g/l) have been demonstrated at very short residence times (6-8 hours). However, laboratory optimization studies have shown that a 24-hour residence time is near optimal to achieve relatively high rates and still reduce perchlorate from high concentrations (4000-6000 ppm) to near the detection limit in a single-stage reactor system. Therefore, 24 hours should be used as the design basis for future implementations. Actual residence times could vary from less than 12 hours to 48 hours depending on the process effluent, nutrient type, nutrient concentration, and other process conditions.

This technology is adaptable to fixed-film biological processes. Preliminary laboratory studies have shown that this consortium of organisms does form a film that continues to reduce perchlorate. Fixed-film processes, including fluidized bed, have the potential to efficiently treat effluents with low perchlorate concentrations ( $< 100$ -500 ppm). They utilize nutrient more efficiently and may demonstrate shorter hydraulic residence times. For perchlorate concentrations greater than 100-500 ppm, CSTR system is very efficient and is the preferred approach.

## 6. Cost Assessment

### **6.1 Cost Performance**

Costs associated with startup, including labor, planning & contracting, site preparation, construction, permitting or other regulatory requirements, and capital equipment will be recorded as data for interpretation and evaluation. The numbers in the cost table are actual or derived costs based on the Thiokol production-scale facility. The capital cost is an estimate based on equipment costs for an operational design vs. an R&D pilot-scale design.

### **6.2 Operations and Maintenance Costs**

Costs associated with operations and maintenance are based on actual, derived, or estimated costs for operating the Thiokol facility at operating conditions anticipated for their site. Assume the effluent treated is a 5000 ppm perchlorate, 100% brine solution (paragraph 3.2). Effluent treating rates anticipated are 450 gallon per day of 100% brine (continuously diluted to 20%). This operating condition does not result in optimal throughput or cost for this process. The operating cost figure shows that fixed costs (electricity, maintenance, and labor) are at minimum for 2000-4000 ppm perchlorate effluents. This is because maximum throughput can be achieved near 4000 ppm effluents. Nutrient cost is approximately linear with perchlorate reduced. As a result, up to 4000 ppm perchlorate effluents can be treated for less than \$0.10 per gallon.

### **6.3 Demobilization**

Demobilization costs are not applicable for this demonstration because the system will remain in place and operated by Thiokol under the Air Force CRDA. The CRDA provides for government furnished equipment to be transitioned to an existing Air Force production or demilitarization program. Disposal or decommissioning costs in the table are estimates based on disassembly and shipment of the Thiokol prototype. The equipment will not require special handling other than decontamination. No salvage value for equipment is credited.

### **6.4 Life-Cycle Costs**

A life cycle cost evaluation will be based on actual long-term data from the operation of the Thiokol prototype. Quarterly reports will be generated during the term of the CRDA and submitted to the Air Force Activity. The information provided in the quarterly reports will include, as a minimum, the following data: material and energy balances, labor, material and maintenance costs, performance assessments (perchlorate destruction efficiency and estimated treatment cost in \$/gal.), changes in operating parameters (feed and product composition, residence time, temperature, nutrient, etc.), system modifications, and planned or proposed system or operational changes (see Table 11).

**Table 11. Cost Data**  
**Cost Data Table, in \$1000**

Cost Category	Project Phase			
	Start-up	Annual O & M	Demobilization	Life-Cycle
Labor	20	25	-	-
Training	5	2	-	-
Site-Specific Treatability Studies	100	-	-	-
Process Tailoring/Engineering	125	-	-	-
Site Preparation	100	-	-	-
Analysis/monitoring	5	5	-	-
Contracting	10	-	-	-
Permits/Regulatory Requirements	-	-	-	-
Capital Equipment	500-800	-	-	-
Modifications	-	-	-	100
Scheduled Maintenance	-	25	-	-
Consumables	-		-	-
- Nutrient		15		
- Acid, Caustic, Chemicals		2		
- Electricity		15		
Ancillary Equipment	-	-	-	-
Effluent Treatment	-	10	-	-
Equipment Decontamination	-	-	5	-
Equipment Removal	-	-	15	-
Site Restoration	-	-	-	None
Future Liability	-	-	-	None

## **7. Regulatory Issues**

The demonstration of this process at Thiokol required no new permits. Activities associated with modification of the existing industrial wastewater treatment plant were within the scope of existing permits. The shipping of "samples" for treatability studies to Tyndall AFB, required permission from the Florida Department of Environmental Protection coordinated through local and base officials.

## **8. Technology Implementation**

### **8.1 DoD Need**

This technology could be applied at other DoD and propulsion manufacturer facilities that generate similar waste streams. This process was developed to treat perchlorate in the presence of inorganic contaminants. Initial studies indicated that perchlorate can also be reduced in the presence of organic and other energetic materials, but processes for these effluents have not been demonstrated. Recent discoveries indicate that ground water remediation may also be accomplished using the same organism.

### **8.2 Transition**

**8.2.1 Scale-up Issues.** Prototype operation at Thiokol, using the 1600-gallon tank reactor, demonstrated performance equivalent to, or better than, the lab-scale (14-liter) reactor systems. This represents approximately a 500-fold scale-up (1560-gallons/12-liters hydraulic volume) with no apparent problems. Biomass flocking was more efficient in the prototype system. This resulted in improved clarification, increased biosolids in the reactor, and improved performance overall. Much larger processes could be employed using the CSTR approach. Stirred-tank-reactors with 20-30,000 gallon capacities can be shop fabricated. Systems with capacities greater than 100,000 gallons per day could be easily implemented by configuring multiple CSTR's in parallel operation. If BOD reduction of the effluent is required, packaged anaerobic and aerobic process are available that could easily handle the demand.

## 9. Lessons Learned

Lessons learned from previous start-ups were successfully employed for both the Tyndall AFB and Thiokol demonstration. It is important to remove nearly all of the oxygen and minimize dilution of the inoculum in the reactor. This, coupled with excess nutrient (~10 g/l) and low initial perchlorate concentrations (500-1000 ppm), results in effective and rapid reactor inoculation. Once the reactor is inoculated, the need to regulate residual oxygen depends on the concentration of other oxygenates in the feed.

Complexity and degree of control and flexibility were very high for this process compared to a typical biological process. This was by design and partially based on the fact that a research prototype was adapted for this demonstration. Future application of this process could be greatly simplified. Local control with computer monitoring could be employed versus PLC type system. Positive displacement pumps and gravity feed design would further simplify operation.

A better choice of construction materials may result in a more reliable and less expensive implementation. Many of the process vessels are exposed to very caustic or corrosive environments. Even the reactor, though is maintained at a pH near 7.0, is subject to corrosion due to biologically generated organic acids and the addition of acid and caustic for pH control. Many of the original vessels were fabricated from stainless steel (304) and showed signs of significant corrosion in places. However, components and vessels fabricated out of fiberglass reinforced plastic (FRP) and high-density polyethylene (HDPE) performed very well. The drawback to plastic piping for a transportable system is that some breakage occurs during transportation and exposure to solar radiation will cause additional deterioration.

This process was specifically designed to permit the addition of two separate effluent streams. This was to minimize the possibility of causing a chemical reaction that could generate compounds detrimental to the biological process. Also if the potassium containing brine were mixed with ammonium perchlorate, the resulting potassium perchlorate precipitation would cause plugging and potential performance problems. We experienced such a problem during the Tyndall AFB demonstration when two different effluents were inadvertently mixed. Future implementations of this technology should maintain the two feed scenarios if the possibility of mixing reactive feeds exist.

A concentrated nutrient feed was provided as a separate feed to the reactor. It is important to maintain an independent nutrient feed for start-up and upset recovery. The ability to utilize concentrated nutrients results in smaller vessels and lower cost. Caustic sterilization of the nutrient, demonstrated during the Thiokol validation, also reduces chemical costs. Since acid-generating reactions occur in the reactor, the caustic nutrient feed can be manipulated in a way to nearly eliminate acid addition for pH control. Other nutrient sterilization techniques (steam), or direct feeding of dry nutrients, may further reduce operating costs.

Solids recycle to the reactor appears to greatly improve performance and nutrient utilization efficiency. In very dilute effluents, where the minimum nutrient addition is required, it may be possible to employ 100% solids recycle. However, for higher perchlorate and nutrient concentrations, the ability to waste a portion of the organic solids is absolutely necessary. There

appears to be some threshold of solids retention time and concentration that is detrimental to the perchlorate reducing consortium microbes. Additional efforts are required to accurately characterize this threshold.

This prototype system was designed with two reactors, two effluent storage tanks, and a clarifier. This multiple vessel approach is very beneficial. More, small reactors verses one large reactor makes start-up inoculation easier. More reactors provide a margin of safety in case of an upset. A reactor can be quickly re-inoculated from the other reactor or storage tanks. Future applications of this technology should always be designed with a minimum of two parallel process trains.

## 10. References

1. Attaway, H., and Smith, M. 1993. "Reduction of Perchlorate by an Anaerobic Enrichment Culture." *Journal of Industrial Microbiology*. 12: 408-412
2. Wallace, W., Ward, T., Breen, A., Attaway, H. 1996 "Identification of an Anaerobic Bacterium Which Reduces Perchlorate and Chlorate as *Wolinella succinogenes*." *Journal of Industrial Microbiology*. 16: 68-72
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5. HACH Water Analysis Handbook, 1989, HACH Company, Loveland, Colorado, 468-477

**Appendix A**  
**Points of Contact**

PROJECT MANAGER	PRINCIPAL INVESTIGATOR
Mr. James A. Hurley	Edward N. Coppola
Air Force Research Laboratory	Applied Research Associates, Inc
AFRL/MLQ	215 Harrison Ave.
139 Barnes Dr., Suite 2	Panama City, FL 32401
Tyndall AFB, FL 32403-5323	
Phone: (850) 283-6243	Phone: (850) 914-3188
DSN# 523-6243	
FAX: (850) 283-6064	FAX: (850) 914-3189
E-mail: jim_hurley@ccmail.aleq.tyndall.af.mil	E-mail: ecoppola@ara.com

ANALYTICAL COORDINATOR	TYNDALL SITE SUPERVISOR
Glen McDonald	Jeffrey Rine
Applied Research Associates, Inc.	Applied Research Associates, Inc.
215 Harrison Ave.	P.O. Box 40128
Panama City, FL 32401	Bldg. 1142 Mississippi Road
	Tyndall AFB, FL 32403
Phone: (850) 914-3188	Phone: (850) 283-6669
	DSN# 523-6669
FAX: (850) 914-3189	FAX: (850) 286-6979
E-mail: gmcdonald@ara.com	E-mail: jrine@ara.com

THIOKOL TECHNICAL POC	CASE ENGR. PROGRAM MANAGER
Greg Startzell	John McKelvey
Thiokol Corporation	Case Engineering
P.O. Box 689	P.O. Box 6039
Mail Stop 301-A	5301 Great Oak Drive
Thiokol, Utah 84302	Lakeland, FL 33801
Phone: (801) 863-8254	Phone: (941) 687-7580
FAX: (801) 863-4037	FAX: (941) 687-8554
E-mail: startgw@thiokol.com	E-mail: case@caseinc.com

## **Appendix B**

### **Data Archiving and Demonstration Plan**

#### **B.1 Final Report and Demonstration Plan**

This "*Final Report: Operational Implementation of Ammonium Perchlorate Biodegradation,*" and the "*Demonstration Plan: Operational Implementation of Ammonium Perchlorate Biodegradation.*" have been submitted to the Defense Technical Information Center (DTIC) for publication. These documents have unlimited distribution and will be available to the public through the National Technical Information Service (NTIS).

#### **B.2 Program Management File**

All research efforts conducted at the Air Force Research Laboratory, Materials and Manufacturing Directorate have maintained a Program Management File that contains all the relevant documents pertaining to the execution of the program. This file is maintained in accordance with Air Force Instruction (AFI) 61-206, which documents the requirements for developing, managing, and retiring R & D project files. Upon final closeout of this effort, the program management file will be prepared for retirement into the National Archives and retained at Tyndall AFB for approximately three years.

#### **B.3 Demonstration Plan Operational Data**

The operational data collected in accordance with the Demonstration Plan is summarized in the following tables. These tables are referenced in the relevant sections of the Final Report. Data collected prior to, and independent of, the Demonstration Plan that is germane to the use of *Wollinella succinogenes* HAP1 in degrading perchlorate will be retained as required in the Program Management File. This data will become a permanent part of the archived file as described in B.2.



Table 3. Summary of Operational Data: R-1400 on Thiokol Brine Effluent

Date	nutr. setpt.	Nutrient			Perchlorate			water setpt.	Water avg. flow GPH	Total flow GPH	Calculated res. time hours	Calculated nut. ratio g nut./g AP	Temp. °F	Comments
		avg. flow GPH	avg. conc. g/l	perchl. setpt.	avg. flow GPH	conc. in ppm	conc. out ppm							
07/10/97	3.30	3.30	4.95	2.80	2.80	1001	0	23.9	23.9	30.00	24.0	4.95	91.2	AP feed = 10,725; DBY = 45 g/l
07/11/97	3.30	3.30	4.95	2.80	2.80	1001	0	23.9	23.9	30.00	24.0	4.95		
07/12/97	2.67	2.67	4.01	3.00	3.00	1073	0	24.33	24.3	30.00	24.0	3.73	90.2	
07/13/97	2.67	4.26	6.29	3.00	3.20	1127	0	24.33	23.0	30.46	23.6	5.59	90.0	
07/14/97	4.00	4.23	4.21	3.00	2.93	414	0	23	23.0	30.16	23.9	10.15	89.0	Initiate brine & 75% whey (30 g/l)
07/15/97	4.00	4.31	4.24	3.00	3.21	449	0	23	23.0	30.52	23.6	9.44	89.3	(4266 ppm ClO4-; 271 g/l TDS)
07/16/97	4.00	4.17	4.13	3.00	3.13	441	0	23	23.0	30.30	23.8	9.37	89.1	
07/17/97	4.00	4.27	4.20	3.00	3.21	449	149	23	23.0	30.48	23.6	9.35	89.1	
07/18/97	4.00	4.00	4.00	3.00	3.00	427	238	23	23.0	30.00	24.0	9.38	87.9	
07/19/97	5.00	5.28	5.04	3.00	3.14	426	315	23	23.0	31.42	22.9	11.83	86.4	Nitrogen sparge turned off
07/20/97	6.00	6.08	6.23	3.60	0.21	31	0	23	23.0	29.29	24.6	203.60	86.1	Glycol circulating pump turned on
07/21/97	6.00	6.68	6.30	3.00	2.12	284	0	23	23.0	31.80	22.6	22.16	91.4	
07/22/97	6.00	6.00	5.60	3.00	3.14	417	0	23	23.0	32.14	22.4	13.44	91.8	
07/23/97	6.00	4.58	4.53	3.00	2.77	389	0	23	23.0	30.35	23.7	11.63	90.9	
07/24/97	4.00	4.00	4.00	3.00	3.00	427	0	23	23.0	30.00	24.0	9.38	90.7	
07/25/97	4.00	4.14	4.11	3.00	3.10	437	0	23	23.0	30.24	23.8	9.39	94.2	
07/26/97	4.00	4.00	4.00	3.00	3.00	427	0	23	23.0	30.00	24.0	9.38	91.2	
07/27/97	4.00	4.22	4.17	3.00	3.16	444	56	23	23.0	30.38	23.7	9.39	89.6	Initiate 50% DBY (30 g/l)
07/28/97	4.00	4.18	4.13	3.00	3.15	443	0	23	23.0	30.33	23.7	9.33	91.2	
07/29/97	4.00	4.27	4.24	3.00	2.95	440	0	23	23.0	30.22	23.8	9.63	94.7	Initiate 100% DBY (30 g/l)
07/30/97	4.00	4.20	4.16	3.00	3.09	440	58	23	23.0	30.29	23.8	9.45	94.7	
07/31/97	4.00	4.27	4.21	3.00	3.19	440	0	23	23.0	30.46	23.6	9.56	94.2	

Table 4. Summary of Operational Data: R-1700 on Thiokol Brine Effluent

Date	Nutrient				Perchlorate			Water setpt.	Total GPH	Calculated res. time hours	Calculated nut. ratio g nut./g AP	Temp. °F	Comments	
	nutr. setpt.	avg. flow GPH	avg. conc. g/l	perchl. setpt.	avg. flow GPH	conc. in ppm	conc. out ppm							
07/10/97	7.20	7.2	4.98	6.10	6.10	1007	0	51.7	51.7	65.0	24.0	4.95	89.5	AP feed = 10,725; DBY = 45 g/l
07/11/97	7.20	7.2	4.98	6.10	6.10	1007	0	51.7	51.7	65.0	24.0	4.95		
07/12/97	5.78	5.78	4.00	6.50	6.50	1073	0	52.7	52.7	65.0	24.0	3.73	89.9	
07/13/97	5.78	8.98	5.90	6.50	6.81	1066	0	52.7	52.7	68.5	22.8	5.53	89.5	
07/14/97	8.70	8.90	4.10	6.50	6.42	421	0	49.8	49.3	65.1	24.0	9.75	89.3	Initiate brine & 75% whey (30 g/l)
07/15/97	8.70	9.12	4.15	6.50	6.94	450	0	49.8	49.5	65.9	23.7	9.24	90	(4266 ppm ClO <sub>4</sub> <sup>-</sup> ; 271 g/l TDS)
07/16/97	8.70	8.22	3.82	6.50	6.60	436	0	49.8	49.8	64.6	24.1	8.76	90	
07/17/97	8.70	9.01	4.12	6.50	6.87	446	0	49.8	49.8	65.7	23.8	9.22	90.1	
07/18/97	8.70	8.70	4.02	6.50	6.50	427	64	49.8	49.8	65.0	24.0	9.41	89.9	
07/19/97	8.70	5.83	2.81	6.50	6.67	457	319	49.8	49.8	62.3	25.0	6.15	88.9	Nitrogen sparge turned off
07/20/97	8.70	9.09	4.16	6.50	6.73	438	0	49.8	49.8	65.6	23.8	9.50	88.3	Glycol circulating pump turned on
07/21/97	8.70	9.26	4.23	6.50	6.68	433	0	49.8	49.8	65.7	23.7	9.75	91.2	
07/22/97	8.70	8.70	4.00	6.50	6.68	437	0	49.8	49.8	65.2	23.9	9.16	92	
07/23/97	8.70	9.32	4.30	6.50	5.92	388	0	49.8	49.8	65.0	24.0	11.07	91.6	
07/24/97	8.70	8.92	4.08	6.50	6.80	443	0	49.8	49.8	65.5	23.8	9.22	90.8	
07/25/97	8.70	8.79	4.04	6.50	6.69	437	0	49.8	49.8	65.3	23.9	9.24	93.5	
07/26/97	8.70	8.70	4.02	6.50	6.50	427	4	49.8	49.8	65.0	24.0	9.41	91.6	
07/27/97	8.70	8.94	4.11	6.50	6.56	429	0	49.8	49.8	65.3	23.9	9.58	91.2	Initiate 50% DBY (30 g/l)
07/28/97	8.70	4.67	2.29	6.50	6.74	470	0	49.8	49.8	61.2	25.5	4.87	90.9	
07/29/97	8.70	8.84	4.08	6.50	6.3	414	17	49.8	49.8	64.9	24.0	9.87	92.9	Initiate 100% DBY (30 g/l)
07/30/97	8.70	6.86	3.25	6.50	6.65	448	0	49.8	49.8	63.3	24.6	7.25	96.3	
07/31/97	8.70	8.95	4.19	6.50	5.34	355	0	49.8	49.8	64.1	24.3	11.79	93.1	

Table 4. Summary of Operational Data: R-1700 on Thiokol Brine Effluent

Date	Nutrient				Perchlorate			Water setpt.	Total GPH	Calculated res. time hours	Calculated nut. ratio g nut./g AP	Temp. °F	Comments	
	nutr. setpt.	avg. flow GPH	avg. conc. g/l	perchl. setpt.	avg. flow GPH	conc. in ppm	conc. out ppm							
07/10/97	7.20	7.2	4.98	6.10	6.10	1007	0	51.7	51.7	65.0	24.0	4.95	89.5	AP feed = 10,725; DBY = 45 g/l
07/11/97	7.20	7.2	4.98	6.10	6.10	1007	0	51.7	51.7	65.0	24.0	4.95		
07/12/97	5.78	5.78	4.00	6.50	6.50	1073	0	52.7	52.7	65.0	24.0	3.73	89.9	
07/13/97	5.78	8.98	5.90	6.50	6.81	1066	0	52.7	52.7	68.5	22.8	5.53	89.5	
07/14/97	8.70	8.90	4.10	6.50	6.42	421	0	49.8	49.3	65.1	24.0	9.75	89.3	Initiate brine & 75% whey (30 g/l)
07/15/97	8.70	9.12	4.15	6.50	6.94	450	0	49.8	49.5	65.9	23.7	9.24	90	(4266 ppm ClO <sub>4</sub> <sup>-</sup> ; 271 g/l TDS)
07/16/97	8.70	8.22	3.82	6.50	6.60	436	0	49.8	49.8	64.6	24.1	8.76	90	
07/17/97	8.70	9.01	4.12	6.50	6.87	446	0	49.8	49.8	65.7	23.8	9.22	90.1	
07/18/97	8.70	8.70	4.02	6.50	6.50	427	64	49.8	49.8	65.0	24.0	9.41	89.9	
07/19/97	8.70	5.83	2.81	6.50	6.67	457	319	49.8	49.8	62.3	25.0	6.15	88.9	Nitrogen sparge turned off
07/20/97	8.70	9.09	4.16	6.50	6.73	438	0	49.8	49.8	65.6	23.8	9.50	88.3	Glycol circulating pump turned on
07/21/97	8.70	9.26	4.23	6.50	6.68	433	0	49.8	49.8	65.7	23.7	9.75	91.2	
07/22/97	8.70	8.70	4.00	6.50	6.68	437	0	49.8	49.8	65.2	23.9	9.16	92	
07/23/97	8.70	9.32	4.30	6.50	5.92	388	0	49.8	49.8	65.0	24.0	11.07	91.6	
07/24/97	8.70	8.92	4.08	6.50	6.80	443	0	49.8	49.8	65.5	23.8	9.22	90.8	
07/25/97	8.70	8.79	4.04	6.50	6.69	437	0	49.8	49.8	65.3	23.9	9.24	93.5	
07/26/97	8.70	8.70	4.02	6.50	6.50	427	4	49.8	49.8	65.0	24.0	9.41	91.6	
07/27/97	8.70	8.94	4.11	6.50	6.56	429	0	49.8	49.8	65.3	23.9	9.58	91.2	Initiate 50% DBY (30 g/l)
07/28/97	8.70	4.67	2.29	6.50	6.74	470	0	49.8	49.8	61.2	25.5	4.87	90.9	
07/29/97	8.70	8.84	4.08	6.50	6.3	414	17	49.8	49.8	64.9	24.0	9.87	92.9	Initiate 100% DBY (30 g/l)
07/30/97	8.70	6.86	3.25	6.50	6.65	448	0	49.8	49.8	63.3	24.6	7.25	96.3	
07/31/97	8.70	8.95	4.19	6.50	5.34	355	0	49.8	49.8	64.1	24.3	11.79	93.1	

**Table 5. Summary of Operational Data: R-1400 on Thiokol Concentrated AP Effluent**

Date	Nutrient				Perchlorate			Water	Total	Calculated	Calculated	Temp.	Comments	
	nutr. setpt.	avg. flow GPH	avg. conc. g/l	AP setpt.	avg. flow GPH	conc. in ppm	conc. out ppm	water setpt.	avg. flow GPH	flow GPH	res. time hours	nut. ratio g nut./g AP		°F
08/01/97	12.00	3.74	6.07	3.00	0.98	4360	0	23.00	23.0	27.72	26.0	1.39	95.0	AP feed = 123,333; DBY = 45 g/l
08/02/97	12.00	12.78	16.05	3.00	0.06	206	0	23.00	23.0	35.84	20.1	77.72	97.0	
08/03/97	12.00	12.63	19.42	1.50	0.13	548	84	16.50	16.5	29.26	24.6	35.45	94.8	Pechlorate feed plugged
08/04/97	12.00	13.45	18.41	1.50	2.92	10956	535	16.50	16.5	32.87	21.9	1.68	95.7	
08/05/97	12.00	12.61	18.49	1.50	1.58	6349	2711	16.50	16.5	30.69	23.5	2.91	93.0	
08/06/97	12.00	9.70	16.62	1.50	0.06	282	4114	16.50	16.5	26.26	27.4	58.99	94.7	
08/07/97	12.00	9.71	44.91	1.50	0.01	127	2856	16.50	0.0	9.73	74.0	354.28	97.4	Initiate 60 g/l DBY
08/08/97	9.00	7.64	18.42	1.50	0.75	3716	1328	19.50	16.5	24.89	28.9	4.96	99.4	Recycle from T-1503
08/09/97	6.00	6.33	12.50	1.00	1.05	4263	1570	23.00	23.0	30.38	23.7	2.93	92.6	
08/10/97	6.00	6.38	12.58	1.00	1.05	4256	2158	23.00	23.0	30.43	23.7	2.96	94.6	
08/11/97	6.00	6.36	12.54	1.00	1.07	4337	2802	23.00	23.0	30.43	23.7	2.89	90.6	Recycle from T-1500
08/12/97	6.00	5.12	10.59	1.00	0.88	3743	2193	23.00	23.0	29.00	24.8	2.83	95.8	Sparge N2 from bottle
08/13/97	6.00	6.38	12.61	1.00	0.97	3942	2272	23.00	23.0	30.35	23.7	3.20	95.7	Recycle from T-1500/1503
08/14/97	6.00	6.33	12.51	1.00	1.03	4184	1730	23.00	23.0	30.36	23.7	2.99	95.4	
08/15/97	6.00	6.34	12.52	1.00	1.05	4261	2214	23.00	23.0	30.39	23.7	2.94	95.0	Add inoculum
08/16/97	6.00	6.28	12.43	1.00	1.04	4230	2371	23.00	23.0	30.32	23.7	2.94	94.2	
08/17/97	6.00	4.81	9.82	1.00	1.57	6591	1295	23.00	23.0	29.38	24.5	1.49	95.7	
08/18/97	9.00	9.13	18.18	1.50	1.51	6179	3602	19.50	19.5	30.14	23.9	2.94	94.0	
08/19/97	9.00	9.59	18.77	1.50	1.57	6315	3836	19.50	19.5	30.66	23.5	2.97	94.0	Recycle from T-1500

**Table 5. Summary of Operational Data: R-1400 on Thiokol Concentrated AP Effluent**

Date	Nutrient				Perchlorate			Water	Total	Calculated	Calculated	Temp.	Comments	
	nutr. setpt.	avg. flow GPH	avg. conc. g/l	AP setpt.	avg. flow GPH	conc. in ppm	conc. out ppm	water setpt.	avg. flow GPH	flow GPH	res. time hours	nut. ratio g nut./g AP		°F
08/20/97	9.00	9.52	18.67	1.50	1.58	6368	1182	19.50	19.5	30.60	23.5	2.93	95.8	Temperature spike to 105.5 F
08/21/97	9.00	9.07	18.06	1.50	1.57	6424	4123	19.50	19.5	30.14	23.9	2.81	98.9	Recycle from T-1500
08/22/97	9.00	10.30	19.51	1.50	1.88	7319	3973	19.50	19.5	31.68	22.7	2.67	96.6	Initiate air sparge at 10 scfh
08/23/97	12.00	3.07	8.70	1.90	2.00	11652	4611	16.10	16.1	21.17	34.0	0.75	98.1	Recycle from T-1500; low pH
08/24/97	12.00	10.67	22.25	1.90	2.00	8574	5098	16.10	16.1	28.77	25.0	2.60	94.5	
08/25/97	12.00	5.29	13.57	1.90	2.00	10546	3817	16.10	16.1	23.39	30.8	1.29	96.9	
08/26/97	12.00	8.49	19.16	1.90	2.00	9277	5444	16.10	16.1	26.59	27.1	2.07	94.2	
08/27/97	12.00	7.79	18.05	1.90	2.00	9527	5596	16.10	16.1	25.89	27.8	1.89	93.8	Nutrient valve plugged
08/28/97	12.00	12.69	24.73	1.90	2.00	8011	5915	16.10	16.1	30.79	23.4	3.09	93.6	Recycle from T-1500/1503
08/29/97	12.00	12.43	24.42	1.90	2.01	8117	4227	16.10	16.1	30.54	23.6	3.01	98.3	
08/30/97	12.00	4.82	12.63	1.90	1.98	10664	3733	16.10	16.1	22.90	31.4	1.18	98.7	Initiate 75% cheese whey
08/31/97	12.00	6.72	16.24	1.90	2.00	9938	3765	16.10	16.1	24.82	29.0	1.63	95.6	Nutrient valve plugged
09/01/97	12.00	12.01	23.94	1.90	1.99	8154	5486	16.10	16.1	30.10	23.9	2.94	113.1	High temp.; nutrient valve plugged
09/02/97	12.00	14.91	27.14	1.90	1.95	7297	5680	16.10	16.1	32.96	21.8	3.72	104.3	High temp.
09/03/97	12.00	4.25	12.48	1.90	0.08	483	5036	16.10	16.1	20.43	35.2	25.84	99.3	
09/04/97	12.00			1.90				16.10						
09/05/97	12.00			1.90				16.10						

**Table 6. Summary of Operational Data: R-1700 on Thiokol Concentrated AP Effluent**

Date	nutr. setpt.	Nutrient		Influent Perchlorate			Effluent	Total	Calculated	Calculated	Temp.	Comments
		avg. flow GPH	total flow GPH	avg. flow GPH	conc. in ppm	cal. conc. ppm	conc. ppm	flow GPH	res. time hours	nut. ratio g nut./g AP	°F	
08/03/97	4.00	4.05	16.68	29.26	84	74	0	33.31	46.8		95.9	
08/04/97	4.00	4.13	17.58	32.87	535	475	0	37.00	42.2		98.3	
08/05/97	4.00	4.08	16.69	30.69	2711	2393	0	34.77	44.9		100.1	
08/06/97	4.00	3.19	12.89	26.26	4114	3668	2729	29.45	53.0		101.8	
08/07/97	4.00	3.16	12.87	9.73	2856	2156	21	12.89	121.0		104.5	Initiate 60 g/l DBY
08/08/97	3.00	2.60	10.24	24.89	1328	1202	24	27.49	56.7		104	
08/09/97	2.00	2.03	8.36	30.38	1570	1472	249	32.41	48.1		103.9	Agitator fail; pH jump to >10
08/10/97	2.00	2.07	8.45	30.43	2158	2021	933	32.50	48.0		106.2	
08/11/97	2.00	2.03	8.39	30.43	2802	2627	1354	32.46	48.1		100.4	
08/12/97	2.00	1.89	6.81	29.00	2193	2072	823	30.69	50.8		100.2	Sparge from nitrogen bottle
08/13/97	2.00	2.07	8.45	30.35	2272	2127	578	32.42	48.1		101	
08/14/97	2.00	2.04	8.37	30.36	1730	1621	12	32.40	48.1		103.4	
08/15/97	2.00	2.06	8.40	30.39	2214	2073	80	32.45	48.1		103.5	
08/16/97	2.00	2.00	8.28	30.32	2371	2224	7	32.32	48.3		103.7	
08/17/97	2.00	2.15	6.96	29.38	1295	1207	40	31.53	49.5		103	
08/18/97	3.00	2.52	11.65	30.14	3602	3324	3	32.66	47.8		104.1	
08/19/97	3.00	3.09	12.68	30.66	3836	3485	87	33.75	46.2		106.6	



**Table 6. Summary of Operational Data: R-1700 on Thiokol Concentrated AP Effluent**

Date	Nutrient		Influent Perchlorate		Effluent conc.	Total flow	Calculated res. time	Calculated nut. ratio	Temp.	Comments	
	nutr. setpt.	avg. flow GPH	total flow GPH	avg. flow GPH							conc. in ppm
08/03/97	4.00	4.05	16.68	29.26	84	74	0	33.31	46.8	95.9	
08/04/97	4.00	4.13	17.58	32.87	535	475	0	37.00	42.2	98.3	
08/05/97	4.00	4.08	16.69	30.69	2711	2393	0	34.77	44.9	100.1	
08/06/97	4.00	3.19	12.89	26.26	4114	3668	2729	29.45	53.0	101.8	
08/07/97	4.00	3.16	12.87	9.73	2856	2156	21	12.89	121.0	104.5	Initiate 60 g/l DBY
08/08/97	3.00	2.60	10.24	24.89	1328	1202	24	27.49	56.7	104	
08/09/97	2.00	2.03	8.36	30.38	1570	1472	249	32.41	48.1	103.9	Agitator fail; pH jump to >10
08/10/97	2.00	2.07	8.45	30.43	2158	2021	933	32.50	48.0	106.2	
08/11/97	2.00	2.03	8.39	30.43	2802	2627	1354	32.46	48.1	100.4	
08/12/97	2.00	1.89	6.81	29.00	2193	2072	823	30.69	50.8	100.2	Sparge from nitrogen bottle
08/13/97	2.00	2.07	8.45	30.35	2272	2127	578	32.42	48.1	101	
08/14/97	2.00	2.04	8.37	30.36	1730	1621	12	32.40	48.1	103.4	
08/15/97	2.00	2.06	8.40	30.39	2214	2073	80	32.45	48.1	103.5	
08/16/97	2.00	2.00	8.28	30.32	2371	2224	7	32.32	48.3	103.7	
08/17/97	2.00	2.15	6.96	29.38	1295	1207	40	31.53	49.5	103	
08/18/97	3.00	2.52	11.65	30.14	3602	3324	3	32.66	47.8	104.1	
08/19/97	3.00	3.09	12.68	30.66	3836	3485	87	33.75	46.2	106.6	

**Table 6. Summary of Operational Data: R-1700 on Thiokol Concentrated AP Effluent**

Date	nutr.	Nutrient		Influent Perchlorate			Efluent	Total	Calculated	Calculated	Temp.	Comments
		setpl.	avg. flow GPH	total flow GPH	avg. flow GPH	conc. in ppm	cal. conc. ppm	conc. ppm	flow GPH	res. time hours	nut. ratio g nut/g AP	
08/20/97	3.00	3.08	12.60	30.60	1182	1074	0	33.68	46.3		107.3	
08/21/97	3.00	3.07	12.14	30.14	4123	3742	0	33.21	47.0		110	
08/22/97	3.00	3.84	14.14	31.68	3973	3543	0	35.52	43.9		109.5	
08/23/97	4.00	4.13	7.20	21.17	4611	3858	51	25.30	61.7		111.7	
08/24/97	4.00	4.09	14.76	28.77	5098	4463	0	32.86	47.5		111.9	
08/25/97	4.00	4.11	9.40	23.39	3817	3247	735	27.50	56.7		108.5	
08/26/97	4.00	4.13	12.62	26.59	5444	4712	300	30.72	50.8		109.5	
08/27/97	4.00	4.09	11.88	25.89	5596	4833	700	29.98	52.0		108.5	
08/28/97	4.00	4.08	16.77	30.79	5915	5223	1156	34.87	44.7		108.1	
08/29/97	4.00	4.07	16.50	30.54	4227	3730	960	34.61	45.1		107.4	
08/30/97	4.00	4.11	8.93	22.90	3733	3165	383	27.01	57.8		108.9	Initiate 75% cheese whey
08/31/97	4.00	4.08	10.80	24.82	3765	3233	0	28.90	54.0		112.4	High temp
09/01/97	4.00	4.00	16.01	30.10	5486	4842	0	34.10	45.7		116.6	High temp
09/02/97	4.00	4.07	18.98	32.96	5680	5056	1955	37.03	42.1		114.2	High temp
09/03/97	4.00	1.35	5.60	20.43	5036	4724	2939	21.78	71.6		106.8	
09/04/97	4.00											
09/05/97	4.00											



Table 8. Anions Data for R-1400 in Parallel and Series

Date	Time	Probe ClO4- (ppm)	Dionex ClO4- (ppm)	Cl- (ppm)	ClO3- (ppm)	NO3- (ppm)	SO4-2 (ppm)	PO4-2 (ppm)	NO2- (ppm)	NH4+ (ppm)
7/2/97	N/A	26		607	--	--	177	1096	59	--
7/3/97	AM	120	100	457	--	--	109	260	--	--
7/3/97	PM	43	0	320	--	--	537	349	62	--
7/11/97	PM	29	0	3973	--	--	98	159	--	--
7/14/97	PM	25	0	13019	--	--	221	76	--	--
7/15/97	PM	36	0	14578	--	--	283	79	--	64
7/16/97	PM	53	0	16179	--	--	320	100	--	44
7/17/97	AM	275	149	16432	--	--	278	86	--	25
7/18/97	AM	298	229	18568	--	--	330	74	10	--
7/19/97	AM	329	315	17577	--	--	289	82	--	--
7/21/97	AM	15	0	9885	--	--	297	104	--	--
7/22/97	PM	27	0	14073	--	--	366	100	--	--
7/23/97	AM	20	0	15103	--	--	525	230	--	--
7/24/97	AM	15	0	16439	--	--	480	226	--	47
7/25/97	AM	25	0	18719	--	--	527	278	--	--
7/26/97	AM	19	0	16162	--	--	405	167	--	--
7/27/97	AM	68	56	16241	--	--	367	140	--	--
7/28/97	PM	25	0	16647	--	--	417	167	--	--
7/29/97	PM	7	0	17420	--	--	421	179	--	--
7/30/97	AM	77	58	16276	--	--	386	153	--	--
7/31/97	AM	4	0	16321	--	--	428	169	--	13

Table 8. Anions Data for R-1400 in Parallel and Series

Date	Time	Probe ClO4- (ppm)	Dionex ClO4- (ppm)	Cl- (ppm)	ClO3- (ppm)	NO3- (ppm)	SO4-2 (ppm)	PO4-2 (ppm)	NO2- (ppm)	NH4+ (ppm)
8/1/97	AM	8	0	15922	--	--	391	165	--	--
8/2/97	AM	8	0	15906	--	--	370	142	--	--
8/3/97	AM	118	84	9826	--	--	919	494	--	--
8/4/97	AM	614	535	6168	--	--	1096	945	--	--
8/5/97	AM	2872	2711	3223	--	--	1134	659	--	--
8/6/97	AM	4432	4114	1996	--	--	939	697	--	1619
8/7/97	AM	3353	2856	2567	21	--	1003	777	--	1526
8/8/97	AM	--	1328	1930	83	--	1025	1133	--	--
8/9/97	AM	--	1570	2867	--	--	689	828	--	--
8/10/97	AM	--	2158	2417	72	--	874	1158	--	--
8/11/97	AM	3200	2802	4235	77	--	1610	1423	--	--
8/12/97	AM	2270	2193	2090	--	--	963	1195	--	--
8/13/97	AM	2598	2272	1403	46	19	678	1176	--	--
8/14/97	AM	2078	1730	1644	--	12	772	770	--	1286
8/15/97	AM	--	2214	846	--	--	627	282	--	--
8/16/97	AM	--	2371	823	--	--	658	311	--	--
8/17/97	AM	--	1295	3548	--	--	1291	736	--	--
8/18/97	AM	3765	3602	845	--	--	580	223	--	--

Table 8. Anions Data for R-1400 in Parallel and Series

Date	Time	Probe ClO4- (ppm)	Dionex ClO4- (ppm)	Cl- (ppm)	ClO3- (ppm)	NO3- (ppm)	SO4-2 (ppm)	PO4-2 (ppm)	NO2- (ppm)	NH4+ (ppm)
8/19/97	AM	4212	3836	698	-	-	888	421	-	-
8/20/97	AM	1420	1182	1471	41	62	556	468	-	-
8/21/97	AM	4512	4123	770	-	56	397	463	28	1843
8/22/97	AM	4477	3973	873	-	52	426	453	-	-
8/23/97	AM	-	4611	818	112	-	459	646	12	-
8/24/97	AM	-	5098	1347	36	53	301	367	41	-
8/25/97	AM	4565	3817	1499	42	-	412	466	-	-
8/26/97	AM	6767	5444	1398	45	57	308	364	-	-
8/27/97	AM	6600	5596	-	-	-	-	-	-	-
8/28/97	AM	7183	5915	-	-	-	-	-	-	2341
8/29/98	AM		4227							
8/30/98	AM		3733							
8/31/98	AM		3765							
9/1/98	AM		5486							
9/2/97	AM	6083	5680	-	-	-	-	-	-	-
9/3/97	AM	5567	5036	-	-	-	-	-	-	-
9/4/97	AM	2782	2236	-	-	-	-	-	-	2179
9/5/97	AM	2608	2049	-	-	-	-	-	-	-
9/9/97	AM	1338	906	-	-	-	-	-	-	-
9/10/97	AM	420	304	-	-	-	-	-	-	-

Table 9. Anions Data for R-1700 in Parallel and Series

Date	Time	Probe ClO4- (ppm)	Dionex ClO4- (ppm)	Cl- (ppm)	ClO3- (ppm)	NO3- (ppm)	SO4-2 (ppm)	PO4-2 (ppm)	NO2- (ppm)	NH4+ (ppm)
7/2/97	N/A	-		-	-	-	-	-	-	-
7/3/97	AM	-		-	-	-	-	-	-	-
7/3/97	PM	-		-	-	-	-	-	-	-
7/11/97	PM	21	0	4,112	-	-	41	92	-	-
7/14/97	PM	26	0	13,160	-	-	200	63	-	-
7/15/97	PM	21	0	14,048	-	-	246	79	-	62
7/16/97	PM	21	0	15,164	-	-	289	88	-	50
7/17/97	AM	66	0	15,917	-	-	275	94	-	32
7/18/97	AM	116	64	18,805	-	-	311	93	-	-
7/19/97	AM	309	319	16,420	-	-	270	90	-	-
7/21/97	AM	18	0	16,311	-	-	289	116	-	-
7/22/97	PM	27	0	21,676	-	-	599	111	-	-
7/23/97	AM	20	0	21,607	-	-	502	262	-	-
7/24/97	AM	15	0	21,487	-	-	628	225	-	47
7/25/97	AM	25	0	19,891	-	-	478	189	-	-
7/26/97	AM	23	4	17,144	-	-	439	129	-	-
7/27/97	AM	5	0	14,977	-	-	401	132	-	-
7/28/97	PM	20	0	17,398	-	-	441	137	-	-
7/29/97	PM	31	17	17,893	-	-	334	95	-	-
7/30/97	AM	6	0	17,583	-	-	366	135	-	-
7/31/97	AM	4	0	18,835	-	-	474	133	-	10

Table 9. Anions Data for R-1700 in Parallel and Series

Date	Time	Probe ClO4- (ppm)	Dionex ClO4- (ppm)	Cl- (ppm)	ClO3- (ppm)	NO3- (ppm)	SO4-2 (ppm)	PO4-2 (ppm)	NO2- (ppm)	NH4+ (ppm)
8/1/97	AM	5	0	18,142	-	-	395	156	-	-
8/2/97	AM	6	0	14,961	-	-	367	169	-	-
8/3/97	AM	6	0	18,417	-	-	476	236	-	-
8/4/97	AM	10	0	18,893	-	-	623	428	-	-
8/5/97	AM	14	0	17,189	-	-	605	598	-	-
8/6/97	AM	2,962	2729	6,594	-	-	744	582	-	675
8/7/97	AM	84	21	14,633	-	-	404	720	-	822
8/8/97	AM	-	24	10,495	-	-	532	1290	-	-
8/9/97	AM	-	249	8,174	96	-	559	954	-	-
8/10/97	AM	-	933	5,786	-	-	2338	1425	-	-
8/11/97	AM	1,598	1354	1,497	74	-	708	1178	-	-
8/12/97	AM	958	823	3,815	-	31	1342	1365	-	1,317
8/13/97	AM	660	578	3,155	-	13	1173	1438	-	-
8/14/97	AM	28	12	2,761	-	-	963	1346	-	-
8/15/97	AM	-	80	2,156	-	-	960	441	-	-
8/16/97	AM	-	7	1,700	-	-	811	687	-	-
8/17/97	AM	-	40	1,840	-	-	797	412	-	-
8/18/97	AM	10	3	1,933	-	-	774	507	-	-
8/19/97	AM	129	87	1,872	-	-	870	462	-	-
8/20/97	AM	11	0	1,943	-	-	708	559	-	-
8/21/97	AM	13	0	2,018	53	69	395	631	-	1,921

Table 9. Anions Data for R-1700 in Parallel and Series

Date	Time	Probe ClO4- (ppm)	Dionex ClO4- (ppm)	Cl- (ppm)	ClO3- (ppm)	NO3- (ppm)	SO4-2 (ppm)	PO4-2 (ppm)	NO2- (ppm)	NH4+ (ppm)
8/22/97	AM	13	0	2,080	-	76	429	538	-	-
8/23/97	AM	-	51	2,309	-	75	506	696		
8/24/97	AM	-	0	2,399	-	-	462	728		
8/25/97	AM	793	735	2,347	54	-	499	680	-	-
8/26/97	AM	408	300	2,580	-	-	466	748	-	-
8/27/97	AM	889	700	-	-	-	-	-	-	-
8/28/97	AM	1,413	1156	-	-	-	-	-	-	2,373
8/29/97			960							
8/30/97			383							
8/31/97			0							
9/1/97			0							
9/2/97	AM	2,300	1955	-	-	-	-	-	-	-
9/3/97	AM	3,528	2939	-	-	-	-	-	-	-
9/4/97	AM	2,862	2247	-	-	-	-	-	-	1,917
9/5/97	AM	2,627	2090	-	-	-	-	-	-	-
9/6/98										
9/7/98										
9/8/98										
9/9/97	AM	422	347	-	-	-	-	-	-	-
9/10/97	AM	20	0	-	-	-	-	-	-	-

Table 10. Thiokol Operational and Analytical Data

Date	Day	Feed Conc. (ppm)	TDS (ppm)	NO3 (ppm)	AP Feed NO3 (ppm)	SO4 (ppm)	Effluent Conc. (ppm)	NO3 (ppm)	Effluent NO3 (ppm)	SO4 (ppm)	R-1400					
											pH	Temp	AP	Nutr. (gph)	H2O	RT (hrs.)
12/17/97	1										7.10	86.2	2.4	4.0	23.6	30.5
12/18/97	2	22500	147500								7.02	87.8	2.0	2.7	15.3	47.1
12/19/97	3	22500	147500								6.93	82.7	2.0	2.7	15.3	47.1
12/20/97	4	22500	147500										2.0	2.7	15.3	47.1
12/21/97	5	22500	147500								7.27	81.0	1.4	2.7	15.9	45.3
12/22/97	6	22500	147500								7.76	77.4	1.4	3.0	15.9	45.3
12/23/97	7	23500	147000								7.11	84.2	1.4	3.0	15.9	45.3
12/24/97	8	23500	147000								6.67	88.0	1.4	5.0	15.9	45.3
12/25/97	9	23500	147000								7.27	79.7	1.6	2.0	16.5	43.6
12/26/97	10	23500	147000								7.24	79.4	1.8	2.0	16.6	43.4
12/27/97	11	5200	230000	9000	5000	4000					6.43	88.5	0.8	4.3	14.8	48.6
12/28/97	12	5200	230000								6.41	87.0	0.8	4.4	14.8	48.6
12/29/97	13	20400	173000	5500	2700	2100					6.23	87.1	0.8	4.0	14.8	48.6
12/30/97	14	9500	269000	8400	4200	3300					6.38	83.5	0.6	3.5	15.6	46.2
12/31/97	15	9500	269000								6.35	83.2	0.8	3.6	15.6	46.2
1/1/98	16	35900	55900								6.46	83.3	0.9	3.0	15.6	46.2
1/2/98	17	35900	55900								6.49	85.4	1.0	3.1	15.6	46.2
1/3/98	18	35900	55900								6.61	82.3	1.0	3.1	15.9	45.3
1/4/98	19	35900	55900								6.60	84.0	na	na	na	
1/5/98	20	35900	55900								6.46	83.8	na	na	na	
1/6/98	21	35900	55900								6.48	85.6	1.1	3.1	15.8	45.6
1/7/98	22	93000	316000								6.90	88.0	2.8	7.0	10.0	72.0
1/8/98	23	93000	316000								7.05	88.2	0.4	3.1	16.5	43.6
1/9/98	24	93000	316000				12.50				6.69	82.2	0.6	3.8	19.8	36.4
1/10/98	25	93000	316000				7.80				6.78	83.8	0.8	4.2	18.3	39.3
1/11/98	26	93000	316000				23.00				6.75	84.3	0.8	4.2	18.3	39.3
1/12/98	27	93000	316000				17.70				6.84	84.0	0.8	4.2	18.3	39.3
1/13/98	28	93000	316000				23.17				6.64	83.0	0.4	4.0	18.3	39.3
1/14/98	29	89700	226000				12.30				6.87	84.5	0.8	5.0	20.0	36.0
1/15/98	30	89700	226000				32.90				6.89	84.4	0.8	5.0	20.0	36.0



Table 10. Thiokol Operational and Analytical Data

Date	Day	Feed Conc. (ppm)	TDS (ppm)	NO3 (ppm)	AP Feed NO3 (ppm)	SO4 (ppm)	Effluent Conc. (ppm)	NO3 (ppm)	Effluent NO3 (ppm)	SO4 (ppm)	R-1400					
											pH	Temp	AP	Nutr. (gph)	H2O	RT (hrs.)
1/16/98	31	89700	226000				25.00				6.89	81.6	0.5	3.0	14.4	50.0
1/17/98	32	80000	171000				25.70				6.90	80.3	0.5	3.0	14.4	50.0
1/18/98	33	80000	171000				3.10						0.5	3.0	14.4	50.0
1/19/98	34	80000	171000				22.00				6.88	79.4	0.5	3.0	14.4	50.0
1/20/98	35	80000	171000				50.80				6.84	81.4	0.5	3.0	14.4	50.0
1/21/98	36	80000	171000				5.30				6.95	82.4	0.5	3.0	14.4	50.0
1/22/98	37	80000	171000				8.60				6.83	84.6	0.7	3.3	14.4	50.0
1/23/98	38	56500	147000				16.00				6.91	86.2	0.7	3.5	16.0	45.0
1/24/98	39	56500	147000				9.00				6.82	86.2	0.7	3.5	16.0	45.0
1/25/98	40	56500	147000				24.60				7.08	83.1	0.7	3.8	18.0	40.0
1/26/98	41	40000	171000				9.40				6.85	81.5	0.7	3.8	18.0	40.0
1/27/98	42	40000	171000				13.60				6.82	88.7	1.0	3.2	13.0	55.4
1/28/98	43	40000	171000				127.00				6.84	86.8	0.7	2.4	14.9	48.3
1/29/98	44	20000	205000				12.00				6.95	88.5	1.8	3.2	19.8	36.4
1/30/98	45	20000	205000				30.00				7.04	83.0	1.0	3.2	19.8	36.4
1/31/98	46	20000	205000				8.00				6.88	85.7	1.0	3.2	17.0	42.4
2/1/98	47	20000	205000				3.70						1.0	3.2	17.0	42.4
2/2/98	48	20000	205000				12.10				7.08	88.1	1.0	3.2	17.0	42.4
2/3/98	49	22500	147500				8.70				7.18	91.0	1.0	3.2	17.0	42.4
2/4/98	50	22500	147500				8.50				6.85	84.9	1.0	3.2	17.0	42.4
2/5/98	51	22500	147500				11.10				6.83	87.0	2.7	3.0	17.0	42.4
2/6/98	52	22500	147500				7.00				7.11	86.2	2.7	4.5	15.0	48.0
2/7/98	53	22500	147500				18.00				6.83	86.3	1.8	3.4	14.8	48.6
2/8/98	54	23500	147000				8.00				6.84	86.2	1.6	3.5	12.9	55.8
2/9/98	55	23500	147000				18.80				7.06	82.8	1.0	3.5	12.9	55.8
2/10/98	56	23500	147000				15.00				7.02	84.5	0.8	1.8	12.5	57.6
2/11/98	57	23500	147000				36.00				6.88	87.8	0.7	1.8	10.0	72.0
2/12/98	58	5200	230000	9000	5000	4000	20.00				6.82	87.8	0.6	2.5	12.0	60.0
2/13/98	59	20400	173000	5500	2700	2100	47.00				6.82	86.7	0.6	2.8	12.0	60.0
2/14/98	60	9500	269000	9400	4200	3300	8.20				6.85	88.1	0.6	3.0	11.4	63.2



Table 10. Thiokol Operational and Analytical Data

Date	Day	Feed Conc. (ppm)	TDS (ppm)	NO3 (ppm)	AP Feed NO3 (ppm)	SO4 (ppm)	Effluent Conc. (ppm)	NO3 (ppm)	Effluent NO3 (ppm)	SO4 (ppm)	R-1400					
											pH	Temp	AP	Nutr. (gph)	H2O	RT (hrs.)
2/15/98	61	9500	269000				26.30				7.05	86.6	0.6	3.0	11.4	63.2
2/16/98	62	9500	269000				17.38				6.88	88.6	0.6	3.3	11.4	63.2
2/17/98	63	9500	269000				65.00				6.83	88.4	0.6	4.0	11.4	63.2
2/18/98	64	9500	269000				22.40				6.95	90.1	0.6	4.0	11.4	63.2
2/19/98	65	4500	260000	8800	3400	2900	14.70				6.82	89.2	0.6	4.0	11.4	63.2
2/20/98	66	4500	260000				22.10				5.64	90.2	0.6	4.0	11.4	63.2
2/21/98	67	4500	260000				26.44				6.91	93.1	1.0	2.8	20.2	35.6
2/22/98	68	4500	260000				17.40				7.05	82.3	1.0	2.8	20.2	35.6
2/23/98	69	4500	260000				24.00				6.92	83.3	0.9	2.8	20.2	35.6
2/24/98	70	4500	260000				19.50				6.91	83.5	0.9	2.8	20.2	35.6
2/25/98	71	4500	260000				13.60				7.15	84.0	0.9	3.1	20.2	35.6
2/26/98	72	2840	217000	2700	8200	1800	8.30				6.98	81.7	0.9	3.1	20.2	35.6
2/27/98	73	2840	217000				13.00				7.08	83.9	0.9	3.1	20.2	35.6
2/28/98	74	2840	217000				8.00				7.16	80.6	0.9	3.4	20.2	35.6
3/1/98	75	2840	217000													
3/2/98	76	2840	217000				10.50				7.10	75.8	1.0	3.2	19.8	36.4
3/3/98	77	2840	217000	10400		2400	5.10				7.12	82.4	1.0	3.2	19.8	36.4
3/4/98	78	2840	217000				13.50				7.08	90.1	1.1	3.7	22.0	32.7
3/5/98	79	2840	217000				7.75				6.90	90.0	1.1	3.7	22.0	32.7
3/6/98	80	4750	254000	10585	3575	2250	17.50				7.39	101.7	1.1	3.7	22.0	32.7
3/7/98	81	4750	254000				11.70				6.93	88.3	1.1	3.7	22.0	32.7
3/8/98	82	4750	254000				12.65				7.36	85.6	1.1	3.7	22.0	32.7
3/9/98	83	5000	254000	9500	3920	3900	22.05	875.0	0.0	125.0	6.98	85.9	1.1	3.7	20.0	36.0
3/10/98	84	5000	254000				10.50				7.20	87.9	1.1	3.7	20.0	36.0
3/11/98	85	5000	254000				9.50				6.99	89.3	1.1	3.7	20.0	36.0
3/12/98	86	3400	230000	16000	5500	3800	9.00	750.0	220.0		7.00	87.6	1.1	4.2	20.0	36.0
3/13/98	87	3400	230000				20.40				7.30	87.3	1.1	4.2	20.0	36.0
3/14/98	88	3400	230000				5.65				7.09	86.4	1.1	4.2	20.0	36.0
3/15/98	89	5650	265000	10875	3410	2500	15.55	750.0	16.5	125.0	6.95	86.8	1.1	4.2	20.0	36.0
3/16/98	90	5650	265000				2.00				7.53	80.6	1.1	3.3	23.3	30.9

Table 10. Thiokol Operational and Analytical Data

Date	Day	Feed Conc. (ppm)	TDS (ppm)	NO3 (ppm)	AP Feed NO3 (ppm)	SO4 (ppm)	Effluent Conc. (ppm)	NO3 (ppm)	Effluent NO3 (ppm)	SO4 (ppm)	R-1400					
											pH	Temp	AP	Nutr. (gph)	H2O	RT (hrs.)
3/17/98	91	5650	265000				14.00						5.0	3.3	23.0	31.3
3/18/98	92	5650	265000				16.50				6.88	85.4	1.1	3.3	23.0	31.3
3/19/98	93	5650	265000				7.30				6.93	87.4	1.1	3.3	23.0	31.3
3/20/98	94	5000	218000	11250	5390	1750	6.20				7.35	84.9	1.1	3.3	23.0	31.3
3/21/98	95	5000	218000				11.00				7.03	79.4	1.1	3.3	23.0	31.3
3/22/98	96	5000	218000				11.30				7.31	84.4	1.1	3.3	23.0	31.3
3/23/98	97	2600	240000	10800	4750	1800	8.20				7.33	87.0	1.1	3.3	23.0	31.3
3/24/98	98	2600	240000				16.50				7.01	86.5	1.1	3.3	23.0	31.3
3/25/98	99	2600	240000				24.00				6.93	86.7	1.1	3.3	23.0	31.3
3/26/98	100	4000	264000	10200	4700	2800	19.00				7.44	87.0	1.1	3.3	23.0	31.3
3/27/98	101	4000	264000				21.70				7.14	101.8	1.1	3.3	23.0	31.3
3/28/98	102	4000	264000				6.00	750.0	0.0	0.0	7.31	89.1	1.1	3.7	22.6	31.9
3/29/98	103	4000	264000				3.70				7.02	88.1	1.1	3.7	22.6	31.9
3/30/98	104	2940	210000	8250	3190	250	9.20				7.01	89.2	0.8	2.7	16.5	43.6
3/31/98	105	2940	210000													

Table 10. Thiokol Operational and Analytical Data

Date	Day	R-1700						Nutr. Used (gal)	Effl. Treated (gal)
		pH	Temp	AP	Nutr. (gph)	H2O	RT (hrs.)		
12/17/97	1	7.10	86.7	3.5	5.8	34.1	46.9	238	148
12/18/97	2	6.92	87.6	4.3	5.8	33.2	48.2	170	128
12/19/97	3	6.70	82.2	3.0	5.8	34.5	46.4	234	71
12/20/97	4			3.0	5.8	34.5	46.4	323	128
12/21/97	5	7.20	82.3	1.0	3.0	20.5	78.0	156	62
12/22/97	6	7.33	80.4	1.0	3.0	20.5	78.0	170	69
12/23/97	7	7.25	83.8	1.0	3.0	20.5	78.0	220	61
12/24/97	8	7.14	85.3	1.0	3.0	20.5	78.0	160	97
12/25/97	9	7.17	81.9	2.0	4.0	19.2	83.3	160	103
12/26/97	10	7.16	80.9	2.2	4.0	19.3	82.9	230	75
12/27/97	11	6.88	83.6	1.2	6.1	17.7	90.4	282	5
12/28/97	12	6.63	90.5	1.2	6.0	17.3	92.5		
12/29/97	13	6.84	88.7	1.2	4.0	10.0	160.0	424	14
12/30/97	14	6.71	84.6	0.9	4.0	18.1	88.4	194	41
12/31/97	15	6.60	84.3	0.9	4.0	18.3	87.4	201	46
1/1/98	16	6.46	83.3	0.9	3.0	15.6	46.2	58	196
1/2/98	17	6.49	85.4	1.0	3.1	15.6	46.2	57	181
1/3/98	18	6.61	82.3	1.0	3.1	15.9	45.3	60	181
1/4/98	19	6.60	84.0					53	160
1/5/98	20	6.46	83.8					55	162
1/6/98	21	6.48	85.6	1.1	3.1	15.8	45.6	55	167
1/7/98	22	6.90	88.0	2.8	7.0	10.0	72.0	132	323
1/8/98	23	7.05	88.2	0.4	3.1	16.5	43.6	20	350
1/9/98	24	6.69	82.2	0.6	3.8	19.8	36.4	33	336
1/10/98	25	6.78	83.8	0.8	4.2	18.3	39.3	51	343
1/11/98	26	6.75	84.3	0.8	4.2	18.3	39.3	45	296
1/12/98	27	6.84	84.0	0.8	4.2	18.3	39.3	48	313
1/13/98	28	6.64	83.0	0.4	4.0	18.3	39.3	35	339
1/14/98	29	6.87	84.5	0.8	5.0	20.0	36.0	64	470
1/15/98	30	6.89	84.4	0.8	5.0	20.0	36.0	63	364

Table 10. Thiokol Operational and Analytical Data

Date	Day	R-1700						Nutr. Used (gal)	Effl. Treated (gal)
		pH	Temp	AP	Nutr. (gph)	H2O	RT (hrs.)		
1/16/98	31	6.89	81.6	0.5	3.0	14.4	50.0	45	324
1/17/98	32	6.90	80.3	0.5	3.0	14.4	50.0	33	265
1/18/98	33			0.5	3.0	14.4	50.0	76	500
1/19/98	34	6.88	79.4	0.5	3.0	14.4	50.0	41	237
1/20/98	35	6.84	81.4	0.5	3.0	14.4	50.0	75	252
1/21/98	36	6.95	82.4	0.5	3.0	14.4	50.0	54	272
1/22/98	37	6.83	84.6	0.7	3.3	14.4	50.0	57	287
1/23/98	38	6.91	86.2	0.7	3.5	16.0	45.0	58	282
1/24/98	39	6.82	86.2	0.7	3.5	16.0	45.0	65	335
1/25/98	40	7.08	83.1	0.7	3.8	18.0	40.0	54	283
1/26/98	41	6.85	81.5	0.7	3.8	18.0	40.0	86	262
1/27/98	42	6.82	88.7	1.0	3.2	13.0	55.4	56	378
1/28/98	43	6.84	86.8	0.7	2.4	14.9	48.3	65	231
1/29/98	44	6.95	88.5	1.8	3.2	19.8	36.4	84	283
1/30/98	45	7.04	83.0	1.0	3.2	19.8	36.4	80	273
1/31/98	46	6.88	85.7	1.0	3.2	17.0	42.4		
2/1/98	47			2.1	7.0	40.0	40.0	291	85
2/2/98	48	6.91	85.5	2.1	7.0	40.0	40.0	276	80
2/3/98	49	6.90	84.7	3.2	7.5	37.5	42.7	279	111
2/4/98	50	6.92	83.5	3.0	8.0	37.5	42.7	293	108
2/5/98	51	6.90	84.5	5.9	6.0	37.0	43.2	301	98
2/6/98	52	6.90	85.5	5.5	10.0	30.0	53.3	323	155
2/7/98	53	6.90	91.0	3.9	7.3	32.2	49.7	313	152
2/8/98	54	6.88	85.2	3.5	7.5	28.0	57.1	244	24
2/9/98	55	6.90	81.9	2.0	7.5	28.0	57.1	177	126
2/10/98	56	6.89	84.8	1.6	3.8	13.0	123.1	154	57
2/11/98	57	6.91	89.0	1.2	3.8	10.0	160.0	154	57
2/12/98	58	6.91	89.4	1.2	5.5	12.0	133.3	163	51
2/13/98	59	6.89	88.8	1.0	5.8	12.0	133.3	187	36
2/14/98	60	6.89	89.8	1.0	6.6	12.0	133.3	277	51

Table 10. Thiokol Operational and Analytical Data

Date	Day	pH	Temp	AP	R-1700			Nutr. Used (gal)	Effl. Treated (gal)
					Nutr. (gph)	H2O	RT (hrs.)		
3/17/98	91			10.0	7.1	50.0	32.0	298	92
3/18/98	92	7.01	81.8	2.4	7.1	50.0	32.0	281	211
3/19/98	93	6.99	84.0	2.4	7.1	50.0	32.0	257	90
3/20/98	94	6.89	81.2	2.4	7.1	50.0	32.0	208	92
3/21/98	95	6.90	83.0	2.4	7.1	50.0	32.0	271	95
3/22/98	96	7.03	82.6	2.4	7.1	50.0	32.0	273	96
3/23/98	97	6.89	83.4	2.4	7.1	50.0	32.0	268	94
3/24/98	98	6.97	82.7	2.4	7.1	50.0	32.0	271	93
3/25/98	99	6.94	82.5	2.4	7.1	45.0	35.6	268	93
3/26/98	100	6.93	84.4	2.4	7.1	45.0	35.6	283	98
3/27/98	101	6.93	94.7	2.4	7.1	45.0	35.6	284	99
3/28/98	102	6.91	86.7	2.4	8.0	44.1	36.3	327	104
3/29/98	103	6.93	86.0	2.4	8.0	44.1	36.3	255	84
3/30/98	104	6.93	85.6	1.7	5.8	35.8	44.7		
3/31/98	105								

Table 10. Thiokol Operational and Analytical Data

Date	Day	pH	Temp	AP	R-1700			Nutr. Used (gal)	Effl. Treated (gal)
					Nutr. (gph)	H2O	RT (hrs.)		
2/15/98	61	6.92	87.7	1.0	6.6	12.0	133.3	275	43
2/16/98	62	6.89	90.3	1.0	7.1	12.0	133.3	277	31
2/17/98	63	6.94	89.7	1.0	7.1	12.0	133.3	251	30
2/18/98	64	6.88	90.4	0.8	7.1	12.0	133.3	286	32
2/19/98	65	6.98	89.8	0.8	7.1	12.0	133.3	293	31
2/20/98	66	7.02	90.2	1.0	7.1	12.0	133.3	305	47
2/21/98	67	6.97	93.3	2.1	6.1	22.0	72.7	286	36
2/22/98	68	6.90	86.9	2.1	6.1	22.0	72.7	267	63
2/23/98	69	6.96	86.3	1.9	6.1	22.0	72.7	208	70
2/24/98	70	6.98	86.7	1.9	6.1	22.0	72.7	247	81
2/25/98	71	6.90	84.4	1.7	6.1	30.0	53.3	244	69
2/26/98	72	6.91	82.6	1.7	6.1	30.0	53.3	242	70
2/27/98	73	6.94	84.7	1.7	6.1	30.0	53.3	234	48
2/28/98	74	6.97	82.8	1.7	6.4	25.0	64.0	250	69
3/1/98	75								
3/2/98	76	6.95	78.8	2.0	7.0	40.0	40.0	509	139
3/3/98	77	6.88	81.4	2.0	7.3	40.0	40.0	268	74
3/4/98	78	6.92	89.6	2.4	8.1	45.0	35.6	275	81
3/5/98	79	6.95	89.0	2.2	8.1	45.0	35.6	201	89
3/6/98	80	7.34	101.1	2.2	8.1	45.0	35.6	73	60
3/7/98	81	7.00	88.1	2.2	8.1	45.0	35.6	314	89
3/8/98	82	6.99	85.0	2.2	8.1	45.0	35.6	362	106
3/9/98	83	6.93	84.5	2.2	8.1	42.0	38.1	251	73
3/10/98	84	6.91	87.2	2.2	8.1	42.0	38.1	166	87
3/11/98	85	6.95	88.7	2.2	8.1	42.0	38.1	293	90
3/12/98	86	6.96	87.3	2.2	9.1	42.0	38.1	308	89
3/13/98	87	6.96	87.3	2.2	9.1	42.0	38.1	366	96
3/14/98	88	6.97	84.9	2.2	9.1	42.0	38.1	359	93
3/15/98	89	6.97	85.9	2.2	9.1	42.0	38.1	313	91
3/16/98	90	6.99	86.7	2.4	7.1	50.5	31.7	326	82