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Environmental Technology Verification Report

Evaluation of BioClean USA, LLC Biological Degreasing System for the Recycling of Alkaline Cleaners

Prepared by

CTC Concurrent Technologies Corporation

Under a Cooperative Agreement with

EPA U.S. Environmental Protection Agency

ETV ETV ETV

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Evaluation of BioClean USA, LLC Biological Degreasing System for the Recycling of Alkaline Cleaners

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Foreword

The Environmental Technology Verification (ETV) Program has been established by EPA to evaluate the performance characteristics of innovative environmental technologies across all media and to report this objective information to the states, buyers, and users of environmental technology. EPA's Office of Research and Development (ORD) has established a five-year pilot program to evaluate alternative operating parameters and determine the overall feasibility of a technology verification program. ETV began in October 1995 and will be evaluated through September 2000, at which time EPA will prepare a report to Congress containing results of the pilot program and recommendations for its future operation.

EPA's ETV Program, through the National Risk Management Research Laboratory (NRMRL), has partnered with *CTC* to establish the Environmental Technology Verification Program P2 Metal Finishing Technologies (ETV-MF) Center. The ETV-MF Center was initiated to identify promising and innovative metal finishing pollution prevention technologies through EPA-supported performance verifications. The following report describes the verification of the performance of the BioClean USA, LLC Biological Degreasing System for recycling of alkaline cleaners in the metal finishing industry.

ACRONYM & ABBREVIATION LIST

amp	Ampere(s)
CFM	Cubic Feet per Minute
CFU	Colony Forming Units
COC	Chain of Custody
CTC	Concurrent Technologies Corporation
Cu	Copper
DCM	Dichloromethane
DI	Deionized
EPA	U.S. Environmental Protection Agency
ETV	Environmental Technology Verification
ETV-MF	Environmental Technology Verification Program P2 Metal Finishing Technologies
ETV-MF QMP	ETV-MF Quality Management Plan
ft^2	Square Feet
g	Gram(s)
g/l	Grams per Liter
GC-FID	Gas Chromatograph-Flame Ionization Detector
HOL	High Oil Load
HP	Horsepower
hr	Hour(s)
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectroscopy
ID	Identification
IDL	Instrument Detection Limit
kWh	Kilowatt-hour
l	Liter(s)
l/hr	Liters per Hour
lb	Pound(s)
LOL	Low Oil Load
MDL	Method Detection Limit
ml	Milliliter(s)
NWTPH-DX	Northwest Total Petroleum Hydrocarbon – Extended Diesel
O&M	Operating and Maintenance
PARCCS	Precision, Accuracy, Representativeness, Comparability, Completeness, Sensitivity
P2	Pollution Prevention
QA	Quality Assurance
QC	Quality Control
RPD	Relative Percent Difference
SOL	Spiked Oil Load
TOC	Total Organic Carbon
TP	Test Period
TS	Total Solids
TSA	Triticase Soy Agar

TSS	Total Suspended Solids
V	Volt(s)
YME	Yeast Malt Extract Agar
Zn	Zinc
$/yr	Cost per Year
$/l	Cost per Liter
$/lb	Cost per Pound

Acknowledgements

This is to acknowledge Dan Groseclose, Marion Rideout, Jim Totter, and Valerie Whitman of *CTC* for their help in preparing this document. *CTC* also acknowledges the support of all those who helped plan and implement the verification activities and prepare this report. In particular, a special thanks to Alva Daniels, EPA ETV Center Manager, and Lauren Drees, EPA Quality Assurance Manager. *CTC* also expresses sincere gratitude to Timothy Callahan, of BioClean USA, LLC, the manufacturer of the Biological Degreasing System, for his participation in and support of this program, and his ongoing commitment to improve metal finishing operations. *CTC* also thanks National Manufacturing, Sterling, Illinois, for the use of their facilities, and specifically the contributions of Dick Hall and Jackie Molina.

THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM

ETV

U.S. Environmental Protection Agency

CTC Concurrent Technologies Corporation

Concurrent Technologies Corporation

ETV JOINT VERIFICATION STATEMENT

TECHNOLOGY TYPE:	**MICROBIOLOGICAL OIL DIGESTION**
APPLICATION:	**AQUEOUS CLEANING APPLICATIONS**
TECHNOLOGY NAME:	**BioClean Biological Degreasing System**
COMPANY:	**BioClean USA, LLC**
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The United States Environmental Protection Agency (EPA) has created the Environmental Technology Verification Program (ETV) to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved, cost-effective technologies. ETV seeks to achieve this goal by providing high-quality, peer-reviewed data on technology performance to those involved in the design, distribution, financing, permitting, purchase, and use of environmental technologies.

ETV works in partnership with recognized standards and testing organizations, stakeholder groups consisting of buyers, vendor organizations, and states, with the full participation of individual technology developers. The program evaluates the performance of innovative technologies by developing test plans that are responsive to the needs of stakeholders, conducting field or laboratory tests (as appropriate), collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

The ETV P2 Metal Finishing Technologies Program (ETV-MF), one of 12 technology focus areas under the ETV Program, is operated by Concurrent Technologies Corporation, in cooperation with EPA's National Risk Management Research Laboratory. The ETV-MF Program has evaluated the performance of a bath maintenance technology for the removal of oil and other organic contaminants. This verification statement provides a summary of the test results for the BioClean Biological Degreasing System.

VERIFICATION TEST DESCRIPTION

The BioClean Biological Degreasing System (BioClean System) was tested, under actual production conditions, at the National Manufacturing Company in Sterling, IL. Alkaline cleaning is performed on metal parts at different times during the manufacturing process to remove oils, coolants and other metalworking fluids prior to electroplating. The verification test evaluated the ability of the BioClean System to remove oils at three soil loading rates (High Oil Load – HOL, Low Oil Load – LOL, and Spiked Oil Load – SOL).

Testing was conducted during three distinct 3-day test periods:

- During the first test period (HOL – high oil load), the unit was operated with a normal soil loading rate from National Manufacturing's three zinc barrel plating lines. The soil was introduced into the system from metal parts.
- During the second test period (LOL – low oil load), the unit was operated on only one of National Manufacturing's zinc barrel plating lines. The soil was introduced into the system from metal parts.
- During the third test period (SOL – spiked oil load), the unit was operated with no metal parts entering the system. The soil was introduced into the system by adding three aliquots, in a short time frame, of a commonly used oil in National Manufacturing's process. The oil was added in its pure form.

Grab samples were collected from various parts of the system (cleaner tanks, BioClean holding tank and separator) during each test run. Historical operational data were taken from the testing facility in order to compare the labor and costs involved the parts cleaning operation.

TECHNOLOGY DESCRIPTION

The BioClean System employs an alkaline cleaning solution and control system that utilizes microbes in the solution to consume the oil/grease that is removed from parts during the cleaning process in the metal finishing industry. The system operates at relatively low temperatures (104ºF – 131ºF) (40ºC - 55ºC) and a pH range of 8.8 - 9.2, which is a viable habitat for these microorganisms. The cleaning process actually takes place in two separate operations. When parts come in contact with the solution, the oil and impurities are emulsified into micro-particulates. The particulates are then consumed by microorganisms, which are present in the bath or spray. The microbe consumption of the oil present in the bath, as its food source, results in the production of CO_2 as a by-product.

The Sterling facility has four plating lines that use a combination of rack and barrel plating technologies. Three of the four lines are zinc barrel plating, and the fourth is a multi-purpose (rack and barrel) line. The cleaning solutions from the four separate cleaning baths are pumped continuously into a holding tank that feeds the BioClean System. After BioClean treatment the cleaning solution is returned, by gravity, into the holding tank and then pumped back into the cleaner tanks. This operation is run in a continuous mode with level guards on the cleaner tanks that prevent overfilling. As a result of the dynamics of the BioClean process and the re-circulation of the bath solution, the consumption of oil by the microbes occurs throughout the BioClean Biological Degreasing System.

VERIFICATION OF PERFORMANCE

During verification testing, the oil concentration in the BioClean System was measured at the beginning and end of each test run at the separator and at each cleaner bath within the test area (baths 1-3 for HOL, bath 3 only for LOL and SOL). Oil entering the system was estimated by measuring the amount of oil on a representative set of parts and comparing the characteristics (part size, geometry, presence of threads) of the parts being cleaned during testing to similar parts from the measured part set. Oil concentration was measured using a modified EPA 8015 procedure (the modifications are changes to the extraction solvent and Gas Chromatograph column and settings).

The performance of the BioClean System was similar during LOL and HOL testing. During the LOL test period, the BioClean System cleaned 28,549 ft^2 of metal parts that contained an estimated oil quantity of 79.7 kg. The greatest microbial growth response was seen during the HOL test. During that test period, the BioClean System cleaned 98,546 ft^2 of metal parts that contained an estimated oil quantity of 219.5 kg. Although a significant amount of oil was introduced into the system on the parts, the oil concentration in the BioClean System remained constant. The SOL test was conducted with no parts running through the Zinc Barrel Plating Line #3 and with its cleaning bath being fed into the BioClean System. The oil was introduced into the system in a short time frame through three aliquot additions. The system was spiked during the first hour of SOL Test Day #1. A total of 9.6 kg of oil was added to Cleaning Bath #3. The other cleaning baths were isolated from the BioClean System during the SOL test. An oil removal efficiency for the SOL is not presented in **Table i**, since the result would be less than the theoretical lower limit of zero. This may be due to the fact that the microbe concentration during the SOL test was approximately 1 percent of that during the HOL and LOL tests, and the oil was added as a bolus. It may be that oil must be emulsified prior to consumption by microbes. Additionally, the spike amount was relatively low with respect to the original oil content of the system. This result would also indicate the imprecision in determining oil consumption. A summary of the oil removal efficiency at the tested load rates is presented in **Table i**. The results of these short duration tests, however, do not reflect the fact that the BioClean System continues to digest oil during periods when production is not occurring.

	Initial Oil Content (Kg)	Oil Added (kg)	Final Oil Content (kg)	Oil Consumed (kg)	Removal Efficiency %
High Oil Load	52.1	219.5	156.9	114.7	42
Low Oil Load	25.9	79.7	60.9	44.7	42
Spiked Oil Load	52.0	9.6	76.6	-15.0	Not Calculated

Table i. Oil Removal Efficiency

Energy Use. Because the BioClean cleaner is maintained at 120-125°F as opposed to the previous soak clean temperature of 140-145°F there is a savings in the utility costs of the preplate cleaning cycle. A comparison of the energy requirements for the BioClean System versus the previous soak cleaner used at National Manufacturing is shown in **Table ii**. The heating costs were calculated using the formulae found in the Metal Finishing Guidebook and Directory chapter on immersion heaters. BioClean auxiliary equipment includes pumps and heaters for the BioClean separator and holding tank.

	BioClean (kWhr)	Soak Cleaner (kWhr)
Heat Required for Startup	12,300	17,200 (4 hr cycle, 50 cycle/yr)
Heat Required for Surface Loss	35,900	88,100
Heat Required for Tank Wall Loss	13,500	20,300
BioClean Aux. Equipment.	34,100	0
Total	95,800	125,600
Savings	29,800	
Savings ($.07/kWhr)	$2,086/year	

Table ii. Energy Requirements

Waste Generation. A waste generation analysis was performed using current data and historical records of the four plating processes that utilize the BioClean System at National Manufacturing. Implementation of the BioClean System has eliminated the requirement for periodic replacement of the alkaline soak clean baths; instead, an annual tank cleaning operation loses approximately 20 percent of each BioClean bath's contents. Additionally, the dump and remake frequency for the four electrocleaner baths was changed from eight times annually to four times per year. Overall, the amount of waste requiring treatment due to bath replacement is reduced from 34,400 gallons to 6940 gallons annually.

Operations and Maintenance Labor. Operating and Maintenance (O&M) labor requirements for the BioClean System were monitored during testing. The O&M labor requirement for the equipment was observed to be two hrs/wk. O&M tasks performed during the verification test include daily inspections of the unit and weekly cleaning of the tank and membrane. Daily unit supervision includes checking: the function of the air blower, the circulation of the degreasing baths through the Separator Module, the function of the metering pumps, the chemical drums for replenishment, the pH value, and the temperature value. Weekly maintenance includes checking the function of the level guards, cleaning and calibrating the pH electrode in the Separator Module, and removing the sludge at the bottom of the Separator.

Cost Analysis. A cost analysis of the BioClean System was performed using current cost factors and historical records from National Manufacturing. The installed capital cost (1998) of the unit was $47,569 (includes $27,625 for the BioClean unit, plus $19,944 for installation to four work-centers). The annual cost savings associated with the BioClean System at National Manufacturing is $86,192. The projected payback period is less than a year (0.6 yrs).

SUMMARY

The test results and a review of historical operating records at National Manufacturing show that the BioClean System provides an environmental benefit by eliminating the need for alkaline bath disposal, thereby extending the bath life and reducing the amount of liquid and solid wastes produced by the cleaning operation. The economic benefit associated with this technology is low operating and maintenance labor and reduced chemical costs, and a payback period of less than a year (0.6 yrs). As with any technology selection, the end user must select appropriate cleaning equipment and chemistry for a process that can meet their associated environmental restrictions, productivity, and cleaning requirement.

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1.0 INTRODUCTION

1.1 ETV Purpose and Program Operation

The BioClean USA, LLC Biological Degreasing System was tested by Concurrent Technologies Corporation (*CTC)* under the EPA Environmental Technology Verification Program for P2 Metal Finishing Technologies (ETV-MF). The purpose of this report is to present the results of the verification test.

The BioClean System was tested to evaluate and characterize the operation of the system through measurement of various process parameters. Testing was conducted at a National Manufacturing Company facility. National Manufacturing has two facilities that utilize BioClean Systems: Rock Falls, IL (704,000 ft^2), and Sterling, IL (550,000 ft^2). The Sterling facility utilizes a BioClean Separator Module I, and Rock Falls utilizes a Module II. The Sterling facility was chosen for the verification test because it employs a Module I, which is the larger of the two units and also more automated. National Manufacturing has been making hardware since 1901 and recently installed the BioClean System on their Rack/Barrel Plating Lines in Sterling. The BioClean System is being used to: (a) consume oil, coolants, and other metal working fluids that are removed from metal parts during the cleaning process, and (b) recycle the cleaner back to the cleaning tank.

The Sterling facility has four plating lines that use a combination of rack and barrel plating technologies. Three of the four lines are used for zinc barrel plating, and the fourth is a multi-purpose (rack and barrel) line. Materials plated on the fourth line include nickel, brass, and chromium.

The physical location of the BioClean System is on a mezzanine on the fourth floor in their Barrel/Rack Plating Department. BioClean Separator Module I is self-enclosed, and its holding tank for the feed streams is open to the atmosphere. The unit is fed with used alkaline cleaner from each of the three-zinc barrel plating lines.

The BioClean System at the Sterling, IL facility is set up as a continuous, closed system. Spent alkaline cleaner is continuously fed into a holding tank, and treated cleaner is continuously returned to the cleaning tank.

2.0 TECHNOLOGY DESCRIPTION AND OPERATING PROCESS

2.1 Technology Theory of Operation

The idea of using microbes to consume oil is not revolutionary. For over 40 years microbes have been utilized to consume oil from oil spills. The BioClean System combines this idea with a cleaner. Most conventional alkaline cleaning solutions would immediately kill the oil-consuming microbes, because of high operating temperatures or high pH. The BioClean chemistry was constructed around the characteristics of the microbe.

The BioClean System employs a mild alkaline bath or spray that operates at relatively low temperatures between 104ºF and 131ºF (40ºC – 55ºC) and in a pH range of 8.8 – 9.2, which is a viable habitat for oil digesting microorganisms. The cleaning solution contains biodegradable compounds (nonylphenol-free) that help to keep the cleaner stable. The cleaning process takes place in two separate operations. When parts come in contact with the solution, the oil and impurities are emulsified into micro-particulates. The particulates then are consumed by microorganisms, which are present in the bath or spray. The microbial consumption of the oil results in the production of CO_2 as a by-product.

The primary equipment component of the BioClean System is the separator module, which is a self-contained system that provides an environment conducive to microbial growth. The BioClean Separator Module I was utilized during verification testing (**Figure 1**). Within the separator module, the solution temperature, pH level, and additions of biodegradable compounds are controlled. The cleaning solution is circulated continually between the cleaning tank and the separator module. The separator's automated control system constantly monitors the bath solution and maintains a preset concentration by adding chemical solution as needed.

The chemical solutions include the BioClean 20/100 cleaner, BioClean T-Booster, and pH+/pH- buffer solutions. The BioClean 20/100 cleaner is used to break the bond between the part and the oil and then form a molecule around the oil particle. The BioClean T-Booster is a surfactant that aids the cleaning process. The pH- contains phosphoric acid and nutrients for the microbes. The pH+ contains sodium hydroxide and nutrients for the microbes. The pH-/pH+ solutions are used to maintain the cleaning solution pH, as well as supply nutrients for the microbes. The microbes ingest the oil first, but if the oil concentration in the cleaning solution is low, the microbes eat what is available. To prevent the microbes from eating the BioClean 20/100 cleaner or T-Booster, nutrients are added in the buffer solutions as a supplementary food source.

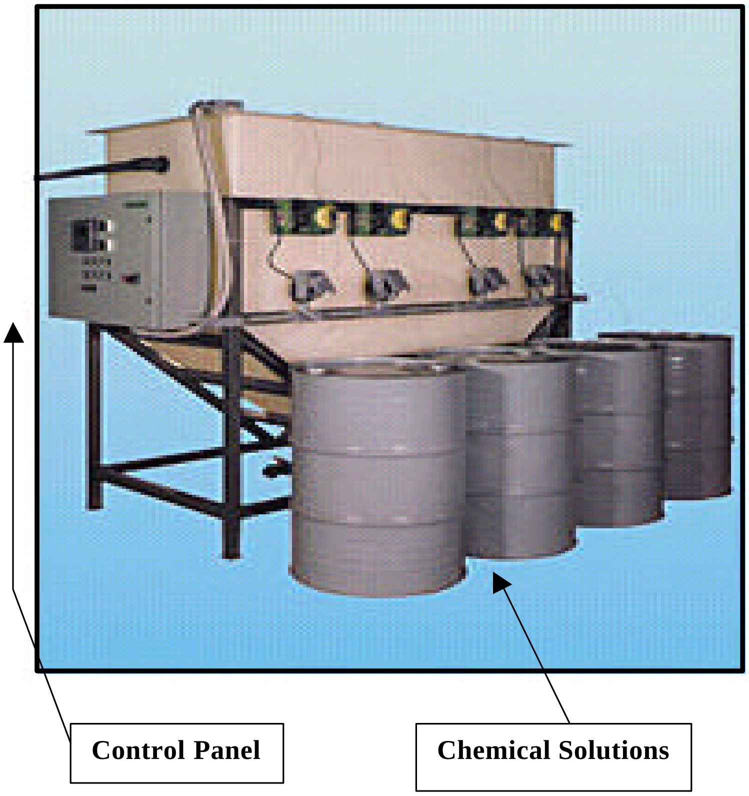


Figure 1. BioClean Separator Module I

The separator control system also uses a blower to aerate the solution to provide oxygen, which is needed by aerobic microorganisms. The microbial population is naturally occurring, and its living habitation is maintained in the BioClean separator. The microbes also are self-controlling. In theory, as the volume of oil increases, the organisms should multiply in direct proportion.

2.2 Equipment Description

The purpose of the BioClean system is to remove soils from parts prior to surface finishing operations. Specifically, this system is designed to remove oils and other lubricants used in parts manufacturing operations.

Figure 2 shows a schematic of the BioClean Separator Module I. The module is self contained and consists of a process tank, lamella separator, blower, transfer pump, primary heat control with a temperature controller, relay and temperature probe, a back-up heater, four chemical metering pumps, a high level guard, pH-meter and electrode, and control panel. The lamella (inclined plates) effectively increases the settling surface area to equal that of a much larger clarifier resulting in a much smaller floor space requirement.

The temperature and pH of the solution are controlled in the separator. The temperature set point is selected on the separator control panel and automatically maintained with either steam or electric heating. The steam and electric heaters can be run in AUTO or MANUAL modes of operation. The desired pH is also set on the control panel and automatically maintained with the pH-/pH+ metering pumps. The pH-/pH+ metering pumps can be run in AUTO or MANUAL modes. The separator module not only controls pH and temperature, but also the amount of BioClean 20/100 and T-Booster added. The flow rates of the chemical metering pumps for the BioClean 20/100 and T-Booster are set based on production (type of parts being cleaned and rate at which they are processed). The flow rate for these metering pumps, at varying production rates, was established during the installation and start-up of the BioClean System at the Sterling, IL facility. Subsequently, National Manufacturing has developed standard operating procedures for setting these metering pumps for varying production demands.

The desired concentration of 20/100 cleaner in the separator is five percent. As a result, a chemical analysis is performed, at a minimum, weekly to determine the 20/100 cleaner concentration. The make-up solutions are added manually into the separator. BioClean USA, LLC recommends a 4:1, 20/100 cleaner to T-Booster ratio.

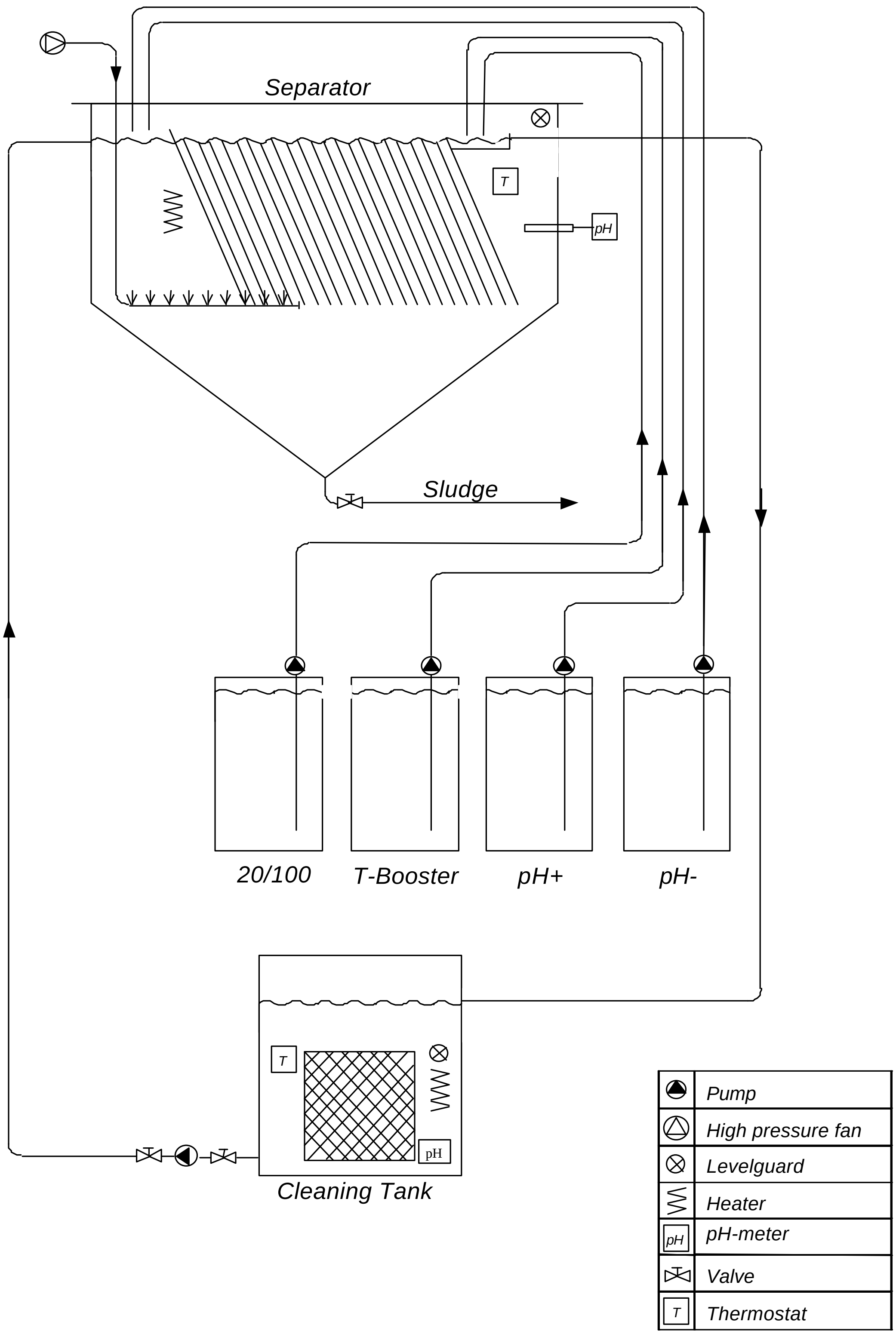


Figure 2. Biological Cleaning System (Module I) Schematic

2.1.1 Equipment List and Utility Requirements

Equipment and utility requirements are identified in **Table 1**.

EQUIPMENT		
No. Required	**Type of Equipment**	**Comments**
One (1)	Stress-relieved polypropylene tank	Stainless steel frame (volume is 1,780 liters)
One (1)	Lamella filter	Made of PVC
One (1)	Blower	
One (1)	Transfer pump	1-inch internal thread pipe connection
One (1)	Primary heat control	Temperature controller, relay, temperature probe, and level guard
Four (4)	Metering pumps with adjustable flow rates	For BioClean cleaner, T-Booster, pH-, and pH+
One (1)	pH meter and electrode	
SPARE PARTS		
One (1)	Electric supplemental heater	5000 Watts
REQUIRED UTILITIES		
Utilities include: Electrical • BioClean separator module 220 Volts, Single Phase, 30 Amperes. Air • 3 – 5 cubic feet per minute		

Table 1. BioClean Equipment List and Utility Requirements

Although BioClean's unit comes standard with an air blower, National Manufacturing tied the BioClean System into one of the existing process blowers at their Sterling, IL facility that draws atmospheric air from the roof. National Manufacturing also uses an electric heater for the primary heating source in the BioClean Separator instead of steam. Primary heating of the alkaline cleaner occurs in the cleaning tanks. During the entire verification test, the separator electric heating element only operated for three hours. However, the process temperature remained within the specified operating range throughout the testing period.

2.3 Test Site Installation

The metal finishing site selected for testing the BioClean System is the National Manufacturing Company's facility in Sterling, IL. The BioClean equipment used for these tests was already installed and had been in operation for twelve to eighteen months. **Figure 3** shows a schematic plan of the BioClean System installed at the test site.

The Sterling facility has four plating lines that use a combination of rack and barrel plating technologies. **Figure 4** shows the plating process flow diagram. Three of the four lines are used for zinc barrel plating, and the fourth is a multi-purpose (rack and barrel) line. The rack/barrel production lines clean a variety of parts. The base metals include die cast zinc, cold rolled steel, stainless steel, and solid brass. The cleaning cycle for the four plating lines includes the following steps: alkaline cleaner, electroclean, rinse, rinse, acid neutralization, rinse, and final rinse. This sequence also is the cleaning cycle employed for most plating lines. The cleaning solutions from the four separate alkaline cleaning tanks are pumped continuously into a holding tank that feeds the BioClean System. After BioClean treatment, the alkaline cleaner is returned, by gravity, into the holding tank and then pumped back into the cleaning tanks (see **Figure 3**). This operation is run in a continuous mode with level guards on the cleaning tanks to prevent overfilling. If the level guard in the cleaning tank is tripped, a solenoid valve will shut off flow to that particular tank. Also, each cleaning tank has a recirculation tank that is used to recirculate the bath solution. The cleaning bath solution is pumped from the recirculation tank to the BioClean holding tank and from the holding tank back to the recirculation tank. As a result of the recirculation of the alkaline cleaner, the consumption of oil by the microbes occurs in the cleaning tank, recirculation tank, separator, and holding tank.

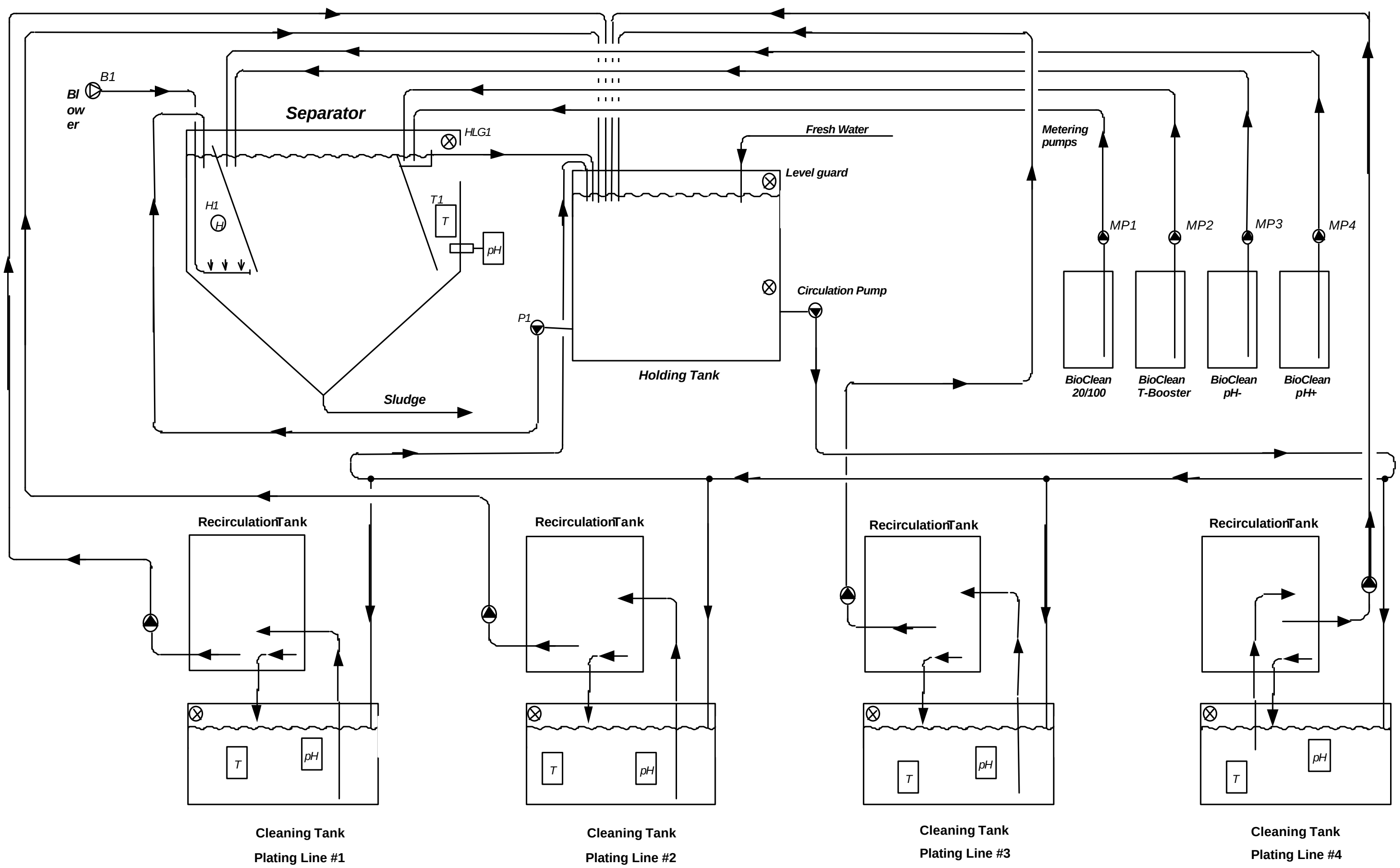


Figure 3. National Manufacturing Company Cleaning Process Flow at Their Sterling, IL, Facility

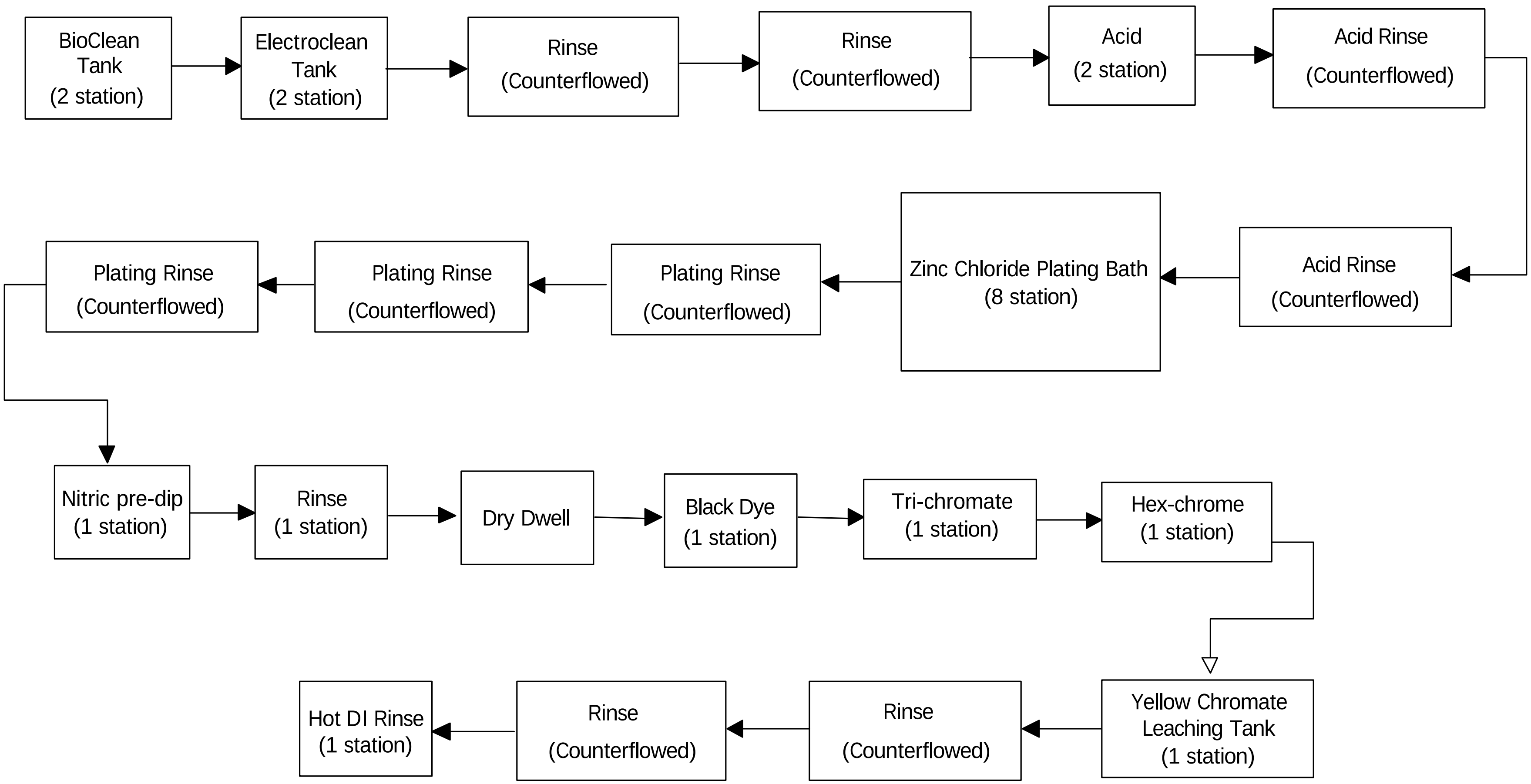


Figure 4. National Manufacturing Company Plating Line Process Flow at Their Sterling, IL, Facility

3.0 METHODS AND PROCEDURES

3.1 Experimental Design

The experimental design of this verification test was developed to provide accurate information regarding the performance of the BioClean System. The impact of field operations, as they relate to data validity, was minimized as much as possible through the use of standard sampling and analytical methodology. The experiment design does not attempt to determine optimum process operating parameters, but to determine if the process is an effective pollution prevention technology as operated at the test site.

3.2 Test Objectives

The overall objective of this project was to verify performance parameters that will enable a potential purchaser to determine if the BioClean Biological Degreasing System is appropriate and feasible for their particular application. Specific test objectives included the following:

1) Determining the oil removal efficiency of the BioClean System when processing specific types of soiled parts, with known oil load, at manufacturer recommended process conditions.
2) Quantifying the biological populations (bacteria and fungi) at selected locations within the BioClean System. (While only bacteria digest the oil in the BioClean System, conditions exist that can foster fungi growth. Therefore, samples were collected and analyzed for both bacteria and fungi. This information was to be used to assess the microbial response to the oil loading, as well as the potential health and safety risks in various stages of the cleaning process.)
3) Determining the addition rate of BioClean cleaner, T-Booster, and pH buffer solutions during observed operating conditions. This information was to be used to estimate operating costs for the BioClean System.
4) Quantifying the energy required to operate the system. Primary energy users include the bath heater, and transfer pumps. This information was to be used to help estimate operating costs for the BioClean System.
5) Quantifying the environmental benefit by determining the reduction in bath disposal frequency.

The verification testing and sampling were performed largely in accordance with the "Verification Test Plan, Evaluation of BioClean USA, LLC Biological Degreasing System for the Recycling of Alkaline Cleaners" [Ref. 1]. The original test plan required strict control of amount of oil being introduced into the system. Because of the extremely large volume and wide variety of parts cleaned daily, this degree of control was not possible under production conditions. Additionally, treating the system as a whole for oil consumption calculations proved to be misleading. Since different components of the BioClean System proved to have different concentrations of oil and different volumes, oil consumption calculations were constructed around each system component and then combined.

Testing was performed according to the equipment manufacturer's recommendations for normal operation. Also, oil loading was determined with input from the equipment manufacturer, so that verification testing would not upset National Manufacturing's production due to overloading the BioClean System. Test results and operational data were used to generally characterize the performance of the BioClean System and to construct an economic model of operating costs. The operating conditions were varied by adjusting the oil load rate into the system. Tests included high, low, and spiked oil loads. The original intent was to use a medium oil load instead of a spiked oil load. However, since controlling the oil load entering the cleaner was not possible, the spiked oil was substituted to investigate the response to an oil bolus. The spiked oil load was conducted during production downtime to avoid an adverse effect on product quality.

3.2.1 Oil Removal Efficiency

The BioClean Separator Module I was tested to determine the efficiency of oil digestion by microbes (oil removal efficiency) when processing specific types of soiled parts, with known oil load, under normal operating conditions. This objective was accomplished by performing a consumption calculation for oil for each component of the system under investigation and summing the results to achieve an overall oil consumption. Specific methods of calculation are presented in section 4.4.

3.2.2 Microbial Population

The biological populations (bacteria and fungi) were quantified at selected locations within the BioClean System. This information was used to assess the microbial response to the oil loading, as well as the potential health and safety risks at various stages of the cleaning process.

3.2.2.1 Microbial Response to Oil Loading

According to BioClean USA, LLC, the microbes used in the BioClean System are self-controlling; consequently, the microbial concentration increases as more oil is introduced into the system. Aqueous samples from the BioClean System were collected and analyzed to verify this relationship between the oil and microbial concentration.

3.2.2.2 Microbial Air Concentration

According to BioClean USA, LLC, the microbes used in the BioClean System are safe for human contact and are comparable to those used in the food, dairy, and brewing industries. The microbes are alleged to be naturally occurring and exist in water. A microbiological study was performed to assess the potential health and safety risk associated with the aerosolized microbes in various stages of the cleaning process. Air samples were collected and sent to a microbiological laboratory to quantify and identify the species of bacteria and fungi that were present. The analysis showed that the concentration of microbes in the system was below OSHA guidelines except in the headspace above the separator module, where the airstream was artificially concentrated.

3.2.3 Operational Requirements and Costs

An overall evaluation of the operation and maintenance requirements for the BioClean System was undertaken in order to estimate operating costs. The addition rates of the BioClean cleaner, T-Booster, and pH buffer solutions were observed during testing, as well as the energy required to operate the system. Primary energy users include the electric bath heater and transfer pumps.

National Manufacturing provided historical information on their previous treatment method, before the installation of BioClean. These data were used to compare their previous treatment method (bath dumping and sludge disposal) with their current method, which utilizes the BioClean System. Historical information provided by National Manufacturing included frequency of bath dumps, chemical costs, waste disposal costs, and associated labor costs.

3.2.4 Environmental Benefit

The BioClean System was evaluated to quantify the environmental benefit that results from its use by determining the reduction in bath disposal frequency. In general, bath disposal was reduced from eight times a year to none. Twenty percent of the bath is lost each year during tank cleaning.

3.3 Test Procedure

National Manufacturing designs and manufactures hardware, such as doorknobs, hinges, staple plates, coat and hat hooks, bolts, and chest handles. They make over 3,000 different products, using “just in time delivery” business practices. Their operations contain virtually the entire supply chain, beginning with the design of the product with computer aided tools, fabrication operations (stamped, die cast, or formed), plating, painting, galvanizing, and packaging. Parts arrive at the Plating Department from a number of different departments. Consequently, large quantities of parts are being plated each hour (19 barrels/hour on each line) and they are rarely identical, which makes it impractical to control the oil load in order to get a consistent load into the BioClean System. **Figure 5** shows a bin of hinges waiting to be loaded into a plating barrel.

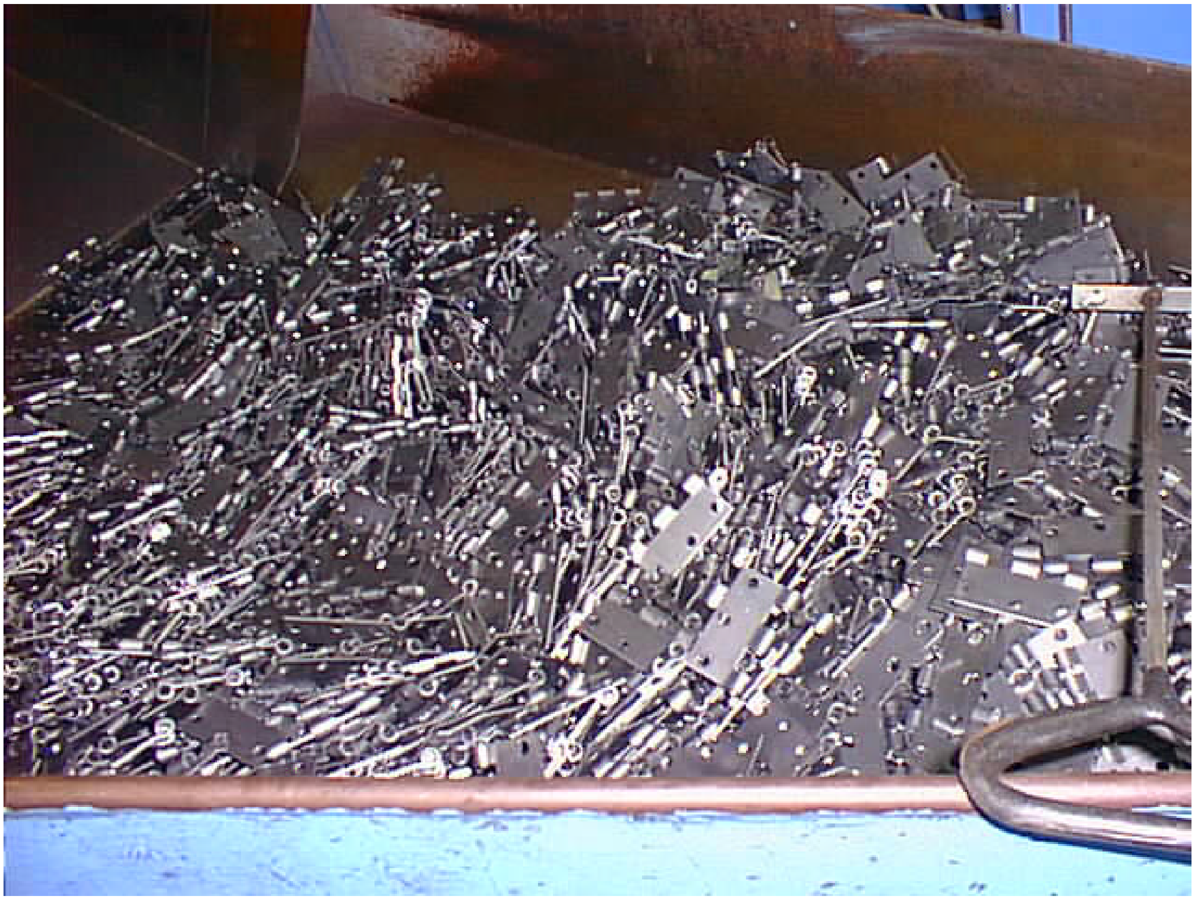


Figure 5. Bin of Hinges Prior to Plating

The test strategy, as outlined in the verification test plan [Ref. 1], was to evaluate the BioClean System performance at three different oil loads. This was accomplished as follows:

- To evaluate the effectiveness of the BioClean System at a high oil loading (HOL), three plating lines were monitored and sampled during an eight-hour shift over a three-day period.
- To evaluate the effectiveness of the BioClean System at a low oil loading (LOL), one plating line was monitored and sampled during an eight-hour shift over a three-day period.
- To understand how the microbes react with a known increase in oil concentration over time, one cleaning bath was spiked with a known amount

of oil, and was monitored and sampled during an eight-hour shift over a three-day period. This condition is referred to as spiked oil loading (SOL). Each of these test strategies and associated procedures are outlined in the ensuing sections.

3.3.1 High Oil Load – Test #1

To evaluate the effectiveness of the BioClean System during high oil loading, the following procedure was conducted:

1) All four cleaning baths were isolated from the BioClean System for one day. During this time the system was allowed to stabilize, with no additional oil introduced into the system.
2) The morning of HOL Test Day #1, initial samples were taken from the Cleaning Tanks #1 – #3, BioClean Separator Effluent, and BioClean Separator Waste Sludge.
3) After initial sampling, three of the cleaning tanks (Lines #1 – #3) were allowed to flow back into the BioClean System, while Line #4 remained isolated. Line #4 is the multi-finish rack/barrel plating line. Because Line #4 plates a variety of metal parts with a variety of finishes (e.g., nickel, brass, chromium) and consequently utilizes a variety of chemical baths, the verification testing focused just on the lines that were similar (i.e., Lines #1 – #3, which are Zinc Barrel Plating Lines).
4) Once cleaner flow to the BioClean System was observed, the system was monitored eight hours a day over a three-day period. While the BioClean System was being monitored, National Manufacturing continued with their normal production on these plating lines.
5) Operational data were taken hourly, and analytical and microbial sampling occurred throughout the day, as outlined in the verification test plan [Ref. 1].

3.3.2 Low Oil Load – Test #2

To evaluate the effectiveness of the BioClean System during low oil loading, the following procedure was conducted:

1) All four cleaning baths were isolated from the BioClean System for one day. During this time the system was allowed to stabilize, with no additional oil introduced into the system.
2) The morning of LOL Test Day #1, initial samples were taken from the Cleaning Tank #3, BioClean Separator Effluent, and BioClean Separator Waste Sludge.
3) After initial sampling, Cleaning Tank #3 was allowed to flow back into the BioClean System, while the other three lines (Lines #1, #2,

and #4) remained isolated. Because the other three lines were isolated from the BioClean System, an overall LOL was obtained.

4) Once cleaner flow to the BioClean System was observed, the system was monitored eight hours a day over a three-day period. While the BioClean System was being monitored, National Manufacturing continued with their normal production on these plating lines.
5) Operational data were taken hourly, and analytical and microbial sampling occurred throughout the day, as outlined in the verification test plan [Ref. 1].

3.3.3 Spiked Oil Load – Test #3

To assess the microbial response to an increase or decrease in oil loading, the following procedure was conducted:

1) All four cleaning baths were isolated from the BioClean System for one day. During this time the system was allowed to stabilize, with no additional oil introduced into the system.
2) Initial samples were taken of Cleaning Tank #3, BioClean Separator Effluent, and BioClean Separator Waste Sludge at the start of SOL Test Day #1.
3) After initial sampling, Cleaning Tank #3 was spiked with a known type and quantity of oil in order to reach 1.5 percent total oil concentration in the bath. The oil was added, in its pure form, directly into the cleaning tank. An empty, rotating barrel remained in the bath for agitation.
4) The cleaner was allowed to flow back into the BioClean System, while the other three lines (Lines #1, #2, and #4) remained isolated. Once cleaner flow to the BioClean System was observed, the system was monitored eight hours a day over a three-day period. While the BioClean System was being monitored, there were no parts being plated on Line #3.
5) Operational data were taken hourly, and analytical and microbial sampling occurred throughout the day, as outlined in the verification test plan [Ref. 1].

3.3.4 Determination of Oil Load

The amount of oil on each particular part is a function of the part's surface area, machining detail, and production rate. Some parts manufactured at National Manufacturing contain more oil than others because of threading and surface grooves. In order to estimate the amount of oil being introduced into the BioClean System, the oil was solvent-extracted from a representative sample of parts.

A total of twenty different representative oil-coated parts were collected prior to cleaning from the zinc barrel plating lines. The oil, from five pieces of each type of part, was extracted using the following procedure. The parts were rinsed with a known amount of acetone three separate times, in order to ensure complete oil removal. The rinses were combined into one sample container for analysis. The parts were categorized by geometry and the presence or absence of threads. The amount of oil in the acetone was quantified using EPA Method 8015 (modified) (**Appendix A**), and the average amount of oil per square foot of part surface was calculated for each category. The amount of oil carried in per square foot was then assumed to be the same for all parts in a category. Production records from National Manufacturing were used to determine the types and amounts of parts processed during testing, and to estimate the amount of oil introduced. The production data and solvent extract data used to determine the oil loads are presented in **Appendix B.**

3.3.5 Critical and Non-Critical Measurements

Measurements were taken during testing to assess the BioClean System's performance. They are classified below as either critical or non-critical. Critical measurements are those necessary to achieve project objectives. Non-critical measurements are those related to process control or general background readings. These data were used to determine the system oil consumption, the efficiency of oil/grease removal, operation and maintenance requirements, and cost effectiveness for a given set of operating conditions.

Critical Measurements:

- Chemical additions: quantity and frequency
    - BioClean cleaner (volume (ml) of each addition, time of each addition)
    - BioClean T-Booster (volume (ml) of each addition, time of each addition)
    - pH+ and pH- solutions (volume (ml) of each addition, time of each addition)
- Biological (bacteria and fungi) concentration (colony forming units (CFU))
- Oil on parts (part clusters)
- Microbe concentration
- Metal concentration (Cu, Zn)
- Total Suspended Solids (TSS) and Total Solids (TS)
- Total Organic Carbon (TOC)
- Production throughput rates (parts/hour, pounds/hour, surface area/unit of time)
- Bath aeration rate (air in cubic feet per minute (CFM))

- Operation and maintenance (O&M) labor requirements
- Solution processing rate and chemical characteristics of feed and product solutions (cleaning chemical and contaminants)
- Waste volumes, characteristics, and costs
- Separator flow rate to the holding tank (volume/time)
- Cleaning bath flow rate to the holding tank (volume/time)

Non-Critical Measurements:

- Temperature (°F) and pH
  - BioClean separator
  - Cleaning tanks
- Fresh water usage (volume/time)

Total suspended solids, total solids, metals, temperature, and pH data were collected to verify the system was operating normally.

3.4 Quality Assurance/Quality Control

Quality Assurance (QA) and Quality Control (QC) activities were performed according to the applicable section of the Environmental Technology Verification Program Metal Finishing Technologies Quality Management Plan (ETV-MF QMP) [Ref. 2].

3.4.1 Data Entry

Documentation of the verification testing events was facilitated through the use of a field laboratory book, data sheets, and chain of custody (COC) forms.

A bound, field laboratory book was used to record test methodology, observations, equipment problems, and pertinent operational data. Each page was dated and signed by the individual responsible for entries. Errors had one line drawn through them and were initialed and dated. Grab sample identification and collection day/times were recorded on specially prepared data log sheets and inserted into the logbook. Test data collection forms were used to record hourly operational data. A COC form accompanied samples that were collected and shipped to the off-site laboratories for analysis. Copies of these forms were kept at the field site through the duration of the testing. Once the laboratories received the samples, the COC was signed and dated. Original COC forms accompanied laboratory sample analysis reports.

3.4.2 Sample Collection and Analysis

Samples were collected for chemical and microbial analysis at AMTest Laboratories and U.S. Micro-Solutions, respectively. Sample collection and analysis were performed according to the procedures outlined in the verification test plan [Ref. 1]. Samples or process measurements were collected according to the frequency given in **Table 2**. The microbial sampling frequency is presented in **Table 3**. The analytical methods used for analyzing the samples are standard and/or modified EPA methods as listed in **Table 4**.

Parameter	Frequency	Cleaning Baths	Separator Effluent	Electroclean Bath	Waste Sludge
Temperature	Hourly	1	1	0	0
pH	Hourly	1	1	0	0
Oil Concentration	Daily	1 – 3	1 – 3	2	2/TP
TSS	Daily	1 – 3	2 – 3	2	2/TP
TS (%)	Daily	1 – 3	2 – 3	2	2/TP
Metals (Cu, Zn)	Daily	1 – 3	2 – 3	2	2/TP
TOC	Test Period (TP)	0	0	0	2

Table 2. Chemical Sampling/Data Collection Schedule

Alkaline Cleaner					
Parameter*	Frequency	Separator Inlet	Separator Effluent	Waste Sludge	Make-up Water
TSA	Daily	3	3	2/TP	1/TP
YME	Daily	3	3	2/TP	1/TP
Air Samples					
Parameter*	Frequency	Separator Tank	Holding Tank	Cleaning Tank	Control (outside or indoor air)
TSA	Per Test Period	10	10	10	1/day
YME	Per Test Period	10	10	10	1/day

*TSA = Triticase Soy Agar YME =Yeast Malt Extract Agar

Table 3. Microbial Sampling/Data Collection Schedule

3.4.3 Internal Quality Control Checks

Raw data generated and collected by the laboratory analysts at the bench and/or sampling site were noted with date, time, sample identification (ID), instrument ID, analyst ID, raw signal or processed signal, and/or quantitative observations. Unusual or nonstandard observations also were noted, as necessary.

A Test Data Collection Form was used for recording operational data on-site, and COC forms were used to accompany shipment of samples to the respective laboratories. Data were scrutinized according to the QA objectives in the BioClean Verification Test Plan [Ref. 1].

The Laboratory Managers at AMTest and U.S. Micro-Solutions have reviewed the final results for adequacy to the QA objectives. They also assembled data packages that include all QC and raw data results, calculations, electronic printouts, conclusions, and laboratory sample tracking information. The packages were reviewed and checked to ensure that all tracking, sample treatments, and calculations were performed correctly. Microbe analyses do not have data for precision or accuracy because matrix spikes/matrix spike duplicates are impossible to perform on this sort of determination.

3.4.4 Calculation of Laboratory Data Quality Indicators

Analytical performance requirements are expressed in terms of precision, accuracy, representability, comparability, completeness, and sensitivity (PARCCS). Calculations of data quality indicators are discussed in this section.

3.4.4.1 Precision

Precision is a measure of the agreement or repeatability of a set of replicate results obtained from duplicate analyses made under identical conditions. To satisfy the precision objectives, the replicate analyses must agree within defined percent deviation limits. The analytical laboratory performed precision evaluations on laboratory duplicates for oil, metals, TSS, TS, and TOC. All of the results were within the selected precision limits. The results of the precision calculations are summarized in **Table 4.**

Flow rates for BioClean 20/100, T-Booster, separator influent, and effluent were determined using the stopwatch/bucket method. The method was performed three times at each sampling point, and one ambiguous data point was discarded.

Critical Measurements	Method	Method of Determination	Units	Method Detection Limit (MDL)		Precision (RPD)		Accuracy (% Recovery)		% Completeness	
				Limit	Actual	Limit	Actual	Limit	Avg. Actual	Limit	Actual
OIL	EPA 8015 (mod.) (W & S)	GC-FID	mg/l	200	200	≤ 30	16.1	50–150	86.9	95	100
Metals (Cu, Zn)	EPA 200.7/200.9 (W) EPA 3050/6010 (S)	ICP-AES	mg/l	0.001	0.001	≤ 35	8.9	80–120	93	95	97
TSS/TS	EPA 160.2/160.3 (W) EPA 160.2/160.4 (S)	Gravimetric	mg/l	1.0	1.0	≤ 30	12.3	80–120	90.3	95	100
TOC	EPA 9060 (S)	Conventional	mg/l	1.0	1.0	≤ 30	1.3	80–120	146	95	83
Microbial Concentration	Serial Dilutions (W & S)	Serial Dilutions (by agar streak method)	CFU/ml	1.0	1.0	N/A	N/A	N/A	N/A	N/A	N/A
	Visual (A)	Visual	CFU/m^3	1.0	1.0	N/A	N/A	N/A	N/A	N/A	N/A
Chemical Additions:											
BioClean 20/100	Stopwatch/Bucket	N/A	ml/min	N/A		≤ 10	≤ 10	± 5	± 5	90	90
BioCleanT-Booster	Stopwatch/Bucket	N/A	ml/min	N/A		≤ 10	≤ 10	± 5	± 5	90	90
pH+	Flow Meter	N/A	ml/min	N/A		N/A	N/A	± 5	± 5	90	90
pH-	Flow Meter	N/A	ml/min	N/A		N/A	N/A	± 5	± 5	90	90
Separator Flow Rates:											
Influent	Stopwatch/Bucket	N/A	ml/min	N/A		≤ 10	≤ 10	± 5	± 5	90	90
Effluent	Stopwatch/Bucket	N/A	ml/min	N/A		≤ 10	≤ 10	± 5	± 5	90	90
Temperature	Thermocouple	N/A	ºF(ºC)	N/A		N/A	N/A	N/A	N/A	100	100

(W) Water (S) Sludge (A) Air N/A – not applicable (see section 3.4.3)

Table 4. Summary of QA Objectives

3.4.4.2 Accuracy

Accuracy is a measure of the agreement between an experimental determination and the true value of the parameter being measured. Analyses with spiked samples were performed to determine percent recoveries as a means of checking method accuracy. QA objectives are satisfied for accuracy if the average recovery is within selected goals. All results were within the selected limits, except Total Organic Carbon (TOC). Out of the six samples that were submitted to the laboratory for TOC testing, the laboratory chose one for spiked matrix testing (AMTest I.D. 00-A005894; Sample I.D. HOL3-139). The one sample chosen was tested three separate times and each percent recovery result was the same, namely 146 percent. The result is above the QA objective limit of 80 – 120 percent. The laboratory did not change the spiked amount during each repetitive test for accuracy, so it is not possible to quantify the matrix effect. TOC was not used in the evaluation of the BioClean System, since the determinations of oil concentration provided a better indication of the soil load and the data reported did not meet the quality assurance requirements for accuracy. The results of the average accuracy calculations are summarized in **Table 4.**

3.4.4.3 Completeness

Completeness is defined as the percentage of measurements judged to be valid compared to the total number of measurements made for a specific sample matrix and analysis. Completeness is calculated using the following formula:

$$\text{Completeness (\%)} = \frac{\text{Valid Measurements}}{\text{Total Measurements}} \times 100\%$$

QA objectives are satisfied if the percent completeness is 90 percent or greater. All measurements made during this verification project have been determined to be valid, except for the TOC sample mentioned above in the Accuracy section. All other measurements were above 90 percent. Results are summarized in **Table 4**.

3.4.4.4 Comparability

Comparability is another qualitative measure designed to express the confidence with which one data set may be compared to another. Sample collection and handling techniques, sample matrix type, and analytical method all affect comparability. Comparability was achieved during this verification test by the use of consistent methods during sampling and analysis and by traceability of standards to a reliable source.

3.4.4.5 Representativeness

Representativeness refers to the degree to which the data accurately and precisely represents the conditions or characteristics of the parameter represented by the data. For this verification project, representativeness was achieved by executing consistent sample collection and mixing procedures.

3.4.4.6 Sensitivity

Sensitivity is the measure of the concentration at which an analytical method can positively identify and report analytical results. The sensitivity of a given method is commonly referred to as the detection limit. Although there is no single definition of this term, the following terms and definitions of detection were used for this project.

- **Instrument Detection Limit** (IDL) is the minimum concentration that can be measured from instrument background noise.
- **Method Detection Limit** (MDL) is the statistically determined minimum concentration of an analyte that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero as determined in the same or a similar matrix. (The MDLs for this verification project are shown in **Table 4**.)

Duplicate and spike duplicate analyses were performed on one out of every ten samples. Sample splitting occurred in the laboratory.

3.4.5 Monitoring/Sampling Equipment

The instruments used to perform analytical methods were calibrated according to the analytical laboratory's quality assurance plan.

An air flow meter was installed in the piping from the blower to the separator. The meter was calibrated according to the procedures and frequency of the equipment manufacturer requirements.

Although pH and temperature are non-critical measurements and are automatically controlled at the separator module, these measurements were checked daily with a pH analyzer and digital thermometer. The following equipment was used, on-site, during the verification testing:

- Cole Palmer Chemcadet pH analyzer (Model No. 5986-60)
- Control Company NIST traceable digital thermometer (-58ºF to 302ºF; 0.1ºF increment)

A two-point calibration of the pH meter was performed each day the instrument was in use. Certified pH buffers in the expected range were used. The values of the two buffers used for calibration and the efficiency of the probe (calculated from the values of the two buffers) were recorded daily in a field laboratory book.

The thermometer and the temperature controller readings on the separator module never varied more than ± five percent, so the separator's back-up temperature controller was not utilized. However, the electric heating element used in the separator module failed twice during the testing. It was replaced with a new unit once during the testing. The process temperature remained within the specified operating range throughout the test period.

An Anderson N6 Single Stage Viable Particulate Sampler was used for bio-aerosol sampling. This sampler was calibrated for 28.3 liters/minute. Anderson recommends that the sampler be calibrated once per year. The sampler was calibrated prior to use during the verification test.

4.0 RESULTS AND DISCUSSIONS

Chemical analysis data supporting the discussion below is included in **Appendices B** and **D.**

4.1 High Oil Load

The plating processes at the Sterling facility were allowed to run normally during the HOL test, with three cleaning baths being fed into the BioClean System. A daily average part production of 750,000 pieces was run through the plating system. The system was monitored for eight hours every day over a three-day period. A summary of the oil added during the HOL test is presented in **Table 5.** The table includes the number of barrels and total surface area of parts processed each day, as well as the estimated oil introduced into the system from the parts.

Test Date	Barrels/Day	Total Surface Area of Parts (ft^2)	Oil feed Bath #1 (g)	Oil feed Bath #2 (g)	Oil feed Bath #3 (g)	Total Oil Added (g)
4/11/00	715	35,738	43,600	31,300	22,400	
4/12/00	690	34,491	13,200	65,800	7,700	
4/13/00	566	28,317	11,400	15,000	9,100	
Total	1,971	98,546	68,200	112,100	39,200	219,500

Table 5. Oil Added During HOL Test

4.2 Low Oil Load

The plating processes also were allowed to run normally during the LOL test, with only one cleaning bath (Bath #3) being fed into the BioClean System. A daily average part production of 107,500 pieces was run through the plating system. The system was monitored for eight hours each day over a three-day period. A summary of the oil added during the LOL test is presented in **Table 6**. This table also includes the number of barrels and total surface area of parts processed each day, as well as the estimated oil from each of the cleaning baths introduced into the system from the parts.

Test Date	Barrels/Day	Total Surface Area of Parts (ft^2)	Oil feed Bath #3 (g)
5/2/00	193	9,634	28,900
5/3/00	223	11,130	19,700
5/4/00	156	7,785	31,100
Total	572	28,549	79,700

Table 6. Oil Added During LOL Test

4.3 Spiked Oil Load

The SOL test was conducted with no parts running through the zinc barrel Plating Line #3, and with its cleaning bath being fed into the BioClean System. The oil was introduced into the system in a short time frame through three aliquot additions. The system was spiked during the first hour of LOL Test Day #1. A total of 9,600 g of oil were added to Cleaning Bath #3. The system was monitored for eight hours each day over a three-day period. The other cleaning baths were isolated from the BioClean System during the SOL test.

4.4 Oil Removal Efficiency

The goal of the BioClean project is to verify performance. This is measured in terms of the efficiency of the BioClean System in removing oil from the alkaline cleaner. Essentially, this is the sum of the amount of oil consumed divided by the amount of oil added to the system. Within this calculation, the volume of the recirculation tank is included in the volume of the cleaning bath, the volume of the holding tank is included in the separator system, and recirculation piping volumes are negligible. The oil removal efficiency equation for the BioClean System is shown below.

Oil Removal Efficiency (%) =

$$\frac{\text{Amount added + initial – final}}{\text{Amount Added + initial}} = \frac{A + \Sigma V_i X_{i,s} - \Sigma V_i X_{i,f}}{A + \Sigma V_i X_{i,s}} \times 100\%$$

where:

V_i	=	Volume (l)
i=1	=	Volume of Cleaning Bath #1
i=2	=	Volume of Cleaning Bath #2
i=3	=	Volume of Cleaning Bath #3
i=4	=	Volume of Separator System
$X_{i,s}$	=	Starting oil concentration at point i (g/l)
$X_{i,f}$	=	Final oil concentration at point i (g/l)
A	=	Mass of oil at added (g)

For example, for the LOL, using the data from **Table 7**:

$$\frac{79{,}700 + (2080)(7.0) + (2840)(4.0) – (2080)(17) – (2840)(9.0)}{79{,}700 + 25{,}900} \text{ X } 100\% = 42\%$$

The calculated oil removal efficiencies for the BioClean System verification test during the high and low oil loads are shown in **Table 7**.

The oil concentrations at each sampling point at the beginning and end of each test run were multiplied by the sampling point specific volumes to determine the initial and final mass of oil within the system. The "oil added" refers to the oil coming into the system on the metal parts for HOL and LOL tests, and the oil that was added to the system in its neat form for the SOL test. The BioClean System oil removal efficiencies were calculated based on oil consumption of the system during distinct 3-day test periods. These calculations were performed for each oil load test. Because of the biological nature of the system, calculating oil removal over a three-day period would be more representative of the system than day to day removals in order to give the microbes time to respond.

For the HOL test, **Table 7** shows that 42 percent of the oil introduced into the system was consumed by the microbe population during the three-day test. The system during the HOL test consisted of the BioClean Separator and holding tank, Cleaning Baths #1 – #3, and associated piping. The table includes each of the components that make up the system and their respective volumes, starting oil concentration, and the oil concentration at the end of the test.

For the LOL test, **Table 7** shows that again 42 percent of the oil introduced into the system was consumed by the microbe population during the three-day test. The system during the LOL test consisted of the BioClean Separator and holding tank, Cleaning Bath #3, and associated piping.

For the SOL test, a computed –24 percent oil removal efficiency indicates an apparent increase in oil beyond the amount of the oil addition. The theoretical lower limit for oil removal efficiency is zero, so it is not reported in **Table 7**. The system during the SOL test consisted of the BioClean Separator and holding tank, Cleaning Bath #3, and associated piping. The oil was added as a bolus. For an unknown reason, the microbe concentration for this test was approximately 1 percent of that during the HOL and LOL tests, and it may be that oil must be emulsified to be available to the microbes, so consumption of oil could be negligible during this test. Additionally, the spike amount was relatively low with respect to the original oil content of the system. These results indicate the imprecision inherent in calculating the oil consumption to any degree of accuracy.

The low quantity of oil in the sludge is shown in section 4.7.3. The only other exit route for oil from the system is dragout on the parts, and supporting evidence of this (i.e., excessive cleaner disposal) is not apparent in National Manufacturing historical waste disposal records.

These three-day test results do not reflect the fact that the microbes continue to digest oil during the rest of the week and non-production periods (weekends). If evaluated over a seven-day period with no production on the weekend, the oil removal could appear to be higher to explain the lack of oil exiting the system through the sludge and lack of cleaner disposal.

	Location	Volume (l)	Oil Concentration Initial (g/l)	Oil Concentration Final (g/l)	Oil Mass Initial (g)	Oil Mass Final (g)	Oil Added (g)	Oil Consumed by Microbes, (g)	Removal Efficiency (%)
High Oil Load	Bath #1	2,080	4.0	12.0	8,320	24,960			
	Bath #2	3,410	5.0	16.0	17,050	54,560			
	Bath #3	2,080	6.0	29.0	12,480	60,320			
	Separator	2,840	5.0	6.0	14,200	17,040			
	Total				52,050	156,880	219,539	114,709	42

	Location	Volume (l)	Oil Concentration Initial (g/l)	Oil Concentration Final (g/l)	Oil Mass Initial (g)	Oil Mass Final (g)	Oil Added (g)	Oil Consumed by Microbes, (g)	Efficiency (%)
Low Oil Load	Bath #3	2,080	7.0	17.0	14,560	35,360			
	Separator	2,840	4.0	9.0	11,360	25,560			
	Total				25,920	60,920	79,700	44,700	42

	Location	Volume (l)	Oil Concentration Initial (g/l)	Oil Concentration Final (g/l)	Oil Mass Initial (g)	Oil Mass Final (g)	Oil Added (g)	Oil Consumed by Microbes, (g)	Efficiency (%)
Spiked Oil Load	Bath #3	2,080	10.0	15.0	20,800	31,200			
	Separator	2,840	11.0	16.0	31,200	45,400			
	Total				52,000	76,600	9,600	-15,000	Not Calculated

Table 7. Oil Removal Efficiency

4.5 Microbial Response

One of the objectives of the verification test was to determine, if possible, how the population of the bacteria, in solution, would vary in response to changes in oil concentration. During the high, low and spiked oil load test runs, samples were collected for oil analysis. Samples also were collected and analyzed for the total quantity of bacteria present in units of colony forming units per milliliter (CFU/ml) of solution.

The following graphs show the bacteria and oil concentrations at the BioClean™ separator effluent for samples taken during he three test runs. Note that the bacteria concentrations are total bacteria in solution. No attempt was made to determine which particular species found were involved in oil digestion. Such an analysis was determined to be more of a research and development project and not within the scope of the verification test.

As with all biological systems, there is some time lag for microbes to grow in response to the introduction of nutrients. There appears to be a growth time lag of approximately 32 hours in **Figure 6** (HOL) before the bacteria start to grow in response to introduction of oil. In **Figure 7** (LOL) the bacteria concentration unexplainably rises, drops, then rises again.

In **Figure 8** (SOL) there is no apparent correlation between bacteria and microbe concentration. Remember this test involved pouring pure oil into the cleaning tank. Bacteria may require the oil to be emulsified by the cleaner before consumption can occur.

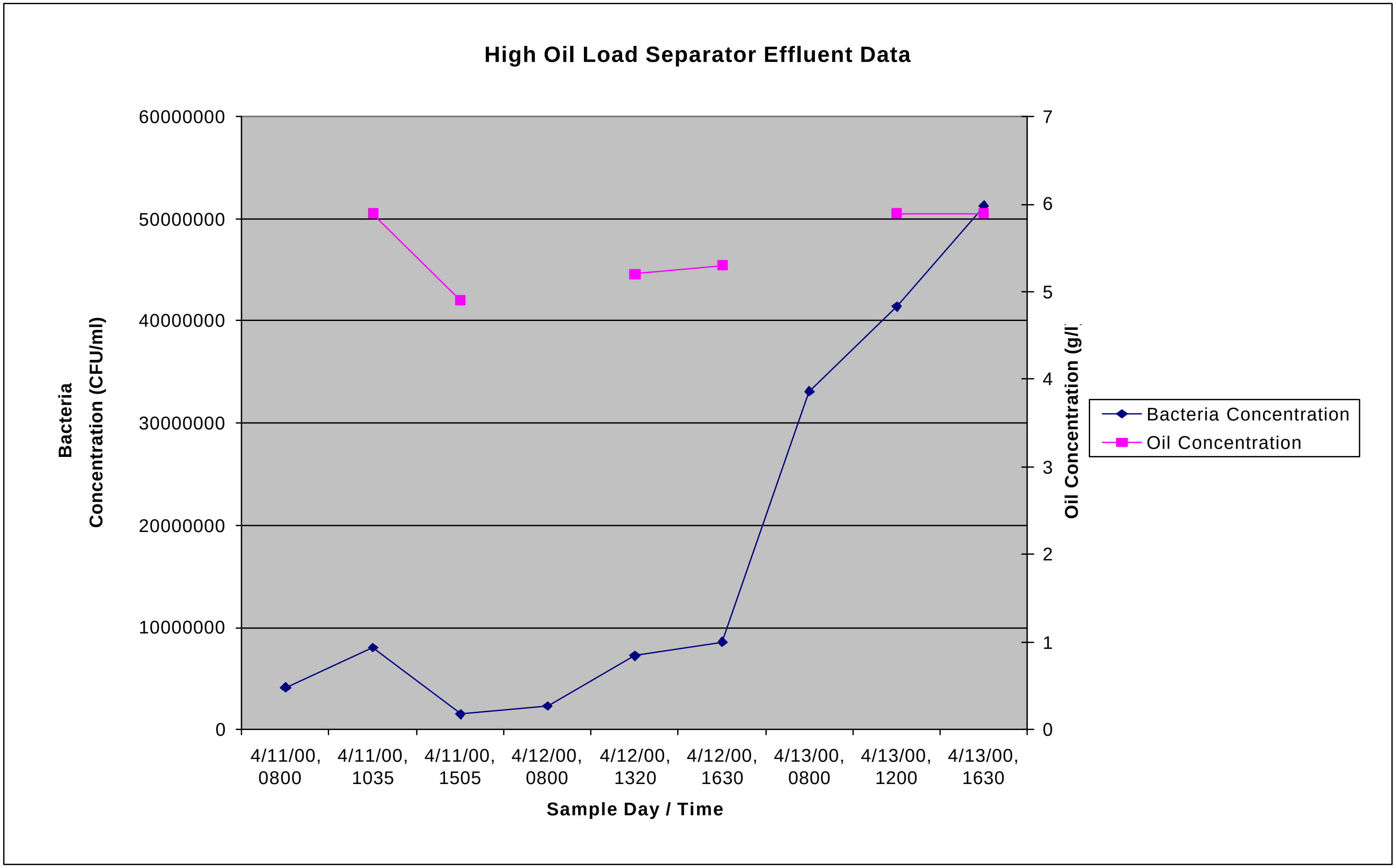


Figure 6: High Oil Load Separator Effluent Data

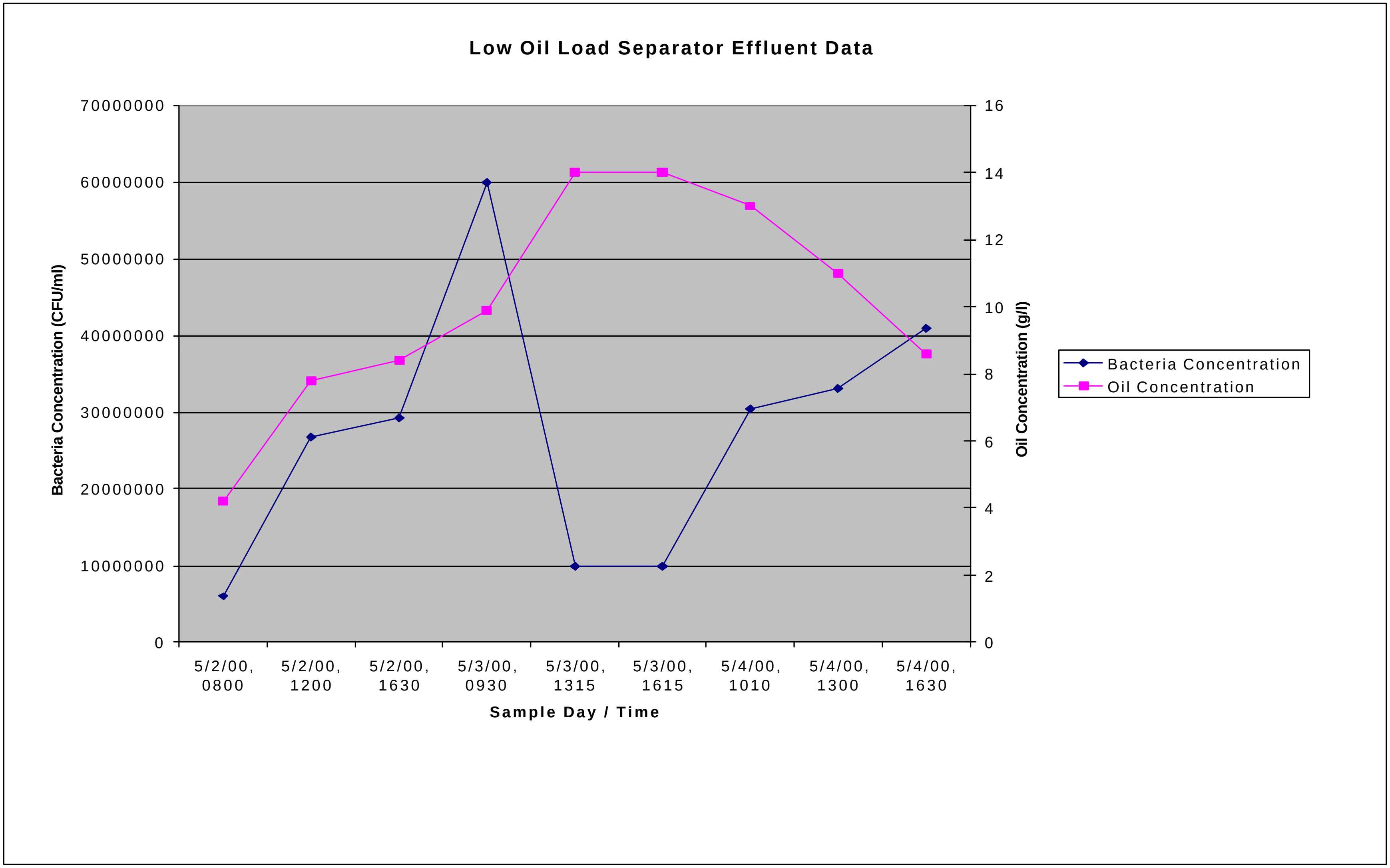


Figure 7: Low Oil Load Separator Effluent Data

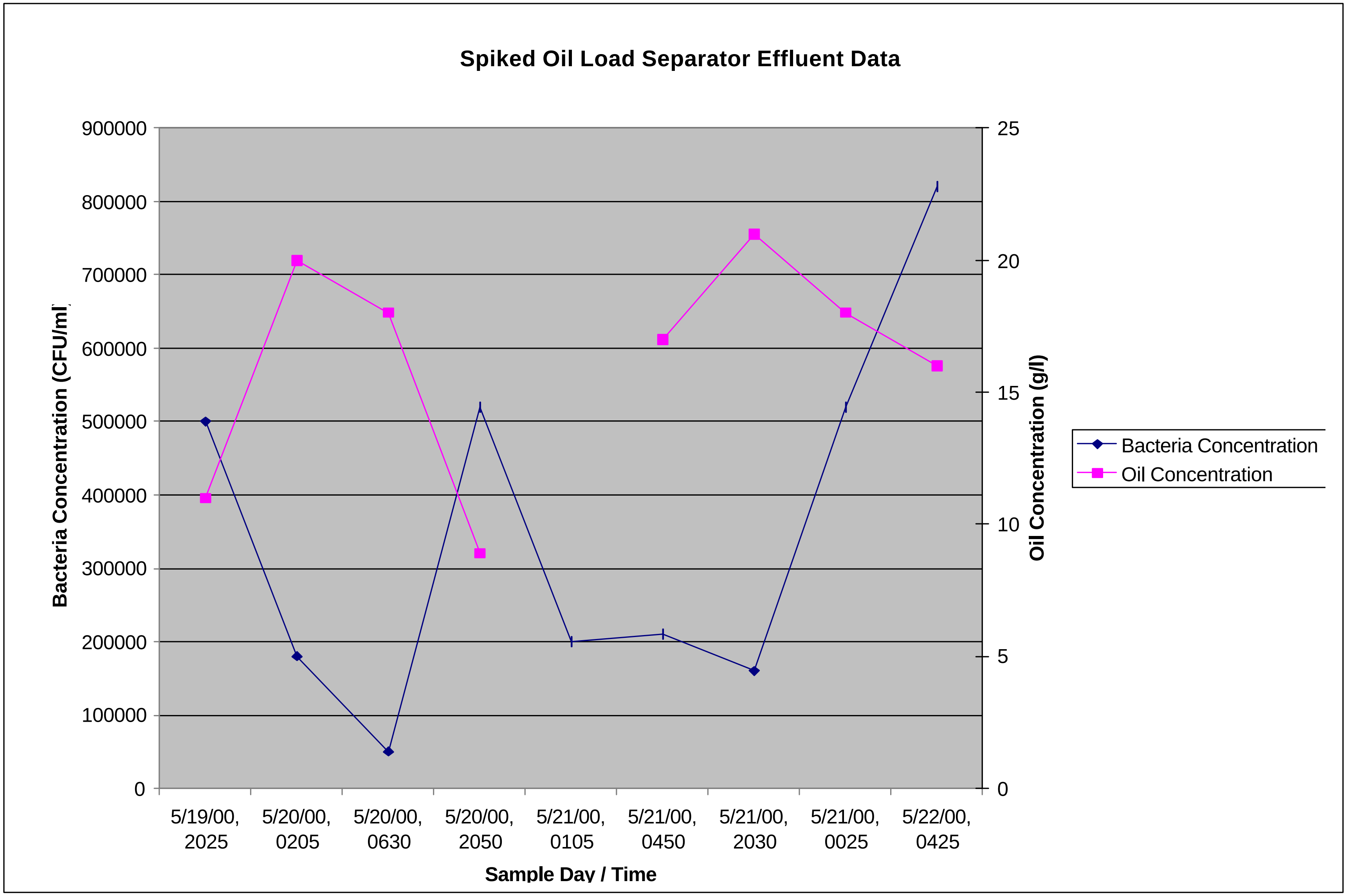


Figure 8: Spiked Oil Load Separator Effluent Data

4.6 Air Sampling

The BioClean System is designed to provide an environment in which oil digesting bacteria and other microorganisms thrive. The National Institute for Occupational Safety and Health has found that 5 percent of indoor air quality problems can be traced to microbial contamination [Ref. 3]. Microbial contamination can cause allergic reactions and infections. Symptoms may include chills, fever, muscle ache, chest tightness, headache, cough, sore throat, diarrhea, and nausea. The U.S. Occupational Safety and Health Administration (OSHA) has issued guidance [Ref. 3] that concentrations of 1,000 colony forming units per cubic meter (CFU/cubic meter) of air may be an indicator of contamination. However, levels in excess of this amount do not necessarily imply that the conditions are unsafe or hazardous. The type and concentrations of the airborne microorganisms will determine the hazard. Air sampling was performed at various points around the BioClean System and away from the system to determine bacteria and fungi concentrations. In the BioClean System, bacteria digest the oil. However, conditions exist in the separator that can foster fungal growth. Therefore, samples for fungi were also collected and analyzed.

4.6.1 Bacteria

During the high oil load air testing, samples were taken from the areas near cleaning baths 1, 2, and 3; the BioClean Separator; and the Holding Tank. One sample (HOL2-74, taken at the BioClean Separator) was not included in the analysis. Its value (230 CFU/cubic meter) was so different from the others for that point that it was rejected as being erroneous. A sample of outside air was collected and determined to be 18 CFU/cubic meter. Other samples of ambient air/blanks were not obtained due to time constraints. Results of the high oil load air samples are summarized in **Table 8**:

Sampling Point	Average Concentration (CFU/cubic meter)	Range CFU/cubic meter
Cleaning Tank 1	378	128-459
Cleaning Tank 2	477	327-531
Cleaning Tank 3	165	150-194
BioClean™ Separator	4,558	2862-5353
Holding Tank	140	35-212

Table 8. HOL Bacteria Air Sampling Results

Only the air samples collected from the exit of the BioClean Separator exceeded the OSHA indicator value of 1,000 CFU/cubic meter. The reason for the high concentration is that the BioClean separator has a cover with two six inch diameter holes, and sampling occurred at one of the holes. The airstream at that point becomes artificially concentrated because the separator's headspace does not receive the dilution from normal ventilation, as seen at all other sampling points.

During the low oil load-testing phase, samples were collected from the Cleaning Tank #3 area and the BioClean Separator. One sample (LOL-18 from Cleaning Tank #3, 5,212 CFU/cubic meter) was rejected as erroneous. The average concentration from Cleaning Tank #3 was 298 CFU/cubic meter, and the average from the BioClean Separator was 1,910 CFU/cubic meter. Outside air samples during the LOL test averaged 700 CFU/cubic meter. The disparity of airborne bacteria concentrations between the high oil load and low oil load tests indicate the variability of airborne bacteria in ambient air.

The samples were characterized also for the type of bacteria present, although no attempt was made to quantify the concentrations of any individual species. *Bacillus spp*, *Micrococcus spp*, *Corynebacterium spp*, and *Micrococcus luteus* were most often identified. These species are gram positive bacteria usually isolated from a variety of environmental sources. Although usually considered harmless, they may become a source of infection in immuno-compromised individuals [Ref. 4].

4.6.2 Fungi

During the high oil load testing, only one air sample (HOL-45 = 1,572 CFU/cubic meter) taken in the vicinity of cleaning tank 2 exceeded the OSHA indicator of 1,000 CFU/cubic meter. The average results of testing are shown as **Table 9**:

Sampling Point	Average Concentration (CFU/cubic meter)	Range CFU/cubic meter
Cleaning Tank 1	254	124-459
Cleaning Tank 2	842	468-1572
Cleaning Tank 3	359	230-424
BioClean™ Separator	240	71-885
Holding Tank	319	150-786

Table 9. HOL Fungi Air Sampling Results

No samples of ambient air were taken during high oil load testing due to time constraints.

During the low oil load testing, samples from outside air and indoor air away from the BioClean™ System exceeded OSHA guidelines. The average results are presented as **Table 10**:

Sampling Point	Average Concentration (CFU/cubic meter)	Range CFU/cubic meter
Cleaning Tank 3	461	12-1873
BioClean™ Separator	695	436-824
Holding Tank	1,263	247-1862
Outdoor Air	1,219	306-2332
Indoor Air	889	660-1119

Table 10. LOL Fungi Air Sampling Results

The samples were characterized for the type of fungi present. The most common species found were *Cladosporium spp*, *Penicillium spp*, *Alternaria spp*, *Fusarium spp*, and *Epicoccum spp*. In general all fungal spores can cause asthma and rhinitis, in addition to species-specific infections. However, health risks associated with fungal growth in an indoor environment have yet to be documented [Ref. 4].

At present there is no dose-response curve for health effects due to exposure to many of these airborne biologicals. Therefore, no threshold exposure limit has been accepted either in the scientific literature or regulatory community. The only area where the OSHA indicator values are routinely exceeded is the vicinity of the BioClean separator and holding tank. Maintenance activities inside the BioClean separator and holding tank may need to be evaluated for the need for personal protective equipment.

4.7 Economic Evaluation

An economic evaluation was prepared comparing National Manufacturing's previous method of cleaning bath treatment and disposal against their current method, which utilizes the BioClean System.

4.7.1 Chemical Costs

Prior to installing the BioClean System, National Manufacturing used an aqueous soak cleaner, which operated at 140-145°F, followed by an electrocleaner, on all four plating lines. These baths were maintained by analysis, and the baths were dumped and remade eight times per year. The associated raw chemical costs for the soak clean/electroclean system are presented below:

	Soak Cleaner	Electrocleaner	Total Annual Cleaner Costs
Annual make-up costs:	$12,460	$10,496	$ 22,956
Annual replenishment costs:	$52,470	$34,876	$ 87,346
Subtotals	$65,110	$45,372	$110,482

Table 11. Associated Raw Chemical Costs

After installing the BioClean System, no disposal of the cleaner solution due to oil saturation was required. The BioClean tanks require only annual cleaning (approximately 20 percent of the bath is replaced during cleaning), and the electroclean bath dump frequency was reduced to four times a year with the following associated chemical costs:

	BioClean Cleaner	Electrocleaner
Annual make-up costs:		$5,284
Annual replenishment costs:	$6994	$25,584
Total	$6994	$30,832
Total Annual Cleaner Costs		$37,376

Table 12. Associated Chemical Costs

The annual savings in direct cleaner costs is $110,482 - $37,376 = $73,106. The above costs on an annual basis were developed using purchasing data from National Manufacturing. During high and low oil load testing, the BioClean 20/100 solution was metered at 2.08 l/hr, and the T-Booster at 0.25 l/hr. During the high oil load a total of 5,270 ml of pH+ was added along with 300 ml of pH- with production of 98,546 square feet. During the low oil load test, 1,556 ml of pH+ was added while processing 28,550 square feet (no pH- was added during low oil load testing). In the spiked oil load test, the 20/100 feed rate was 0.62 l/hr, the T-Booster feed rate was 0.16 l/hr, and 771 ml of pH+ was added (no pH- was used).

4.7.2 Energy Costs

Because the BioClean cleaner is maintained at 120-125°F, as opposed to the previous soak clean temperature of 140-145°F, there is a savings in the utility costs of the preplate cleaning cycle. The heating costs were calculated using the formulae found in the Metal Finishing Guidebook and Directory [Ref. 5] chapter on immersion heaters. BioClean auxiliary equipment includes pumps and heaters for the BioClean Separator and holding tank.

	BioClean (kWh)	Soak Cleaner (kWh)
Heat Required for Startup	12,300	17,200 (4-hr cycle, 50 cycle/yr)
Heat Required for Surface Loss	35,900	88,100
Heat Required for Tank Wall Loss	13,500	20,300
BioClean Aux. Equipment.	34,100	0
Total	95,800	125,600
Savings	29,800	
Savings (at $.07/kWh)	$2,086/year	

Table 13. Heating Costs

4.7.3 Waste Disposal

Seven gallons of bottoms, with the following average composition, were collected from the BioClean Separator during each test period.

Oil	20.6 g/l
Total Solids	130 g/l
Total Suspended Solids	117 g/l
Total Organic Carbon	43.4 g/l
Zinc	10.5 g/l
Copper	0.11 g/l

Table 14. Average Composition

The amounts and concentrations of these materials are negligible with respect to the BioClean System mass and energy calculations. Note that the oil content of the bottoms would be approximately 545g, which is negligible with respect to the estimated amount of oil added to the cleaning baths by incoming parts.

A waste generation analysis was performed using current data and historical records of the four plating processes that utilize the BioClean System at National Manufacturing. Implementation of the BioClean System has eliminated the requirement for periodic replacement of the alkaline soak clean baths; instead, an annual tank cleaning operation loses approximately 20 percent of each BioClean bath's contents. Additionally, the dump and remake frequency for the four electrocleaner baths was changed from eight times annually to four times per year. Overall, the amount of waste requiring treatment due to bath replacement is reduced from 34,400 gallons to 6940 gallons annually.

The separator bottoms were disposed of in the verification test site's on-site waste treatment facility, and can be assumed to be negligible in terms of the total annual waste generation there. Waste disposal costs prior to the BioClean System installation for the combination soak clean/electroclean system from historical records were $8,800 per year, as compared to $4,000 per year for the BioClean/electroclean system, which corresponds to a savings of $4,800/year.

4.7.4 Labor

Daily preventative maintenance labor observed during testing included checking the function of the air blower, circulation of the cleaning baths through the separator, function of the metering pumps, chemical level in the replenishment pumps, pH value, and temperature value. Weekly maintenance tasks included checking the level probes, cleaning and calibrating the separator pH probe, and removing the sludge from the bottom of the separator. These tasks required a total of two labor-hours per week.

Regardless of tank size or content, a bath change in the preplate cleaning process requires eight labor-hours. Prior to the BioClean System installation, eight cleaning baths were changed eight times annually. The annual labor hours required to change the baths were:

8 baths x 8 changes/year/bath x 8 hours/change = 512 labor-hours.

The BioClean system, with its reduced bath change frequency, requires the following annual labor hours:

4 BioClean cleaner baths x 1 change/year/bath x 8 hours/change +
4 electroclean baths x 4 changes/year/bath x 8 hours/change = 160 labor-hours.

To the BioClean labor requirements, the additional preventative maintenance burden of 104 man-hours/year must be added (two labor-hour/week x 52 weeks/year). The total preventative maintenance burden for the BioClean system, therefore, is 104 + 160 = 264 labor-hours/year.

National Manufacturing assumes labor costs (with burden) to be $25/labor-hour, so the total annual labor savings is:

(512 - 264 labor-hours/year) x $25/labor-hour = $6,200/year.

4.7.5 Cost Summary

Total savings seen in the operation of the BioClean System annually are:

	Savings
Chemical Usage	$73,106
Energy	$ 2,086
Waste Disposal	$ 4,800
Labor	$ 6,200
Total	$86,192/yr

Table 15. Operational Savings

The installed cost of the BioClean System at National Manufacturing was $47,569; the simple return on the investment (payback) was 0.6 years

4.7.6 Performance Summary

The data collected during the short three-day test runs were not sufficient to accurately quantify the oil removal efficiency or demonstrate a predictable response by the microbes to oil. No evidence of significant quantities of oil exiting the system through any pathway (i.e., sludge, dragout, cleaner disposal) leads to the conclusion that the BioClean System does provide significant environmental and cost benefits. The environmental benefit is achieved by eliminating the need for alkaline bath disposal, thereby extending the bath life and reducing the amount of liquid and solid wastes produced by the cleaning operation. The economic benefit associated with this technology is low operating and maintenance labor and reduced chemical costs, and a payback period of less than a year (0.6 yrs). As with any technology selection, the end user must select appropriate cleaning equipment and chemistry for a process that can meet their associated environmental restrictions, productivity, and cleaning requirement.

4.8 AUDITS

A technical systems audit was conducted by a *CTC* QA representative on May 5, 2000. The primary finding was the absence of a method to document approved changes to the verification test plan. A test plan deviation documentation method was implemented in later test plans. None of the findings or observations impacted the results of the verification test.

A data quality review was conducted by the *CTC* QA Manager on October 10, 2000. The QA Manager noted the blank spike recovery and blank spike duplicate for the oil analysis were low. However, they were within the acceptable range of 50 to 150 percent recovery. Lessons learned from the data quality review will be incorporated into future test projects.

5.0 REFERENCES

Note: References 1 and 2 are available by accessing the ETV-MF Program Internet website at: www.etv-mf.org.

1. Concurrent Technologies Corporation, "*Environmental Technology Verification Program for Metal Finishing Pollution Prevention Technologies, Verification Test Plan, Evaluation of BioClean USA, LLC Biological Degreasing System for the Recycling of Alkaline Cleaners*" (February 4, 2000).

2. Concurrent Technologies Corporation, "*Environmental Technology Verification Program, Metal Finishing Technologies Quality Management Plan*" (December 9, 1998).

3. Occupational Safety and Health Administration (OSHA). *OSHA Technical Manual, Section 3, Chapter 2: Indoor Air Quality Investigation* (1999).

4. Murray, P. R., E. J. Baron, M. A. Pfaller, F. C. Tenover, and R. H. Yolken,. Manual of Clinical Microbiology, 6th Edition, American Society for Microbiology, Washington, D.C. (1995).

5. Richards, Tom, "*Immersion Heating*", Metal Finishing, Vol. 98, No. 1, pages 755 - 766 (2000).

APPENDIX A

OIL ANALYSIS

EPA METHOD 8015 (MODIFIED)

(NORTHWEST TOTAL PETROLEUM HYDROCARBON – EXTENDED DIESEL)

(NWTPH-DX)

NWTPH-DX
Diesel Range Organics In Soil And Water

Summary

The NWTPH-DX Method adapts EPA SW-846 Methods 3540 and 8000 and covers the quantitative analysis of semi-volatile petroleum products in soils. The method involves extracting the sample with methylene chloride and injecting a portion of the extract into a gas chromatograph equipped with a flame ionization detector. This method specifies criteria for the identification and quantitation of semi-volatile petroleum products. When the type of fuel is unknown #2 diesel is used to quantitate the sample. The reporting limit is 25 mg/kg for soil and 0.10 mg/l for water samples eluting from the jet fuels range to the diesel #2 range. For petroleum products eluting after diesel #2, the reporting limits are 100 mg/kg for soil and 0.20 mg/l for water (assuming 100% total solids for soil).

Equipment and Reagents

Gas Chromatograph
Flame Ionization Detector
Column: J & W DB-5 30M x 0.32mm with 0.25µm film thickness capillary column
Maxima Data System
Analytical Balance accurate to at least 0.001g
Horn Sonicator
Volumetric Flasks, 10 ml Ground Glass Stoppered
150 ml beakers
Sodium Sulfate
Methylene Chloride
K-D Equipment (refer to K-D section)
Nitrogen evaporator
Sulfuric Acid, concentrated
Silica Gel cartridges
Various Petroleum products for standards

Collection Requirements

All samples should be collected in I-Chem containers and preserved at 4 degrees Celsius until extracted. The holding time from the date of collection to extraction is 14 days for soils and preserved water. For unpreserved water, the holding time is seven days. Preservation is accomplished by adjusting pH to about 2 using a 1:1 HC1 aqueous solution.

Standards

Fuel Stock Standard

Choose the appropriate fuel for comparison to the sample fingerprint. Weigh approximately 0.10 g into a 10-ml volumetric flask and dilute to volume with Dichloromethane (DCM). Label and record the exact concentration.

Calculate the concentration as follows:

$$\text{Stock Conc. } (\mu g/ml) = \frac{\text{weight diesel (g)} \times 1{,}000{,}000\ (\mu g/g)}{10\ ml}$$

Calibration Standard

An initial characterization and evaluation of these "neat" formulated products using the modified Method 8015 was performed by the analytical laboratory (AMTest, Inc. of Redmond, WA). Modifications to the standard 8015 method involved slight changes in the ramp time within the gas chromatographic program, which were within the proscribed acceptable method modifications. Each type of organic soil evaluated yielded a characteristic chromatographic signature. Based on the information received, no one particular organic soil product is known to dominate over the others. Using the aliquots from the neat solutions of the different formulated products, a mixed reference standard was created and a range of calibration concentration standards derived. Results are reported in milligrams/liter (mg/L).

Prepare calibration standards from the stock diesel standard at concentrations of 25, 50, 200, and 300 μg/ml by adding appropriate volumes to a 10-ml volumetric flask and diluting to volume with methylene chloride. For fuels heavier than diesel #2, prepare standards at concentrations of 50, 100, 150, 300, and 400 ug/ml.

To calculate volume (μl) of stock standard to add to 10-ml vol. flask use the equation below:

$$\text{Volume Diesel Stock } (\mu l) = \frac{\text{Cal. Std. Conc. } \mu g/ml \times 1000\ \mu l \times 10\ ml}{\text{Diesel Stock } \mu g/ml}$$

Dilute the flask to 10 ml with DCM.

Stock Surrogate Standard

Make up a surrogate of bromofluorobenzene and 2-fluorobiphenyl that contains approximately 8,000 μg/ml by weighing about 0.080 g of the surrogate compounds into a 10-ml volumetric flask and filling to volume with methylene chloride.

Working Surrogate Spike (800 mg/ml)

Add the appropriate volume of the stock standard to a 10-ml volumetric flask that has been filled with 5 ml of methylene chloride, taking care not to add the surrogate standard solution into the solvent without contacting the neck of the flask. Fill the flask to volume, stopper and mix. Store at 4 ^{o}C.

$$\text{Volume Surrogate Stock } (\mu l) = \frac{800\ \mu g/ml \times 10\ ml \times 1{,}000\ \mu l}{\text{Surrogate Stock Conc. } \mu g/ml}$$

GC/FID PARAMETERS FOR FUEL SCANS

Instrument Parameters:

Column:	J & W DB-5 30M x 0.32mm with 0.25 µm film thickness, capillary column
Injection Sample Volume:	2 µl
Injector Temperature:	290 ^{o}C
Ion Block Temperature:	300 ^{o}C
Initial Temperature:	35 ^{o}C
Initial Time:	5 minutes
Initial Rate:	10 degrees/min
Final Temperature:	300 ^{o}C
Final Time:	5 minutes
Purge Valve On Time:	1.5 minutes
Purge Valve Off Time:	36 minutes
Purge Valve On Time:	1.5 minutes
Purge Valve Off Time:	36 minutes
Hydrogen Flow:	25 – 30 ml/min
Air Flow:	300 – 400 ml/min
Make-up Gas Flow:	30 ml/min
Carrier Gas:	Helium
Helium Carrier gas Head Pressure:	12 psi

Sample Extraction Soil

Accurately weigh approximately 20 grams of soil (note that if the sample is hydrated, more than 20 grams are needed) and 20 grams of anhydrous sodium sulfate and place into a 150-ml beaker and mix completely with a spatula. The mixture should have a grainy texture. If it forms a clump, add more sodium sulfate, grind to a grainy texture and note this in the extraction log. Add 100 µl of working surrogate spike and 50 ml of methylene chloride; sonicate this for 3 minutes utilizing the horn sonicator. (Refer to horn sonicator instructions at the end of this SOP if unfamiliar with the operation of the instrument).

Allow the mixture to stand. Collect the extract in a 250 ml Kuderna-Danish (K-D) flask to which is connected a 10-ml concentrator tube and a sodium sulfate drying apparatus.

Repeat the extraction twice more using 50 ml of methylene chloride and add the extract to the same K-D flask. Attach a 3-ball Snyder column and concentrate the extract to a final volume of 10 ml. If the extract is highly colored or forms a precipitate, a dilution may be necessary to stay within the calibration range. If samples need to be cleaned up, refer to clean-up procedure at the end of this method.

Store the samples at 4 ^{o}C in a glass vial until ready for analysis.

Water Extraction Procedure

Pour 500 ml of the sample into a 2-liter separatory funnel. Adjust the pH to 2 if needed. Add 200 µl of surrogate working standard. Extract the sample with 50 ml of DCM. Pour the extract through sodium sulfate into a K-D set up. Extract the sample twice more with 50 ml DCM, adding the extract to the K-D set up. Concentrate the sample to 5 – 10 ml on a steam bath. Remove the ampule and continue to concentrate on a N-Vap to below 2 ml. Adjust the final volume to 2 ml in a volumetric flask. Clean up the sample if needed using the procedure at the end of the SOP.

Determine the Total Solids Percentage of soil sample.

<u>GC Run to include the following:</u>

1. Five point calibration curve
2. 10% duplicates
3. Surrogate std (100 µg/l working surrogate spike to 10 ml)
4. Mid standard check every ten samples analyzed
5. End standard check at the end of each run.

<u>Data Validation:</u>

1. Continuing calibration checks and end checks must fall +/- 15 percent of the known value of the standard
2. Surrogate recoveries must be between 50 percent - 150 percent
3. Standard curve must have a minimum correlation of 0.99

Sample Calculations

The retention time range windows for integration must be adjusted to incorporate the majority of the components of the petroleum product of interest. If an exact match cannot be made, a standard is chosen that closely represents the sample. In all cases, the selected retention time window used for quantitation must, at a minimum, include any unresolved envelope of compounds as well as all discrete components peaks with an area greater than or equal to 10 percent of the largest peak. These must be integrated to the baseline as a group.

Be sure to subtract the area of the surrogates if the surrogate falls within the retention time window.

Adjustments of retention time windows may be made if interferences are present, i.e, overlap of oil into diesel area.

$$\text{Sample Conc. (mg/kg)} = \frac{\text{Sample conc. }\mu\text{g/ml} \times V \times DF}{\text{Sample weight} \times TS}$$

where: V = Final Volume of extract
DF = Dilution Factor
TS = Decimal percent solids of sample

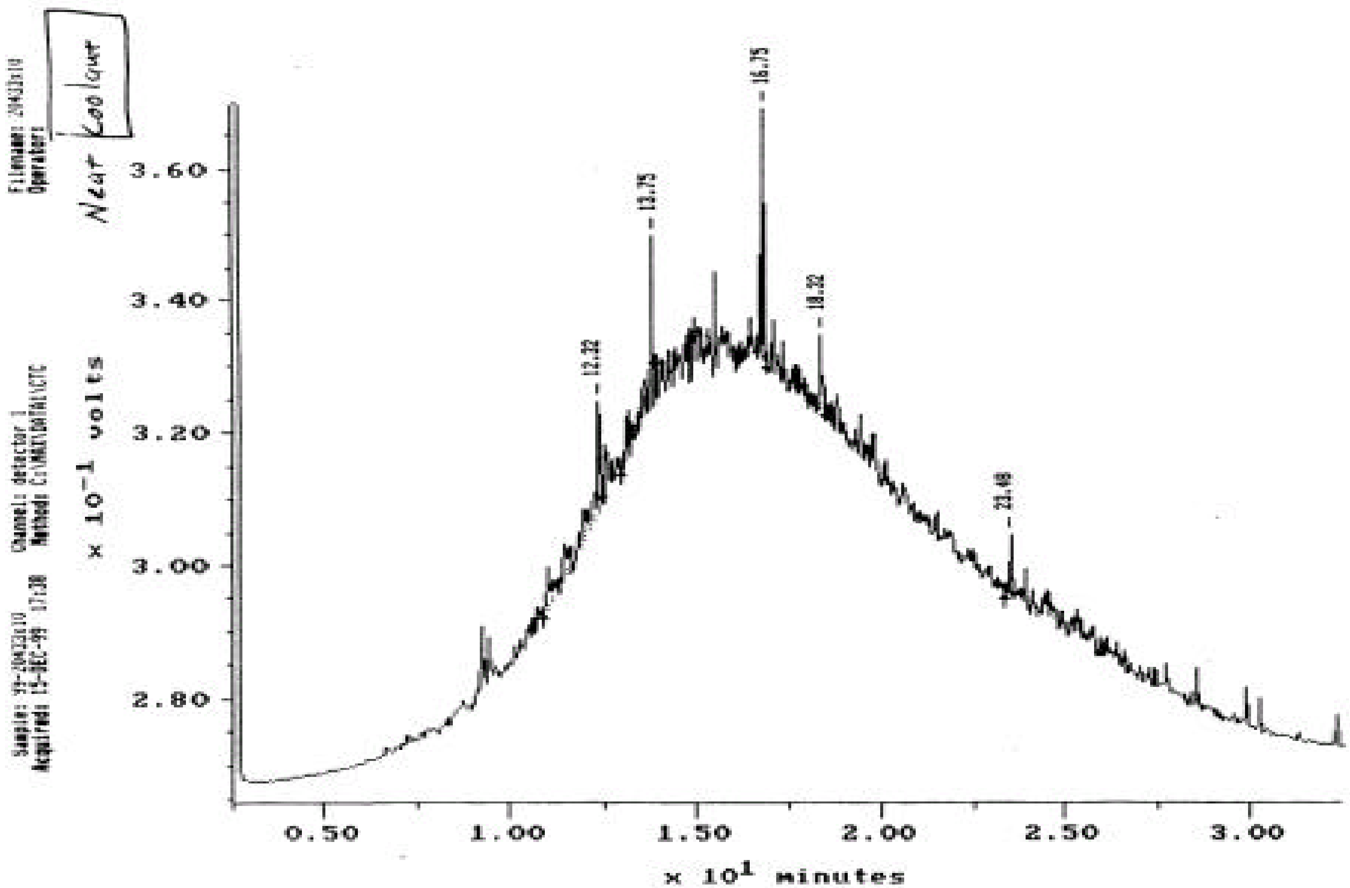
Horn Sonicator Settings

Sonicator Type: Ultrasonic, Inc. Model W-385 (475 watt) with No. 207, ¾-inch Tapped Disruptor Horn
Settings: 3 minutes
Output Control Knob: 10
Mode: Pulse
Percent Duty Knob: 50 percent

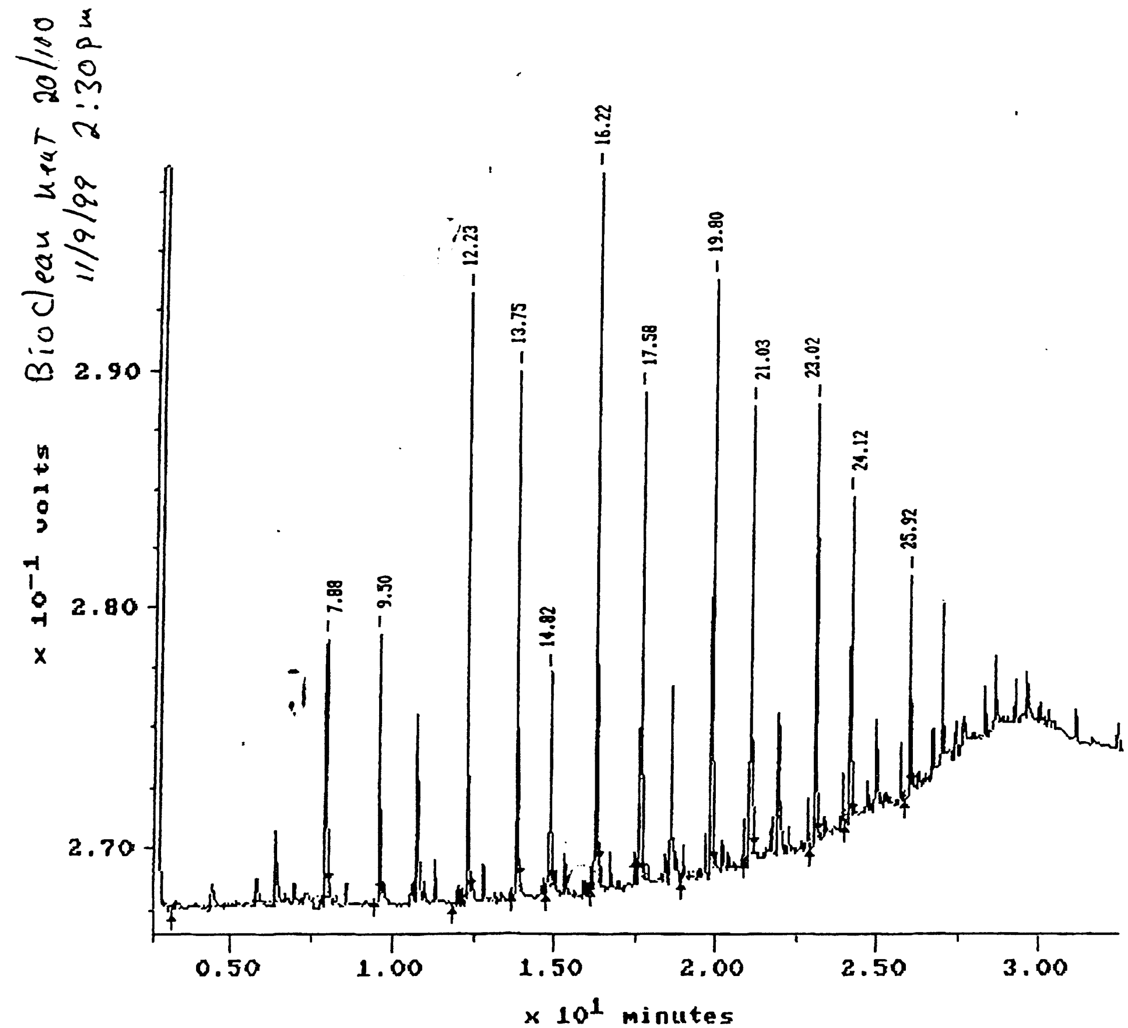
Sample Clean-up Procedure

When samples contain a significant amount of naturally occurring non-petroleum organics, e.g. leaf litter, bark, etc., which may contribute to biogenic interference, the following clean-up technique may be employed to assist in their reduction or elimination.

1. Transfer 2 ml of the sample extract to a 4-ml vial
2. Add 0.3 to 0.5 ml concentrated sulfuric acid to the vial and shake for one minute
3. Allow the phases to separate and transfer the upper layer to another 4-ml vial
4. Add about .4 g of silica to the vial and shake
5. Repeat the procedure a second time; transfer the cleaned extract to an auto sampler vial for analysis
6. If the clean-up affects the analyte of interest, clean the standards in the same way as the samples



3.60
3.40
3.20
3.00
2.80
x 10^{-1} volts
0.50
1.00
1.50
2.00
2.50
3.00
x 10^{1} minutes
12.32
13.75
16.75
18.32
23.48


BioClean neat 20/100
11/9/99 2:30pm
x 10^-1 volts
2.90
2.80
2.70
7.88
9.30
12.23
13.75
14.82
16.22
17.58
19.80
21.03
23.02
24.12
25.92
0.50
1.00
1.50
2.00
2.50
3.00
x 10^1 minutes

APPENDIX B

Solvent Extraction and Oil Loading Data

SOLVENT EXTRACTION OF PARTS

SAMPLE I.D.	PART DESCRIPTION	QUANTITY OF ACETONE (L)	AVG. PART WEIGHT (lb./part)	PART SURFACE AREA (ft2)	AVG. OIL(g)/ft2	AVG. OIL IN ONE BARREL (lbs.)	SOLVENT EXTRACT ANALYSIS (mg/l oil)
21901121	ubolt - threaded	0.35	0.0541	0.025	2.300	0.25	820
117.3H.1	flat corner iron	0.9	0.0519	0.03	0.096	0.01	16
.30S1H1AJ	plastic staple zinc	0.7	0.0453	0.035	0.032	0.00	8
.22….5	bar	0.7	0.1069	0.042	0.013	0.00	4
20762..1	heavy open s-hook	0.7	0.0646	0.025	0.022	0.00	3.9
113.2HL1J	90 degree corner	0.9	0.0406	0.049	0.059	0.01	16
.27….1	large part, eye holes	0.95	0.3605	0.23	0.990	0.11	1200
2169E3B1	LH HOOKBOLT	0.6	0.0653	0.0238	0.010	0.48	270
WRP121DC	SCREWS/RO/PHIL/D BLK	0.6	0.0107	0.0069	0.000	0.00	0.8
171….1C	6 1/2 PULL HANDL3E	0.6	0.1778	0.1198	0.011	0.57	320
LH.D1H.C.	LAG SCREW/HEX MD	0.6	0.0195	0.01	0.003	0.14	77
PHT.5..1	PIN STYLE HEAVY TIGHT	0.6	0.1015	0.034	0.001	0.06	34
B36..H125	CHAIN BOLTS	0.6	0.1717	1	0.002	0.11	60
1134…1	1134 ASSEMBLY/DEAD BLAC	0.6	0.261	0.256	0.004	0.19	110
MCCD3	1/4 - 20x3 BOLT/CGE/S4	0.6	0.0374	0.015	0.014	0.71	400
C5805625	4x4 PLATE	1.8	0.4565	0.192	0.004	0.19	110
.30S1B1A	1 - 1/8x1 PLT STAPLE ZINC	0.6	0.0261	0.2	0.000	0.01	4.9
20533H.1	3 1/2 TARP HOOK	0.6	0.1199	0.045	0.005	0.23	130
.32.6..1	HASP FRAME	0.9	0.2333	0.17	0.003	0.13	74
PLBULH.1	PIN STL LT BUTTON HEAD	0.6	0.0044	0.0043	0.001	0.06	30

* 5 representative parts were taken for each sample I.D.
* A total of 50 ft2 is placed in each barrel for plating.
* 19 barrels/hr. are plated on each line
* National runs 5 days/week with 2 - 8 hr. shifts, occasionally there is a need to run a 3rd shift

National Manufacturing Production
4/11/00 - 4/13/00

Date Started	Part	Part Description	Threaded Finish/Size	Start Time	Quantity Completed	Surface Area ft2	Average Weight	Total Surface Area Processed ft2	# barrels	# barrels each day	Estimated Oil (lbs.)/barrel	Estimated Oil (lbs.)	Estimated Oil for the day (lbs.)
4/11/2000	115.2J.1J	Corner Iron	No/medium	4:45A	174,000	0.045	0.0603	7830	157	**236**	0.23	36.0	**50**
	836.H12J5	Cane Bolt	No/large	11:00A	3,479	.5000	0.717	1740	35		0.11	3.7	
	195.42.1	Turnbuckle Rod	No/large	1:15P	19,550	0.07	0.1152	1369	27		0.23	6.3	
	195.42.2	Turnbuckle Rod	No/large	4:45P	6,500	0.07	0.1071	455	9		0.23	2.1	
	175.3H.2J	Handle Plate	No/medium	6:00P	6,700	0.0604	0.204	405	8		0.23	1.9	
4/12/2000	175.3H.2J	Handle Plate	No/medium	5:00A	6,366	0.0604	0.204	385	8	**210**	0.23	1.8	**17**
	115.2J.1J	Corner Iron	No/medium	5:45A	134,880	0.045	0.0603	6070	121		0.01	0.8	
	20400..1	Ceiling Hook	Yes/large	10:45A	18,952	0.05	0.01364	948	19		0.01	0.1	
	195.42.2	Turnbuckle Rod	No/large	12:15P	39,219	0.07	0.1071	2745	55		0.23	12.6	
	20533H.1	Tarp Hook	No/medium	9:15P	7,600	0.045	0.1199	342	7		0.23	1.6	
4/13/2000	20533H.1	Tarp Hook	No/medium	8:30A	36,490	0.045	0.1199	1642	33	**109**	0.23	7.6	**20**
	20140..1	Screw Eye	Yes/large	11:00A	31,079	0.035	0.0936	1088	22		0.00	0.0	
	195.42.2	Turnbuckle Rod	No/large	12:30P	15,513	0.07	0.1071	1086	22		0.23	5.0	
	195.42.1	Turnbuckle Rod	No/large	4:30P	23,160	0.07	0.1152	1621	32		0.23	7.5	
4/11/2000	2217L..1	Grip Clip	No/medium	7:00A	25,188	0.057	0.0345	1436	29	**227**	0.01	0.2	**69**
	32113..B	Pulley Frame	No/medium	10:00A	2,400	0.327	0.5717	785	16		0.19	3.1	
	32173..2	Pulley Hook	No/small	11:15A	5,900	0.055	0.1442	325	6		0.23	1.5	
	32173..1	Pulley Scissor	No/small	11:45A	3,900	0.055	0.1406	215	4		0.23	1.0	
	32113..B	Pulley Scissor	No/small	12:00P	2,137	0.327	0.5717	699	14		0.19	2.7	
	32153..1	Pulley Scissor	No/small	2:00P	2,360	0.055	0.1402	130	3		0.23	0.6	
	508.1..2J	Cabinet hinge	No/small	2:20P	33,000	0.0078	0.008	257	5		0.00	0.0	
	508.1..1J	Cabinet hinge	No/small	2:40P	33,000	0.0078	0.0095	257	5		0.00	0.0	
	176….1	Handle Plate	No/medium	3:00P	2,138	0.192	0.4463	410	8		0.19	1.6	
	508.3..2J	Cabinet hinge	No/medium	4:40P	19,859	0.105	0.0707	2085	42		0.57	23.6	
	508.3..1J	Cabinet hinge	No/medium	5:50P	19,706	0.105	0.0748	2069	41		0.57	23.4	
	504.3..1	Door hinge	No/medium	6:50P	10,000	0.0775	0.1343	775	16		0.23	3.6	
	504.3..2	Door hinge	No/medium	8:00P	10,000	0.0775	0.127	775	16		0.23	3.6	
	1134….AJ	Gate Latch	No/medium	10:00P	4,350	0.256	0.261	1114	22		0.19	4.3	
4/12/2000	1134….AJ	Gate Latch	No/medium	5:00A	1,054	0.256	0.261	270	5	**315**	0.19	1.0	**145**
	.30S1B1AJ	Staple Plate	No/small	5:15A	26,820	0.02	0.0261	536	11		0.71	7.6	
	835.J181J	Bracket	No/medium	6:00A	4,000	0.0825	0.0806	330	7		0.01	0.0	
	32112..9	Pulley Sheave	No/medium	6:50A	44,930	0.0436	0.1356	1959	39		0.00	0.1	
	2169E3B1	Hook bolt	Yes/large	1:30P	8,530	0.0238	0.0653	203	4		0.48	1.9	
	20148..1	Screw Eye	Yes/large	2:00P	53,224	0.01	0.0152	532	11		0.14	1.4	
	25391L.1	Hanger	No/large	2:40P	20,005	0.036	0.0365	720	14		0.00	0.1	
	.32S…2J	Spring	Yes/large	3:35P	228,021	0.016	0.0013	3648	73		0.71	51.4	
	288.3..1	Hasp	No/large	4:20P	20,045	0.095	0.0461	1904	38		0.57	21.5	
	288.3..2	Hasp	No/large	5:00P	20,045	0.095	0.044	1904	38		0.57	21.5	
	504.3..1	Door hinge	No/medium	5:40P	10,000	0.0775	0.1343	775	16		0.23	3.6	
	504.3..2	Door hinge	No/medium	7:00P	10,000	0.0775	0.127	775	16		0.23	3.6	
	508.2..1J	Cabinet hinge	No/medium	8:25P	50,000	0.022	0.0361	1100	22		0.71	15.5	
	508.2..2J	Cabinet hinge	No/medium	9:45P	50,000	0.022	0.0352	1100	22		0.71	15.5	
4/13/2000	286.4..5	Pin	No/medium	5:00A	45,000	0.019	0.0419	855	17	**309**	0.71	12.1	**33**
	504.3..1	Door hinge	No/medium	6:00A	56,181	0.0775	0.1343	4354	87		0.23	20.0	

National Manufacturing Production
4/1100 – 4/13/00
High Oil Load Oil Added

Date Started	Part Number	Part Description	Threaded Finish/Size	Start Time	Quantity Completed	Surface Area ft2	Average Weight	Total Surface Area Processed ft2	# barrels	# barrels each day	Estimated Oil (lbs.)/barrel	Estimated Oil (lbs.)	Estimated Oil for the day (lbs.)
4/13/2000	508.2H.2J	Cabinet hinge	No/medium	6:45A	29,218	0.04	0.043	1169	23		0.00	0.1	
	508.2H.1J	Cabinet hinge	No/medium	7:45A	29,120	0.04	0.0471	1165	23		0.00	0.1	
	284.3..1	Gate Hinge	No/medium	8:45A	76,000	0.035	0.0425	2660	53		0.00	0.2	
	284.3..2	Gate Hinge	No/medium	11:00A	74,200	0.04	0.0488	2968	59		0.00	0.2	
	284.3..1	Gate Hinge	No/medium	2:00P	47,334	0.035	0.0425	1657	33		0.00	0.1	
	504.3..1	Door hinge	No/medium	3:40P	8,200	0.0775	0.1343	636	13		0.00	0.1	
4/11/2000	.21….1C	Case	No/small	6:00A	3,695	0.13	0.2007	480	10	**252**	0.57	5.4	**96**
	1134…AC	Gate Latch	No/medium	6:35A	0	0.256	0.261	0	0		0.19	0.0	
	..2….C4	Pull/handle	No/medium	10:35A	1,418	0.077	0.0926	109	2		0.01	0.0	
	171….1C	Pull/handle	No/large	10:55A	3,500	0.1198	0.1778	419	8		0.57	4.7	
	.24A…C4	Latch	No/small	11:40A	3,000	0.135	0.896	405	8		0.57	4.6	
	834.2H.BC	Case	No/medium	11:45A	4,500	0.075	0.0472	338	7		0.01	0.0	
	1134…AC	Gate Latch	No/medium	12:05P	16,680	0.256	0.261	4270	85		0.19	16.6	
	.21….5C	Latch bar	No/medium	10:00A	9,333	0.042	0.1382	392	8		0.00	0.0	
	.21A…1J	Latch	No/small	10:40A	15,000	0.059	0.1001	885	18		0.23	4.1	
	.21….1C	Case	No/small	11:40A	40,850	0.13	0.2007	5311	106		0.57	60.0	
4/12/2000	1134….AC	Gate Latch	No/medium	7:30A	5,529	0.256	0.261	1415	28	**165**	0.19	5.5	**29**
	1134…2C	Strike	No/medium	9:30A	10,000	0.069	0.0715	690	14		0.01	0.1	
	286.6..C4	Gate Hinge	No/large	10:05A	3,600	0.282	0.364	1015	20		0.19	3.9	
	286.6..CO	Gate Hinge	No/large	12:00P	945	0.282	0.364	266	5		0.19	1.0	
	1134…AC	Gate Latch	No/medium	9:00	18,975	0.256	0.261	4858	97		0.19	18.9	
4/13/2000	836.H125C	Cane Bolt	No/large	6:00A	7,818	0.149	0.717	1165	23	**148**	0.13	3.0	**25**
	WRP121HC.	Screw	Yes/small	11:30A	120,000	0.0081	0.0121	972	19		0.00	0.0	
	WRP121DC.	Screw	Yes/small	12:20P	206,127	0.0069	0.0107	1422	28		0.00	0.0	
	TTP71H.C.	Tap Screw	Yes/small	1:35P	60,000	0.005	0.0068	300	6		0.00	0.0	
	LH.D1H.C.	Lag	Yes/small	1:55P	20,000	0.01	0.0195	200	4		0.14	0.5	
	286.6..CO	Gate Hinge	No/large	10:00A	1,455	0.282	0.364	410	8		0.19	1.6	
	MS850A.C.	Stud	No/medium	10:45A	21,520	0.062	0.1395	1334	27		0.23	6.1	
	MB50A..HC	Axle	No/medium	1:25P	50,000	0.017	0.0391	850	17		0.71	12.0	
	836.H125C	Cane Bolt	No/large	2:25P	5,124	0.149	0.717	763	15		0.13	2.0	
					2,238,422 pieces			**35,738 ft^2**		**715**	***4/11/2000***	**lbs.**	**214**
								34491 ft^2		**690**	***4/12/2000***	**lbs.**	**191**
								28317 ft^2		**566**	***4/13/2000***	**lbs.**	**78**
							Total	**98,546 ft^2**				**lbs.**	**484**
								2					**219,539g**

National Manufacturing Production
4/1100 – 4/13/00
Low Oil Load Oil Added

Date Started	Part Number	Part Description	Threaded/ Size	Start Time	Quantity Completed	Surface Area	Average Weight	Total Surface Area Processed	# barrels	Total # Barrels	Estimated Oil (lbs.)/barrel	Estimated Oil (lbs.)	Estimated Oil for the day (lbs.)
5/2/2000	2060…J*	Hitch Ring	No/medium	6:45 AM	29,044	0.106	0.2586	3079	62	**193**	0.57	35.0	64
	2062E3DJ*	Lag Eyebolt	Yes/large	11:45 AM	7,895	0.268	0.2389	2116	42		0.19	8.2	
	2160F6.1	Eyebolt	Yes/large	12:30 PM	73,998	0.06	0.2015	4440	89		0.23	20.5	
5/3/2000	2160F6.1	Eyebolt	Yes/large	4:45 AM	5,000	0.06	0.2015	300	6	**223**	0.23	1.4	42
	2168F5B1	Hookbolt	Yes/large	5:30 AM	15,758	0.045	0.1478	709	14		0.23	3.2	
	2160F4.1	Eyebolt	Yes/mediu m	7:30 AM	67,107	0.06	0.2015	4026	81		0.23	18.6	
	.76.4..1	Spring	No/large	1:00 PM	25,875	0.215	0.1878	5563	111		0.11	12.1	
	2160H101	Eyebolt	Yes/large	8:15 PM	4,254	0.125	0.5287	532	11		0.57	6.2	
5/4/2000	2160H101	Eyebolt	Yes/large	7:00 AM	1,044	0.125	0.5287	131	3	**156**	0.57	1.7	68
	171…1J	Handle	No/medium	7:15 AM	39,400	0.1198	0.1778	4720	94		0.57	53.1	
	2160F4.1	Eyebolt	Yes/mediu m	12:45 PM	48,908	0.06	0.2015	2934	59		0.23	13.6	
					322,083	pieces	5/2/2000	9634 ft^2		**572**			**174**
							5/3/2000	11130 ft^2					
							5/4/2000	7785 ft^2					
							Total	28550 ft^2					79,700 g

APPENDIX C

Bulk Microbe Analysis Results

BioClean Bulk Microbial Analysis –
High Oil Load

SAMPLE I.D.	SAMPLE LOCATION	SAMPLE TIME	SAMPLE DATE	TSA (CFU/ml)	YME (CFU/ml)	MAC (CFU/ml)	Total (CFU/ml)
HOL1-020	Separator Inlet	1035	4/11/00	4,700,000	650	400,000	5,100,650
HOL1-021	Separator Inlet	1335	4/11/00	4,400,000	200	290,000	4,690,200
HOL1-022	Separator Inlet	1505	4/11/00	1,500,000	70	272,000	1,772,070
HOL1-023	Separator Effluent	1035	4/11/00	4,100,000	220	100,000	4,200,220
HOL1-024	Separator Effluent	1335	4/11/00	8,000,000	250	308,000	8,308,250
HOL1-025	Separator Effluent	1505	4/11/00	1,500,000	150	305,000	1,805,150
HOL1-026	Waste Solids Sludge (test start)	0934	4/11/00	450,000	100	260,000	710,100
HOL1-027	Algae in Holding Tank	1335	4/11/00	2,000,000 CFU/gram	1,000 CFU/gram	50,000 CFU/gram	
HOL2-065	Separator Inlet	1000	4/12/00	800,000	< 1	92,000	892,000
HOL2-066	Separator Inlet	1320	4/12/00	2,700,000	6,000	10,000	2,716,000
HOL2-067	Separator Inlet	1515	4/12/00	2,600,000	5	200,000	2,800,005
HOL2-068	Separator Effluent	1000	4/12/00	2,300,000	< 1	70,000	2,370,000
HOL2-069	Separator Effluent	1320	4/12/00	7,200,000	10	226,000	7,426,010
HOL2-070	Separator Effluent	1515	4/12/00	8,600,000	20	10,000	8,610,020
HOL3-110	Separator Inlet	0830	4/13/00	24,700,000	10	600,000	25,300,010
HOL3-111	Separator Inlet	1100	4/13/00	12,700,000	10	250,000	12,950,010
HOL3-112	Separator Inlet	1500	4/13/00	31,100,000	< 1	350,000	31,450,000
HOL3-113	Separator Effluent	0830	4/13/00	33,100,000	< 1	500,000	33,600,000
HOL3-114	Separator Effluent	1100	4/13/00	41,400,000	< 1	520,000	41,920,000
HOL3-115	Separator Effluent	1500	4/13/00	51,300,000	< 1	420,000	51,720,000
HOL3-116	Make-up Water			300	30		330
HOL3-118	Waste Solids Sludge (test end)	1500		16,000,000	30	580,000	16,580,030
HOL3-177	Oil Coating			< 1	< 1		
HOL3-178	Oil Coating			69,000,000	20	1300000	
HOL3-179	Oil Coating			100	20		

BioClean Bulk Microbial Analysis –
Low Oil Load

SAMPLE I.D.	SAMPLE LOCATION	SAMPLE TIME	SAMPLE DATE	TSA (CFU/ml)	YME (CFU/ml)	MAC (CFU/ml)	BULK TOTAL (CFU/ml)
LOL1-002	Make-up Water	1315	5/3/00	< 1	10		10
LOL1-009	Separator Inlet	0815	5/2/00	12,000,000	< 1	950,000	12,950,000
LOL1-010	Separator Inlet	1230	5/2/00	113,900,000	10	920,000	114,820,010
LOL1-011	Separator Inlet	1500	5/2/00	12,600,000	20	1,400,000	14,000,020
LOL1-012	Separator Effluent	0815	5/2/00	6,000,000	10	300,000	6,300,010
LOL1-013	Separator Effluent	1230	5/2/00	26,700,000	15	1,600,000	28,300,015
LOL1-014	Separator Effluent	1500	5/2/00	29,200,000	10	930,000	30,130,010
LOL1-015	Waste Solids Sludge	0815	5/2/00	8,100,000	10	260,000	8,360,010
LOL1-016	Compressor Cond.	1500	5/3/00	1,300	10		1,310
LOL2-044	Separator Inlet	0930	5/3/00	18,900,000	< 1	1,100,000	20,000,000
LOL2-045	Separator Inlet	1315	5/3/00	14,200,000	< 1	500,000	14,700,000
LOL2-046	Separator Inlet	1500	5/3/00	18,900,000	10	2,300,000	21,200,010
LOL2-047	Separator Effluent	0930	5/3/00	60,000,000	< 1	3,600,000	63,600,000
LOL2-048	Separator Effluent	1315	5/3/00	10,000,000	< 1	6,000,000	16,000,000
LOL2-049	Separator Effluent	1500	5/3/00	10,000,000	10	2,300,000	12,300,010
LOL3-076	Separator Inlet	1015	5/4/00	7,400,000	10	330,000	7,730,010
LOL3-077	Separator Inlet	1305	5/4/00	4,800,000	15	40,000	4,840,015
LOL3-078	Separator Inlet	1530	5/4/00	5,900,000	30	160,000	6,060,030
LOL3-079	Separator Effluent	1010	5/4/00	30,400,000	50	2,100,000	32,500,050
LOL3-080	Separator Effluent	1300	5/4/00	33,100,000	100	1,300,000	34,400,100
LOL3-081	Separator Effluent	1530	5/4/00	41,000,000	110	1,600,000	42,600,110
LOL3-103	Waste Solids Sludge	1530	5/4/00	130,000,000	120	3,900,000	133,900,120

BioClean Bulk Microbial Analysis – Spiked Oil Load

SAMPLE I.D.	SAMPLE LOCATION	SAMPLE TIME	SAMPLE DATE	TSA (CFU/ml)	YME (CFU/ml)	MAC (CFU/ml)	BULK TOTAL (CFU/ml)
SOL1-012	Separator Inlet	2022	5/19/2000	10	20	10	40
SOL1-013	Separator Inlet	0208	5/20/2000	3,000	< 5	200	3,200
SOL1-014	Separator Inlet	0630	5/20/2000	30,000	10	3,100	33,110
SOL1-015	Separator Effluent	2025	5/19/2000	500,000	< 5	400,000	900,000
SOL1-016	Separator Effluent	0205	5/20/2000	180,000	< 5	2,000	182,000
SOL1-017	Separator Effluent	0630	5/20/2000	50,000	20	2,000	52,020
SOL1-018	Waste Solids Sludge	2025	5/19/2000	10,000,000 CFU/gram	< 5 CFU/gram	200 CFU/gram	10,000,205 CFU/gram
SOL2-028	Separator Inlet	2050	5/20/2000	169,000	< 5	400,000	569,000
SOL2-029	Separator Inlet	0055	5/21/2000	100	< 5	10	110
SOL2-030	Separator Inlet	0450	5/21/2000	5,000	< 5	900	5,900
SOL2-031	Separator Effluent	2050	5/20/2000	520,000	< 5	3,000	523,000
SOL2-032	Separator Effluent	0105	5/21/2000	200,000	< 5	1,000	201,000
SOL2-033	Separator Effluent	0450	5/21/2000	210,000	< 5	2,000	212,000
SOL3-043	Separator Inlet	2030	5/21/2000	300,000	< 5	300,000	600,000
SOL3-044	Separator Inlet	0025	5/22/2000	16,000	< 5	4,400	20,400
SOL3-045	Separator Inlet	0430	5/22/2000	237,000	< 5	275,000	512,000
SOL3-046	Separator Effluent	2030	5/21/2000	160,000	< 5	4,000	164,000
SOL3-047	Separator Effluent	0030	5/22/2000	520,000	< 5	2,000	522,000
SOL3-048	Separator Effluent	0425	5/22/2000	820,000	< 5	1,000	821,000
SOL3-049	Waste Solids Sludge	0430	5/22/2000	1,600,000	< 5	96,000	1,696,000
SOL3-052	Neat Oil - Towerdraw G943	n/a	n/a	< 10	220	< 10	240
SOL3-053	Make-up Water (hose by process line)	0230	5/20/2000	860	10	< 10	880

APPENDIX D

Bulk Chemical Analysis

BioClean Chemical Analysis, High Oil Load

Sample ID	Sample Location	Sample Date	Sample Time	Oil (g/l)	TS (mg/l)	TSS (mg/l)	TOC (mg/l)	Copper (mg/l)	Zinc (mg/l)
HOL1-001	Waste solids sludge	4/11/2000	0934	28.0	170,000	150,000	19,000	<0.1	5,400
HOL1-004	Cleaner bath 1	4/11/2000	0948	4.0	17,000	3,900		3.6	120
HOL1-005	Cleaner bath 1	4/11/2000	1256	6.0	13,000	8,300		10.0	540
HOL1-006	Cleaner bath 1	4/11/2000	1700	5.0	15,000	7,200		8.6	500
HOL1-008	Cleaner bath 2	4/11/2000	0950	5.0	14,000	4,800		4.0	140
HOL1-009	Cleaner bath 2	4/11/2000	1255	6.0	20,000	8,700		7.8	380
HOL1-010	Cleaner bath 2	4/11/2000	1700	5.0	17,000	8,000		10.0	640
HOL1-012	Cleaner bath 3	4/11/2000	0953	6.0	18,000	6,700		6.1	560
HOL1-013	Cleaner bath 3	4/11/2000	1300	6.0	27,000	8,500		6.2	190
HOL1-014	Cleaner bath 3	4/11/2000	1700	6.0	17,000	8,700		10.0	690
HOL1-018	Separator effluent	4/11/2000	1700	5.0	13,000	6,600		12.0	840
HOL2-049	Cleaner bath 1	4/12/2000	1310	4.0	11,000	11,000		26.0	450
HOL2-051	Cleaner bath 1	4/12/2000	1600	3.0	11,000	10,000		22.0	360
HOL2-055	Cleaner bath 2	4/12/2000	1600	5.0	12,000	10,000		23.0	360
HOL2-059	Cleaner bath 3	4/12/2000	1600	3.0	11,000	10,000		22.0	350
HOL2-061	Separator effluent	4/12/2000	1320	5.0	11,000	10,000		24.0	330
HOL2-063	Separator effluent	4/12/2000	1600	5.0	10,000	8,700		23.0	300
HOL3-094	Cleaner bath 1	4/13/2000	1130	3.0	11,000	7,700		19.0	260
HOL3-096	Cleaner bath 1	4/13/2000	1630	12.0	10,000	6,500		16.0	150
HOL3-098	Cleaner bath 2	4/13/2000	1130	29.0	12,000	7,500		19.0	270
HOL3-100	Cleaner bath 2	4/13/2000	1630	16.0	11,000	7,800		17.0	270
HOL3-102	Cleaner bath 3	4/13/2000	1130	4.0	12,000	9,900		21.0	350
HOL3-104	Cleaner bath 3	4/13/2000	1630	29.0	11,000	7,900		18.0	280
HOL3-106	Separator effluent	4/13/2000	1130	6.0	9,400	5,400		19.0	280
HOL3-108	Separator effluent	4/13/2000	1630	6.0	11,000	6,900		16.0	220

BioClean Chemical Analysis, Low Oil Load

Sample ID	Sample Location	Sample Date	Sample Time	Oil (g/l)	TS (mg/l)	TSS (mg/l)	TOC (mg/l)	Copper (mg/l)	Zinc (mg/l)
LOL1-001	Waste solids sludge	5/2/2000	0815	20.0	110,000	98,000	37,000	220.0	17,000
LOL1-003	Cleaning bath 3	5/2/2000	0815	7.0	15,000	7,400		11.0	350
LOL1-004	Cleaning bath 3	5/2/2000	1230	10.0	14,000	7,100		11.0	290
LOL1-005	Cleaning bath 3	5/2/2000	1630	8.0	15,000	6,700		11.0	320
LOL1-006	Separator effluent	5/2/2000	0815	4.0	13,000	4,300		11.0	190
LOL1-007	Separator effluent	5/2/2000	1230	8.0	14,000	4,700		10.0	240
LOL1-008	Separator effluent	5/2/2000	1630	8.0	14,000	5,600		11.0	220
LOL2-037	Cleaning bath 3	5/3/2000	1203	11.0	14,000	7,400		10.0	300
LOL2-039	Cleaning bath 3	5/3/2000	1315	11.0	12,000	8,400		13.0	370
LOL2-040	Cleaning bath 3	5/3/2000	1615	13.0	14,000	7,400		11.0	350
LOL2-041	Separator effluent	5/3/2000	0930	10.0	15,000	6,600		9.4	250
LOL2-042	Separator effluent	5/3/2000	1315	14.0	13,000	7,300		12.0	290
LOL2-043	Separator effluent	5/3/2000	1615	14.0	12,000	7,100		10.0	300
LOL3-070	Cleaning bath 3	5/4/2000	1015	12.0	11,000	6,000		10.0	370
LOL3-071	Cleaning bath 3	5/4/2000	1305	17.0	15,000	7,200		8.9	310
LOL3-072	Cleaning bath 3	5/4/2000	1630	17.0	14,000	6,800		12.0	390
LOL3-073	Separator effluent	5/4/2000	1010	13.0	14,000	6,300		9.1	220
LOL3-074	Separator effluent	5/4/2000	1300	11.0	15,000	7,800		8.1	250
LOL3-075	Separator effluent	5/4/2000	1630	9.0	12,000	6,100		12.0	310

BioClean Chemical Analysis, Spiked Oil Load

Sample ID	Sample Location	Sample Date	Sample Time	Oil (g/l)	TS (mg/l)	TSS (mg/l)	TOC (mg/l)	Copper (mg/l)	Zinc (mg/l)
SOL1-001	Waste solids sludge	5/19/2000	2025	9.0	120000	130000	48000	190	12000
SOL1-002	Holding Tank	5/19/2000	2025	15.0	12000	8800		7.3	250
SOL1-003	Cleaner bath 3	5/19/2000	2020	10.0	13000	8800		7	250
SOL1-004	Cleaner bath 3	5/20/2000	0208	13.0	13000	9400		5.7	220
SOL1-005	Cleaner bath 3	5/20/2000	0630	13.0	14000	9500		5.6	250
SOL1-009	Separator effluent	5/19/2000	2025	11.0	13000	7500		4.8	160
SOL1-010	Separator effluent	5/20/2000	0205	20.0	13000	8200		5.7	210
SOL1-011	Separator effluent	5/20/2000	0630	18.0	14000	9300		6.2	230
SOL2-019	Cleaner bath 3	5/20/2000	2050	13.0	14000	8900		4.6	230
SOL2-020	Cleaner bath 3	5/21/2000	0055	12.0	14000	9000		4.1	210
SOL2-021	Cleaner bath 3	5/21/2000	0450	14.0	14000	8900		3.1	170
SOL2-025	Separator effluent	5/20/2000	2050	9.0	14000	9100		4.9	220
SOL2-026	Separator effluent	5/21/2000	0105	Sample jar broke in transit					
SOL2-027	Separator effluent	5/21/2000	0450	17.0	15000	8500		2	180
SOL3-034	Cleaner bath 3	5/21/2000	2030	9.0	14000	8100		3.9	190
SOL3-035	Cleaner bath 3	5/22/2000	0030	12.0	13000	9700		6.3	240
SOL3-036	Cleaner bath 3	5/22/2000	0430	15.0	13000	9700		6.3	240
SOL3-040	Separator effluent	5/21/2000	2030	21.0	14000	7700		3.5	160
SOL3-041	Separator effluent	5/22/2000	0025	18.0	14000	8400		5.3	200
SOL3-042	Separator effluent	5/22/2000	0425	16.0	14000	8900		6	240
SOL3-050	Holding Tank	5/22/2000	0425	22.0	14000	8300		6.1	220
SOL3-051	Waste solids sludge	5/22/2000	0430	9.0	100000	61000	39000	130	7300