U.S. ENVIRONMENTAL PROTECTION AGENCY

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

Revision 0

February 4, 2000

Concurrent Technologies Corporation is the Verification Partner for the EPA ETV Metal Finishing Pollution PreventionTechnologies Pilot under EPA Cooperative Agreement No. CR826492-01-0.

U.S. Environmental Protection Agency
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

Prepared by: ETV-MF Program and
BioClean USA, LLC

TITLE: EVALUATION OF BIOCLEAN USA, LLC BIOLOGICAL DEGREASING SYSTEM FOR THE RECYCLING OF ALKALINE CLEANERS

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Environmental Technology Verification Program For Metal Finishing Pollution Prevention Technologies Verification Test Plan for the Evaluation of BioClean USA, LLC Biological Degreasing System for the Recycling of Alkaline Cleaners.

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1.0 INTRODUCTION

The purpose of this test plan is to document the objectives, procedures, equipment, and other aspects of testing that will be utilized at National Manufacturing Company, during verification testing an aqueous biological degreasing and recycling system manufactured by BioClean USA, LLC. BioClean's Biological Degreasing System is a pollution prevention technology designed for conventional soak or spray cleaning operations in the metal finishing industry. This test plan has been prepared to evaluate the performance of the technology in conjunction with the U.S. Environmental Protection Agency's (EPA's) Environmental Technology Verification for Metal Finishing Pollution Prevention Technologies (ETV-MF) Program. The objective of this program is to identify promising and innovative pollution prevention technologies through EPA-supported performance verifications. The results of the verification test will be documented in a verification report which will provide objective performance data to metal finishers, environmental permitting agencies, and consultants.

BioClean USA, LLC, Bridgeport, Connecticut, distributes an alkaline cleaning solution and control system that utilizes microbes in the solution to consume the organic soils that are removed from parts during the cleaning process. This technology is used in soak or spray cleaning operations. The BioClean technology was developed in Europe, and is now being distributed in the US, Canada, and Mexico. Installed applications include cleaning operations for powder coating, plating and anodizing lines (barrel and rack), metal stamping, and forming operations.

National Manufacturing Company, where the technology has been installed and operating for approximately 13 months, was selected by BioClean as the test site for this technology. The National Manufacturing Company designs and manufactures hardware, such as doorknobs, hinges, staple plates, coat and hat hooks, bolts, chest handles, and numerous others. Their operations virtually contain 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. Their metal finishing operations consist of rack and barrel production lines for the plating of the following metals: zinc, brass, nickel, and chromium.

This project will evaluate the ability of BioClean's system to extend the bath life in National Manufacturing's alkaline cleaning operation. Evaluating and verifying the performance of BioClean's system will be accomplished by collecting operational data and in-process samples for analysis. The resultant test data will be used to prepare a mass balance and determine the efficiency of oil removal under specific operating conditions. An important component of the verification testing of a microbiological alkaline cleaner recycling system is the quantification of the biological populations (bacteria and fungi) at selected locations within the aqueous system. The characterization of the microbial population (to assess microbial response to the oil) along with the quantification of bio-aerosol generation within the work area (to assess potential health and safety risks) during various stages of the BioClean process provides important information to project managers, technicians, operators, and risk assessors regarding the process variables. Also important to evaluate is the environmental benefit of each verified technology. The benefit of an alkaline cleaner recycling system can be quantified by determining the reduction of bath dumps over a period of time. The BioClean system was installed prior to initiation of verification testing so this benefit will not be verified but will be obtained from historical data collected by National Manufacturing.

The test plan described in this document has been structured to allow the above objectives to be met using sound scientific principles. This document explains testing plans with respect to areas such as test methodology, procedures, parameters, and instrumentation. Section 5.0 describes Quality Assurance/Quality Control requirements of this task that will ensure the accuracy of data. Also presented within this document are data interpretation procedures and hypothesized results. Worker health and safety considerations are covered in Section 8.0.

This test plan will be maintained at the test site, and verification testing will be conducted in strict adherence to the test plan requirements.

1.1 Background

The surface finishing and assembly industries require that oils, coolants, and other metal-working fluids be removed from parts prior to finishing, assembly, or packaging. Cleaning stamped, diecast, or formed parts prior to subsequent processing has become increasingly difficult and expensive due to more stringent environmental requirements and a dramatic rise in waste treatment and disposal costs. For example, environmental protection authorities are continuously strengthening regulations concerning the use and disposal of trichloroethylene and chemicals containing nonylphenol etoxylate, which the cleaning and finishing industry uses to clean organic soil off stamped, formed, and mechanical parts prior to finishing (e.g., plating, painting, and powder coating). Until recently, waste reduction in surface preparation operations focused on conserving these organic solvent cleaners, because for years the metal finishing industry relied on organic solvents for cleaning metal parts. During the 1980's, however, environmental concerns for health and disposal consequences increased, and metal finishers began to turn to other options for their cleaning operations. With the metal finishing industry moving away from solvent technology, aqueous cleaning has emerged as a viable alternative [Ref. 1].

The tank life of alkaline cleaners is limited by the buildup of oil, grease, and other contaminants in the bath. When contaminants in the bath begin to sacrifice product quality, it becomes necessary to discard the cleaning solution. Although the alkaline cleaners do not carry all of the risks and liabilities associated with the disposal of waste organic solvents, most cleaners are difficult to treat, because they contain components that can be difficult to break down. Periodic replacement of the bath results in high costs due to disposal and replacement chemicals. Hence, an efficient and environmentally friendly cleaning and recycling system that can extend the usable life of the cleaning bath will have the desirable effects of consistent performance, life extension of solutions down-line, waste reduction, reduction in labor costs, reduction in raw material costs, and ultimately, lower costs of operation.

2.0 TECHNOLOGY DESCRIPTION

2.1 Theory of Operation

The idea of using microbes to consume oil is not revolutionary. For over 40 years they have been utilized to consume oil from oil spills. BioClean's 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. BioClean's chemistry was constructed around the characteristics of the microbe.

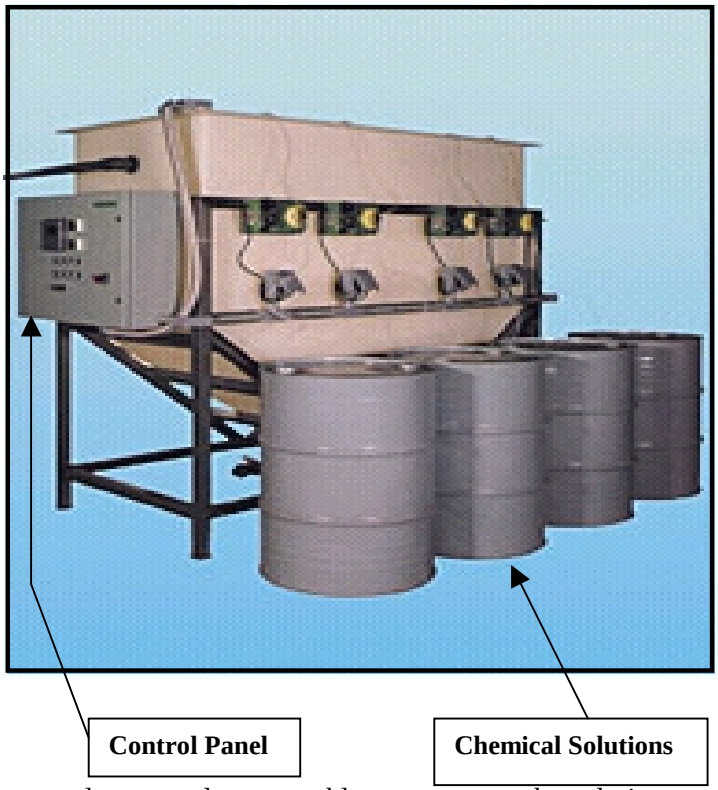
The BioClean system employs a mild alkaline bath or spray that operates at relatively low temperatures (104ºF – 131ºF) (40ºC - 55ºC) and a pH range of 8.8 - 9.2, which is a viable habitat for these microorganisms. The cleaning solution contains biodegradable compounds (nonylphenol-free), which help to keep the cleaner stable. 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 a CO_2 and water as 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. BioClean's Separator Module I is the unit that will be utilized during verification testing (***Figure 1***). Within the separator module the solution temperature, pH, and 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 and maintains the bath solution at 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 forms 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 organic soils 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 biological degreasing system. The microbes are also self controlling. As the volume of oil increases, the organisms multiply in direct proportion.

2.2 Commercial Status

BioClean's system has reportedly been operating for more than 20 years in Europe. The microbiological cleaning technology was originally developed in Sweden, a known leader in environmental reform. This technology is now being sold and installed in the US, Canada, and Mexico.

2.3 Pollution Prevention Classification

BioClean's system is a bath maintenance technology. Bath maintenance refers to a range of pollution prevention practices and technologies that preserve or restore the operating integrity of metal finishing process solutions, thereby extending their useful lives. Due to the rising costs of chemicals, energy, and treatment/disposal, and increasingly more stringent environmental requirements, bath maintenance has become a greater priority to metal finishing companies, and the methods and technologies they employ have increased in sophistication. Today, firms are willing to expend significant amounts of capital and operating funds for equipment and methods that primarily reduce the disposal frequency of their baths. In addition to extending bath life, solution maintenance often improves the operating efficiency and effectiveness of a process solution and therefore has a positive impact on production rates and finish quality [Ref 2].

2.4 Environmental Significance

BioClean's system employs microbes to consume oils and grease found in aqueous cleaning operations. The technology reportedly increases the life of the cleaner baths. The cleaner's chemistry breaks the bond between the part and oil and then forms a molecule around the oil particle. This reduces or eliminates the presence of oil floating on the surface of the cleaners or sequential tanks. Destruction of the organic soil can eliminate other cleaning steps or at least significantly increase their life and reduce the bath disposal frequency.

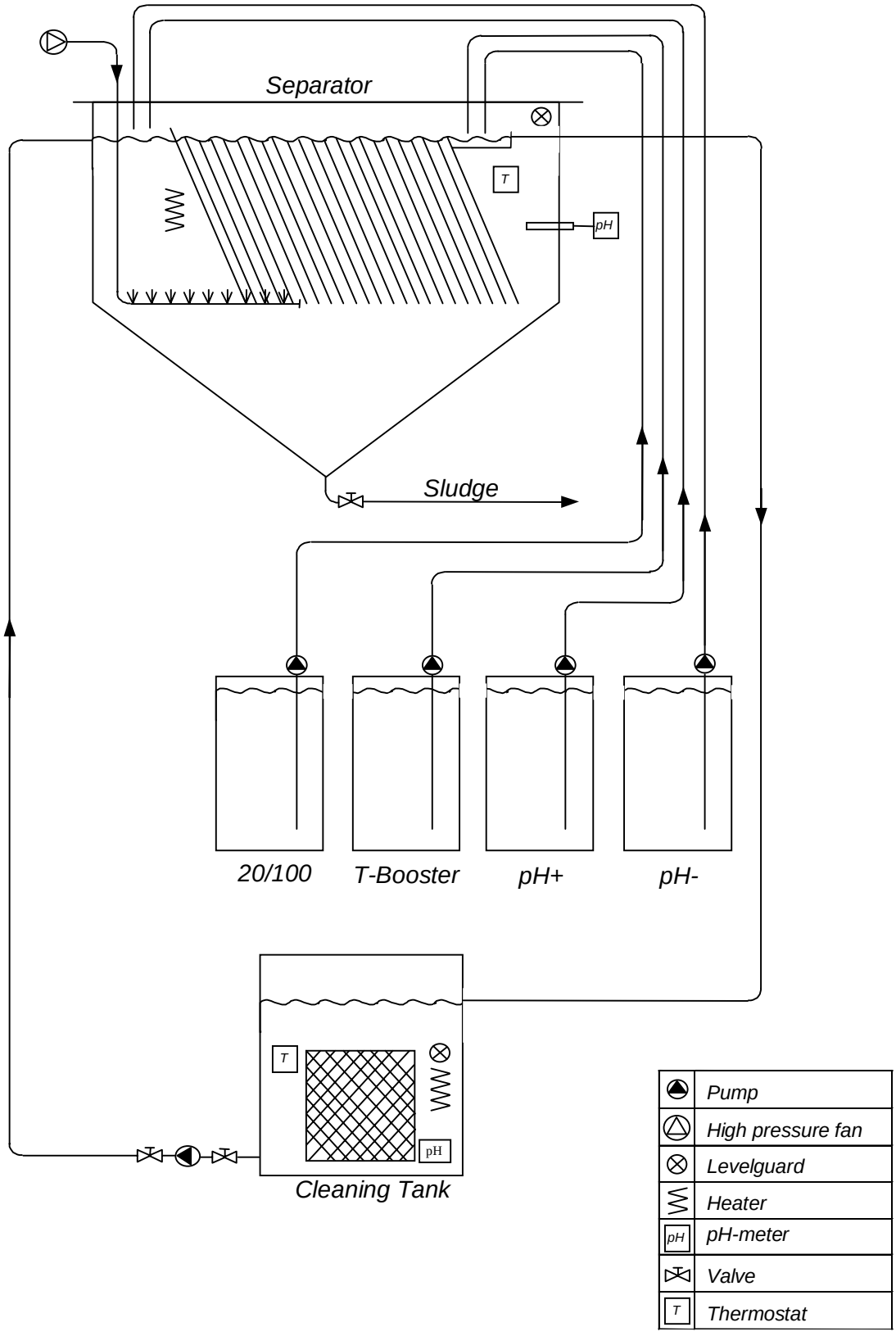
The microbiological degreasing system is mostly used as the first cleaning step, and it can be used in dip, spray, or ultrasonic applications. Conventional cleaning systems require scheduled maintenance, which entails line shut down, loss of production time, and hazardous chemical disposal. BioClean's cleaning solution contains nonylphenol-free, biodegradable compounds, which eliminates the posed toxicity concern found in some industrial detergents. Also, its systems have reportedly been operating for more than 20 years without bath dumping, producing only small amounts of non-hazardous residue.

3.0 PROCESS DESCRIPTION

3.1 Equipment and Flow Diagram

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-heater, four chemical metering pumps, a high level guard, pH-meter and electrode and control panel.

Figure 2: Biological Degreasing System (Module 1) Schematic



The pH-/pH+ metering pumps can be run in AUTO or MANUAL. The separator control panel also has ON and OFF switches for the blower, circulation pump, and BioClean 20/100 and T-Booster up heater, four chemical metering pumps, a high level guard, pH-meter and electrode, and control panel.

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. The desired pH is also set on the control panel and metering pumps. The level guard, if tripped, has an audible alarm, which can be disabled on the control panel.

The separation module not only controls pH and temperature, but also the amount of BioClean 20/100 and T-Booster added. The flow-rates of these chemical metering pumps 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 National Manufacturing. 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 5 percent. As a result, a chemical analysis is performed monthly to determine the 20/100 cleaner concentration. The make-up solutions are added manually into the separator. The manufacturer recommends a 4:1 20/100 cleaner to T-Booster ratio.

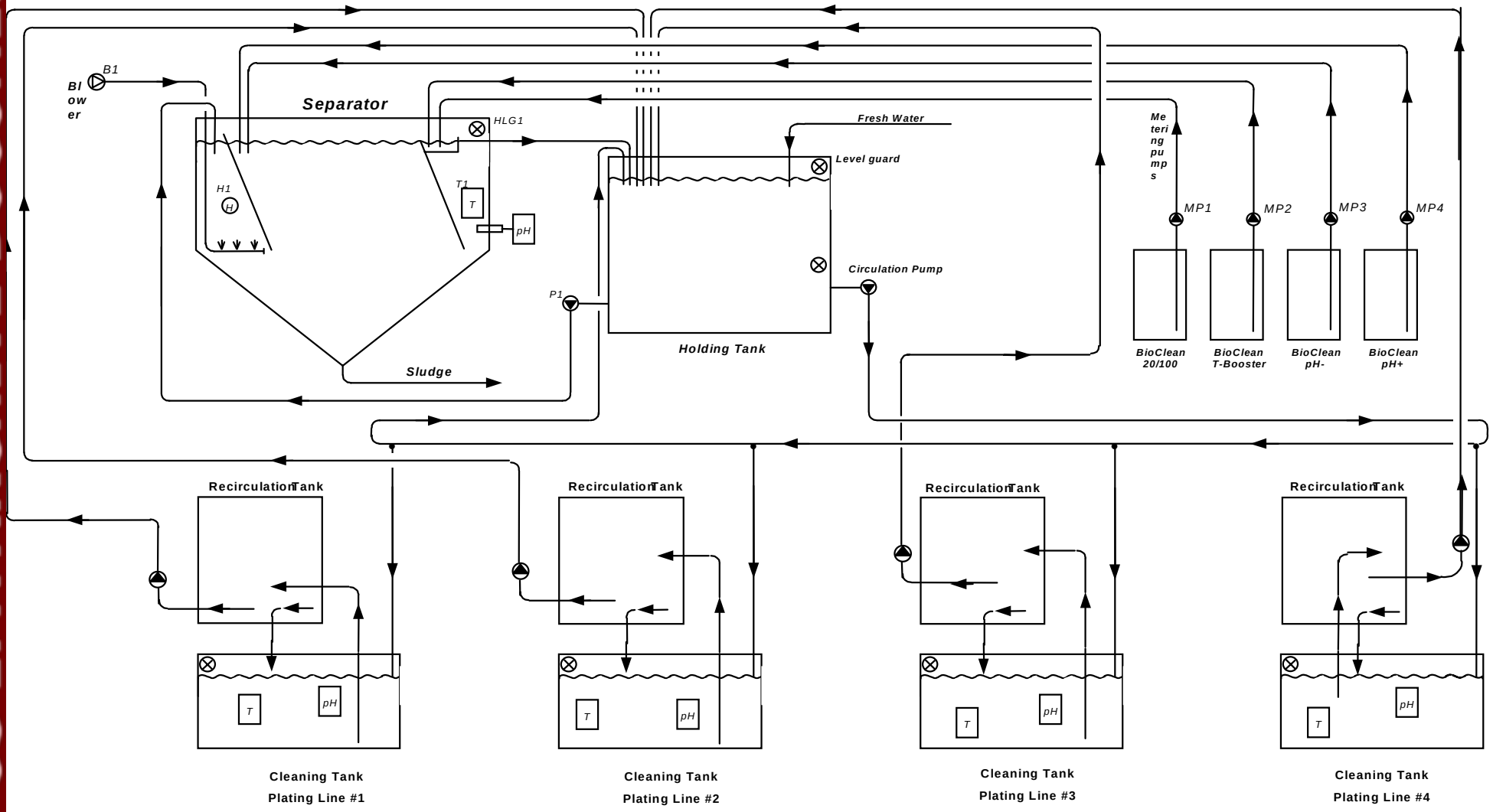
3.2 Testing Site

The metal finishing site selected for testing the BioClean system is the National Manufacturing Company. National Manufacturing has two facilities that utilize BioClean's systems, Rock Falls, Illinois (704,000 square feet), and Sterling, Illinois (550,000 square feet). National's Sterling facility utilizes BioClean's Separator Module I, and Rock Falls utilizes Module II. The Sterling facility was chosen as the test site because it employs Module I, which is the larger of the two units and also more automated.

The Sterling facility has four plating lines that use a combination of rack and barrel plating technologies (see ***Appendix A*** for plating process flow). Three of the four lines are 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 rack/barrel production lines at Sterling clean a variety of parts. The base metals include zinc die cast, cold rolled steel, stainless steel, and solid brass. The cleaning cycle for the four plating lines includes the following steps: degrease, electroclean, rinse, rinse, acid, and rinse, rinse. This is also the cleaning cycle for most plating lines. 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 a 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 that 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 re-circulation tank that is used to re-circulate the bath solution. The bath solution is pumped from the re-circulation tank to the BioClean holding tank. The re-circulation tank can also be used to isolate a bath from the BioClean separator for a short period of time.

Figure 3: National Manufacturing Company Cleaning Process Schematic



Isolation of the cleaning tank will play an important role in the BioClean verification at National Manufacturing. National Manufacturing's plating line plates the parts that are in demand at that particular time, which consequently means that there is not a consistent method of obtaining a steady oil load into the BioClean system. The advantage of isolating the cleaning tanks from each other, during the BioClean verification, is that it gives an opportunity to control, as much as possible, the soil load into the BioClean Separator.

4.0 EXPERIMENTAL DESIGN

4.1 Test Goals and Objectives

The overall goal of this project is to establish the technical and economic performance parameters that will enable a potential user to determine if the BioClean Biological Degreasing System is appropriate and feasible under their specific operating conditions. The objective of testing is to generate the analytical data and performance observations required to support these technology verification efforts.

The following are statements of specific project objectives:

- Determine the cleaning effectiveness and organic soil removal efficiency of the BioClean system when processing specific types of soiled parts, with known oil load, at manufacturer recommended process conditions.
- Determine the addition rate of BioClean cleaner, T-Booster, and pH buffer solutions during observed operating conditions. This information will be used to estimate operating costs for the BioClean system.
- Quantify the biological populations (bacteria and fungi) at selected locations within the BioClean system. This information will 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 BioClean process.
- Quantify the energy required to operate the system. Primary energy users include the bath heater, transfer pumps, and the air blower. This information will be used to help estimate operating costs for the BioClean system.
- Quantify the environmental benefit by determining the reduction in bath disposal frequency.

These objectives will be used to determine the system mass balance, the efficiency of organic soils removal, operation and maintenance requirements, and cost effectiveness for a given set of operating conditions.

4.2 Critical and Non-Critical Measurements

Measurements that will be taken during testing are classified below as either critical or non-critical. Critical measurements are those that are necessary to achieve project objectives. Non-critical measurements are those related to process control or general background readings.

Operational data will be collected on the unit's performance during treatment of solutions of known alkaline cleaner solution concentrations, and organic soil load. The following operational data will be collected:

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)
- Organic soil on parts (incoming & outgoing)
- Metal concentration (Cu, Zn)
- Total Suspended Solids (TSS)/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))
- O&M labor requirements
- Solution processing rate & chemical characteristics of feed & product solutions (cleaning chemical & contaminants)
- Waste volumes, characteristics, & 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)

These data will be used to determine the system mass balance, the efficiency of organic soil removal, operation and maintenance requirements, and cost effectiveness for a given set of operating conditions.

Historical Data:

National Manufacturing historical data on alkaline cleaner bath disposal frequency prior to installation of the BioClean system will be collected and provided in the verification report to determine the environmental benefit.

4.3 Test Matrix

The Bioclean System will be evaluated on its ability to efficiently remove oil at three specific soil-loading rates. The specific tests planned are described below and listed in ***Table 1***.

Initial Testing

Representative part samples (five) will be collected at each soil load rate (low, medium, and high). Part samples will be taken directly from the process point (barrel and/or rack). In order to quantify oil content on the representative part samples the oil from the parts will be removed through solvent extraction (immersion into a known volume of acetone) and the acetone rinse analyzed using a modified organics and hydrocarbon gas chromatographic method (EPA Method 8015, modified). The acetone rinse will be collected and transferred into one-liter amber glass sampling containers and returned to

the analytical laboratory. At the lab, the acetone rinse will be evaluated for the indicated initial control parameters in ***Table 1***. The initial part sampling process will be performed three times and an average oil quantity calculated for each soil load rate category. This average oil quantity will be used as the soil feed rate (by category) into the system.

The three soil load rates correspond directly to the type of part being cleaned. Some parts manufactured at National Manufacturing contain more oil than others because of threads and surface grooves. Only three types of parts will be processed during testing, and they will be classified as low, medium, or high soil load. National Manufacturing will determine the testing schedule.

Another set of representative part samples (five) will be collected to determine the residual oil remaining after the BioClean system. The same test methodology stated above will be used.

Verification Testing at Low, Medium and High Soil Loads

Each test will be conducted at normal operating parameters recommended by BioClean, USA. The key operating data, as discussed in Section 4.2, will be recorded onto the form shown in ***Figure 4*** at hourly intervals. Analytical and microbiological testing of samples will be conducted to evaluate the indicated parameters in ***Table 1***.

4.4 Operating Procedures

National Manufacturing personnel will perform normal operation and maintenance activities during testing. These activities will be observed and noted by an ETV-MF representative.

The BioClean System will be operated 24 hrs/day for 5 to 7 days per week. The exact number of days is dependent on the schedule at National Manufacturing. Each test run will consist of a minimum of 3 days.

4.5 Sampling, Process Measurements, and Testing Procedures

4.5.1 Sampling Responsibilities & Procedures

Samples will be taken at the frequency listed in ***Table 1*** for each location. The appropriate sampling container will be used as outlined in ***Tables 2 - 6*** for each test parameter. Each laboratory sample bottle will be labeled with the date, time, sample ID number, and test parameters required. Sampling will begin once the unit has been operating normally for a period of at least one-hour. Sample preparation methods are described in each individual analytical method.

Samples to be analyzed at an off-site laboratory will be accompanied by a chain of custody form. The samples will be transported in appropriate sample transport containers (e.g., coolers with packing and blue ice) by common carrier. The transport containers will be secured with tape to ensure sample integrity during the delivery process to the analytical laboratory. The Project Manager or designee will perform sampling, labeling, and ensure that samples are properly secured and transported to the appropriate laboratory. AMTest, Inc. in Redmond, WA, will perform analytical testing, and U.S. Micro-Solutions in Greensburg, PA, will perform microbiological testing.

SAMPLE LOCATION	SOIL LOAD	NUMBER OF SAMPLES	FREQUENCY	TEST DURATION	TEST PARAMETERS
Before BioClean (feed) (low, medium and high load)	Initial Control	5 (for each soil load)	3 times (for each soil load)	N/A	Oil Concentration Total Suspended Solids (TSS)/Total Solids (TS) Metals
After BioClean (product) (low, medium and high load)	Initial Control	5 (for each soil load)	3 times (for each soil load)	N/A	Oil Concentration TSS/TS Metals
Cleaning Tank	Low Medium High	4 (for each soil load)	2/day (for each soil load)	3 days (for each soil load)	Oil Concentration (for each soil load) TSS/TS Metals
Separator Effluent (BioClean Unit)	Low Medium High	1 (for each soil load)	2/day (for each soil load)	3 days (for each soil load)	Oil Concentration (for each soil load) TSS/TS Metals
Separator Effluent (BioClean Unit)	Low High	1 (for each soil load)	1/hr. for 10 hrs. (for each soil load)	3 days (for each soil load)	Biological Population
Waste Solids – Sludge (BioClean Unit)	Low Medium High	1 (for each soil load)	1 sample at the end of each soil load	3 days (for each soil load)	Oil Concentration Metals Weight Total % Solids Total Organic Carbon (TOC)
Waste Solids - Sludge (BioClean Unit)	Low High	1 (for each soil load)	1/hr. for 10 hrs. (for each soil load)	3 days (for each soil load)	Biological Population
Cleaning Tank	Low High	1 (for each soil load)	1/hr. for 10 hrs. (for each soil load)	3 days (for each soil load)	Biological Population

Table 1: TEST MATRIX

TEST #_____ OPERATION: Parts Cleaning

DATE:_____ BATH TYPE: Alkaline Cleaner

SAMPLE DATA			BIOCLEAN SEPARATOR PARAMETERS						CLEANING TANK PARAMETERS			
Sample #	**Time**	**Sample Location**	**Temp. (^{0}F)**	**pH- (mL)**	**pH+ (mL)**	**20/100 (mL)**	**T-Booster (mL)**	**Flowrate (L/hr)**	**H20 (L/hr)**	**Flowrate (L/hr)**	**pH**	**Temp. (^{0}F)**

Figure 4: TEST DATA COLLECTION FORM

ANALYTE	METHOD	SAMPLE BOTTLE	PRESERVATION METHOD
Oil	EPA Method 8015, modified	1 liter Glass	Cool to 39ºF (4ºC)
TSS/%TS	EPA Method 160.2/160.3	500 ml High Density Polyethylene (HDPE)	Cool to 39ºF (4ºC)
Metals	EPA Method 200.7/200.9	500 ml HDPE	pH<2

Table 2: AQUEOUS SAMPLES

ANALYTE	METHOD	SAMPLE BOTTLE	PRESERVATION METHOD
Oil	EPA Method 8015, modified	16 ounce glass	Cool to 39ºF (4ºC)
%TS	EPA Method 160.4	16 ounce glass	Cool to 39ºF (4ºC)
Metals (Cu, Zn)	EPA Method SW846 6010	16 ounce glass	Cool to 39ºF (4ºC)
TOC	EPA Method SW846 9060	16 ounce glass	Cool to 39ºF (4ºC)

Table 3: SLUDGE SAMPLES

4.5.2 Process Measurements

Monitoring of the tests will be accomplished by recording key operating data on the Test Data Collection Form (***Figure 4***). Monitoring instrumentation will be calibrated by National Manufacturing, according to the frequency required by the manufacturer. See Section 4.5.6 for specific calibration procedures and frequency.

Electricity use will be measured by determining the power requirements and cycle times of pumps and other powered devices. National Manfacaturing will provide the cost of labor, electricity, and other data needed for cost analysis.

Separator and cleaning bath flow rates for this demonstration will be measured by allowing the BioClean holding tank influent solution fill a graduated cylinder or beaker while monitoring with a stopwatch. The same procedure will be used to measure the separator effluent stream, as well as the BioClean 20/100 and T-Booster flow rates. Flow rate will then be calculated using the following equations:

$$\frac{\text{Volume (milliliters)}}{\text{Time (second)}} = \text{Flow rate (mL/sec.)} \quad \text{(a)}$$

$$\text{Flow rate (mL/sec.)} \times 60 \text{ sec./min.} = \text{Flow rate (mL/min.)} \quad \text{(b)}$$

4.5.3 Testing Parameters & Procedures

4.5.3.1 pH/Buffer Control

The pH of the cleaning solution and the separator effluent is continuously monitored and automatically controlled. The pH-increasing chemical (pH+) contains sodium hydroxide, and the decreasing chemical (pH-) contains phosphoric acid.

4.5.3.2 Temperature

Temperature is continuously monitored by the BioClean control system. An immersion heater typically maintains the bath temperature at 120°F (49°C) and can be adjusted as desired.

BioClean does not recommend bath temperatures exceeding 135°F (57°C), as higher temperatures will pasteurize the bath, killing the bacteria.

4.5.3.3 Oil Concentration (content and type)

The amount and type of oil coating the parts to be cleaned could affect the performance of the BioClean system. A representative sample of oil on the parts will be collected and analyzed using the modified organics and hydrocarbon gas chromatographic method, EPA Method 8015 (modified).

A one-liter sample will be collected for oil analysis in aqueous and sludge samples using Method 8015 (modified). Typically, a one-liter volume is extracted prior to Method 8015 analysis because of the presumption of low level concentration. Preliminary samples were taken to verify the efficacy of Method 8015 to be used during the BioClean Verification Test. The samples under consideration have been observed to contain extremely high concentrations upon receipt at the laboratory. Laboratory sample splits (duplicates) were also analyzed and internal QA/QC matrix spikes were run from the sample splits. Approximately 100 mLs per sample aliquot proved to be sufficient (500 mLs were used in assaying less highly impacted samples).

4.5.3.4 Related Parameters of Interest

Other parameters may also be of interest in evaluating the relative performance of the BioClean System. Specifically, the amount of total solids suspended in solution (TSS) and total solids (TS) in the effluent stream will be determined (TSS by EPA Method 160.2, TS by EPA Method 160.3). Certain metals are known to inhibit the viability of microbial populations, specifically copper (Cu) and zinc (Zn), among others. Both metals may be present as an artifact of the manufacturing and/or plating process. Subsequently, copper and zinc were selected as target analytes of interest. Copper

and zinc will be measured, using EPA Method 200.7/200.9, to determine if their presence correlates with lower than expected microbial population or oil removal efficiency. Method 200.7 utilizes a charge injection device (CID) capable of simultaneous analysis and Method 200.9 utilizes a graphite furnace (GF) detector that is capable of individual metal analyses. Depending on the metal under consideration and/or the concentration range in the samples one method may be more sensitive than the other method. Due to the complex nature of the samples collected matrix interference may possibly affect analyses. Consequently, the 200.7 method will be performed first followed by the 200.9 method, should analytical sample conditions so warrant. If this happens, the laboratory will indicate the reason(s) for use of Method 200.9 in the data package.

4.5.3.5 Sludge Composition

The quantity and composition of the sludge, which forms in the bottom of the BioClean Separator, will enable the ETV-MF team to determine the operating costs of the BioClean system. Sludge samples will be taken before and after each soil-loading test. If a sufficient sludge quantity is collected then it will be characterized according to the test parameters in ***Table 1***. If there's not a sufficient quantity of sludge collected for each individual soil load then a composite sample will be prepared for characterization that will be comprised of the entire volume of sludge collected. The analytical laboratory results will determine if individual samples are sufficient or if a composite sample is required. Before sludge samples are collected in sampling containers the piping from the conical shaped separator will be drained as much as possible in order to ensure a representative sample. The sludge quantity will be determined by simply dewatering and weighing it, expressed as percent total solids (EPA Method 160.4). Organic soil concentration will be determined (EPA Method 8015, modified). Total organic carbon (TOC) content (EPA Method 9060, SW846) and specific metals (copper, zinc) concentration (EPA Method 6010, SW846) will be determined.

4.5.3.6 Biological Testing

The presence of surface biological contamination (bacteria and/or fungi) or airborne biological contaminants in indoor air (bio-aerosols), may affect product quality as well as worker health. The assay of the microbial content of the workplace (ambient air, work surfaces, raw materials, etc.) has become increasingly more significant in the past decade as the need for "contamination-free" environments has become more apparent.

The ASHRAE (American Society for Heating, Refrigerating and Air-Conditioning Engineers) defines acceptable indoor air quality as "air in which there are no known contaminants at harmful concentrations and with which a substantial majority (usually 80%) of the people exposed do not express dissatisfaction." Bio-aerosols have been conclusively associated with hypersensitivity syndromes and sick building syndrome (SBS). These hypersensitivity syndromes result from exposure to materials in the environment (antigens) that stimulate a specific immunologic response. Most of these workplace/building-related antigens are assumed to be of fungal or bacterial origin.

An important component of the verification testing of an aqueous biological degreasing system is the quantification of the biological populations (bacteria and fungi) at selected locations within the aqueous system. The characterization of the biological population (to assess response to the oil loading) along with the quantification of bio-aerosol generation within the work area (to assess potential health and safety risks) during various stages of the BioClean process provides important information to project managers, technicians, operators, and risk assessors regarding the process variables.

4.5.3.6.1 Biological Concentrations Sampling & Analysis

The concentrations of biological populations (bacteria and fungi) shall be evaluated by collecting representative samples (50-100 ml) from a number of locations in the BioClean Separator Module I, including bulk samples of the separator waste solids, separator effluent, and cleaning tank. The bulk samples of solid and liquid process materials shall be collected in sterile 50-ml vials, sealed and then shipped overnight to an approved microbiology laboratory for analysis. The holding period for biological samples is 24-hours. U. S. Micro-Solutions (Greensburg, PA) shall analyze samples for the predominant genera and concentration of bacteria and fungi.

4.5.3.6.2 Bulk Bacteria Sampling & Analysis

Bulk samples of the alkaline cleaning solution shall be collected in sterile 50-ml screw-cap vials from a series of sampling points. The sampling points for the bulk bacteria samples (***Table 4***) shall include the separator inlet, separator effluent, and separator waste sludge. Twenty samples shall be collected from each sampling point at a rate of one sample per hour with approximately ten samples collected at each location

during *low* oil-grease loads in the BioClean system and ten bulk samples collected at each sampling location during *high* oil-grease loads on the system. Sampling during the *low* and *high* oil-grease loads will provide a good minimum and maximum biological characterization representation in the BioClean system. Assuming the bacterial population response is linear, collecting samples at high and low oil loading is sufficient to characterize the bacterial population response. For this reason samples will not be taken during *medium* oil-grease loading. Two bulk sample "blanks" shall be collected prior to beginning each series of bulk samples of high and low oil-grease loads on the system: one bulk sample of the makeup water for the aqueous cleaner and one bulk sample of the organic soil coating on the parts to be cleaned. These blanks shall provide the concentrations of bacteria/fungi present at background levels in the BioClean system. The organic soil coating on the parts to be cleaned will be analyzed for biological contamination by swabbing the part(s) with a sterile swab. The swab collecting the sample is placed in a sterile tube and stored prior to analysis.

The bulk solution samples shall be collected in sterile 50-ml vials, sealed and then shipped overnight to U.S. Micro-Solutions for analysis. Serial dilutions (i.e., 1:10, 1:100, 1:1,000) of the bulk samples shall be made with sterile water, and tryticase soy agar (TSA) plates shall be inoculated with the serial dilutions for the isolation and identification of the bacteria. The agar plates are incubated at 95^{o}F (35^{o}C) for three days and 81^{o}F (27^{o}C) for two days. Bacteria counts shall be performed, and colony-forming units (CFU)/gram of bulk sample and/or CFU/ml of liquid sample shall be calculated.

4.5.3.6.3 Bulk Fungi Sampling & Analysis

Bulk samples of the alkaline cleaning solution shall be collected for the identification and quantification of fungi following the aforementioned protocol. The bulk samples of the alkaline cleaning solution analyzed for fungi shall be collected in sterile 50-ml screw-cap vials from the separator inlet, separator effluent, and separator waste sludge (***Table 4***). The twenty samples collected (10 samples during low oil-grease loads and 10 samples during high oil-grease loads) shall

SAMPLE STREAM LOCATION	TEST TYPE (Oil Load)	NUMBER OF SAMPLES	FREQUENCY	TEST PARAMETER	TEST METHOD
Separator Inlet	Low High	10 low 10 high	1 sample/hour	CFU/ml	Serial Dilutions: TSA YMA
Separator Effluent	Low High	10 low 10 high	1 sample/hour	CFU/ml	Serial Dilutions: TSA YMA
Separator Waste Sludge	Low High	10 low 10 high	1 sample/hour	CFU/ml	Serial Dilutions: TSA YMA
Makeup Water (for aqueous cleaner)	Blank	1 low 1 high	1 sample/load 1 sample/load	CFU/ml	Serial Dilutions: TSA YMA
Oil Coating	Low High	1 low 1 high	1 sample/load 1 sample/load	CFU/ml	Serial Dilutions: TSA YMA

CFU – colony forming units TSA – tryticase soy agar YMA - yeast malt extract agar

Table 4: BULK BACTERIA & FUNGI SAMPLING & ANALYSIS

be sealed, shipped, and handled as previously described. Bulk samples for fungal analysis shall be serially diluted with sterile water (i.e., 1:10, 1:100, 1:1,000), and yeast malt extract agar (YMA) plates shall be inoculated with the different serial dilutions for the isolation of the fungi. The YMA plates are incubated at 81°F (27°C) for five days. Fungal counts shall be performed and CFU/gram or CFU/ml of sample shall be calculated.

4.5.3.6.4 Airborne Biological Sampling & Analysis

The on-site investigation of biological populations begins outdoors. The outdoor air is never sterile and often contains in excess of 10^5 fungal spores per cubic meter. Fungal spores usually dominate the outdoor air spora, although pollen, bacteria, algae, and insect fragments also are present. It is essential to carefully examine the outdoor environment adjacent to the aqueous cleaning operation for potential sources and to sample the ambient air, as controls for all indoor samples collected during the verification testing. Outdoor air will be sampled for airborne bacteria and fungi according to the frequency shown in Tables 5 and 6.

Airborne microbes (biological aerosols) include viable biological contaminants occurring as particles in the air. These particles can vary in size from viruses less than 0.1 micron in diameter to fungal spores 100 or

more microns in diameter. They occur as single, unattached organisms or as aggregates. Air particle samplers have been generally used to collect and assay aerobic species of bacteria and fungi. Even though many samplers will collect some virus particles, there is no convenient, practical method for the cultivation and enumeration of these particles. The survey shall focus on the quantification and identification of predominant bacteria and fungi and will not address the presence of viral particles in the ambient air surrounding the workplace.

The airborne bacterial and fungi samples shall be collected at three locations (***Tables 5-6***) within the BioClean Separator Module system. The airborne samples shall be collected on sterile agar collection plates using an Andersen N-6 particle sampler. The Andersen sampler is a single-stage, size-selective impactor sampler designed to separate bio-aerosol particles from the air. A vacuum pump draws ambient air over an agar collection plate at the rate of 28.3 liters per minute (l/min). The airborne bacteria are collected on an agar medium appropriate to the microorganisms that may be encountered (TSA). The TSA plate is then removed from the sampler, inverted, incubated, and counted by an accepted method. Sampler stages are disinfected with isopropyl alcohol before and after each impact sample is collected. The TSA air plates are incubated at 95^{o}F (35^{o}C) for three days and 81^{o}F (27^{o}C) for two days. The YMA air plates are incubated at 81^{o}F (27^{o}C) for five days. Microbial counts are then made and the colony forming units per cubic meter of air (CFU/m^{3}) are determined.

The sampling time for airborne biological contaminants using the Anderson N-6 particle sampler is 2 minutes for outdoor air (control) and 4 to 6 minutes for the process environment (indoor air). Sampling time is recorded and multiplied by the sampling rate to determine the volume of air sampled.

SAMPLE STREAM LOCATION	TEST TYPE (Oil Load)	NUMBER OF SAMPLES	FREQUENCY	TEST PARAMETER	TEST METHOD
Separator	Low High	10 low 10 high	1 sample/hour (10 hours x 2)	CFU/m^3	TSA
Holding Tank	Low High	10 low 10 high	1 sample/hour (10 hours x 2)	CFU/m^3	TSA
Cleaning Tank	Low High	10 low 10 high	1 sample/hour (10 hours x 2)	CFU/m^3	TSA
Outside Air	Control	2	1 : test onset 1 : test end	CFU/m^3	TSA

CFU – colony forming units *TSA – tryticase soy agar*

Table 5: AIRBORNE BACTERIA SAMPLING & ANALYSIS

SAMPLE STREAM LOCATION	TEST TYPE (Oil Load)	NUMBER Of SAMPLES	FREQUENCY	TEST PARAMETER	TEST METHOD
Separator	Low High	10 low 10 high	1 sample/hour (10 hours x 2)	CFU/m^3	YMA
Holding Tank	Low High	10 low 10 high	1 sample/hour (10 hours x 2)	CFU/m^3	YMA
Degreasing Tank	Low High	10 low 10 high	1 sample/hour (10 hours x 2)	CFU/m^3	YMA
Outside Air	Control	2	1 : test onset 1 : test end	CFU/m^3	YMA

CFU – colony-forming units *YMA – yeast malt extract agar*

Table 6: AIRBORNE FUNGI SAMPLING & ANALYSIS

4.5.4 Test Procedures

4.5.4.1 Solvent Extraction

Organic soil load on each particular part will be different as a function of the part's surface area, machining detail, and application rate on each part's line. The three soil load rates correspond directly to the type of part being cleaned. Some parts manufactured at National Manufacturing contain more organic soil than others because of threading and surface grooves. For example, parts that have blind holes contain more organic soil than small smooth parts.

Only three types of parts will be observed during this testing and they will be classified as low, medium, or high soil load. Each of the four plating lines may receive one and/or several parts over a given time period. So to better understand the

relative organic soil loading contribution from a particular series of parts, representative "clusters" of parts will be used as characteristic of the range of parts loads to these plating lines. This will entail understanding the manufacturing process and parts loads over the testing period, then defining one or several "characteristic clusters" for each plating line. Once defined, these "part clusters" will be aggregated by randomly sampling parts. National Manufacturing will assist the project team in determining which parts will be sampled, based on their production schedule.

By nature of the experimental conditions and distance from the manufacturing test site and the analytical laboratory, the organic soil on these part clusters will be extracted at the test site. The parts clusters will be rinsed in a solvent such as acetone. Acetone is readily available, stable, and under well-ventilated conditions relatively safe for properly trained personnel to use. Representative aliquots of both the straight acetone and extraction medium will be sent to the analytical laboratory for analysis. At the lab, the acetone rinse will be evaluated for the indicated test parameters in ***Table 1*** using a modified organics and hydrocarbon gas chromatographic method (EPA Method 8015, modified).

The solvent extraction procedure consists of the following steps:

1. Remove five part samples from the barrel/rack finishing line.
2. Put part in a container (beaker or erlenmeyer flask) with a known quantity of acetone. If the part is too large for a beaker or flask then rinse a clean laboratory pan with acetone and place the part in the pan. Using a volumetric beaker and/or flask fill the pan with acetone until the part is covered.
3. Record the amount of acetone used into the laboratory logbook.
4. Let the part lay immersed in the acetone for two minutes.
5. Remove part and place the extraction medium in a properly labeled sampling container (1 L jar) for shipment to the analytical laboratory.

4.5.5 Nonstandard or Modified Test Methods

4.5.5.1 EPA Method 8015, Modified

A gravimetric method for measuring organic soils in aqueous and sludge samples was not chosen for analytical testing. Concerns about possible interference's and "false positives" for organic soil concentration from surfactants and/or

proprietary chemicals within the system under evaluation led to consider alternative analytical methods, or at least to demonstrate the efficacy of standard methods to these materials and sample matrices.

"Neat" samples of the exact formulated lubrication products, which are used on the parts themselves, will be characterized and used as calibration standards in these quantitative analyses. Therefore, by using known dilutions of "neat" standards, calibration curves and reference solutions can be drawn (reference solutions were used for quantification purposes). EPA Method 8015 can then be used to quantify the organic soil in the cleaner separate from the BioClean cleaner compounds.

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 (see ***Appendix B*** for chromatograms). 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).

Analyses of the BioClean proprietary biological enhancement (T-Booster) and cleaning (20/100) solutions were also performed. When compared to the prepared organic soil reference calibration standards, neither sample solution exhibited "false positive" signatures for organic soil concentration. Thus, reported organic soil concentration should be expected as directly related to the formulated organic soil content.

Another reference step evaluated the efficacy of the modified Method 8015 test method for these samples and matrices. Aliquots were analyzed using the modified Method 8015, the conventional freon extraction-gravimetric method, as well as the recently approved EPA Method 1664 (hexane extract). The modified Method 8015 and freon methods yielded comparable results. Although the freon method yielded acceptable results, freon has been phased out as an acceptable material under the Montreal Accord, and hence will not be in use within analytical methods in the very near future. The hexane extraction method did not yield successful extraction results. The aqueous matrix turned milky (akin to liquid gelatin),

requiring several cleanup steps and resulting in poor sample recovery (un-reproducibility of results). Due to the arduous sample preparation that would have been required, these test samples were not carried through to analyses. The cost for such analyses would be prohibitively expensive, so the method was discounted from further consideration in this study.

4.5.6 Calibration Procedures and Frequency

The following procedures will be used to calibrate the instruments/equipment that will be used to collect critical measurements:

1. Instruments used to perform analytical methods will be calibrated according to the analytical laboratory's quality assurance plan.

2. The bioaerosol sampler, Andersen N-6 Single Stage Particulate Sampler, will be calibrated by the equipment manufacturer once before the start of verification testing.

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

4. Although pH and temperature are non-critical measurements and are automatically controlled by the separator module, these measurements will be checked daily with a digital pH and thermometer. The digital pH reader will be calibrated daily before taking the bath reading. If the manual and controlled pH readings are off by $\pm$ 5 percent than the pH controller will be calibrated. If the digital thermometer and temperature controller readings are off by $\pm$ 5 percent than the separator's backup temperature controller will be used for the duration of the testing.

5.0 QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

Quality Assurance/Quality Control activities will be performed according to the applicable section of the Environmental Technology Verification Program Metal Finishing Technologies Quality Management Plan (ETV-MF QMP) [Ref 3].

5.1 Quality Assurance Objectives

One QA objective is to ensure that the process operating conditions and test methods are maintained and documented throughout each test and laboratory analysis of samples. Another QA objective is to use standard test methods for laboratory analyses. The analytical methods that will be used for analyzing the baths and product and/or waste samples are standard EPA methods.

5.2 Data Reduction, Validation, and Reporting

5.2.1 Calculation of Results

The conservation of mass/energy in any isolated system is one of the most fundamental laws in science and engineering. The mass/energy balance can account for the inputs, outputs, consumption, and accumulation in a system. To determine system efficiency, measuring or quantifying all of the elements for a mass balance in an industrial setting is very difficult. The greatest challenge is generally defining the system boundaries and what degree of accuracy is required. Sampling, measurement, and analytical errors preclude absolute precision; however, the mass/energy balance provides us with a fundamental tool for evaluating the performance of environmental technologies where we are generally evaluating some form of efficiency. ***Figure 5*** illustrates the most fundamental form of the material balance equation. Batch systems and continuous systems can both be modeled using this general form. ***Figure 6*** illustrates the material flow into and out of the BioClean separator

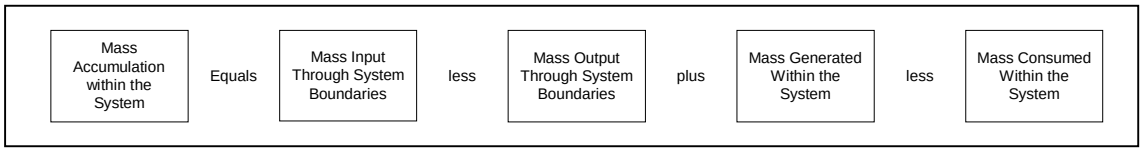


Figure 5: FUNDAMENTAL MATERIAL BALANCE EQUATION

The consumption term in this fundamental material balance equation warrants further discussion because of the complexity of the BioClean technology. Because the technology presented includes biological chemistry, microbial kinetics would need to be evaluated in order to accurately quantify the mass consumed within the system. Some of the parameters that go into the microbial kinetics are oxygen uptake, carbon dioxide and water production. These parameters will be impossible to measure because the BioClean technology is an open system. Since the microbes consume oxygen as they metabolize hydrocarbons to produce carbon dioxide and water, dissolved oxygen must be continuously replenished in the cleaning bath. Dissolved oxygen analysis will

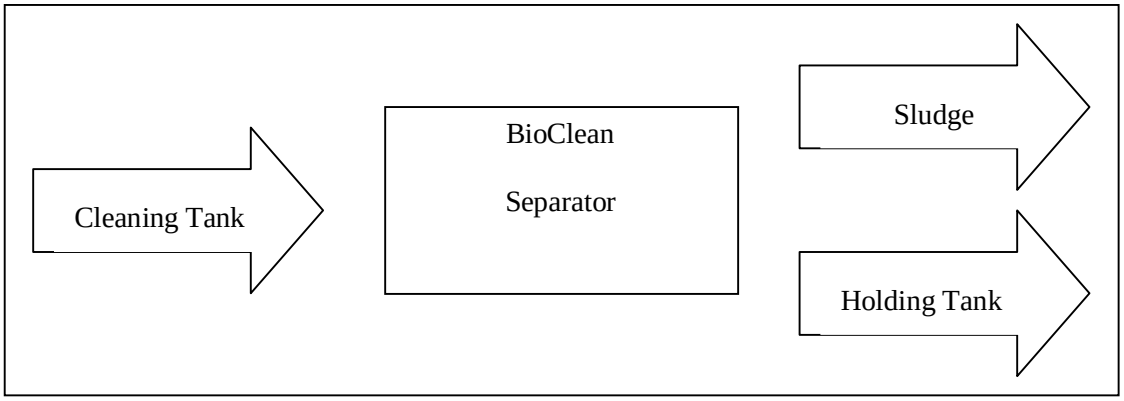


Figure 6: MATERIAL FLOW DIAGRAM FOR BIOCLEAN SYSTEM

determine if the bath contains enough oxygen (as supplied through aeration) to support the microbial population, and also the effect of dissolved oxygen on system performance (or oil consumption/removal efficiency). Dissolved oxygen data will not be accurate enough to calculate microbial kinetics. In addition, it might turn out that microbial growth would not show first-order-kinetics with respect to oil loading or consumption. For example microbial growth may be due to a combination of oil degradation as well as nutrients and surfactants, thus it becomes hard to determine which had the major influence. Focusing on the microbial kinetics that will be needed to quantify the mass consumed within the BioClean system will launch efforts into an R&D path, which deviates from the ETV Program goals. Consequently, quantifying the consumption term is not a project objective, but every attempt will be made to measure it indirectly, if feasible in order to report the mass balance as accurately as possible.

The goal of the BioClean project is to verify performance, and this can generally be measured in terms of the BioClean Separator oil removal (digestion) efficiency. QA objectives will be satisfied if the mass balance is between 50 and 150 percent.

To determine oil removal efficiency, the fundamental material balance equation for the BioClean separator can be simplified to:

Xs + Xh	=	Xi, where
Xs	=	Mass of oil leaving the separator in the sludge
Xh	=	Mass of oil leaving the separator and entering the holding tank
Xi	=	Mass of oil leaving the cleaning tank and entering the separator

Separator oil removal efficiency is determined by:

$$\text{Efficiency \%} = \frac{Xs + Xh}{Xi} \times 100\%$$

5.2.2 Internal Quality Control Checks

Raw Data Handling. Raw data is generated and collected by laboratory analysts at the bench and/or sampling site. These include original observations, printouts, and readouts from equipment for sample, standard, and reference QC analyses. Data is collected both manually and electronically. At a minimum, the date, time, sample ID, instrument ID, analyst ID, raw signal or processed signal, and/or qualitative observations will be recorded. Comments to document unusual or non-standard observations also will be included on the forms, as necessary. The Test Data Collection Form presented in ***Figure 4*** will be used for recording data on-site. The on-site Project Team member will generate chain of custody (C.O.C.) forms and these forms, which will accompany samples during shipment to the respective labs. Raw data will be processed manually by the analyst, automatically by an electronic program, or electronically after being entered into

a computer. The analyst will be responsible for scrutinizing the data according to laboratory precision, accuracy, and completeness policies. Raw data bench sheets and calculation or data summary sheets will be kept together for each sample batch. From the standard operating procedure and the raw data bench files, the steps leading to a final result may be traced. The ETV-MF Program Manager will maintain process-operating data for use in report verification preparation.

Data Package Validation. The generating analyst will assemble a preliminary data package, which shall be initialed and dated. This package shall contain all QC and raw data results, calculations, electronic printouts, conclusions, and laboratory sample tracking information. A second analyst will review the entire package and check sample and storage logs, standard logs, calibration logs, and other files, as necessary, to ensure that all tracking, sample treatments, and calculations are correct. After the package is reviewed in this manner, a preliminary data report will be prepared, initialed, and dated. The entire package and final report will be submitted to the Laboratory Manager.

The Laboratory Manager shall be ultimately responsible for all final data released from the laboratory. The Laboratory Manager or designee will review the final results for adequacy to task QA objectives. If the manager or designee suspects an anomaly or non-concurrence with expected or historical performance values, or with task objectives for test specimen performance, the raw data will be reviewed, and the generating and reviewing analysts queried. If suspicion about data validity still exists after internal review of laboratory records, the manager will authorize a re-test. If sufficient sample is not available for re-testing, a re-sampling shall occur. If the sampling window has passed, or re-sampling is not possible, the manager will flag the data as suspect. The Laboratory Manager signs and dates the final data package. The data analyzed shall be evaluated through and will ensure

Data Reporting. A report signed and dated by the Laboratory Manager will be submitted to the ETV-MF Project Manager. The ETV-MF Project Manager will decide the appropriateness of the data for the particular application. The final report contains the laboratory sample ID, date reported, date analyzed, the analyst, the SOP used for each parameter, the process or sampling point identification, the final result, and the units. The ETV-MF Program Manager shall retain the data packages as required by the ETV-MF QMP [Ref 3].

5.2.3 Calculation of Laboratory Data Quality Indicators

Analytical performance requirements are expressed in terms of precision, accuracy, representability, comparability, completeness, and sensitivity (PARCCS). Summarized below are definitions and QA objectives for each PARCCS parameter. Duplicates and spike duplicates will be performed on one out of every ten samples. Sample splitting will occur in the analytical laboratory.

5.2.3.1 Precision

Precision is a measure of the agreement or repeatability of a set of replicate results obtained from duplicate analyses made under identical conditions. Precision is estimated from analytical data and cannot be measured directly.

The precision of a duplicate determination can be expressed as the relative percent difference (RPD), and calculated as:

$$RPD = \{(|X_1 - X_2|)/(X_1 + X_2)/2\} \times 100 = \left\{ \frac{|X_1 - X_2|}{\frac{(X_1 + X_2)}{2}} \right\} \times 100$$

where, X_1 = larger of the two observed values and X_2 = smaller of the two observed values.

Multiple determinations will be performed for each test on the same test specimen. The replicate analyses must agree within the relative percent deviation limits as specified in ***Table 7***.

Table #7: QA OBJECTIVES

Critical Measurements	Matrix	Method	Reporting Units	Method of Determination	MDL	Precision (RPD)	Accuracy (% Recovery)	Completeness
O&G Concentration	Water	8015, modified	mg/L	GC-FID	200	≤ 30	50-150	95
	Sludge	8015, modified	mg/L	GC-FID	200	≤ 30	50-150	95
Metals (Cu, Zn)	Water	200.7/200.9	mg/L	ICP-CID or ICP-	0.001	≤ 35	80-120	95
	Sludge	3050, 6010	mg/L	GF	0.001	≤ 35	80-120	95
TSS/TS	Water	160.2/160.3	mg/L	Gravimetric	1.0	≤ 30	80-120	95
	Sludge	160.2/160.4	mg/L or % solids	Gravimetric	1.0	≤ 30	80-120	95
TOC	Sludge	9060	mg/L	Conventional	1.0	≤ 30	80-120	95
Microbial Concentration	Water	Serial Dilutions (TSA, YMA)	CFU/ml	Serial dilutions by agar streak method	< 1.0	≤ 75	75-125	90
	Sludge	Serial Dilutions (TSA, YMA)	CFU/ml	Serial dilutions by agar streak method	< 1.0	≤ 75	75-125	90
	Air	Visual	CFU/ m^3	Visual	< 1.0	≤ 75	75-125	90
Chemical Additions:								
BioClean 20/100	Water	Stop watch	ml/min	-	-	≤ 10	±5	90
BioClean T-Booster	Water	Stop watch	ml /min	-	-	≤ 10	±5	90
pH+	Water	Flow meter	ml /min	-	-	-	±5	90
pH-	Water	Flow meter	ml/min	-	-	-	±5	90
Separator Flow rates:								
Influent	Water	Stop watch	ml/min	-	-	≤ 10	±5	90
Effluent	Water	Stop watch	ml/min	-	-	≤ 10	±5	90
Temperature	Water	Thermocouple	°F (°C)	-	-	-	36 (2)	100

5.2.3.2 Accuracy

Accuracy is a measure of the agreement between an experimental determination and the true value of the parameter being measured. Accuracy is estimated through the use of known reference materials or matrix spikes. It is calculated from analytical data and is not measured directly. Spiking of reference materials into a sample matrix is the preferred technique because it provides a measure of the matrix effects on analytical accuracy. Accuracy, defined as percent recovery (P), is calculated as:

$$P = \left[\frac{(SSR - SR)}{SA}\right] x\ 100$$

where: SSR = spiked sample result

SR = sample result (native)

SA = the concentration added to the spiked sample

Analyses will be performed with periodic calibration checks with traceable standards to verify instrumental accuracy. These checks will be performed according to established procedures in the contracted laboratory(s) that have been acquired for the BioClean verification testing. Analysis with spiked samples will be performed to determine percent recoveries as a means of checking method accuracy. QA objectives will be satisfied if the *average* recovery is within the goals described in ***Table 7***.

5.2.3.3 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 is limited by the other PARCCS parameters because data sets can be compared with confidence only when precision and accuracy are known. Comparability will be achieved in the BioClean technology verification by the use of consistent methods during sampling and analysis and by traceability of standards to a reliable source.

5.2.3.4 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:

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

Experience on similar projects has shown that laboratories typically achieve about 90 percent completeness. QA objectives will be satisfied if the percent completeness is 90 percent or greater as specified in ***Table 7***.

5.2.3.5 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 the purposes of this demonstration, representativeness will be achieved by presenting identical analyte samples to the specified lab(s) and executing consistent sample collection and mixing procedures.

5.2.3.6 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 definition of detection will be used for this program.

Instrument Detection Limit (IDL) is the minimum concentration that can be measured from instrument background noise

Method Detection Limit (MDL) is a statistically determined concentration. It is the 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 [because of the lack of information on analytical precision at this level, sample results greater than the MDL but less than the practical quantification limit (PQL) will be laboratory qualified as "estimated"] MDL is defined as follows for all measurements:

$$MDL = t_{(n-1,1-\alpha = 0.99)} \times s$$

where:

	MDL	=	method detection limit
	s	=	standard deviation of the replicate analyses
	$t_{(n-1,1-\alpha = 0.99)}$	=	students t-value for a one-sided 99% confidence level and a standard deviation estimate with n-1 degrees of freedom

Method Reporting Limit (MRL) is the concentration of the target analyte that the laboratory has demonstrated the ability to measure within specified limits of precision and accuracy during routine laboratory operating conditions [This value is variable and highly matrix dependent. It is the minimum concentration that will be reported as "unqualified" by the laboratory].

5.3 Quality Audits

Technical System Audits. An audit will be performed during verification testing by the *CTC* QA Manager according to Section 2.9.3 Technical Assessments of the ETV-MF QMP [Ref 3] to ensure testing and data collection are performed according to the test plan requirements. In addition to the *CTC* Technical System Audit, the EPA Quality Assurance Manager may also conduct an audit to assess the quality of the verification test.

Internal Audits. In addition to the internal laboratory quality control checks, internal quality audits will be conducted to ensure compliance with written procedures and standard protocols.

Corrective Action. Corrective action for any deviations to established quality assurance and quality control procedures during verification testing will be performed according to section 2.10 Quality Improvement of the ETV-MF QMP [Ref 3].

Laboratory Corrective Action. Examples of non-conformances include invalid calibration data, inadvertent failure to perform method specific QA, process control data outside specified control limits, failed precision and/or accuracy indicators, etc. Such non-conformances will be documented on a standard laboratory form. Corrective action will involve taking all necessary steps to restore a measuring system to proper working order and summarizing the corrective action and results of subsequent system verifications on a standard laboratory form. Some non-conformances are detected while analysis or sample processing is in progress and can be rectified in real time at the bench level. Others may be detected only after a processing trial and/or sample analysis is completed. Typically, the Laboratory Manager detects these types of non-conformances. In all cases of non-conformance, the Laboratory Manager will consider sample re-analysis as one source of corrective action. If insufficient sample is available or the holding time has been exceeded, complete re-processing may be ordered to generate new samples if a determination is made by the Task Leader that the non-conformance jeopardizes the integrity of the conclusions to be drawn from the data. In all cases, a non-conformance will be rectified before sample processing and analysis continues.

6.0 PROJECT MANAGEMENT

6.1 Organization/Personnel Responsibilities

The ETV-MF Project Team that is headed by Concurrent Technologies Corporation (*CTC*) will conduct the evaluation of BioClean's system. The ETV-MF Program Manager, Donn Brown, will have responsibility for all aspects of the technology verification, including appointment of a Project Manager, making ETV-MF Project Team personnel assignments, and coordination of technology testing. The Project Manager assigned to the BioClean verification is Dr. A. Gus Eskamani of CAMP, Inc. Dr. Eskamani and/or his staff will be on-site throughout the testing period and will conduct or oversee all sampling and related measurements.

A BioClean representative will assist in operating the system and will be on-call during the test period for response in the event of equipment problems. AMTest Laboratories is responsible for performing chemical analysis of verification test samples. AMTest Laboratories is accredited by the state of Washington Department of Ecology for the analyses identified in this Test Plan. U.S. Micro-Solutions will perform the biological analysis of verification test samples. U.S. Micro-Solutions is accredited by the American Industrial Hygiene Association Environmental Microbiology Proficiency Analytical Testing Program. The Laboratory Manager or designee will be the point of contact.

The ETV-MF Project Manager and National Manufacturing have the authority to stop work when unsafe or unacceptable quality conditions arise. The *CTC* ETV-MF Program Manager will provide periodic assessments of verification testing to the EPA ETV Pilot Manager.

6.2 Schedule/Milestones

The schedule and milestones will be determined mutually by *CTC* and National Manufacturing.

6.3 Documentation/Records

All original documentation generated during verification testing (chain of custody forms, data collection forms, analytical results, etc.) will be maintained at the *CTC* regional office in Largo, FL.

7.0 EQUIPMENT

7.1 Equipment List and Utility Requirements

Equipment and utility requirements are identified in ***Table 8***.

EQUIPMENT		
Number Req'd	**Type of Equipment**	**Comments**
One (1)	Stress relieved polypropylene tank	Stainless steel frame (volume is 1780 liters)
One (1)	Lamella filter	Made of PVC
One (1)	Blower	
One (1)	Transfer pump	1" internal thread pipe connection
One (1)	Primary heat control	Temperature controller, relay, temperature probe, and level guard
Four (4)	Metering pumps w/ adjustable flow rates	For BioClean cleaner, tensides, pH-, and pH+
One (1)	pH meter and electrode	
SPARE PARTS		
One (1)	Electric supplemental heater	5000 Watts
Required Utilities		
Utilities to include: Electrical • BioClean separator module 220 Volt, Single Phase, 30 Amp. Air • 3 – 5 cubic feet per minute		

Table 8: BIOCLEAN EQUIPMENT LIST AND UTILITY REQUIREMENTS

7.2 Monitoring/Sampling Equipment

All monitoring/sampling equipment to be used during testing is identified in ***Table 9.***

Table 9: MONITORING/SAMPLING EQUIPMENT

Equipment	Purpose
Andersen N6 Single Stage Viable Particulate Sampler	Bio-aerosol sampling

8.0 HEALTH AND SAFETY PLAN

This Health and Safety Plan provides guidelines for recognizing, evaluating, and controlling health and physical hazards that could occur during verification testing. More specifically, the Plan specifies for assigned personnel, the training, materials, and equipment necessary to protect them from hazards, and any waste generated. The National Manufacturing Hazcom/PPE Plan/Program will be used throughout the BioClean verification testing.

8.1 Hazard Communication

All personnel assigned to the project will be provided with the potential hazards, signs and symptoms of exposure, methods or materials to prevent exposures, and procedures to follow if there is contact with a particular substance during verification testing. Hazard communication will take place during training and will be reinforced throughout the test period. All appropriate MSDS's will be available for the chemical solutions used during the testing.

8.2 Emergency Response Plan

National Manufacturing has a contingency plan to protect employees, assigned project personnel, and visitors in the event of an emergency at the facility. This plan will be used throughout the project. All assigned personnel will be provided with information about the plan during training.

8.3 Hazard Controls Including Personal Protective Equipment

All assigned project personnel and visitors will be provided with appropriate personal protective equipment (PPE) and any training needed for its proper use, considering their assigned tasks. The use of PPE will be covered during training.

8.4 Lockout/Tagout Program

National Manufacturing's lockout/tagout procedure will be implemented if necessary and will be explained prior to performing such duties.

8.5 Material Storage

Any materials used during the project will be kept in proper containers and labeled according to Federal and State law. Proper storage of the materials will be maintained based on associated hazards. Spill trays or similar devices will be used as needed to prevent material loss to the surrounding area.

8.6 Safe Handling Procedures

All chemicals and wastes or samples will be transported on-site in appropriate covered containers. Emergency spill clean up will be performed according to National Manufacturing procedures.

9.0 WASTE MANAGEMENT

The equipment will be tested on processes already in place and operating at National Manufacturing. Any wastes that may be generated will be no different than those already generated. Therefore, no special or additional provisions for waste management will be necessary.

10.0 TRAINING

It is important that the verification activities performed by the ETV-MF Program be conducted with high quality and with regard to the health and safety of the workers and the environment. By identifying the quality requirements, worker safety and health, and environmental issues associated with each verification test, the qualifications or training required for personnel

involved can be identified. Training requirements will be identified using the Job Training Analysis (JTA) Plan [Ref. 4].

The purpose of this JTA Plan is to outline the overall procedures for identifying the hazards and quality issues and training needs for each verification test project. This JTA Plan establishes guidelines for creating a work atmosphere that meets the quality, environmental, and safety objectives of the ETV-MF Pilot. The JTA Plan describes the method for studying ETV-MF project activity and identifying training needs. The ETV-MF Operation Planning Checklist (***Appendix C***) will be used as a guideline for identifying potential hazards, and the Job Training Analysis Form (***Appendix D***) will be used to identify training requirements. After completion of the form, applicable training will be performed. Training will be documented on the ETV-MF Project Training Attendance Form (***Appendix E***).

11.0 REFERENCES

1. US EPA Office of Research and Development, "*Waste Reduction in the Metal Fabricated Products Industry"* EPA/600/SR-93/144, September 1993.
2. George C. Cushnie Jr., CAI Engineering, *"Pollution Prevention and Control Technology for Plating Operations" NCMS/NAMF*, 1994.
3. Concurrent Technologies Corporation, *"Environmental Technology Verification Program Metal Finishing Technologies (ETV-MF) Quality Management Plan" December 9*, 1998.
4. Concurrent Technologies Corporation, *"Environmental Technology Verification Program Metal Finishing Technologies (ETV-MF) Pollution Prevention Technologies Pilot Job Training Analysis Plan" May 10*, 1999.

12.0 DISTRIBUTION

Alva Daniels, EPA (3)

Timothy Callahan, BioClean USA

Dick Hall, National Manufacturing Company

Jackie Molina, National Manufacturing Company

A. Gus Eskamani, CAMP, Inc. (2)

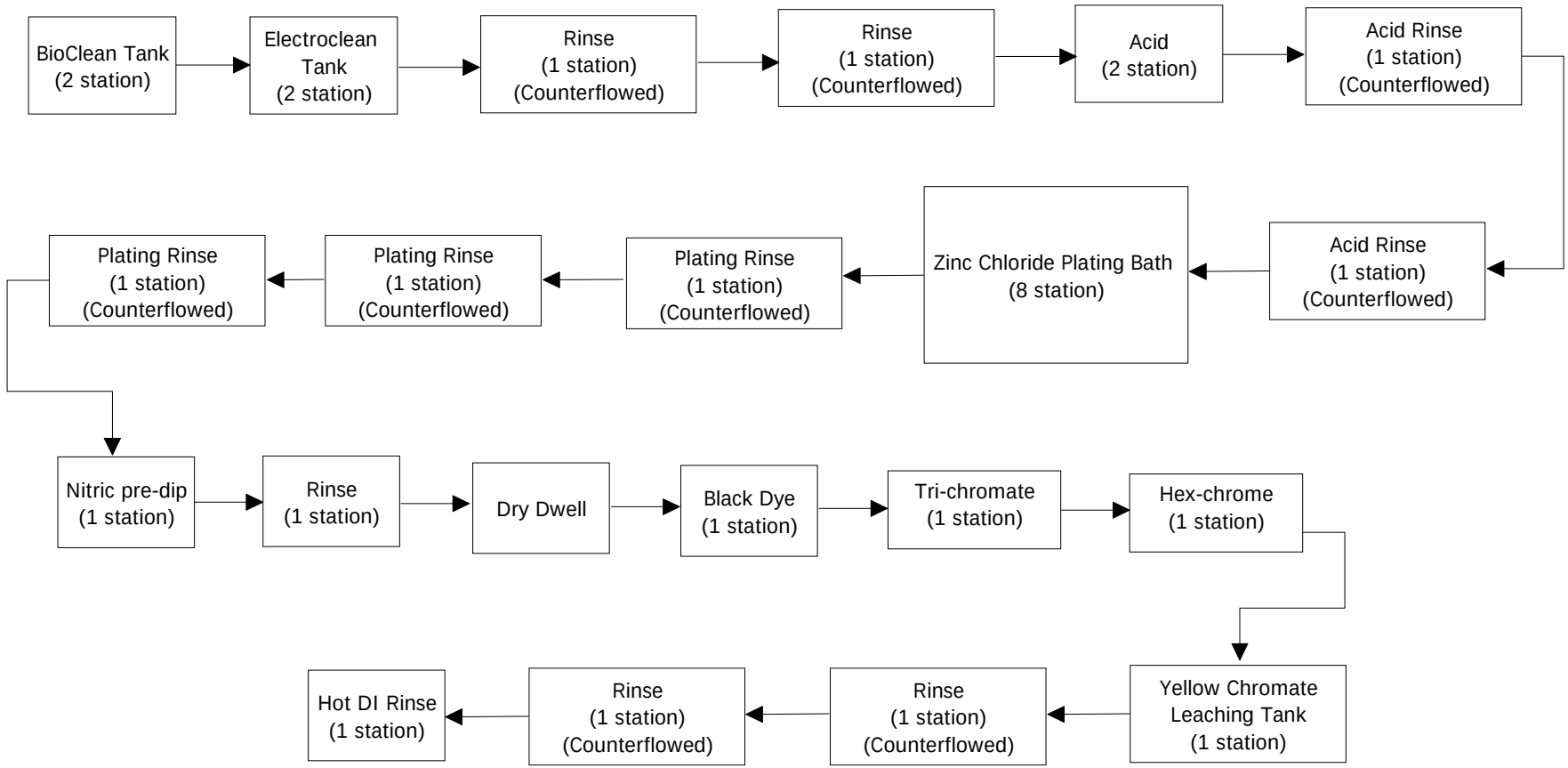
Donn Brown, *CTC* (3)

Clinton Twilley, *CTC*

Appendix A

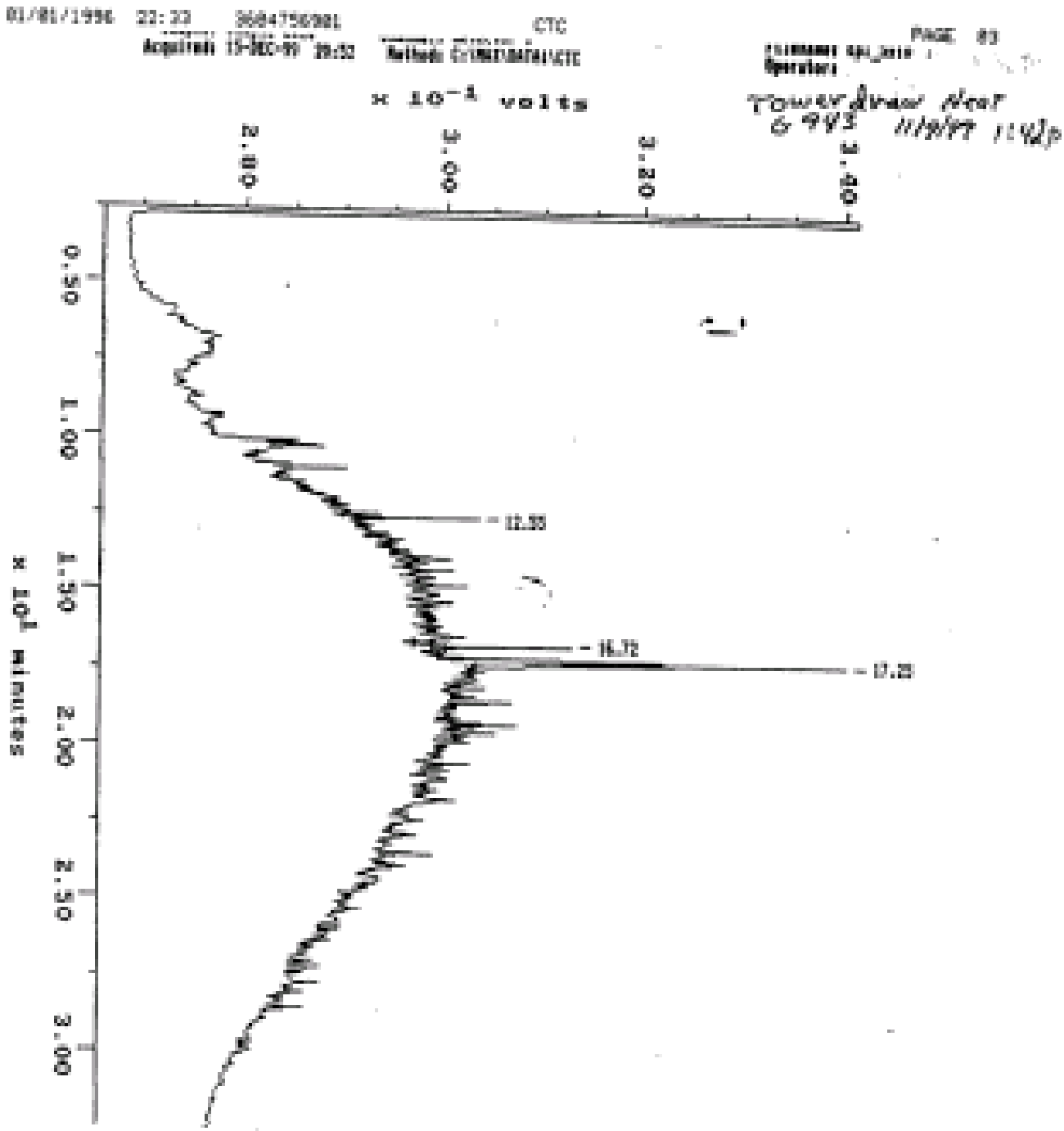
National Manufacturing Plating Process Flow

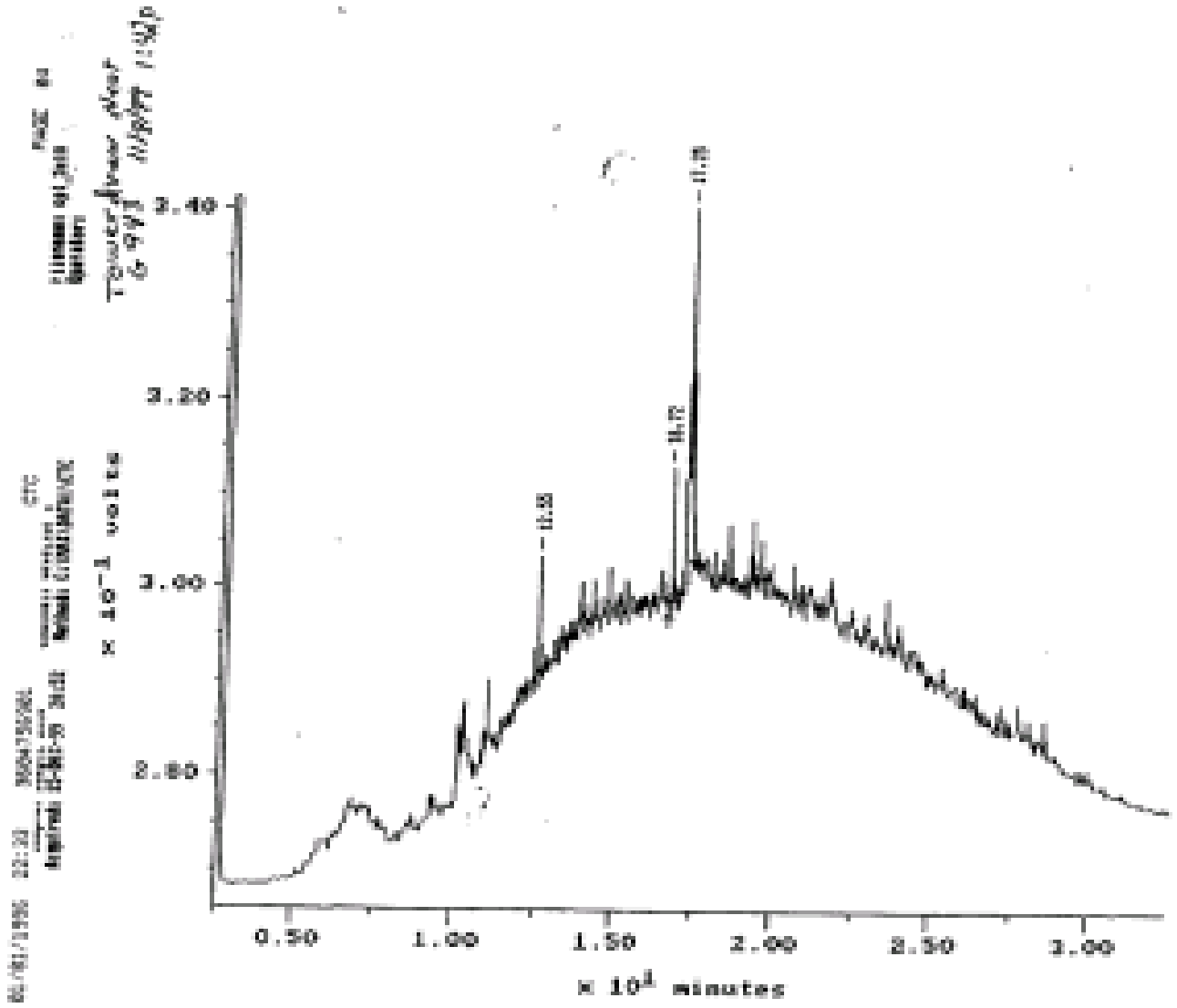
NATIONAL MANUFACTURING STERLING, ILLINOIS
PLATING LINE PROCESS FLOW

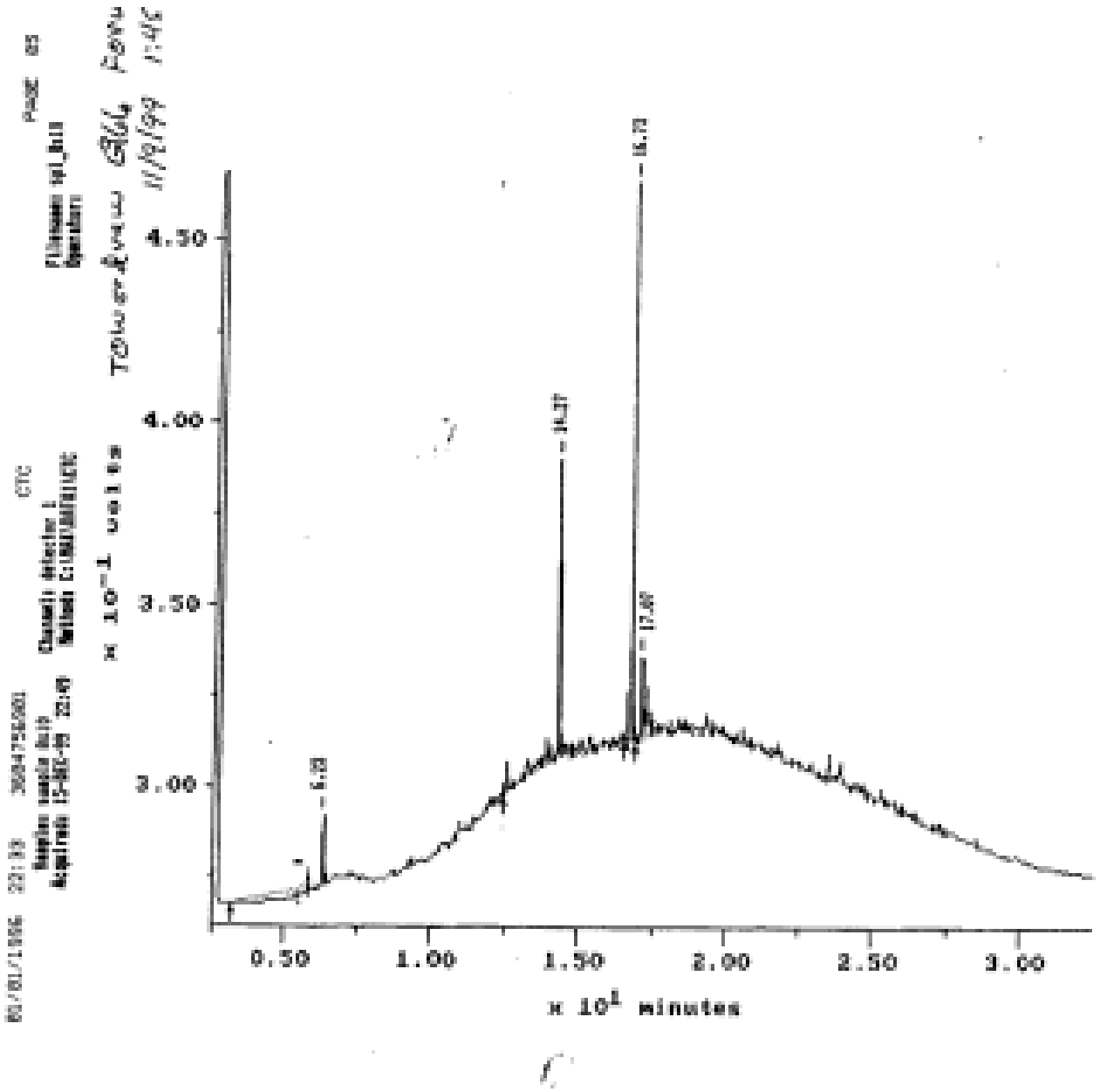


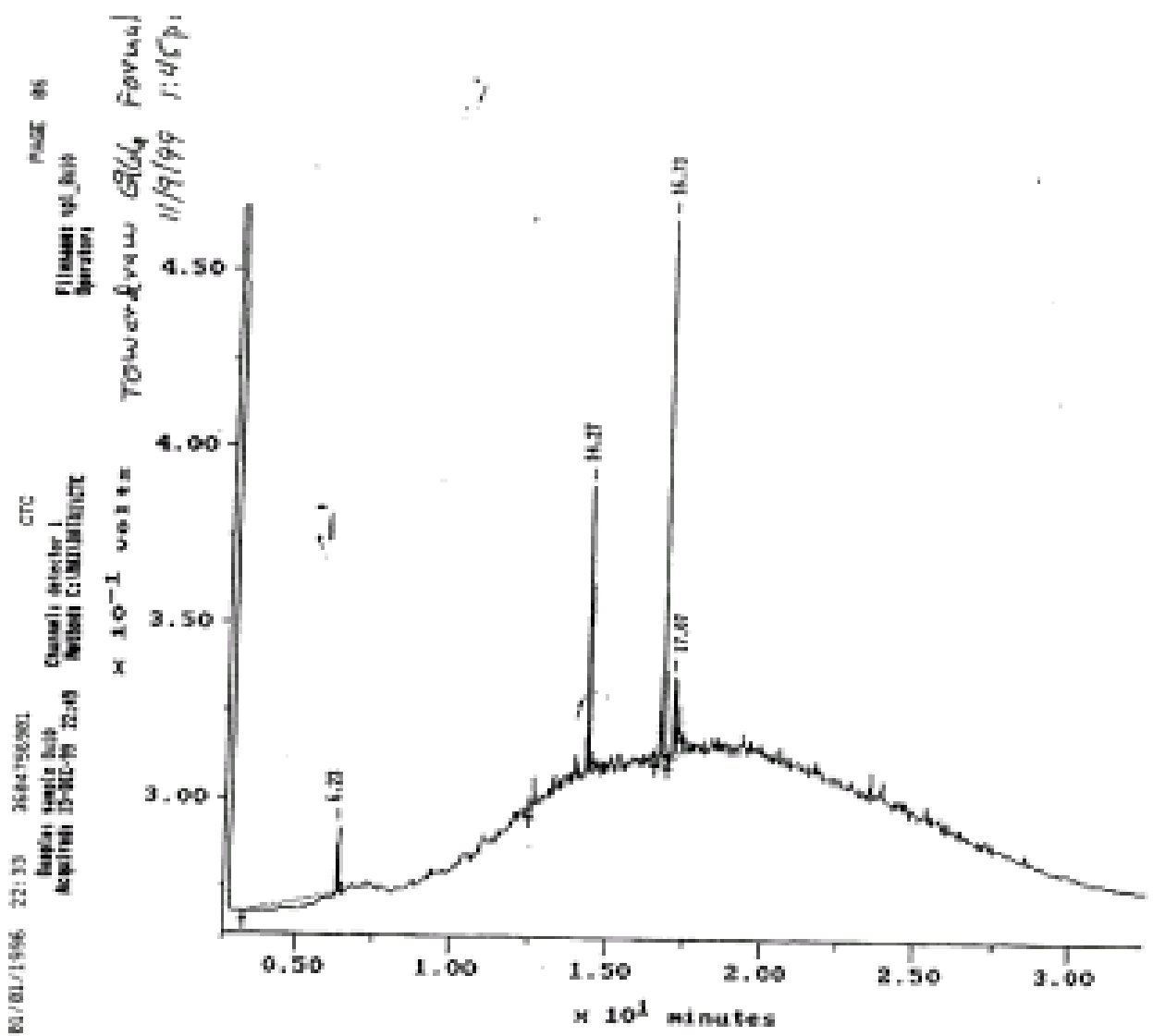
Appendix B

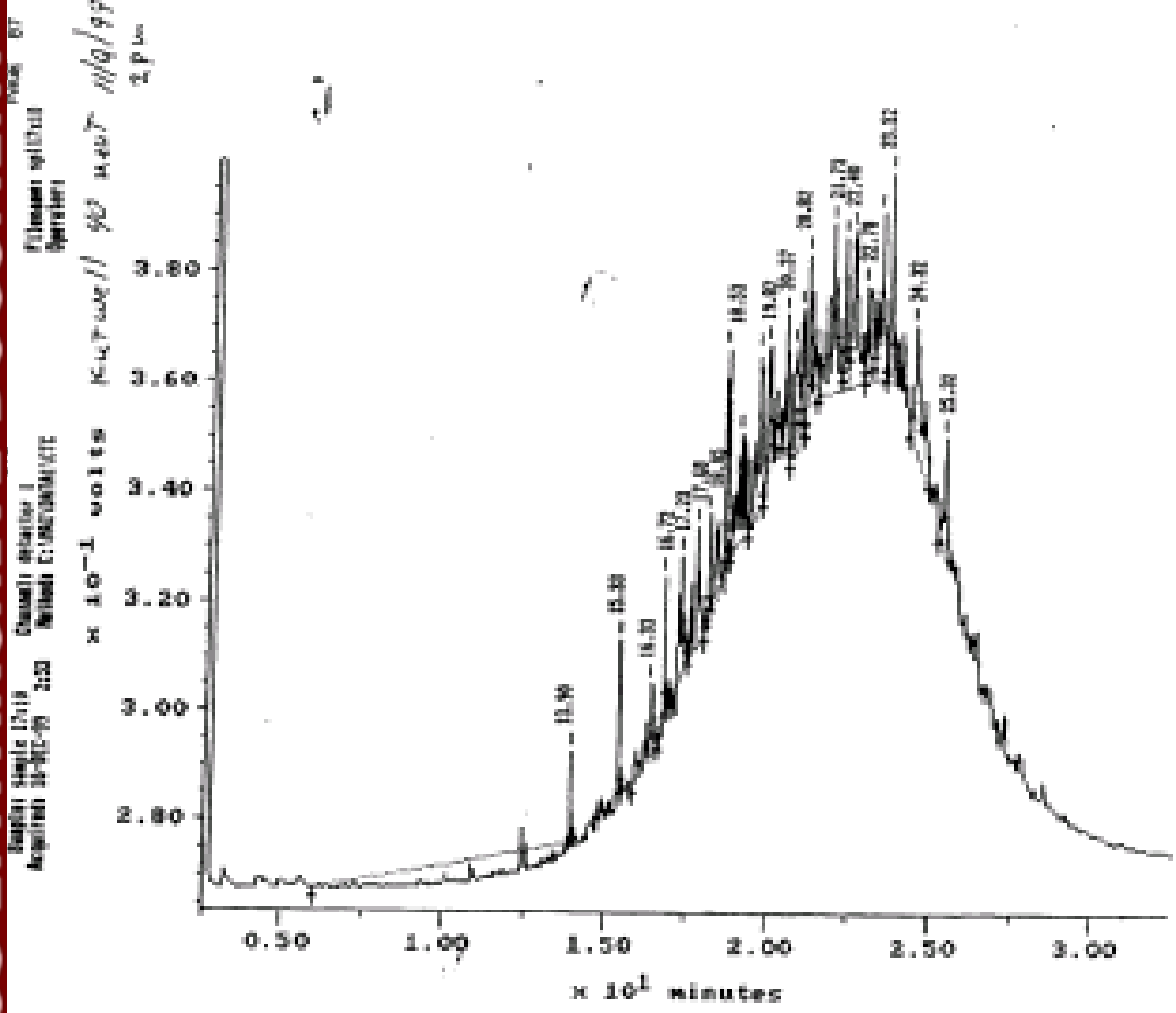
Organic Soil Analysis Chromatograms

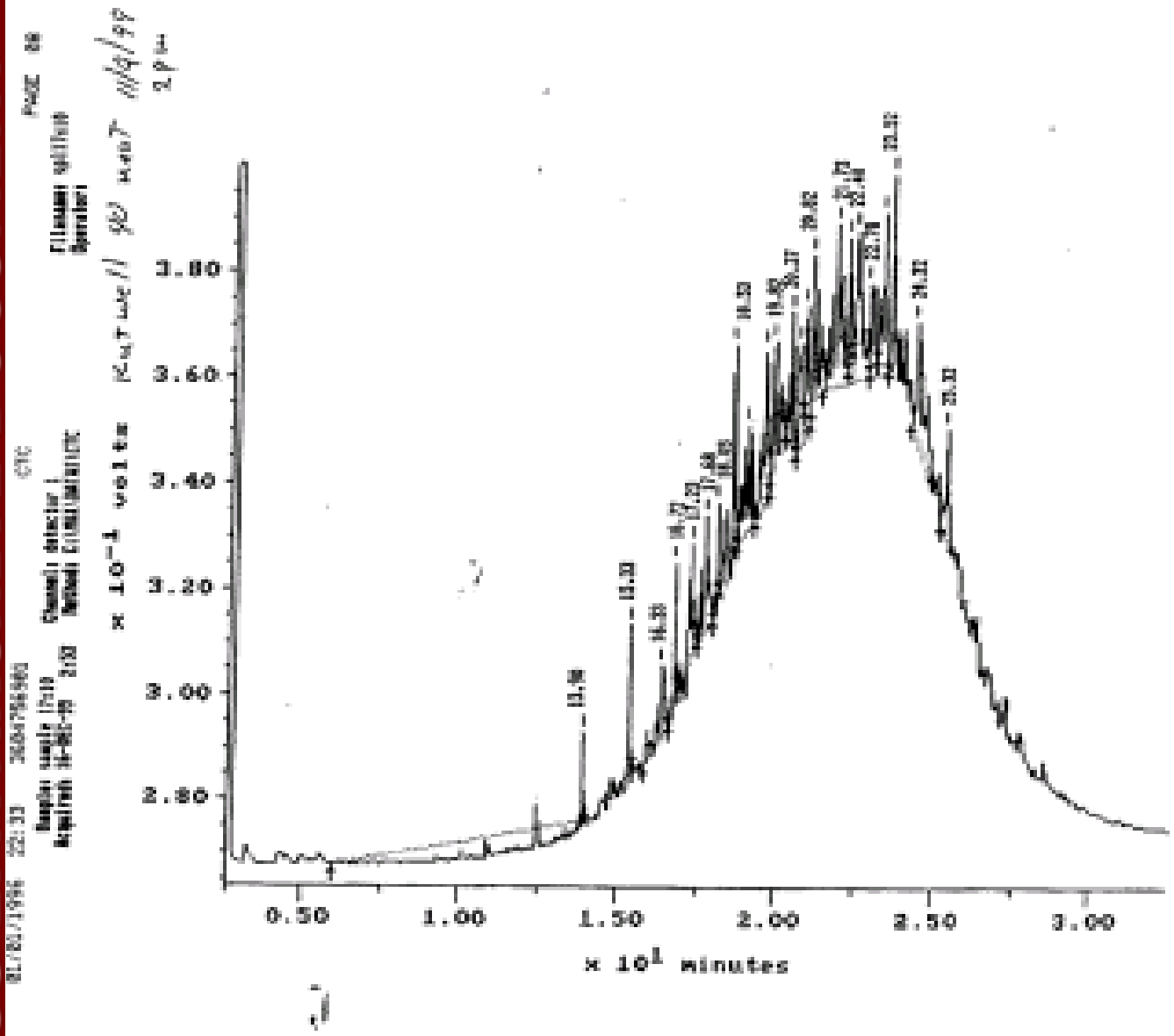


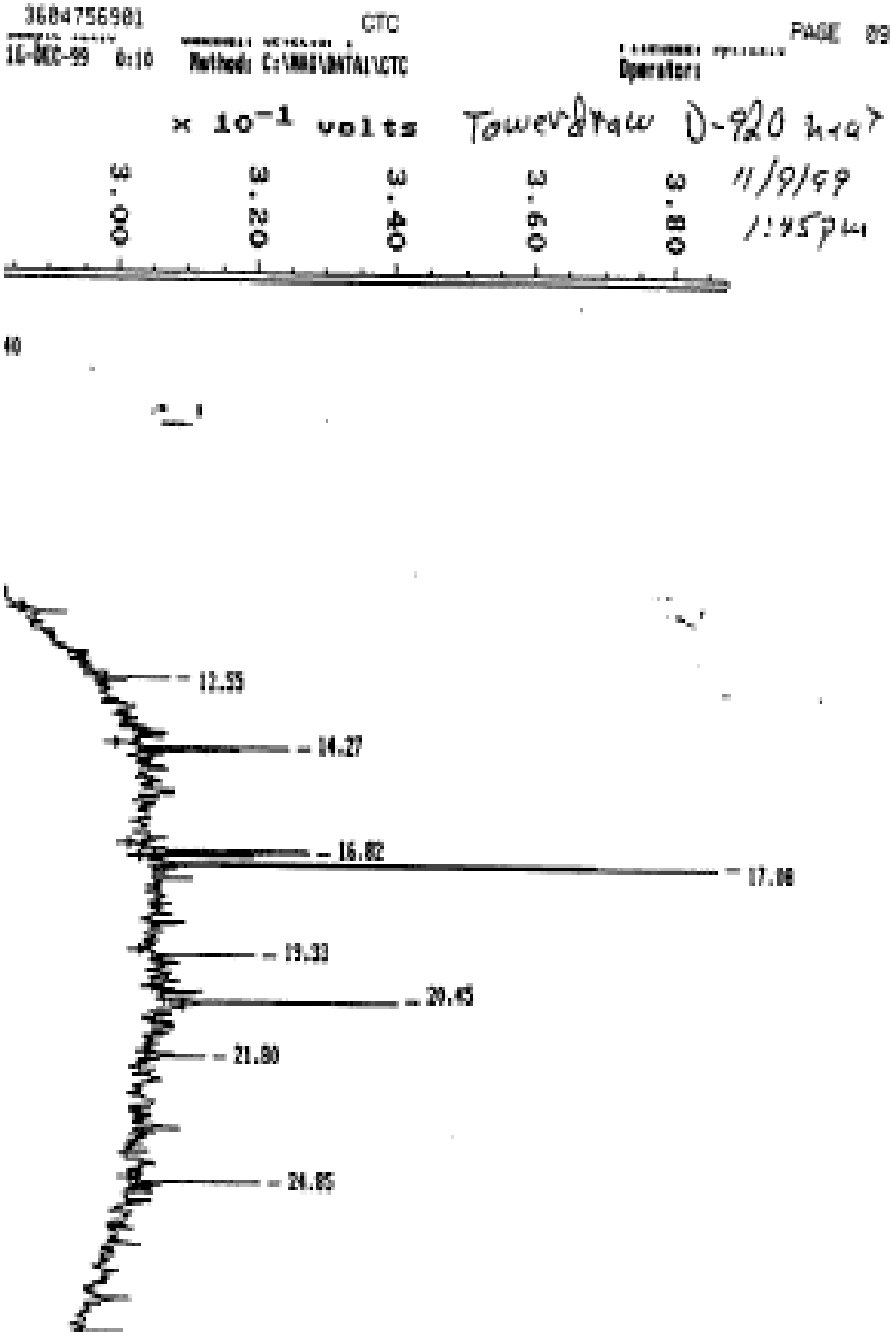


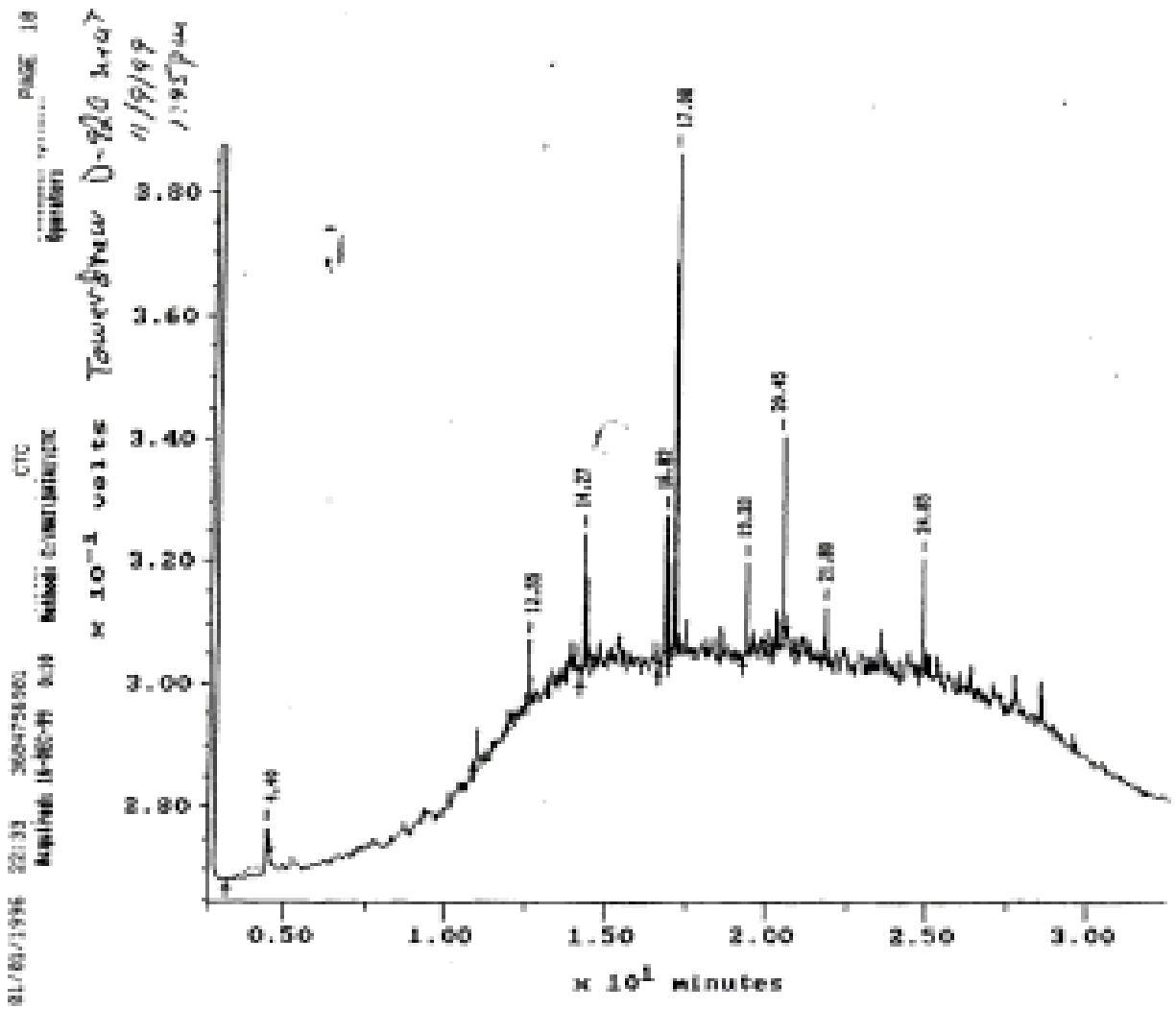


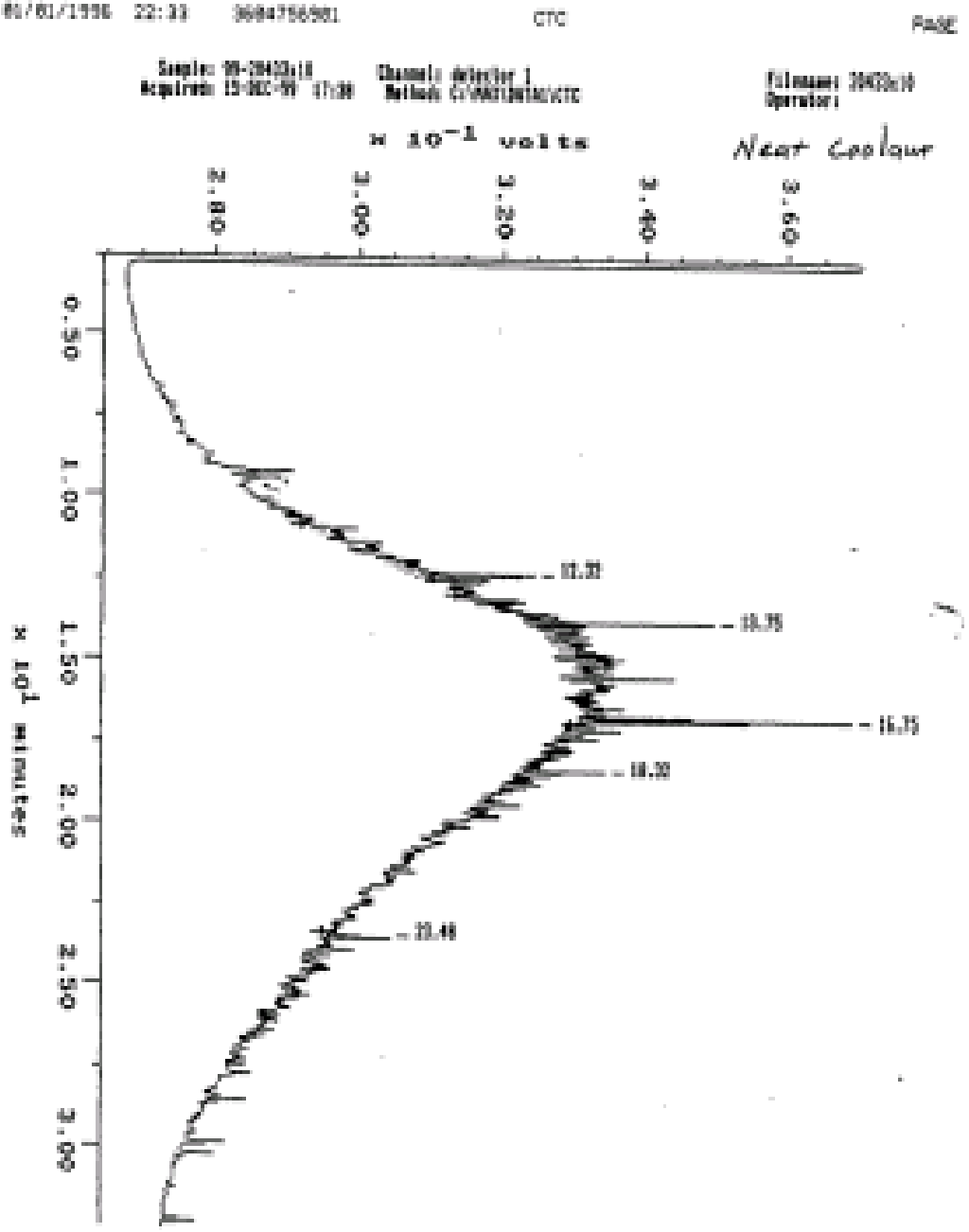


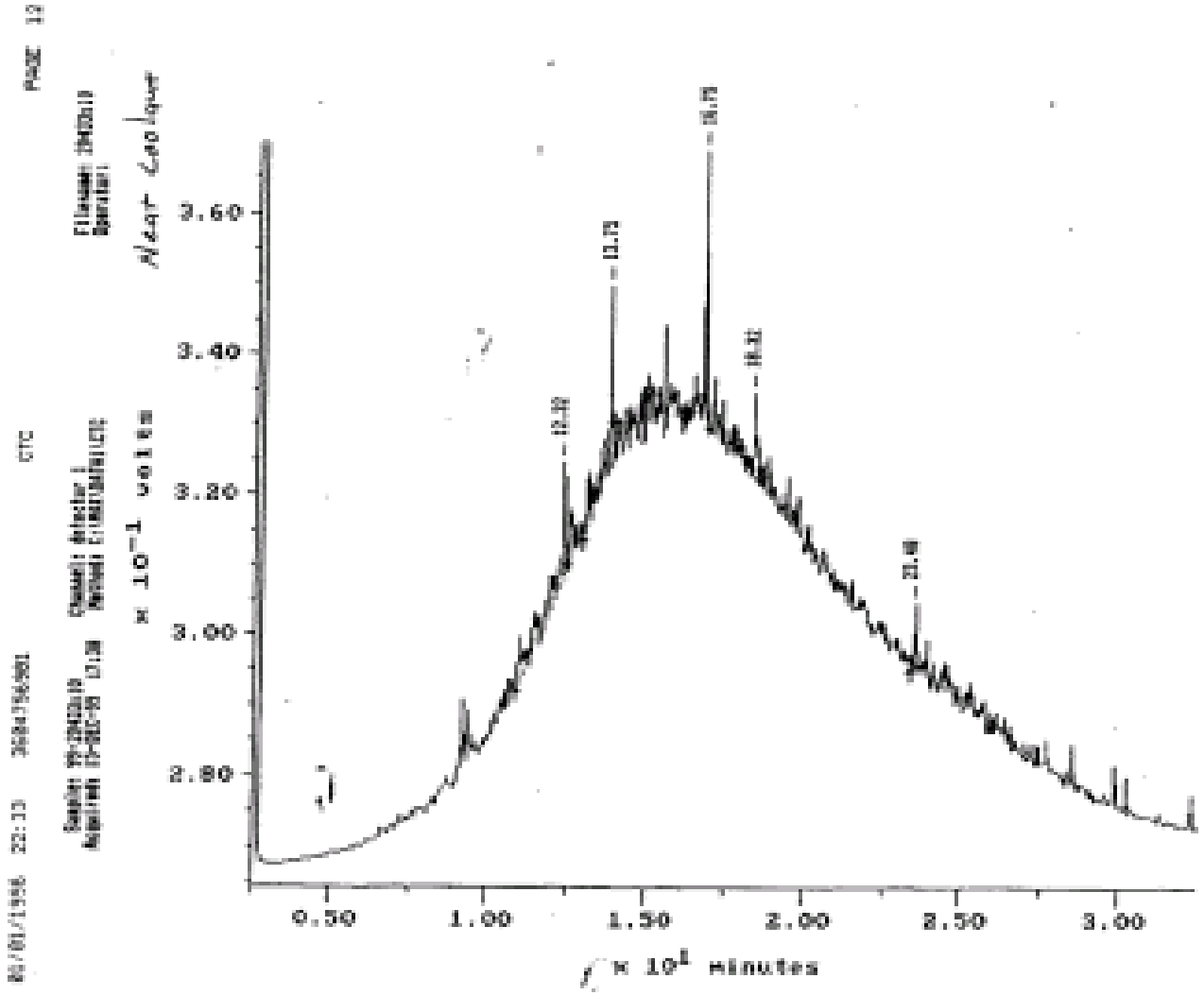


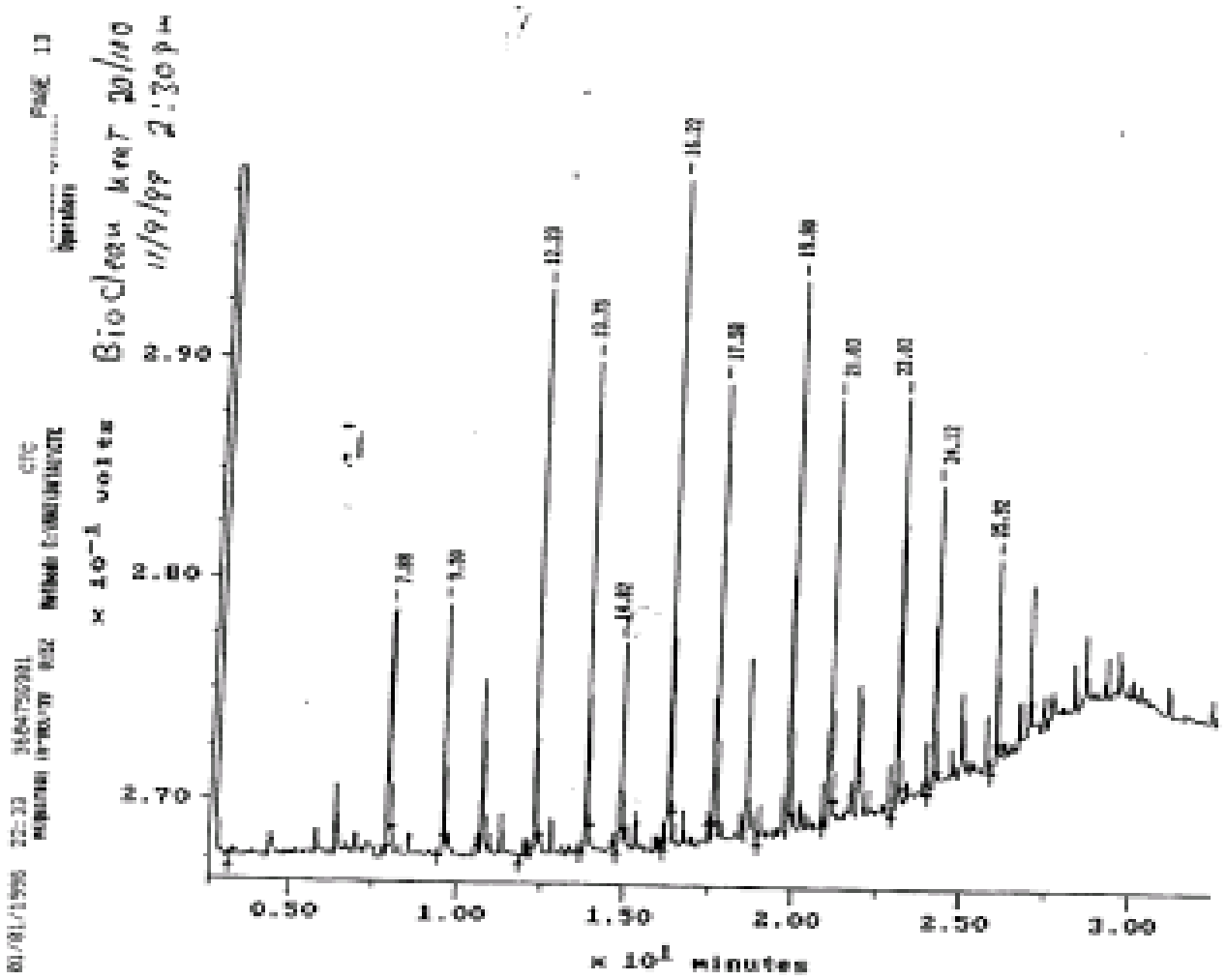


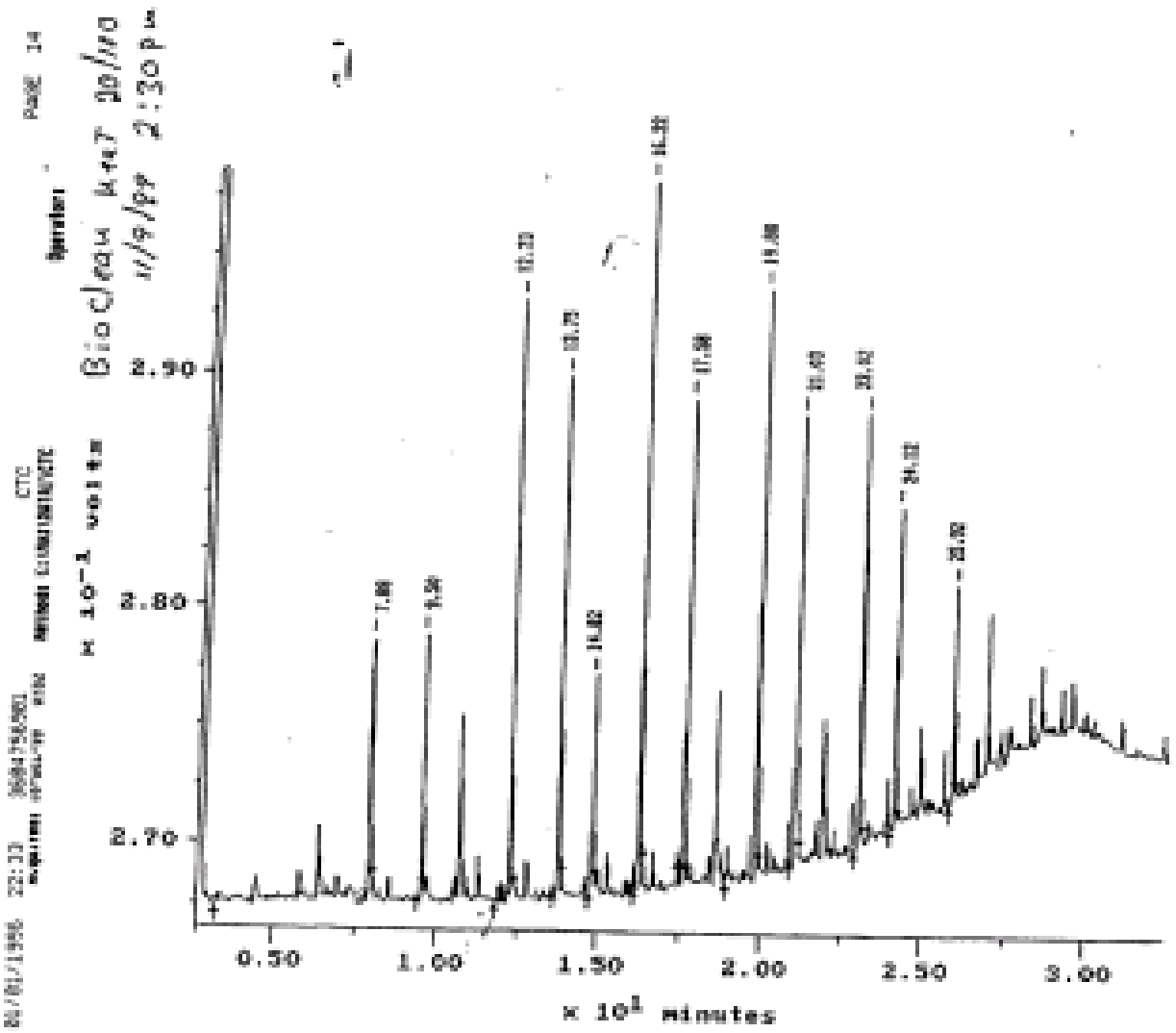


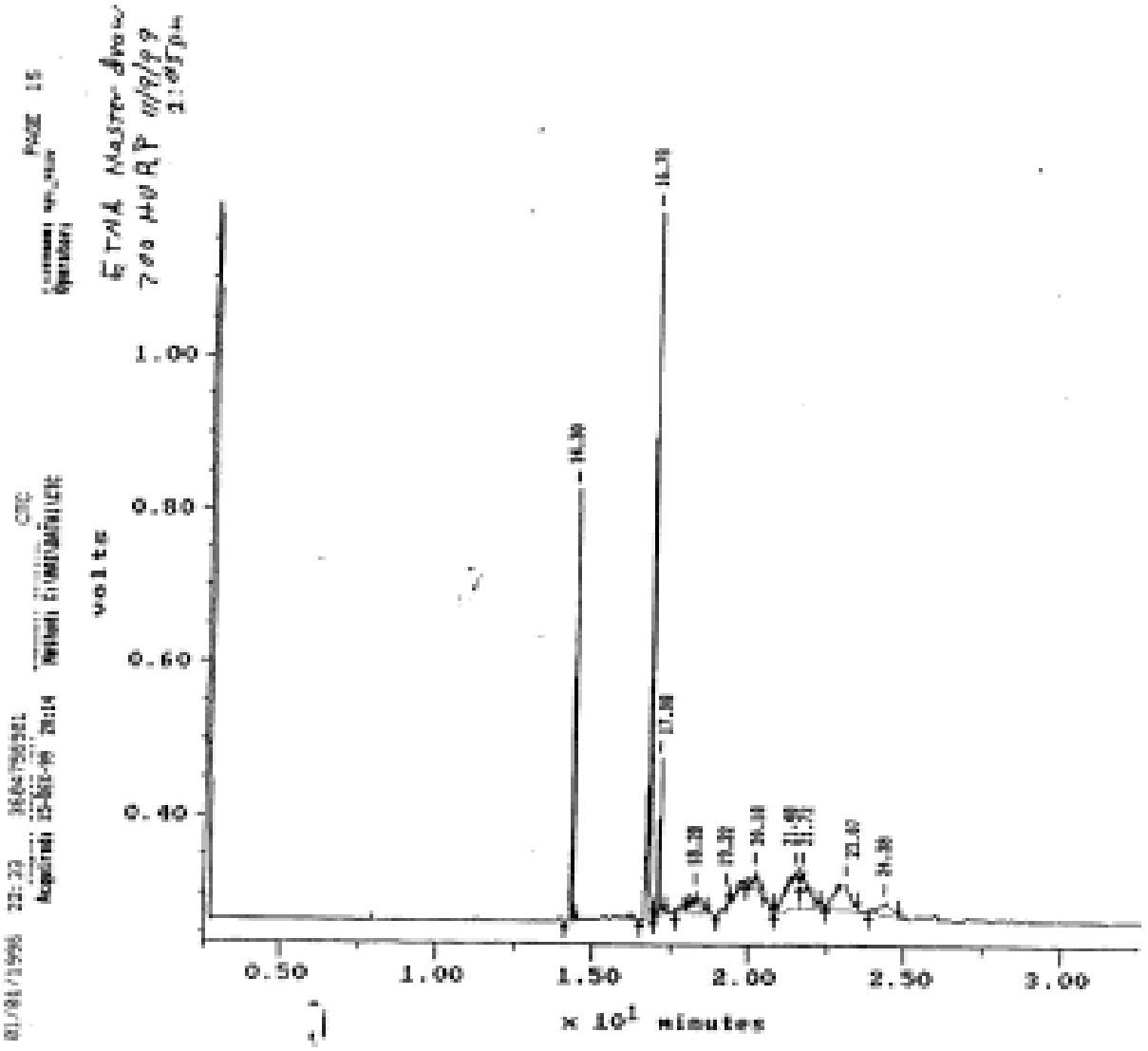


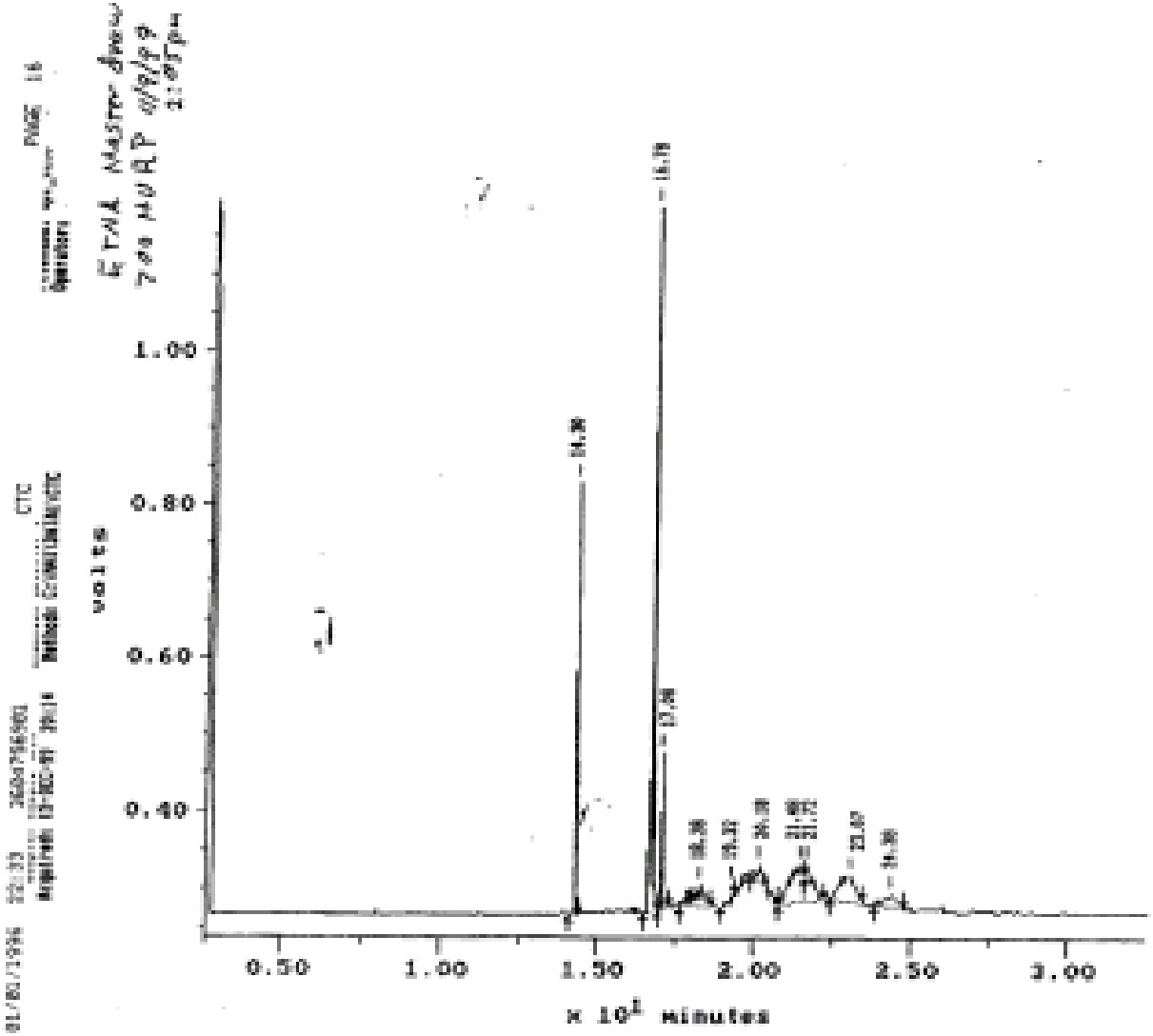












Appendix C
ETV-MF Operation Planning Checklist

The ETV-MF Project Manager prior to initiation of verification testing must complete this form. If a "yes" is checked for any items below, an action must be specified to resolve the concern on the Job Training Analysis Form.

Project Name: Expected Start Date:

ETV-MF Project Manager: _____ _____

Will the operation or activity involve the following:	**Yes**	**No**	**Initials & Date Completed**
1. Equipment requiring specific, multiple steps for controlled shutdown? (e.g. in case of emergency, does equipment require more than simply pressing a "Stop" button to shut off power?) *Special Procedures for emergency shutdown must be documented in Test Plan.*			
2. Equipment requiring special fire prevention precautions? (e.g. Class D fire extinguishers)			
3. Modifications to or impairment of building fire alarms, smoke detectors, sprinklers or other fire protection or suppression systems?			
4. Equipment lockout/tagout or potential for dangerous energy release? *Lockout/tagout requirements must be documented in Test Plan.*			
5. Working in or near confined spaces (e.g., tanks, floor pits) or in cramped quarters?			
6. Personal protection from heat, cold, chemical splashes, abrasions, etc.. *Use Personal Protective Equipment Program specified in Test Plan.*			
7. Airborne dusts, mists, vapors and/or fumes? *Air monitoring, respiratory protection, and /or medical surveillance may be needed.*			
8. Noise levels greater than 80 decibels? *Noise surveys are required. Hearing protection and associated medical surveillance may be necessary.*			
9. X-rays or radiation sources? *Notification to the state and exposure monitoring may be necessary.*			
10. Welding, arc/torch cutting, or other operations that generate flames and/or sparks outside of designated weld areas? *Follow Hot Work Permit Procedures identified in Test Plan.*			
11. The use of hazardous chemicals? *Follow Hazard Communication Program, MSDS Review for Products Containing Hazardous Chemicals. Special training on handling hazardous chemicals and spill clean up may be needed. Spill containment or local ventilation may be necessary.*			
12. Working at a height of six feet or greater?			

ETV-MF OPERATION PLANNING CHECKLIST

The ETV-MF Project Manager prior to initiation of verification testing must complete this form. If a "yes" is checked for any items below, an action must be specified to resolve the concern on the Job Training Analysis Form.

Project Name: _____

ETV-MF Project Manager: _____

Will the operation or activity involve the following:	**Yes**	**No**	**Initials & Date Completed**
13. Processing or recycling of hazardous wastes? *Special permitting may be required.*			
14. Generation or handling of waste?			
15. Work to be conducted before 7:00 a.m., after 6:00 p.m. and/or on weekends? *Two people must always be in the work area together.*			
16. Contractors working in *CTC* facilities? *Follow Hazard Communication Program.*			
17. Potential discharge of wastewater pollutants?			
18. EHS aspects/impacts and legal and other requirements identified?			
19. Contaminants exhausted either to the environment or into buildings? *Special permitting or air pollution control devices may be necessary.*			
20. Any other hazards not identified above? (e.g. lasers, robots, syringes) *Please indicate with an attached list.*			

The undersigned responsible party certifies that all applicable concerns have been indicated in the "yes" column, necessary procedures will be developed, and applicable personnel will receive required training. As each concern is addressed, the ETV-MF Project Manager will initial and date the "initials & date completed" column above.

ETV-MF Project Manager: _____ (Name) _____ (Signature) _____ (Date)

Appendix D
JOB TRAINING ANALYSIS FORM

ETV-MF Project Name:

Basic Job Step	Potential EHS Issues	Potential Quality Issues	Training

ETV-MF Project Manager:_____ _____

Name **Signature**

_____

Date

Appendix E
ETV-MF Project Training Attendance Form

ETV-MF Pilot Project:

Date Training Completed	Employee Name Last First	Training Topic	Test Score (If applic.)

ETV-MF Project Manager: _____