QUALITY ASSURANCE PROJECT PLAN
TUTU SERVICE STATION INVESTIGATION
ST. THOMAS, U.S. VIRGIN ISLANDS

March 1992

Prepared for

Tutu Environmental Investigation Committee

Prepared by

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Prepared by Geraghty & Miller, Inc. Date: March 20, 1991

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QUALITY ASSURANCE PROJECT PLAN
TUTU SERVICE STATION INVESTIGATION
ST. THOMAS, U.S. VIRGIN ISLANDS

1.0 INTRODUCTION

Geraghty & Miller Inc. has prepared this document on behalf of the Tutu Environmental Investigation Committee (TEIC) to establish sampling and analysis protocols and quality assurance and quality control (QA/QC) procedures to be applied to the collection and interpretation of soil and ground-water data for the Tutu site located in St. Thomas, U.S. Virgin Islands. This Quality Assurance Project Plan (QAPP) has been prepared in accordance with the U.S. Environmental Protection Agency (USEPA) document entitled "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" dated December 1980 (USEPA 1980). These procedures must be implemented so that the precision, accuracy, completeness, comparability, and representativeness of the data generated by this investigation can be documented.

The objectives of the Tutu Service Station Investigation are to delineate the potential sources, the horizontal and vertical extent, and potential migration pathways of petroleum hydrocarbon products in soil and ground water in the vicinity of Route 38 within the Tutu Wells site. The USEPA is also concerned about chlorinated hydrocarbon compounds (i.e., tetrachloroethene and its breakdown products) which have been detected in ground water in the Tutu area. To assist the USEPA in evaluating the Tutu area, the TEIC has agreed to install deep monitoring wells (i.e., screened below the water table) and analyze all soil and ground water samples for target compound list (TCL) and other specified volatile organic compounds (VOCs), base neutral and acid (BNA) extractable compounds, target analyte list (TAL) inorganics, and total petroleum hydrocarbons (TPH).

Included in this QAPP are the organization, policies, objectives, field and laboratory investigation procedures, and QA/QC requirements for the investigation activities as outlined in the Work Plan (Geraghty & Miller, Inc. 1992a). A summary of the proposed sampling and laboratory analyses are presented in Table 1-1.

2.0 PROJECT DESCRIPTION

2.1 SITE DESCRIPTION

The Tutu site area is located in the upper Turpentine Run basin in east central St. Thomas, U. S. Virgin Islands (Figure 2-1). Several water supply wells are located in the Turpentine Run basin. During July through September 1987, the U.S. Virgin Islands Department of Planning and National Resources (DPNR) closed 18 wells in the Tutu area due to the presence of volatile organic compounds in well water samples.

Various commercial establishments line the major roads in the area. These establishments include Texaco Tutu service station at the intersection of Highways 38 and 384 and the Esso Tutu service station on Highway 38. Private homes and multi-family housing, such as the Virgin Islands Housing Authority (VIHA) buildings, generally occupy the less heavily traveled roads. Water supply wells were operated by the VIHA, water purveyors, and private concerns for domestic and commercial purposes.

A brief reconnaissance of the area, conducted by Geraghty & Miller in September 1990, revealed that there are other establishments which represent potential sources of constituents of concern to soil and ground water. These establishments include but may not be limited to the following:

- The LAGA building, which formerly housed a textile processing operation.
- A Jeep dealership and repair shop.
- The Ramsay Motor dealership and repair shop.
- The maintenance area of the VIHA complex.
- Tillett Garden, where silk screening and pottery manufacturing and glazing are performed.
- O'Henry dry cleaners.
- The paint store.
- The fire station.

- The Vitelco facility.
- Other small repair shops located on Highway 38.
- Septic tanks.
- The sanitary and storm sewer lines.

2.2 BACKGROUND

The Tutu Environmental Investigation Committee (TEIC), which is comprised of Texaco Caribbean, Inc. (Texaco) and Esso Virgin Islands, Inc. (Esso), retained Geraghty & Miller, Inc. in December 1989 to prepare a work plan for the investigation of soil and ground-water conditions in the vicinity of the Tutu Wells Site, Anna's Retreat, St. Thomas, U.S. Virgin Islands (USVI). The Work Plan was developed to be incorporated in the Administrative Order of Consent (Order) that will be issued by USEPA Region II, pursuant to Subtitle I of the Resource Conservation and Recovery Act (RCRA) and agreed upon by Texaco and Esso. Geraghty & Miller submitted a Draft Work Plan dated January 1991 to the USEPA (Geraghty & Miller, Inc. 1991a). Revised Work Plans, addressing USEPA comments, were submitted on May 30, 1991 (Geraghty & Miller, Inc. 1991b) and October 14, 1991 (Geraghty & Miller, Inc. 1991c).

The Work Plan describes the methodology and procedures to be used during a three component investigation including 1) a soil investigation; 2) a ground-water investigation and, if necessary, 3) a free product investigation. As part of these investigation soil and ground-water samples will be collected for laboratory analysis. The scope of work includes ten soil borings and seventeen ground-water monitoring wells.

In conjunction with the Work Plan, a Health and Safety Plan (HASP) for the Tutu Service Station Investigation has been developed as a separate document (Geraghty & Miller, Inc. 1991d). The HASP has been prepared for use during the field investigation portion of the study. The QAPP discusses QA/QC procedures to be employed for the analytical portion of the Work Plan (Geraghty & Miller, Inc. 1992a).

Quarterly monitoring of supply well water quality within the study area, is being carried out by Soil Tech of San Juan, Puerto Rico and Geraghty & Miller in accordance with a USEPA-approved work plan entitled "Sampling, Analysis, and Monitoring Plan for Wells, Tutu Wells Site, St. Thomas, U.S. Virgin Islands" (Geraghty & Miller, Inc. 1990). A revised sampling, analysis, and monitoring plan (SAMP) was submitted in September 1991 to clarify analytical procedures (Geraghty & Miller, Inc. 1991e). The objectives of the SAMP are to identify, quantify, and monitor the occurrence of gasoline constituents and chlorinated hydrocarbons in supply wells in the vicinity of Route 38 within the Tutu Wells Site. Data from the SAMP quarterly sampling (Geraghty & Miller, Inc. 1991f; 1991g; 1991h; 1992b) will be used in conjunction with data generated from the Work Plan to evaluate hydrogeologic and water quality conditions. In accordance with the SAMP, sampling of supply wells will continue for up to eight quarterly sampling events. The data quality objectives (DQOs) for the supply well sampling are addressed in the SAMP and the ongoing quarterly reports. Therefore, this QAPP only addresses the objectives and quality assurance (QA) requirements of the Tutu Service Stations Investigation Work Plan (Geraghty & Miller, Inc. 1992a).

2.3 TARGET COMPOUNDS AND REPORTING LIMITS

For the purposes of this investigation the target compounds were identified by reviewing results from previous investigations and by incorporating specific target parameters recommended by the USEPA Region II to confirm their presence or absence at the site. Based on the previous investigations conducted at the site, the compounds of interest will include VOCs, BNAs, metals, cyanide, and TPH. The VOCs detected most frequently are: trichloroethene (TCE); 1,2 dichloroethane; tetrachloroethene (PCE); 1,1,2-trichloroethane; vinyl chloride; benzene; and toluene. The ground-water and soil samples collected throughout this investigation will be analyzed for the list of analytes in Tables 2-1 through 2-3 in accordance with the March 1990 USEPA Contract Laboratory Program (CLP) routine analytical services (RAS) protocols (USEPA 1990a; 1990b) and other specified methods.

Reporting limits for the site parameters in ground water and soil are listed in Tables 2-1 through 2-3. The quantitation limits for VOCs, BNAs, and TAL metals and cyanide are as specified in the organic and inorganic CLP statements of work (SOW) (USEPA 1990a; 1990b). The quantitation limits will be met unless sample dilutions or unknown interferences make it necessary to raise them. If quantitation limits are raised, the laboratory will make every effort to achieve reporting limits as low as possible and will report estimated concentrations at less than the quantitation limit.

2.4 SAMPLING NETWORK

The sampling procedures and total number of samples are discussed in detail in the Work Plan (Geraghty & Miller, Inc. 1992a).

2.5 SCHEDULE

The proposed schedule for sampling events and laboratory analyses is included in the Work Plan (Geraghty & Miller, Inc. 1992a).

3.0 PROJECT ORGANIZATION AND RESPONSIBILITY

Geraghty & Miller will be responsible for the overall management of the project, including the supervision of subcontractor activities, the interpretation and evaluation of data, and all field activities.

3.1 PROJECT ORGANIZATION

The primary Geraghty & Miller personnel involved in the project, their addresses and phone numbers are listed below. The location noted in parentheses following the individual's name indicates which Geraghty & Miller office these individuals are primarily affiliated with and where they may be contacted.

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Project QA Officer:	Lidya Gulizia (New Jersey)
Project Chemist/ Data Validator:	Cameron Dunnan (New Jersey)
Field Manager:	Clinton Moffatt (New Jersey)
Project Health and Safety Officer:	Alberto Colberg Nevares (Puerto Rico)

Primary responsibility for project management will be shared by the project officer and the project manager. Through the field manager, they will coordinate on-site field personnel, and provide technical assistance for activities which are directly related to the determination of site conditions.

The evaluation of laboratory data will be the responsibility of the Project Chemist and the Project QA Officer. They will attest to the validity and representativeness of the data. Data will be collected and processed by field hydrogeologists and the Field Manager, and reviewed by the Project Manager and the Project QA Officer. If QA issues requiring special attention are identified, the Project Officer, Project Manager, and the Project QA Officer will identify the appropriate corrective action(s) and implement it(them).

Other technical advisors will be available as needed to provide expertise for various disciplines including hydrogeology, geochemistry, engineering, and other specialty fields. An organizational chart is provided in Figure 3-1.

Analytical services will be provided by Enseco East of Somerset, New Jersey. A project management organizational chart for Enseco East is presented in Figure 3-2. Laboratory analyses of all samples will adhere to the internal QA/QC procedures of Enseco East, which is a participant in the USEPA CLP for organics and is contracted through the CLP for OLM01.1.1. These procedures are detailed in the laboratory QA Program Plan which is included in this document as Appendix A. These procedures meet or exceed USEPA QA/QC requirements under the CLP.

3.2 FIELD ORGANIZATION

Field investigations and activities will be according to the planned programs and schedules shown in the Work Plan (Geraghty & Miller, Inc. 1992a). For sampling work, the selection of the sampling team members will depend on the type and extent of sampling, and will consist of a combination of one or more of the following:

- Project Manager
- Field Manager
- Field Hydrogeologists

- Project QA Officer
- Site Health and Safety Officer

The Project Manager will be responsible for coordination of on-site personnel, and providing technical assistance when required. Daily logs will be maintained describing field activities. An example of a daily log form, as well as other forms that will be used to gather data are contained in the Work Plan (Geraghty & Miller, Inc. 1992a).

Field hydrogeologists will be responsible for sample collection, chain-of-custody, and sample shipment. A Sampling Coordinator will be designated by the Project Manager. This individual will be responsible for all sampling efforts, and for assuring that the necessary shipping materials, packing materials, and sampling equipment are available. Other responsibilities include completing sampling documentation, daily logs, water-sampling logs, soil-core logs, field instrument calibration logs, and chain-of-custody forms. Sample bottles, preservatives (if necessary), and shipping coolers will be provided by the laboratory.

The Project QA Officer will be responsible for the implementation of this QAPP during the field investigations. Adherence to these procedures will facilitate the collection of data of high quality and increase its usability. If the guidelines described in this plan require modifications due to site conditions, changes in the Work Plan, or for any other reasons, the Project QA Officer will be notified, and the changes will be documented and implemented.

The Project and Site Health and Safety Officers will be responsible for assuring that all team members adhere to the site health and safety requirements. Additional responsibilities are detailed in the Health and Safety Plan which has been prepared as a separate document (Geraghty & Miller, Inc. 1991d).

4.0 QUALITY ASSURANCE OBJECTIVES

The overall QA objective is to ensure that all data collected during field activities are of known and acceptable quality. Data-collection activities will adhere to the QA/QC procedures for the collection, preservation, documentation, and custody of samples. Specific laboratory QA/QC procedures and DQOs are described in the laboratory QA Program Plan (Appendix A). To ensure that data of acceptable quality are obtained, the QA objective parameters (precision, accuracy, completeness, representativeness, and comparability) to be evaluated are detailed below.

4.1 PRECISION

Precision is a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of standard deviation and or relative percent difference. Various measures of precision exist depending upon the " prescribed similar conditions". The precision of an analytical method, field measurement, or sampling technique is measured through duplicate analyses or replicate measurements.

Measurements of the precision of laboratory generated data are necessary to demonstrate the reproducibility of the data. Precision is evaluated by calculating the relative percent difference (RPD) between duplicate analyses. Duplicate analyses, matrix spike duplicates, and duplicate laboratory control samples will be analyzed at the rate specified in the laboratory QA Program Plan (Appendix A) and/or the analytical protocols to be used for the site investigation. Duplicate control samples (blank water spikes) are useful to assess non-matrix-specific precision. Matrix spike duplicates are used to assess matrix-specific precision. This difference is useful in determining the source of inadequate precision and, therefore, makes the corrective action necessary easier to identify.

For VOC, BNA, and TPH analyses, precision will be assessed by calculating RPD between matrix spike and matrix spike duplicate samples. Additionally, in the analysis of

TPH, duplicate laboratory control samples will be analyzed in order to evaluate analytical precision. For the analysis of TAL metals and cyanide, precision will be evaluated by calculating the RPD between a sample and a matrix duplicate sample. The acceptable range of precision for VOC, BNA, metals and cyanide will be within those limits specified by the CLP SOW (USEPA 1990a; 1990b). For analyses of TPH, the RPD will vary by less than twenty percent.

The precision of field measurements for pH, temperature, and specific conductance will be assessed through replicate measurements, and acceptable results will vary by less than 20 percent (RPD). The precision of sampling will be assessed through a comparison of field replicate results. Sampling precision is difficult to quantify and will be assessed qualitatively.

4.2 ACCURACY

Accuracy describes the degree of agreement of a measurement with an accepted reference or "true" value. Accuracy is a measure of the bias in an analytical system or a specified matrix. Analytical accuracy will be determined from (1) the analyses of standard reference materials of known and traceable purity and quality, (2) the analyses of surrogate and system-monitoring compounds (SPCs), and/or (3) the analyses of blank and matrix samples fortified with representative analytes for the analytical fraction.

Analytical accuracy for VOC and BNA analyses will be determined from the analyses of SPCs or surrogates added to the sample prior to analysis. The SPC and surrogate acceptance limits for VOC and BNA analyses have been summarized in Table 3-1. For VOC, BNA and TPH analyses, matrix spike and matrix spike duplicate samples will be analyzed at the intervals specified in the organic CLP SOW (USEPA 1990a) or in the laboratory QA Program Plan (Appendix A). For metals and cyanide, accuracy will be assessed by matrix spike and laboratory control sample analyses. The SPCs, surrogates, and matrix spike compounds for VOC and BNA analyses specified i [illegible] CLP SOW

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(USEPA 1990a) will be used. Accuracy for VOCs, BNAs, metals, and cyanide results will be evaluated using the limit criteria specified in the CLP SOW (USEPA 1990a; 1990b). For TPH analyses, accuracy will be evaluated using a range of 80 to 120 percent recovery as the acceptance limit criteria.

Accuracy of field measurements will be assured through calibration techniques and the measurement of known standards.

4.3 COMPLETENESS

Completeness is a measure of the amount of valid data obtained from any measurement system compared to the amount of data set. Valid data are defined as data generated from samples that arrived at the laboratory intact, properly preserved, in sufficient quantity to perform the requested analyses, and accompanied by a chain-of-custody record. Furthermore, samples must be analyzed within the specified analytical holding times and analyzed with the appropriate and relevant level of quality control (QC) effort. The calculation for completeness will be performed after critical QC criteria have been reviewed and assessments for precision, accuracy, and achieved sensitivity have been performed. These preliminary assessments and the evaluation of the completeness objective will be done at the completion of each of the field investigation tasks described in the Work Plan (Geraghty & Miller, Inc. 1992a) during data validation phases. A discussion of completeness will be provided in the technical memorandums submitted to the USEPA following the completion of each investigatory task.

It is expected that Enseco East will provide data meeting QC acceptance criteria of 95 percent or more for all samples tested using the CLP RAS organic and inorganic protocols. For TPH analyses, although completeness is expected to be high, it may be reduced to below 95 percent due to the analytical methods employed.

As demonstrated in the CLP RAS protocols, precision and accuracy data for multi-component analyses are typically only provided for a subset of the analyte list, or for representative analytes of most analytical protocols and methods. Summaries of precision, accuracy, and completeness objectives for TPH and representative analytes in the RAS protocols in water and soil matrices have been included in Tables 3-2 through 3-4.

The methods selected for this investigation were chosen to achieve a specified detection limit in the samples. In the assessment of the completeness objective, an evaluation of the achieved sample reporting limits or sample sensitivity will be performed. Sensitivity may be defined as the minimum concentration of an analyte that can be measured and reported. In terms of samples, sensitivity is the lowest concentration of analyte(s) that can be reliably achieved within specified limits of method precision and accuracy. The evaluation of sensitivity and the assessment of whether the necessary quantitation limit(s) has been achieved to meet the DQOs will be made by comparing the sample reporting units to the quantitation limits listed in Tables 2-1 through 2-3.

4.4 REPRESENTATIVENESS

This parameter expresses the degree to which data accurately and precisely represent the characteristics of a population, parameter variations at a sampling point, a process condition, or an environmental condition. The representativeness of the data will be assessed in three areas: 1) the number of locations, matrices, and samples sufficient to accurately depict site conditions; 2) the sampling procedures which must be designed so that individual samples accurately represent the chemistry of the matrix from which they were collected; 3) the appropriateness of the analytical method used to the type of sample obtained. Specific sampling procedures to be used so that samples are as representative as possible are described in the sampling plan for the field investigations.

4.5 COMPARABILITY

Comparability expresses the confidence with which one data set can be compared to another. The comparability of the data is assured by using standard sampling and analysis procedures, and data reporting formats. The data will be generated in a manner such that similar data sets can be compared to each other, and individual comparisons can be made within each data set.

5.0 SAMPLING PROCEDURES

Procedures for collecting samples, conducting tests and other measurements are described in the Work Plan (Geraghty & Miller, Inc. 1992a). Also included in the Work Plan is information on sampling procedures, drilling procedures, equipment decontamination, sample documentation, sample shipment, field filtering (if necessary), preservation of samples, and chain-of-custody. Laboratory preservation, container types, and holding time requirements for the parameters to be analyzed are described in Table 5-1.

6.0 SAMPLE CUSTODY

Sample custody is a part of proper laboratory or field operation. Sample custody procedures are designed to provide documentation of preparation, handling and storage, and shipping of samples. Samples collected during this field investigation will be the responsibility of the Field Manager and sampling staff from the time samples are collected to the time when they are relinquished to the laboratory directly, or to a common carrier for daily shipment to the laboratory. Stringent chain-of-custody procedures will be adhered to at all times to document sample possession.

6.1 SAMPLE IDENTIFICATION

Each sample collected will be assigned a unique alphanumeric identification. All samples will begin with a two or three letter alphabetic prefix designating the type of sample (e.g., MW - Monitoring Well; SB - Soil from Boring; SMW - Soil from Monitoring Well boring; PT - Pumping Test). Following the alphabetic prefix will be a hyphen and a one to two digit number identifying the monitoring well or boring number. Deeper monitoring wells have a letter "D" following the monitoring well number. For pumping test and soil boring samples a third code will be added to designate the sequence or depth, respectively, of the sample.

6.1.1 Monitoring Well Samples

Ground-water samples collected from monitoring wells will be labelled according to the well designation. Monitoring wells are designated with the prefix "MW" followed by the well number. Deeper monitoring wells are designated with the suffix "D" (e.g., MW-10D).

6.1.2 Soil Boring Samples

The identification of soil samples collected from borings will begin with "S" followed by the boring or monitoring well designation. Borings where monitoring wells will not be installed are labelled with a "B"; borings where monitoring wells will be installed are labeled with the well identification. The depth interval of the soil sample (in feet below grade) will be indicated in the suffix. Examples of soil sample identifications are as follows:

SB-1 (8-10)	(Soil sample from boring B-1; depth 8 - 10 feet)
SMW-10D (4 - 4.5)	(Soil sample from Monitoring Well MW-10D; depth 4 - 4.5 feet).

6.1.3 Pumping Test Samples

Identification of pumping test samples will begin with the prefix "PT" followed by the designation of the well where the sample was collected. A numeric code will designate the sequence of the sample collection as follows:

PT-MW-10D-1	(1st sample collected)
PT-MW-10D-2	(2nd sample collected)

6.1.4 Field Replicates

Field replicates will be designated using the appropriate alphabetic prefix followed by a three digit number starting with 100. The date of sampling will be recorded on the sample container and the chain-of-custody; however, the time of sampling will not be recorded on the chain-of-custody to keep the sample identity concealed from the laboratory. Field notes maintained by the sampling personnel will record the actual location of field replicates. The following are examples of field replicate sample identifications:

MW-100
MW-101
SB-100
SB-101

6.1.5 Field Blanks

Field blanks will be labeled as "FB" followed by a number in sequence. The sampling personnel will maintain field notes documenting where and how the field blanks are collected. Field blank samples will be identified as follows:

FB-1
FB-2

6.2 FIELD CUSTODY

The sampling staff is responsible for the care and custody of the samples until they are delivered to the contracted laboratory or to the assigned courier. The sample containers used for shipment will be sealed on site by the field sampling crew using nylon strapping tape and chain-of-custody seals. Sample bottles will be kept in the shipping containers except when they are being filled in the field.

Sample shipping and handling procedures will be in compliance with the requirements of the organic and inorganic CLP SOWs (USEPA 1990a; 1990b). The USEPA CLP considers sample holding times to begin at the time the sample is received by the laboratory. Geraghty & Miller will comply with sample holding times beginning from the time of sample collection. The signed and dated chain-of-custody form will be included in the shipping container.

6.3 CHAIN-OF-CUSTODY FORMS

All the sampling and custody paperwork to be completed prior to, during, and after sampling activities is included in the Work Plan in Appendix I (Geraghty & Miller, Inc. 1992a). The Laboratory Task Order (LTO) form is an integral part of the Geraghty & Miller Analytical Quality Assurance and Laboratory Control Program (AQA/LCP). This form must be completed prior to each sampling program and it serves to identify the samples to be collected, the analyses to be performed, the analytical methods to be used, the detection limits to be achieved, the required level of laboratory QA/QC deliverables, and laboratory reporting format. Chain-of-custody forms are to be completed prior to sample shipment, with identification of samples collected, number of bottles filled, and the requested analyses.

6.4 SAMPLE SHIPMENT PROCEDURES

Samples requiring refrigeration will be promptly chilled with ice to an approximate temperature of four degrees Celsius and packaged in an insulated cooler for shipping to the laboratory. To prevent breakage, the sample containers will be packed using vermiculite or bubble-wrap packing. Nylon strapping tape will be used to seal the coolers for transportation to the laboratory. Custody seals will be affixed to the coolers to allow the receiver to quickly identify any tampering which may have taken place during transport. The shipping coolers will be sent out daily to the laboratory for next day delivery via an overnight common carrier.

7.0 CALIBRATION PROCEDURES

All calibration procedures for VOCs, BNAs, metals, and cyanide will be as specified in the CLP SOW (USEPA 1990a; 1990b). For the TPH analyses, calibration will be performed in accordance with the procedures specified in USEPA Method 418.1 of Methods of Chemical Analysis of Water and Wastes (USEPA 1983) and the laboratory QA Program Plan (Appendix A).

Field equipment will be calibrated by trained Geraghty & Miller personnel according to approved manufacturer's specifications and instructions, and the protocols appended to the Work Plan (Geraghty & Miller, Inc. 1992a).

8.0 ANALYTICAL PROCEDURES

Samples collected during the field investigation will be analyzed for TCL VOCs, 1,2-dibromomethane, n-propylbenzene, methyl tert-butyl ether (MTBE), TCL BNAs, TAL metals, cyanide, and TPH. A summary of analytial protocols and methodologies to be used in support of the field investigation may be found in Table 8-1.

All VOC and BNA analyses will be performed in accordance with the USEPA March 1990 CLP organic RAS SOW (USEPA 1990a). The SOW will be modified during VOC analysis in order to analyze and quantitate 1,2-dibromomethane, n-propylbenzene, and MTBE in all field samples.

For TAL metal and cyanide analyses, the USEPA March 1990 inorganic SOW protocols will be used for the quantitation of TAL analytes (USEPA 1990b).

For the analysis of TPH in water samples, preparation and instrumental analysis will be performed in accordance with USEPA Method 418.1 in *Methods of Chemical Analysis of Water and Wastes* (USEPA 1983). Soil samples for TPH analysis will be prepared in accordance with the U.S. Army Corp of Engineers (USACOE) Oil and Grease Method CE81-1 following the infrared (IR) analysis option (Plumb 1981). The extract generated using the USACOE sediment preparation will be analyzed in accordance with the IR instrumental procedure of USEPA Method 418.1 (USEPA 1983) starting at procedural step 7.6.

Analyses of samples in the field using portable instruments will be performed according to the instrument manufacturer's instructions and/or recommendations, and the protocols appended to the Work Plan (Geraghty & Miller, Inc. 1992a).

9.0 DATA VALIDATION, REDUCTION, AND REPORTING

All data collected during this field investigation will be reduced, validated, reported, and evaluated by Geraghty & Miller personnel.

9.1 DATA VALIDATION

Data validation is a process in which analytical data generated by the laboratory are evaluated against a specified set of requirements and specifications. All VOC, BNA, metals, and cyanide data will be validated in accordance with the USEPA Region II data validation guidelines for organic and inorganic analyses performed in accordance with CLP RAS protocols. The guidelines for organics and inorganics are provided in the regional Standard Operating Procedures (SOP) No. HW-6, Revision 8, and No. HW-2, Revision 11, respectively (USEPA 1992a; 1992b).

Data validation of TPH data will be performed qualitatively according to criteria specified in Method 418.1 (USEPA 1983) and in accordance with the following guidelines:

1. Method and/or Reagent Blanks - apparent concentration to be less than the quantitation limit.

2. Precision of Duplicates - Acceptable precision will be demonstrated by calculation of RPD, which will vary by less than 20 percent. For calculated RPD between 20 to 40 percent, all associated sample data for the analytical batch will be estimated and qualified with a "J" flag.

3. Accuracy of Matrix Spikes - Acceptable accuracy will be demonstrated by percent recovery (%R) of matrix spikes in the acceptance window range of 80 to 120 percent. For %R values outside of the 80 to 120 window, but within 50 to 150 %R, associated sample data will be qualified as estimated

concentrations. Professional judgement will be used in applying qualifications to other samples in the sample delivery group (SDG) based on this criterion.

4. Accuracy of Laboratory Control Spikes - %R of blank spikes will be in the window of 80 to 120 percent. For %R values outside of the 80 to 120 window, but within 50 to 150 %R, associated sample data in the SDG will be qualified as estimated concentrations.

9.2 DATA REDUCTION

Data reduction will consist of summarizing the raw field data into a format necessary for interpretation, analysis, and evaluation. The data will be presented in the form of tables, well logs, illustrations, maps, graphs, as deemed appropriate by the Project Manager and/or Project Officer. The laboratory QA Program Plan more clearly defines the specific calculations/equations that are used during the generation of analytical data and any associated reporting limits.

9.3 DATA REPORTING

Laboratory deliverables for VOCs, BNAs, metals, and cyanide will consist of a complete hard copy data package in accordance with the required reporting format for the USEPA March 1990 CLP organic and inorganic RAS protocols (USEPA 1990a; 1990b). For the VOC analyses of 1,2-dibromomethane, n-propylbenzene, and MTBE, a standard Enseco East laboratory datasheet and supporting raw data will be provided. These will be collated into the CLP sample data package and sample summary package for each sample immediately following the VOC Form-I result summaries. For TPH analyses, reporting deliverables will consist of the following (or equivalent) forms from the USEPA March 1990 CLP inorganic RAS protocol (USEPA 1990b):

1. Sample result summary (Form I-IN) - to include sample identification, laboratory identification, sample matrix, date received, sample result corrected for percent solids, percent solids result, concentration units, and sample qualifiers.

2. Summary of initial and continuing calibration verification results [Form II (Part 1)-IN].

3. Summary of blank results, including preparation blank, initial and continuing calibration blanks (Form III-IN).

4. Summaries of matrix spike/matrix spike duplicate recoveries (Form V-IN) and RPD (Form VI-IN).

5. Summary of laboratory control and/or blank spike recoveries (Form VII-IN).

Raw data supporting documentation will be supplied for all TPH analyses and will include, but not be limited to, sample preparation logs, IR spectra of all samples, standards and associated QC, and instrument printouts.

In the Geraghty & Miller technical memoranda and data validation reports, the analytical data for field samples and blanks will be reported in tabular form with sample identifications, matrix, parameters, reporting limits, and concentrations where applicable. These tables will include any qualifiers placed on the data following the validation guidelines and/or by the laboratory. The tables will be consistent with the Geraghty & Miller data presentation format which includes date of sampling, method of analysis, analytical laboratory, type of sample, sample identification, sample result and/or quantitation limits. The Project QA officer is ultimately responsible for the data generated in the field investigation although a number of assigned personnel within Geraghty & Miller will be involved in the process.

10.0 QUALITY CONTROL PROCEDURES

QC procedures will be followed in the field as well as in the laboratory. The laboratory will be responsible for performing QC samples at the specified frequencies in accordance with the USEPA CLP RAS protocols (USEPA 1990a; 1990b), the analytical method and/or the laboratory QA Program Plan (Appendix A). The specific procedures for collecting replicate samples are detailed in the Work Plan (Geraghty & Miller, Inc. 1992a). The specific procedures for the preparation of laboratory QC samples are detailed in the laboratory QA Program Plan (Appendix A).

10.1 LABORATORY QC PROCEDURES

Internal QC checks for laboratory activities in support of VOC, BNA, and inorganic analyses will be carried out as specified by the USEPA CLP organic and inorganic SOWs (USEPA 1990a; 1990b). These include, but are not limited to, laboratory duplicates, method blanks, surrogate and system monitoring compound spikes, matrix spike/spike duplicates, internal standards, and reference standards. The frequency and implementation of these QC checks will occur as specified by the SOWs (USEPA 1990a; 1990b). For TPH analyses, information on the above can be found in the laboratory QA Program Plan (Appendix A).

10.2 FIELD QC PROCEDURES

Field QC procedures will include the collection of contractor-prepared field and trip blanks (prepared from laboratory supplied analyte-free water), field replicates, matrix spikes, matrix spike duplicates, and matrix duplicates. The frequency of these will be as follows:

Trip blanks	1 per cooler of VOC samples.
Field blanks	1 per 20 field samples collected, or one daily if less.
Field replicates	1 per 10 field samples collected for each matrix.

Matrix spikes	1 per 20 field samples collected for each matrix.
Matrix spike duplicates	1 per 20 field samples collected for each matrix (organics only).
Matrix duplicates	1 per 20 field samples collected for each matrix (inorganics only).

A summary of laboratory and field QC samples to be used in support of the field investigation is provided in Table 10-1.

11.0 PERFORMANCE AND SYSTEMS AUDITS

Geraghty & Miller will conduct systems audits to determine that the integrity of chain-of-custody procedures and the adherence to established and documented procedures of sample collection, management and analysis are followed. These audits will be performed at the discretion of the Project QA Officer, the Project Manager, and the Project Officer. These audits will also be performed prior to, or shortly after the systems are operational, and on a regular scheduled basis during the lifetime of the project. The results of these audits will be reported by the Project Manager. This report will serve to notify management of audit results and to identify areas requiring corrective action.

Laboratory systems audits will performed as specified in the CLP SOW (USEPA 1990a; 1990b). The laboratory contracted for this project will be under contractual responsibilities outlined by the Geraghty & Miller AQA/LCP. Prior to signing such a contract, Geraghty & Miller performs a comprehensive laboratory audit which covers all aspects of the operation. Internal laboratory audits are as detailed in the Enseco laboratory QA Program Plan (Appendix A).

12.0 PREVENTATIVE MAINTENANCE PROCEDURES

Preventative maintenance will be performed on field equipment in accordance with manufacturer's specifications. The maintenance of laboratory equipment will be performed according to the CLP SOW (USEPA 1990a; 1990b), the laboratory QA Program Plan, and/or to manufacturer's specifications. Additional laboratory equipment calibration, operation, and maintenance procedures are described in the laboratory QA Program Plan (Appendix A).

13.0 PROCEDURES TO ASSESS DATA PRECISION ACCURACY, AND COMPLETENESS

Laboratory data generated during the field investigations will be assessed for their precision, accuracy, and completeness as described in Section 4.0 of this document. The specific procedures, formulas, and calculations will be in accordance with the CLP SOW (USEPA 1990a; 1990b) and the laboratory QA Program Plan provided in Appendix A. These procedures, formulas, and calculations will be reviewed and assessed during the data validation of the project data.

14.0 CORRECTIVE ACTION

If unacceptable conditions are identified as a result of systems audits at the laboratory and during the field investigations, the Project QA Officer, Project Chemist, and the Project Manager will be responsible for initiating corrective action procedures. The specific conditions or problems will be clearly identified and isolated, cause will be determined, and appropriate corrective action plans implemented. Corrective actions may include, but are not be limited to, the following:

- Reanalyzing samples which fail to meet holding time criteria.
- Resampling and reanalysis.
- Amending sampling procedures and analytical procedures.
- Retraining staff.

After corrective actions are implemented, their effectiveness will be determined and the condition eliminated, or the problem readdressed.

15.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The Project QA Officer will review all aspects of the implementation of this QAPP on a regular basis. Reviews will be conducted at the completion of each field activity and will include an assessment of data quality and the results of system audits.

The laboratory will conduct internal reviews and include QA reports with the analytical data deliverable packages.

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16.0 REFERENCES

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Geraghty & Miller, Inc. 1991a. Tutu Service Station Investigation Work Plan (Draft), St. Thomas, U.S. Virgin Islands. Prepared for the Tutu Environmental Investigation Committee, January 1991.

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U.S. Environmental Protection Agency (USEPA). 1990b. USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, Multi-media, Multi-concentration, SOW No. 3/90, United States Environmental Protection Agency, March 1990.

U.S. Environmental Protection Agency (USEPA). 1992a. Evaluation of Organic Data for the Contract Laboratory Program (CLP), Region II Standard Operating Procedure No. HW-6, Revision No. 8, United States Environmental Protection Agency, January 1992.

U.S. Environmental Protection Agency (USEPA). 1992b. Evaluation of Metals Data for the Contract Laboratory Program (CLP), Region II Standard Operating Procedure No. HW-2, Revision No. 11, United States Environmental Protection Agency, January 1992.

Table 1–1. Summary of Proposed Sampling and Laboratory Analysis Effort, Tutu Service Station Investigation, St. Thomas, U.S. Virgin Islands.

Matrix	Field Task/Sample Source	Estimated Sample Frequency	Estimated Sample Quantity	Analytical Parameters
Ground Water	Development Water	1 sample before and 1 sample after treatment	2	VOCs
	Purge Water	1 sample before and 1 sample after treatment	2	VOCs
	Shallow Monitoring Wells	10 wells 2 sampling rounds	20	VOCs, BNAs, metals cyanide, TPH
	Deep Monitoring Wells	7 wells 2 sampling rounds	14	VOCs, BNAs, metals cyanide, TPH
	Pump Tests	2 wells	8	VOCs
Soil	Soil from Boring	13 borings 1 sample per boring	13	VOCs, BNAs, metals cyanide, TPH
	Soil from Monitoring Well Installation	17 wells 1 sample per well	17	VOCs, BNAs, metals cyanide, TPH

VOCs Volatile organic compounds.
BNAs Base neutral and acid extractable compounds.
TPH Total petroleum hydrocarbons.

Table 2-1. Target Compound List and Reporting Limits for Volatile Organic Compounds, to be Analyzed by USEPA CLP Protocols, in Ground-Water and Soil Samples for the Tutu Service Station Investigation, St. Thomas, U.S. Virgin Islands.

Parameter	CAS Number	Quantitation Limits *		
		Ground-Water (ug/L)	Soil (Low)** (ug/kg)	Soil (Medium)** (ug/kg)
Chloromethane	74-87-3	10	10	1200
Bromomethane	74-83-9	10	10	1200
Vinyl chloride	75-01-4	10	10	1200
Chloroethane	75-00-3	10	10	1200
Methylene chloride	75-09-2	10	10	1200
Acetone	67-64-1	10	10	1200
Carbon disulfide	75-15-0	10	10	1200
1,1-Dichloroethene	75-35-4	10	10	1200
1,1-Dichloroethane	75-34-3	10	10	1200
1,2-Dichloroethene (total)	540-59-0	10	10	1200
Chloroform	67-66-3	10	10	1200
1,2-Dichloroethane	107-06-2	10	10	1200
2-Butanone	78-93-3	10	10	1200
1,1,1-Trichloroethane	71-55-6	10	10	1200
Carbon tetrachloride	56-23-5	10	10	1200
Bromodichloromethane	75-27-4	10	10	1200
1,2-Dichloropropane	78-87-4	10	10	1200
cis-1,3-Dichloropropene	10061-01-5	10	10	1200
Trichloroethene	79-01-6	10	10	1200
Dibromochloromethane	124-48-1	10	10	1200
1,1,2-Trichloroethane	79-00-5	10	10	1200
Benzene	71-43-2	10	10	1200
trans-1,3-Dichloropropene	10061-02-6	10	10	1200
Bromoform	75-25-2	10	10	1200
4-Methyl-2-pentanone	108-10-1	10	10	1200
2-Hexanone	591-78-6	10	10	1200
Tetrachloroethene	127-18-4	10	10	1200
Toluene	108-88-3	10	10	1200
1,1,2,2-Tetrachloroethane	79-34-5	10	10	1200
Chlorobenzene	108-90-7	10	10	1200
Ethyl benzene	100-41-4	10	10	1200
Styrene	100-42-5	10	10	1200
Xylenes (Total)	1330-20-7	10	10	1200
1,2-Dibromomethane	74-95-3	10	10	1200
Methyl tert-butyl ether	1634-04-4	50	50	6000
n-Propylbenzene	103-65-1	10	10	1200

The above reporting limits are those specified in the USEPA March 1990 CLP SOW protocols.

USEPA U.S. Environmental Protection Agency.

CLP Contract Laboratory Program.

ug/kg Micrograms per kilogram.

CAS Chemical Abstracts Service.

* Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on a dry weight basis as required by the CLP contract, will be higher.

** Soil and sediment sample quantitation limits to be used in the final analytical report will be based on the determination of the sample concentration level made from the mandatory pre-screening of the sample matrix.

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Table 2-2. Target Compound List and Reporting Limits for Base Neutral and Acid Extractable Compounds, to be Analyzed by USEPA CLP Protocols, in Ground-Water and Soil Samples for the Tutu Service Station Investigation, St. Thomas, U. S. Virgin Islands.

Parameter	CAS Number	Quantitation Limits*		
		Ground-Water (ug/L)	Soil (Low)** (ug/kg)	Soil (Medium)** (ug/kg)
Phenol	108-95-2	10	330	10000
bis(2-Chloroethyl)ether	11-44-4	10	330	10000
2-Chlorophenol	95-57-8	10	330	10000
1,3-Dichlorobenzene	541-73-1	10	330	10000
1,4-Dichlorobenzene	106-46-7	10	330	10000
1,2-Dichlorobenzene	95-50-1	10	330	10000
2-Methylphenol	95-48-7	10	330	10000
2,2-Oxybis(1-chloropropane)	108-60-1	10	330	10000
4-Methylphenol	106-44-5	10	330	10000
N-Nitroso-di-n-dipropylamine	621-64-7	10	330	10000
Hexachloroethane	67-72-1	10	330	10000
Nitrobenzene	98-95-3	10	330	10000
Isophorone	78-59-1	10	330	10000
2-Nitrophenol	88-75-5	10	330	10000
2,4-Dimethylphenol	105-67-9	10	330	10000
bis(2-Chloroethoxy)methane	111-91-1	10	330	10000
2,4-Dichlorophenol	120-83-2	10	330	10000
1,2,4-Trichlorobenzene	120-82-1	10	330	10000
Naphthalene	91-20-3	10	330	10000
4-Chloroaniline	106-47-8	10	330	10000
Hexachlorobutadiene	87-68-3	10	330	10000
4-Chloro-3-methylphenol	59-50-7	10	330	10000
2-Methylnaphthalene	91-57-6	10	330	10000
Hexachlorocyclopentadiene	77-47-4	10	330	10000
2,4,6-Trichlorophenol	88-06-2	10	330	10000
2,4,5-Trichlorophenol	95-95-4	25	800	25000
2-Chloronaphthalene	91-58-7	10	330	10000
2-Nitroaniline	88-74-4	25	800	25000
Dimethylphthalate	131-11-3	10	330	10000
Acenaphthylene	208-96-8	10	330	10000
2,6-Dinitrotoluene	606-20-2	10	330	10000
3-Nitroaniline	99-09-2	25	800	25000
Acenaphthene	83-32-9	10	330	10000
2,4-Dinitrophenol	51-28-5	25	800	25000
4-Nitrophenol	100-02-7	25	800	25000
Dibenzofuran	132-64-9	10	330	10000
2,4-Dinitrotoluene	121-14-2	10	330	10000
Diethylphthalate	84-66-2	10	330	10000

The above reporting limits are those specified in the USEPA March 1990 CLP SOW protocols.

USEPA U.S. Environmental Protection Agency.
CLP Contract Laboratory Program.
ug/L Micrograms per liter.
ug/kg Micrograms per kilogram.
CAS Chemical Abstracts Service.
* Specific quantitation limits are highly matrix-dependent. The quantitation limits listed are provided for guidance and may not always be achievable. Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on a dry weight basis as required by the CLP contract, will be higher.
** Soil and sediment sample quantitation limits to be used in the final analytical report will be based on the determination of the sample concentration level made from the mandatory pre-screening of the sample matrix.

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Table 2–2. Target Compound List and Reporting Limits for Base Neutral and Acid Extractable Compounds, to be Analyzed by USEPA CLP Protocols, in Ground–Water and Soil Samples for the Tutu Service Station Investigation, St. Thomas, U. S. Virgin Islands.

Parameter	CAS Number	Quantitation Limits*		
		Ground–Water (ug/L)	Soil (Low)** (ug/kg)	Soil (Medium)** (ug/kg)
4–Chlorophenyl–phenyl ether	7005–72–3	10	330	10000
Fluorene	86–73–7	10	330	10000
4–Nitroaniline	100–01–6	25	800	25000
4,6–Dinitro–2–methylphenol	534–52–1	25	800	25000
N–Nitrosodiphenylamine	86–30–6	10	330	10000
4–Bromophenyl–phenylether	101–55–3	10	330	10000
Hexachlorobenzene	118–74–1	10	330	10000
Pentachlorophenol	87–86–5	25	800	25000
Phenanthrene	85–01–8	10	330	10000
Carbazole	86–74–8	10	330	10000
Anthracene	120–12–7	10	330	10000
Di–n–butylphthalate	84–74–2	10	330	10000
Fluoranthene	206–44–0	10	330	10000
Pyrene	129–00–0	10	330	10000
Butylbenzylphthalate	85–68–7	10	330	10000
3,3–Dichlorobenzidine	91–94–1	10	330	10000
Benzo(a)anthracene	56–55–3	10	330	10000
Chrysene	218–01–9	10	330	10000
bis(2–Ethylhexyl)phthalate	117–81–7	10	330	10000
Di–n–octylphthalate	117–84–0	10	330	10000
Benzo(b)fluoranthene	205–99–2	10	330	10000
Benzo(k)fluoranthene	207–08–9	10	330	10000
Benzo(a)pyrene	50–32–8	10	330	10000
Indeno(1,2,3–cd)pyrene	193–39–5	10	330	10000
Dibenz(a,h)anthracene	53–70–3	10	330	10000
Benzo(g,h,i)perylene	191–24–2	10	330	10000

The above reporting limits are those specified in the USEPA March 1990 CLP SOW protocols.

USEPA U.S. Environmental Protection Agency.

CLP Contract Laboratory Program.

ug/L Micrograms per liter.

ug/kg Micrograms per kilogram.

CAS Chemical Abstracts Service.

* Specific quantitation limits are highly matrix–dependent. The quantitation limits listed are provided for guidance and may not always be achievable. Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on a dry weight basis as required by the CLP contract, will be higher.

** Soil and sediment sample quantitation limits to be used in the final analytical report will be based on the determination of the sample concentration level made from the mandatory pre–screening of the sample matrix.

Table 2–3. Target Analyte List and Reporting Limits for Metal Analytes, Cyanide, and Total Petroleum Hydrocarbons to be Analyzed by USEPA CLP Protocols and Other Methodologies, in Ground–Water and Soil Samples for the Tutu Service Station Investigation, St. Thomas, U. S. Virgin Islands.

Parameter	CAS Number	Quantitation Limits*	
		Ground–Water ** (ug/L)	Soil (ug/kg)
Aluminum	7429–90–5	200	200
Antimony	7440–36–0	60	60
Arsenic	7440–38–2	10	10
Barium	7440–39–3	200	200
Beryllium	7440–41–7	5	5
Cadmium	7440–43–9	5	5
Calcium	7440–70–2	5000	5000
Chromium	7440–47–3	10	10
Cobalt	7440–48–4	50	50
Copper	7440–50–8	25	25
Iron	7440–89–6	100	100
Lead	7439–92–1	3	3
Magnesium	7439–95–4	5000	5000
Manganese	7439–96–5	15	15
Mercury	7439–97–6	0.2	0.2
Nickel	7440–02–0	40	40
Potassium	7440–09–7	5000	5000
Selenium	7782–49–2	5	5
Silver	7440–22–4	10	10
Sodium	7440–23–5	5000	5000
Thallium	7440–28–0	10	10
Vanadium	7440–62–2	50	50
Zinc	7440–66–6	20	20
Cyanide	–	10	10
TPH	–	500	20000

USEPA U.S. Environmental Protection Agency.
CLP Contract Laboratory Program.
CAS Chemical Abstracts Service.
ug/L Micrograms per liter.
ug/kg Micrograms per kilogram.
TPH Total petroleum hydrocarbons.
* The quantitation limits for metal analytes and cyanide are equal to the contract required detection limits (CRDLs) in the CLP inorganic statement of work (SOW). The CRDLs are the instrument detection limits obtained in pure water that must be met using the procedure in the CLP SOW for inorganics analysis, March 1990, Exhibit E. The detection limits for samples may be considerably higher depending on the sample matrix.
** The quantitation limits for metal analytes in ground–water samples are for both total and dissolved constituents.

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Table 3–1. Summary of Acceptance Limits for System Monitoring and Surrogate Compounds, to be Analyzed by USEPA CLP Protocols, in Ground–Water and Soil Samples for the Tutu Service Station Investigation, St. Thomas, U.S. Virgin Islands.

Parameter	Matrix	Acceptance Limits (%R)
VOC System Monitoring Compounds		
Toluene–d8	Water	88–110
	Soil	84–138
Bromofluorobenzene	Water	86–115
	Soil	59–113
1,2–Dichloroethane–d4	Water	76–114
	Soil	70–121
BNA Surrogate Compounds		
Nitrobenzene–d5	Water	35–114
	Soil	23–120
2–Fluorobiphenyl	Water	43–116
	Soil	30–115
Terphenyl–d14	Water	33–141
	Soil	18–137
Phenol–d5	Water	10–110
	Soil	24–113
2–Fluorophenol	Water	21–110
	Soil	25–121
2,4,6–Tribromophenol	Water	10–123
	Soil	19–122
2–Chlorophenol–d4	Water	33–110 *
	Soil	20–130 *
1,2–Dichlorobenzene–d4	Water	16–110 *
	Soil	20–130 *

USEPA	U.S. Environmental Protection Agency.
CLP	Contract Laboratory Program.
%R	Percent recovery.
VOC	Volatile organic compound.
BNA	Base neutral and acid extractable compound.
*	Advisory acceptance limits for specified surrogate compounds.

Table 3–2. Summary of Precision, Accuracy, and Completeness for Representative Volatile Organic Compounds, to be Analyzed by USEPA CLP Protocols, in Ground–Water and Soil Samples for the Tutu Service Station Investigation, St. Thomas, U.S. Virgin Islands.

Parameter	Matrix	Precision (RPD)	Accuracy (%R)	Completeness (%)
Benzene	Water	11	76–127	>95
	Soil	21	66–142	>95
Chlorobenzene	Water	13	75–130	>95
	Soil	21	60–133	>95
1,1–Dichloroethene	Water	14	61–145	>95
	Soil	22	59–172	>95
Toluene	Water	13	76–125	>95
	Soil	21	57–139	>95
Trichloroethene	Water	14	71–120	>95
	Soil	24	62–137	>95

USEPA U.S. Environmental Protection Agency.
CLP Contract Laboratory Program.
RPD Relative percent difference.
R Percent recovery.
% Percent.
> Greater than.

PR01301–#2/pac3–1.wk3

TUT 002 1001

Table 3–3. Summary of Precision, Accuracy, and Completeness for Representative Base Neutral and Acid Extractable Compounds, to be Analyzed by USEPA CLP Protocols, in Ground–Water and Soil Samples for the Tutu Service Station Investigation, St. Thomas, U.S. Virgin Islands.

Parameter	Matrix	Precision (RPD)	Accuracy (%R)	Completeness (%)
Acenaphthene	Water	31	46–118	>95
	Soil	19	31–137	>95
4–Chloro–3–methylphenol	Water	42	23–97	>95
	Soil	33	26–103	>95
2–Chlorophenol	Water	40	27–123	>95
	Soil	50	25–102	>95
1,4–Dichlorobenzene	Water	28	36–97	>95
	Soil	27	28–104	>95
2,4–Dinitrotoluene	Water	38	24–96	>95
	Soil	47	28–89	>95
4–Nitrophenol	Water	50	10–80	>95
	Soil	50	11–114	>95
N–Nitroso–di–n–propylamine	Water	38	41–116	>95
	Soil	38	41–126	>95
Pentachlorophenol	Water	50	9–103	>95
	Soil	47	17–109	>95
Phenol	Water	42	12–110	>95
	Soil	35	26–90	>95
Pyrene	Water	31	26–127	>95
	Soil	36	35–142	>95
1,2,4–Trichlorobenzene	Water	28	39–98	>95
	Soil	23	38–107	>95

USEPA	U.S. Environmental Protection Agency.
CLP	Contract Laboratory Program.
RPD	Relative percent difference.
%R	Percent recovery.
%	Percent.
>	Greater than.

Table 3–4. Summary of Precision, Accuracy, and Completeness for Metal Analytes, Cyanide, and Total Petroleum Hydrocarbons, to be Analyzed by USEPA CLP Protocols and Other Methodologies, in Ground–Water and Soil Samples for the Tutu Service Station Investigation, St. Thomas, U.S. Virgin Islands.

Parameter	Method	Matrix	Precision (RPD)	Accuracy (%R)	Completeness (%)
Aluminum	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Antimony	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Arsenic	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Barium	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Beryllium	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Cadmium	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Calcium	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Chromium	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Cobalt	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Copper	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Iron	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Lead	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Magnesium	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Manganese	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Mercury	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Nickel	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Potassium	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Selenium	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Silver	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Sodium	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Thallium	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Vanadium	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Zinc	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
Cyanide	March 1990 CLP RAS	Water/Soil	20 or +/– CRDL (a)	75–125	>95
TPH	USEPA 418.1	Water	20	80–120	>90
TPH	USACOE CE81–1/USEPA 418.1	Soil	20	80–120	>90

USEPA	U.S. Environmental Protection Agency.
CLP RAS	Contract Laboratory Program Routine Analytical Services protocols.
RPD	Relative percent difference.
%R	Percent recovery.
%	Percent.
CRDL	Contract required detection limit.
>	Greater than.
USACOE	U.S. Army Corps of Engineers.
(a)	A control limit of 20 percent RPD shall be used for original and duplicate sample values greater than or equal to five times (5X) the CRDL. A control limit of plus or minus (+/–) the CRDL must be used for sample values less than 5X the CRDL.

Table 5–1. Requirements for Sample Containers, Preservation, and Recommended Holding Times, Tutu Service Station Investigation, St. Thomas, U.S. Virgin Islands.

Analytical Parameter	Matrix	Container	Sample Volume	Preservation	Recommended Maximum Holding Time (a)
VOCs	Soil	Glass with Teflon–lined septum	2–40 mL vials	Cool to 4 degrees Celsius	10 days
BNAs	Soil	Glass with Teflon–lined cap	1–250 mL bottle	Cool to 4 degrees Celsius	7 days to extraction 40 days to analysis
Metals	Soil	Glass with Teflon–lined cap	1–250 mL bottle	Cool to 4 degrees Celsius	6 months for all except Hg; 28 days for Hg
Cyanide	Soil	Glass with Teflon–lined cap	1–250 mL bottle	Cool to 4 degrees Celsius	14 days
TPH	Soil	Glass with Teflon–lined cap	1–250 mL bottle	Cool to 4 degrees Celsius	28 days
VOCs	Water	Glass with Teflon–lined septum	2–40 mL vials	Add HCl to pH<2 Cool to 4 degrees Celsius	7 days unpreserved (aromatics only) 14 days preserved (b)
BNAs	Water	Amber glass with Teflon–lined cap	2–1 L bottles	Cool to 4 degrees Celsius	7 days to extraction 40 days to analysis
Metals	Water	Polyethylene with Teflon–lined cap	1–1 L bottle	Add HNO3 to pH<2 Cool to 4 degrees Celsius	6 months for all except Hg; 28 days for Hg
Cyanide	Water	Polyethylene with Teflon–lined cap	1–1 L bottle	Add NaOH to pH>12 Cool to 4 degrees Celsius	14 days
TPH	Water	Amber glass with Teflon–lined cap	1–1 L bottle	Add HCl to pH<2 Cool to 4 degrees Celsius	28 days

VOCs	Volatile organic compounds.	HCl	Hydrochloric acid.
BNAs	Base neutral and acid extractable compounds.	HNO3	Nitric acid.
TP	Total petroleum hydrocarbons.	NaOH	Sodium hydroxide.
Hg	Mercury.	<	Less than.
mL	Milliliter.	>	Greater than.
L	Liter.		

a Holding times measured from time of sample collection.

b If sample acidification causes effervescence, the sample will not be acidified but will be cooled to approximately 4 degrees Celsius. In this event, the holding time is 7 days from the time of sampling for aromatic compound analysis and 14 days for halogenated VOCs.

Table 8-1. Analytical Methods, Tutu Service Station Investigation, St. Thomas, U.S. Virgin Islands.

Matrix	Parameter	Analythical Method
Soil	VOCs (a)	March 1990 CLP RAS SOW
Soil	TCL BNAs	March 1990 CLP RAS SOW
Soil	TPH	USACOE CE81-1/USEPA 418.1
Soil	TAL Metals and Cyanide	March 1990 CLP RAS SOW
Water	VOCs (a)	March 1990 CLP RAS SOW
Water	TCL BNAs	March 1990 CLP RAS SOW
Water	TPH	USEPA 418.1
Water (b)	TAL Metals and Cyanide	March 1990 CLP RAS SOW

VOCs	Volatile oganic compounds.
CLP RAS SOW	Contract laboratory program routine analytical services statement of work (Organic SOW Document No. OLM01.0 and revisions; Inorganic SOW Document No. ILM01.0)
TCL	Target compound list.
BNAs	Base neutral and acid extractable compounds.
TPH	Total petroleum hydrocarbons.
USACOE	Army Corps of Engineers.
USEPA	U.S. Environmental Protection Agency.
TAL	Target analyte list.

(a) VOCs include analyses for TCL VOCs, 1,2-dibromomethane, n-propylbenzene, and methyl tert-butyl ether.

(b) For TAL in water, two samples will be collected (filtered and unfiltered).

#PR01301-wp2/methods.wp

Table 10–1. Field and Laboratory Quality Control Samples to be Analyzed for the Tutu Service Station Investigation, St. Thomas, U.S. Virgin Islands.

Matrix	Field Task/ Sample Source	Parameter	Estimated Sample Quantity	Estimated Field Blank Quantity	Estimated Trip Blank Quantity	Estimated Field Replicate Quantity
Ground Water	Development Water	VOCs	2	NA	1	NA
	Purge Water	VOCs	2	NA	1	NA
	Shallow Monitoring Wells	VOCs	20	2	2	2
		BNAs	20	2	NA	2
		Metals (total)	20	2	NA	2
		Metals (dissolved)	20	2	NA	2
		Cyanide	20	2	NA	2
		TPH	20	2	NA	2
	Deep Monitoring Wells	VOCs	14	2	2	2
		BNAs	14	2	NA	2
		Metals (total)	14	2	NA	2
		Metals (dissolved)	14	2	NA	2
		Cyanide	14	2	NA	2
		TPH	14	2	NA	2
	Pump Tests	VOCs	8	NA	2	1
Soil	Soil from Boring	VOCs	13	1	NA	2
		BNAs	13	1	NA	2
		Metals	13	1	NA	2
		Cyanide	13	1	NA	2
		TPH	13	1	NA	2
	Soil from Monitoring Well Installation	VOCs	17	1	NA	2
		BNAs	17	1	NA	2
		Metals	17	1	NA	2
		Cyanide	17	1	NA	2
		TPH	17	1	NA	2

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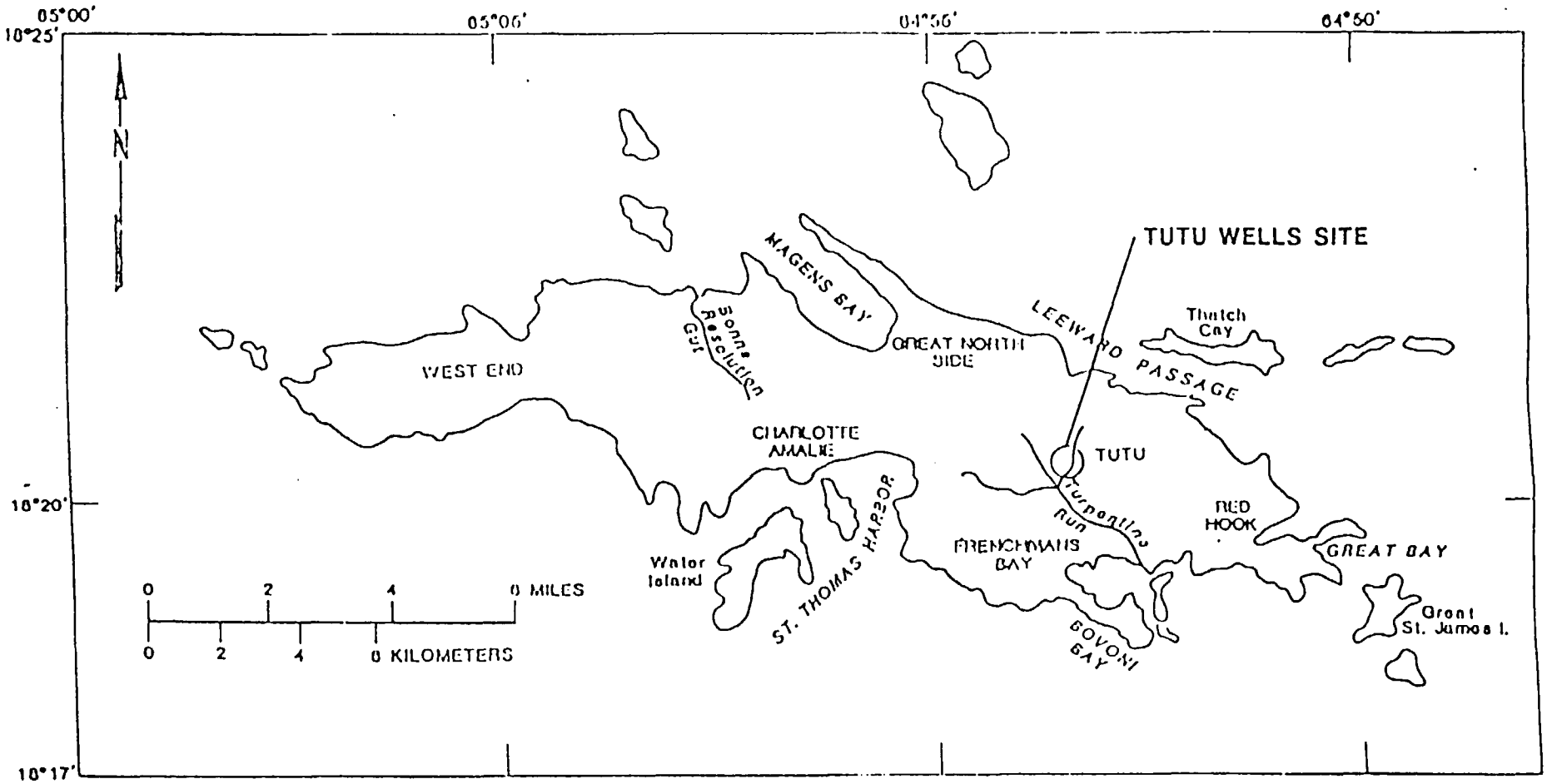
VOCs Volatile organic compounds.
NA Not applicable.
BNAs Base neutral and acid extractable compounds.
TPH Total petroleum hydrocarbons.



SUBJECT

TUTU WELLS SITE
TUTU SERVICE STATION INVESTIGATION, ST. THOMAS, U. S. VIRGIN ISLANDS

PREPARED FOR

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	PROJECT MGR: DANAHY		

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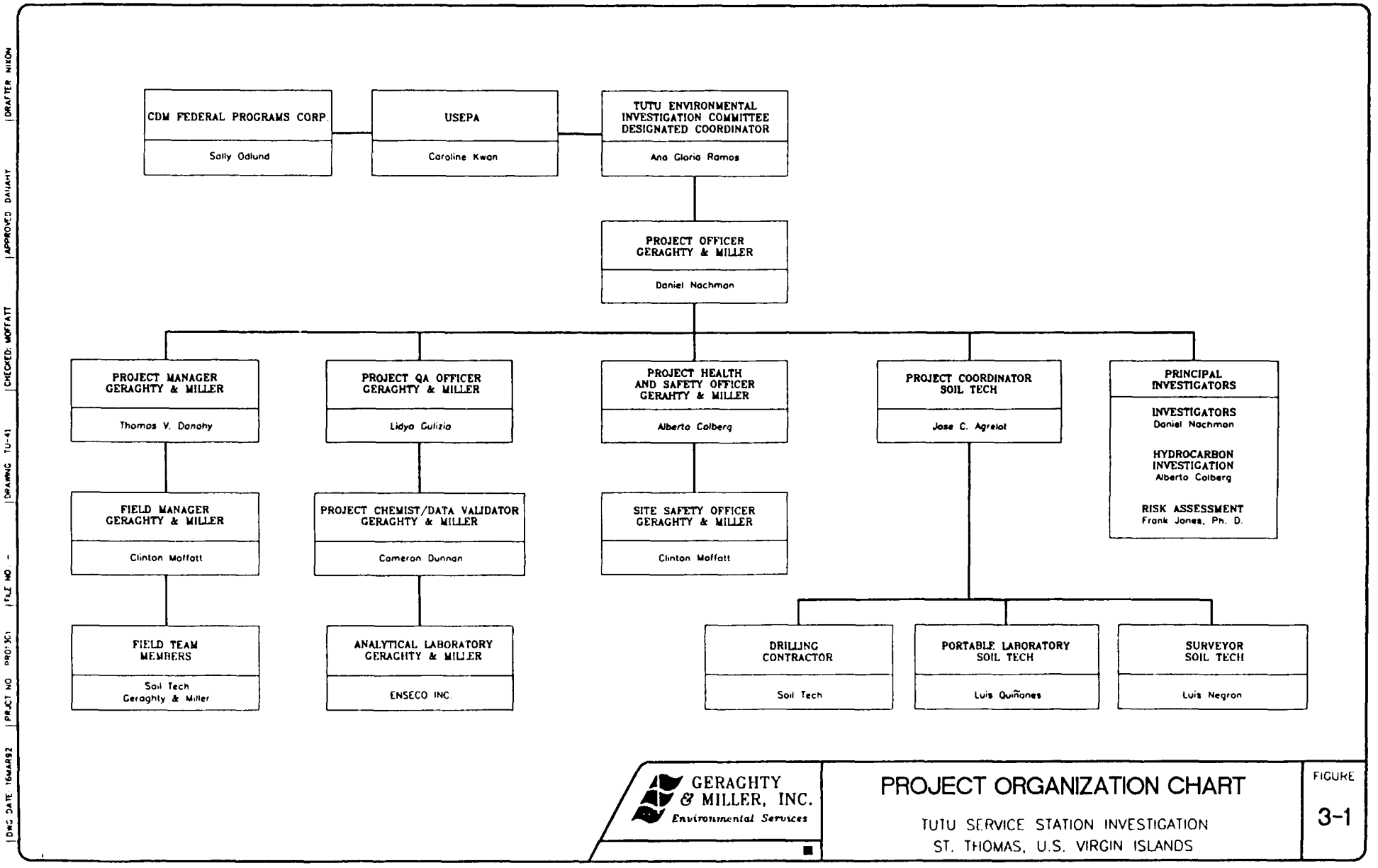

GERAGHTY & MILLER, INC.
Environmental Services

PROJECT ORGANIZATION CHART

TUTU SERVICE STATION INVESTIGATION
ST. THOMAS, U.S. VIRGIN ISLANDS

FIGURE
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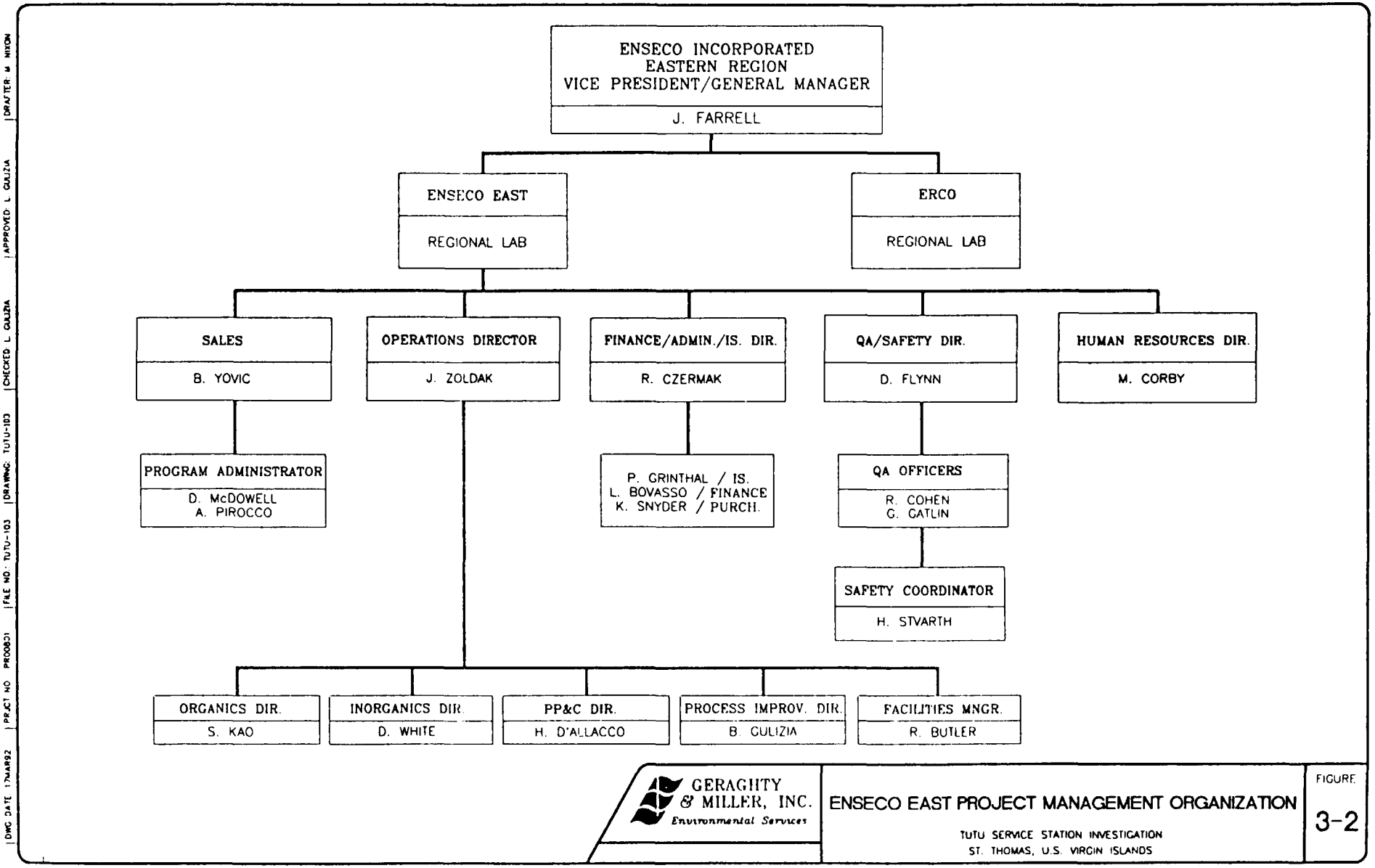
ENSECO INCORPORATED
EASTERN REGION
VICE PRESIDENT/GENERAL MANAGER
J. FARRELL
ENSECO EAST
REGIONAL LAB
ERCO
REGIONAL LAB
SALES
B. YOVIC
OPERATIONS DIRECTOR
J. ZOLDAK
FINANCE/ADMIN./IS. DIR.
R. CZERMAK
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R. BUTLER
GERAGHTY & MILLER, INC.
Environmental Services
ENSECO EAST PROJECT MANAGEMENT ORGANIZATION
TUTU SERVICE STATION INVESTIGATION
ST. THOMAS, U.S. VIRGIN ISLANDS
FIGURE
3-2
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APPENDIX A

ENSECO INCORPORATED QUALITY ASSURANCE PROGRAM PLAN FOR ENVIRONMENTAL CHEMICAL MONITORING

ENSECO INCORPORATED
QUALITY ASSURANCE
PROGRAM PLAN
FOR
ENVIRONMENTAL CHEMICAL MONITORING

Prepared by:
Enseco Incorporated
2200 Cottontail Lane
Somerset, NJ 08875

Revision 3.4
April 1991

Approval:

Margaret S. Sleevi
Director of Quality Assurance

Gary Ward
Director of Quality Assurance
& Technology

Robert S. Hulsy
President

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Appendix I	Enseco Recommended Maximum Holding Times and Sample Collection/Preservation Information
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1. INTRODUCTION

Enseco Incorporated (Enseco) is the largest and most experienced environmental testing laboratory in the United States. The environmental component of Enseco consists of the combined resources of:

- Enseco-Erco Laboratory in Cambridge, Massachusetts,
- Enseco-East in Somerset, New Jersey,
- Enseco-Rocky Mountain Analytical Laboratory in Denver, Colorado,
- Enseco-Houston in Houston, Texas,
- Enseco-California Analytical Laboratory in Sacramento, California,
- Enseco-CRL in Garden Grove, California,
- Enseco-El Monte in El Monte, California,
- Enseco-Santa Maria in Santa Maria, California,
- Enseco-Ventura in Ventura, California, and
- Enseco-Mobile Laboratories headquartered in Garden Grove, California.

Addresses and telephone numbers for these Enseco laboratories are listed in Table 1-1.

This document describes the Enseco Quality Assurance policies and procedures related to chemical monitoring for environmental pollutants.

TABLE 1-1

ENSECO LABORATORY LOCATIONS

Enseco-California Analytical Laboratory
2544 Industrial Boulevard
West Sacramento, CA 95691
(916) 372-1393
Facsimile (916) 372-1059

Enseco-CRL
7440 Lincoln Way
Garden Grove, CA 92641
(714) 898-6370
Facsimile (714) 891-5917

Enseco-East
2200 Cottontail Lane
Somerset, NJ 08875
(201) 469-5800
Facsimile (201) 469-7516

Enseco-El Monte
9537 Telstar Avenue #118
El Monte, CA 91731
(818) 442-8400
Facsimile (818) 442-3758

Enseco-Erco Laboratory
205 Alewife Brook Parkway
Cambridge, MA 02138
(617) 661-3111
Facsimile (617) 354-5258

Enseco-Houston
1420 East North Belt Suite 120
Houston, TX 77032
(713) 987-9767
Facsimile (713) 987-9769

Enseco-Mobile Laboratories
7440 Lincoln Way
Garden Grove, CA 92641
(714) 898-6370
Facsimile (714) 891-5917

Enseco-Rocky Mountain Analytical Laboratory
4955 Yarrow Street
Arvada, CO 80002
(303) 421-6611
Facsimile (303) 431-7171

Enseco-Santa Maria
2325 Skyway Drive, Suite K
Santa Maria, CA 93455
(805) 922-2776
Facsimile (805) 922-5897

Enseco-Ventura
2810 Bunsen Avenue, Unit A
Ventura, CA 93003
(805) 650-0546
Facsimile (805) 650-0756

Enseco, Inc. (Corporate Office)
2200 Cottontail Lane
Somerset, NJ 08875
(201) 469-5800
Facsimile (201) 469-6257

2. QUALITY ASSURANCE POLICY

Enseco is committed to providing quality environmental analytical services to both the public and private sectors. To ensure the production of scientifically sound, legally defensible data of known, documentable and verifiable quality, an extensive Quality Assurance (QA) program has been implemented within Enseco. This program relies on clearly defined objectives, well-documented procedures, a comprehensive audit system, and management support, both Corporate and Divisional, for its effectiveness.

3. PURPOSE AND SCOPE OF DOCUMENT

Purpose

This QA Program Plan presents an overview of the essential elements of the Enseco QA program. Enseco has modeled this plan along EPA guidelines as outlined in "Interim Guidelines and Specifications for Preparing Quality Assurance Program Plans," QAMS-004/80, December 29, 1980 and "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans," QAMS-005/80, February, 1983. Both of these documents have been issued by the Office of Monitoring Systems and Quality Assurance, Office of Research and Development, U.S. Environmental Protection Agency (U.S. EPA). Elements above and beyond those specified in these two documents have been included in this QA Program Plan in order to completely describe the Enseco QA/QC system.

Scope

The Enseco QA program is designed to control and monitor the quality of data generated in Enseco laboratories. The program has four key elements.

- Demonstrating laboratory capability by providing information which documents the overall qualifications of the laboratory to perform environmental analyses;
- Controlling laboratory operations by establishing procedures which measure laboratory and instrument performance on a daily basis;
- Measuring matrix effects to determine the effect of a specific matrix on method performance, and
- Reporting appropriate QC information with the analytical results to enable the end-user to assess the quality of the data.

The specific procedures involved in implementing each aspect of the Enseco program are described in this document. An overview of these QC procedures, along with the section number in which each is discussed, is given in Table 3-1.

The QA/QC policies and procedures described herein are designed to eliminate systematic errors and minimize the occurrence of other errors. However, no QA program, regardless of how elaborate, can eliminate all errors which may occur during an analysis. The QA program forms the framework for minimizing errors and identifying and correcting those errors which do occasionally occur. These QA/QC policies and procedures must be coupled with the professional judgment of the technical staff in interpreting the events surrounding the generation of the final result to ensure that quality data is consistently produced, and decisions and corrective actions are fully documented.

In many instances, Enseco participates with its clients in the preparation and evaluation of project-specific Quality Assurance Project Plans (QAPjP). Typically the elements of the Enseco QAPP are incorporated into these documents. In some instances other requirements may be specified. Each QAPjP must be reviewed and approved by the QA Director of the Enseco facility entering into the client agreement to assure that minimum standards of quality exist by which the work can be evaluated as to its scientific and legal integrity. The QA Director must assure that both the analytical testing objectives and regulatory requirements of the project are met. All requirements in a QAPjP which do not meet the minimum requirements as stated in the Enseco QAPP must be approved by the Corporate Director of QA. In the presence of an approved QAPjP, Enseco laboratories must follow the specific requirements of that project plan which supersedes the Enseco QAPP for any work explicitly associated with that QAPjP.

Table 3-1

ELEMENTS OF QA PROGRAM PLAN

Evaluation Criteria	Operational Elements	Section of QA Plan
LABORATORY QUALIFICATIONS	Facilities/equipment/staff.....	*
	Written SOPs for all laboratory procedures, including:.....	17
	Sample custody.....	7
	Calibration procedures.....	8
	Analytical procedures.....	9
	Data validation.....	10
	Documented QA program.....	1-15
	Laboratory certifications.....	12
LABORATORY PERFORMANCE	Check samples.....	12
	Method blanks.....	11
	Calibration data/calibration verification..	8
	Method detection limits.....	14
MATRIX EFFECTS	Matrix spike/matrix duplicate/ matrix spike duplicate analyses.....	11
	Sample surrogate recoveries.....	11
	Standard additions.....	11
	Field blanks.....	11
	Method detection limits (determined with specific sample matrix).....	11
DATA REPORTING	Data reduction and validation.....	10
	Data reporting.....	10
	Reporting Limits.....	14

* Described in a separate document available from Enseco.

4. DEFINITION OF TERMS

Quality Assurance (QA): the total integrated program for assuring the reliability of data generated in the laboratory.

Quality Control (QC): the routine application of specific, well-documented procedures to ensure the generation of data of known and accepted quality, thus fulfilling the objectives of the QA program.

Quality Assurance Program Plan (QAPP): an assemblage of management policies, objectives, principles, and general procedures outlining the techniques by which the laboratory produces data of known and accepted quality.

Standard Operating Procedure (SOP): a detailed, written description of a procedure designed to systematize and standardize the performance of the procedure.

Quality Assurance Project Plan (QAPjP): an assemblage of detailed procedures describing how the laboratory will generate data that meet the Data Quality Objective (DQOs) of a specific project.

Legally Defensible Data: data which are supported by a QAPP and documentation adequate to reconstruct the analytical process. Legal defensibility is not dependent on the level of deliverables.

Holding Time: the period of time during which a sample can be stored after collection and preservation without significantly affecting the accuracy of the analysis.

Sample Delivery Acceptance: the point in time at which Enseco determines that it can proceed with the analytical work. Sample delivery acceptance follows receipt and inspection of the samples and complete definition of analyses required.

Initiate Preparation: the point in time at which the separation of organic extractable compounds or metals from the sample matrix by solvent extraction or acid digestion is begun.

Initiate Analysis: the point in time at which the sample, extract or digestate is introduced into an instrument or process which complies with the SOP for analysis of the parameter of interest.

5. RESPONSIBILITIES AND AUTHORITIES

Executing an effective QA program in a large and complex multi-laboratory system demands the commitment and attention of both management and staff. The QA effort at Enseco is administered by the Director of Quality Assurance and Technology who manages the Corporate Quality Assurance Office. The Director of QA and Technology reports directly to the President and has the responsibility for overseeing and regulating all laboratory functions (see Figure 5-1). The Corporate QA Director reports to the Director of QA and Technology and has the responsibility of the day-to-day functions of the QA office. The QA Office operates independently of all areas generating analytical data to ensure complete objectivity in the evaluation of laboratory operations.

The implementation of the QA program within each individual Enseco laboratory is administered by the Division QA Director. The QA Director reports to both the Corporate QA Director and to the General Manager, who manages the laboratory. In addition, all scientists within the organization play a vital role in assuring the quality of their work. We believe that the success of Enseco is dependent upon the continued commitment of all within the organization to a strong and viable QA Program. The responsibilities and levels of authority within the organization are described below.

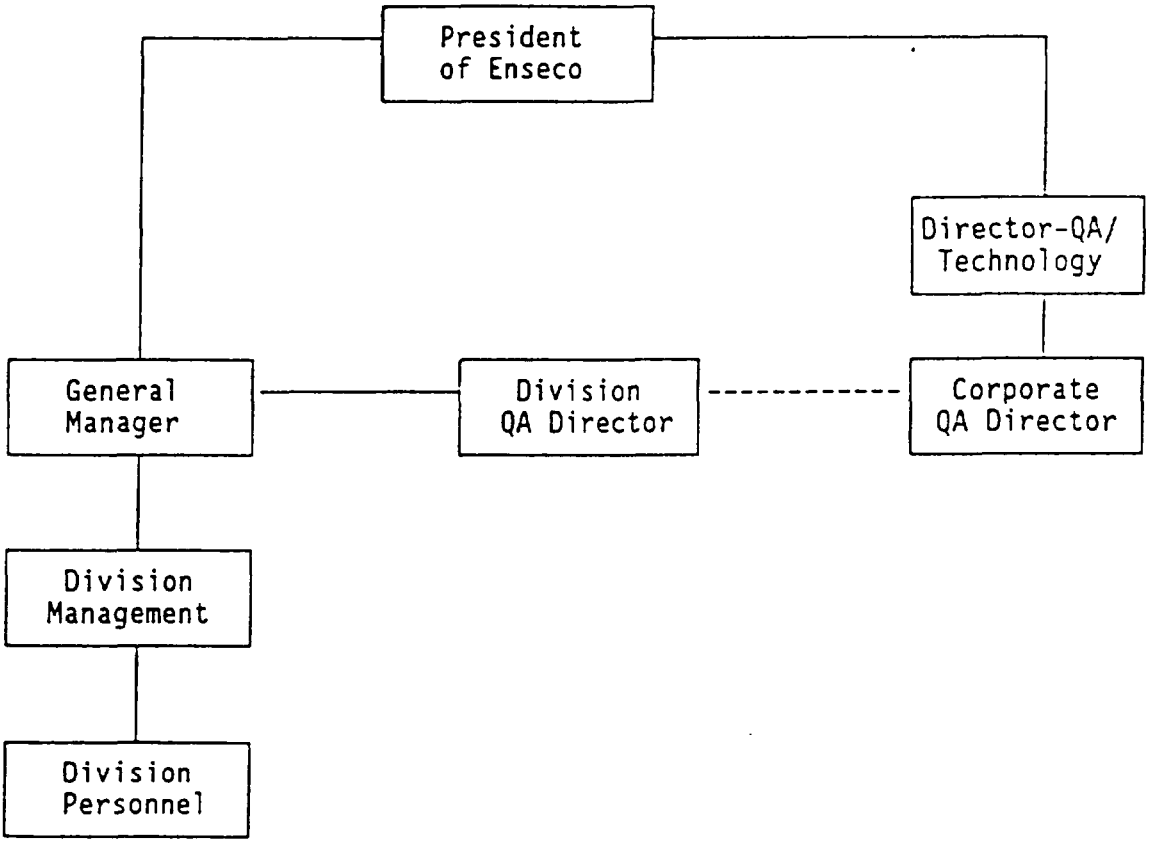
Corporate Quality Assurance Office

Members

The QA effort within Enseco is directed by the Corporate QA Director under the management of the Director of Quality Assurance and Technology to carry out the responsibilities of the department.

Figure 5-1

ENSECO QA ORGANIZATIONAL CHART



Responsibilities

The Corporate QA Director under the direction of the Director of QA and Technology is responsible for:

- Developing and implementing a Corporate QA program that ensures that all data generated in Enseco laboratories are scientifically sound, legally defensible, and of known precision and accuracy;
- Monitoring the QA Plan to ensure compliance with QA objectives in all Enseco laboratories;
- Developing and implementing new QA procedures within the corporation to improve data quality;
- Conducting audits and inspections of all Enseco laboratories on a regular basis, reporting the results of those audits to management, and applying corrective actions as needed to ensure compliance with the Enseco QA Plan;
- Coordinating the distribution of Performance Evaluation (PE) samples to all Enseco laboratories on a routine basis, evaluating the results of those samples, reporting to management, and applying corrective actions as needed to ensure that all Enseco laboratories are able to generate data that meet the data quality objectives defined in the QA Plan;
- Establishing databases that accurately reflect the performance of each of the Enseco laboratories;
- Directing Division QA Directors in the implementation of the Enseco QA Plan within individual facilities;
- Chairing the Enseco QA Committee, a working committee which includes all of the Division QA Directors and deals with QA issues on an ongoing basis;
- Coordinating certification programs within Enseco;
- Conducting seminars on QA issues for both clients and laboratory staff; and
- Promoting sound QA practices within the environmental regulatory and analytical communities.

Authority

Both the Director of QA and Technology and the Corporate QA Director have the authority on issues dealing with data quality and have the authority to require that procedures be amended or discontinued, or analyses suspended or repeated. The Director of QA and Technology and the Corporate QA Director have the authority to suspend or terminate employees on the grounds of dishonesty, incompetence, or repeated non-compliance with QA procedures. In addition, these Corporate Directors have the authority to overrule decisions and actions of the Division QA Directors and must approve the termination or transfer of any Division QA Director. The authority of the Corporate QA Director and the Director of QA and Technology comes directly from the President of Enseco.

Divisional Quality Assurance Departments

Members

Each Divisional QA Department is managed by a QA Director. The QA Director reports directly to the General Manager and indirectly to the Corporate QA Director. The QA Director is supported by a QA staff within the laboratory.

Responsibilities

The Division QA Director is responsible for:

- Implementing Enseco QA policies;
- Monitoring the implementation of the QA Plan within the laboratory to ensure complete compliance with QA objectives;
- Conducting in-house audits to identify potential problems and ensure compliance with written SOPs;

- Performing statistical analyses of QC data and establishing databases that accurately reflect the performance of the laboratory;
- Prescribing and monitoring corrective actions;
- Serving as the in-house client representative on all project inquiries involving data quality issues;
- Monitoring the preparation and verification of analytical standards;
- Assisting chemists in the writing of SOPs;
- Reporting the status of the laboratory QA program to the Corporate QA Director with formal and informal communications;
- Maintaining records and archives of all QC data, PE results, audit comments, and customer inquiries concerning data quality;
- Assuring that the laboratory staff has access to current SOPs;
- Monitoring laboratory performance in the areas of holding times, turn-around times, and meeting contractual obligations;
- Conducting seminars on QA issues for clients and laboratory staff;
- Preparing QA Project Plans when needed;
- Assisting the Corporate QA office in the writing of QA policies and procedures;
- Serving as a member of the Enseco QA Committee; and
- Auditing subcontractors.

Authority

The Division QA Director is the final authority within each laboratory on all issues dealing with data quality. He/she has the authority to require that procedures be amended or discontinued or analyses suspended or repeated. He/she can make recommendations to the General Manager and the Corporate Director of QA regarding suspension or termination of employees for incompetence or non-compliance with QA procedures. The authority of the Division QA Director comes directly from the Corporate Director of QA.

Divisional Management

Members

The managers and supervisors who direct the analytical work at each laboratory are directly responsible for ensuring that all employees reporting to them are complying with the Enseco QA Plan.

Responsibilities

Laboratory management is responsible for:

- Actively supporting the implementation of the Enseco QA Plan within the laboratory;
- Maintaining accurate SOPs and enforcing their use in the laboratory;
- Maintaining a work environment that emphasizes the importance of data quality; and
- Providing management support to the Corporate and Divisional QA departments.

Authority

The managers and supervisors of the laboratory have the authority to accept or reject data based on compliance with well-defined QC criteria. In addition, managers and supervisors, with the approval of the QA department, can accept or reject data that fall outside of established QC guidelines if, in their judgment, there are technical reasons which warrant the acceptance or rejection of the data. These circumstances must be well documented and any need for corrective action identified by the incident must be defined and initiated. The authority of the laboratory management comes directly from the President of Enseco and the General Manager.

Divisional Personnel

Members

All laboratory personnel involved in the generation and reporting of data have a responsibility to understand and follow the Enseco QA Plan.

Responsibilities

Laboratory personnel are responsible for:

- Having a working knowledge of the Enseco QA Plan;
- Ensuring that all work is generated in compliance with the Enseco QA Plan;
- Performing all work according to written SOPs;
- Ensuring that all documentation related to their work is complete and accurate; and
- Providing management with immediate notification of quality problems.

Authority

Laboratory personnel have the authority to accept or reject data based on compliance with well-defined QC criteria. The acceptance or rejection of data that fall outside of established QC guidelines must be approved by laboratory management and the QA department. The authority of the laboratory personnel flows from the General Manager.

6. SAMPLING PROCEDURES

The generation of quality data begins with the collection of the sample, and therefore the integrity of the sample collection process is of concern to the laboratory. Samples must be collected in such a way that no foreign material is introduced into the sample and no material of interest escapes from the sample prior to analysis. To ensure sample integrity, the following must be considered:

- Samples must be collected in appropriate containers. In general, glass containers are used for organic parameters and polyethylene containers for inorganic/metal parameters (see Appendix I);
- The sample containers must be properly cleaned to ensure that the sample is not contaminated during the collection process;
- Samples must be preserved appropriately to minimize the loss of materials of interest due to adsorption, chemical or biological degradation, or volatilization (see Appendix I);
- Appropriate volumes of sample must be collected to ensure that the required detection limits can be met and quality control samples can be analyzed (see Appendix I); and
- Samples must be properly shipped to the laboratory, in the appropriate time frame, to ensure that holding times for the analyses can be met (see Appendix I).

Sample Containers and Preservatives

Enseco can assist in the sample collection process by providing consultation and assistance to clients designing sampling programs. Also Enseco can make available to the client sample containers that are properly cleaned and preserved for use in sample collection. Enseco has had its coolers, sample containers and packaging methods independently tested to demonstrate that Department of Transportation standards are met. Appropriate containers and preservatives, and minimum sample volumes required for analyzing routine organic, metal, and conventional parameters are listed in Appendix I.

Holding Times

EPA has established holding time requirements for some analyses. These holding time requirements are listed in Appendix I, along with container and preservative requirements. As indicated in Appendix I, holding time requirements differ depending on the regulatory program. Enseco follows the holding times given in SW-846, Update I, Federal Register, October 26, 1984 or Methods of Chemical Analysis of Water & Waste, based on the method source, unless otherwise instructed by the client. CLP holding times are followed when CLP protocols are requested by the client. Other holding times can be honored if special arrangements are made with the laboratory.

Enseco is obligated to initiate preparation and/or analysis of the sample within holding times if sample delivery acceptance occurs within 72 hours of sampling or before one-half of the holding time period has expired, whichever is less. (See Section 4 for definition of above terms.)

On occasion, a sample must be reanalyzed to comply with this QA Program Plan. If this reanalysis is conducted outside of the holding time, the laboratory will be considered to have fulfilled its obligation to meet holding times if the first preparation and/or analysis was initiated within the prescribed holding time.

Sample Disposition

All soil samples, sample extracts, and aqueous samples that meet Federal or applicable State definitions as a hazardous waste are incinerated at a RCRA Part B permitted facility or returned to the client. Empty sample containers are disposed of by shredding and incineration. An alternate procedure for disposal of empty containers involves triple rinsing the container, blanking out the label and disposal as a solid waste. Sample disposition procedures meet Federal and State regulations.

7. SAMPLE CUSTODY

Upon receipt by Enseco, samples proceed through an orderly processing sequence specifically designed to ensure continuous integrity of both the sample and its documentation.

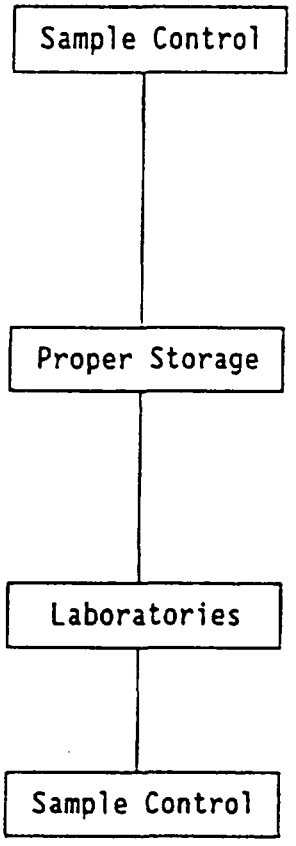
All samples are received by Enseco's Sample Control Group and are carefully checked for label identification, and completed, accurate chain-of-custody records. Photographs document the condition of samples and each sample is then assigned a unique laboratory identification number through a computerized Laboratory Information Management System (LIMS) that stores all identifications and essential information. The LIMS system tracks the sample from storage through the laboratory system until the analytical process is completed and the sample is returned to the custody of the Sample Control Group for disposal. This process is summarized in Figure 7-1. Access to all Enseco laboratories is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

An example of the Enseco Chain-Of-Custody Record used to transmit samples from the client to the laboratory is given in Figure 7-2. The Chain-Of-Custody Record (Interlaboratory Analysis Form) used to transmit samples between laboratories within Enseco is given in Figure 7-3.

Sample bottles provided to the client by Enseco are transmitted under custody.

Figure 7-1

ENSECO SAMPLE PROCESSING FLOW CHART



* Check and document physical condition of sample
* Verify documentation and parameter assignment
* Log into LIMS
* Send acknowledgement letter to client

* Store sample according to preservation guidelines
* Transfer sample to lab with proper documentation

* Document analytical work
* Return unused samples to Sample Control

* Return sample to client or arrange for sample disposal

Enseco
A Corning Company

Rocky Mountain Analytical Laboratory
4955 Yarrow Street
Arvada, CO 80002
303/421-6611 FAX: 303/431-7171

N OF CUSTODY

IENT

COMPANY

SITE

DER

SAMPLE SAFE™ CONDITIONS

PACKED BY

SEAL NUMBER

SEAL INTACT UPON RECEIPT BY SAMPLING COMPANY

CONDITION OF CONTENTS

SEALED FOR SHIPPING BY

INITIAL CONTENTS TEMP °C

SEAL NUMBER

SAMPLING STATUS ☐ Done ☐ Continuing Until

SEAL INTACT UPON RECEIPT BY LAB ☐ Yes ☐ No

CONTENTS TEMPERATURE UPON RECEIPT BY LAB °C

TE	TIME	SAMPLE ID/DESCRIPTION	SAMPLE TYPE	# CONTAINERS	ANALYSIS PARAMETERS	REMARKS

Figure 7.2

CUSTODY TRANSFERS PRIOR TO SHIPPING

UISHED BY (SIGNED)	RECEIVED BY (SIGNED)	DATE	TIME

SHIPPING DETAILS

DELIVERED TO SHIPPER BY

METHOD OF SHIPMENT

AIRBILL NUMBER

RECEIVED FOR LAB

SIGNED

DATE/TIME

ENSECO PROJECT NUMBER

ERLABOR TORY
AIN OF CUSTODY

Enseco
A Corning Company

PAGE OF

O

ITION

ANALYTICAL REQUESTS

SEND RESULTS TO.

ATTENTION.

EXPORT ID

SAMPLE CONDITION UPON RECEIPT

COMMENTS

TEST PRICE

SUBTOTAL

DISCOUNT / SURCHARGE

TOTAL

WRITTEN RESULTS REQUIRED BY (DATE)

VERBAL/FAC RESULTS REQUIRED BY (DATE)

P O No

Q.C. ☐ STANDARD ENSECO ☐ CLP PROTOCOL ☐ PROJECT SPECIFIC

SAMPLE DISPOSAL ☐ ENSECO ☐ RETURN TO CLIENT ☐ PHONE

DETECTION LIMITS ☐ COMMON PRODUCTS ☐ OTHER*

HOLDING TIMES ☐ ENSECO ☐ EPA-CLP ☐ TIER ☐ OTHER*

RAW DATA COPIES NEEDED ☐ YES ☐ NO

CUSTODY SEALS INTACT ☐ YES ☐ NO ☐ WET WEIGHT ☐ DRY WEIGHT

INSTRUCTIONS

RELINQUISHED DATE / TIME

RECEIVED DATE / TIME

Figure 7.3

8. CALIBRATION PROCEDURES AND FREQUENCY

Standard/Reagent Preparation

A critical element in the generation of quality data is the purity/quality and traceability of the standard solutions and reagents used in the analytical operations. Enseco continually monitors the quality of reagents and standard solutions through a series of well-documented procedures.

Primary reference standards and standard solutions used by Enseco are obtained from the National Institue of Standards and Technology, an EPA Cooperator Supplier, or other reliable commercial sources to ensure the highest purity possible. All standards and standard solutions are catalogued to identify the supplier, lot number, purity/concentration, receipt/preparation date, preparer's name, method of preparation, expiration date, and all other pertinent information.

Standard solutions are validated prior to use. Validation procedures can range from a check for chromatographic purity to verification of the concentration of the standard using a standard prepared at a different time or obtained from a different source. Stock and working standards are checked regularly for signs of deterioration, such as discoloration, formation of precipitates, or change in concentration. Care is exercised in the proper storage and handling of standard solutions, and all containers are labeled as to compound, concentration, solvent, expiration date, and preparation data (initials of preparer/date of preparation).

Reagents are examined for purity by subjecting an aliquot or subsample to the analytical method in which it will be used; for example, every lot of dichloromethane (for organic extractables) is analyzed for undesirable contaminants prior to use in the laboratory.

Instrument Calibration and Tuning

Calibration of instrumentation is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet established reporting limits. Each instrument is calibrated with standard solutions appropriate to the type of instrument and the linear range established for the analytical method. The frequency of calibration and calibration verification and the concentration of calibration standards are determined by the manufacturer's guidelines, the analytical method, or the requirements of special contracts.

Gas Chromatography/Mass Spectrometry (GC/MS)

Each day prior to analysis of samples, the instrument is tuned with bromofluorobenzene (BFB) for volatile compounds and decafluorotriphenylphosphine (DFTPP) for semivolatile compounds or other tune criteria as specified by the method used. No samples are analyzed until the instrument has met the tuning criteria of the method.

In general, the instrument is then calibrated for all target compounds. An initial calibration curve is produced to define the working range to establish criteria for identification. This initial calibration is evaluated on a daily basis to ensure that the system is within calibration. If the daily standard does not meet the established criteria, the system is recalibrated.

Chromatography

The field of chromatography involves a variety of instrumentation and detection systems. While calibration standards and acceptance criteria vary depending on the type of system and analytical methodology required for a specific analysis, the general principles of calibration apply uniformly. Each chromatographic system is calibrated prior to performance of analyses. Initial calibration consists of determining the working range, establishing limits of detection, and establishing retention time windows. The calibration is checked on a daily basis to ensure that the system remains within specifications. In addition, continuing calibrations are performed at frequencies required by the method used. If the calibration checks do not meet established criteria, corrective action is taken which may include recalibration and reanalysis of samples. The procedures include examination of instrument performance and analysis information, consultation with the Supervisor and a decision path to determine if recalibration and reanalysis of samples back to the previous acceptable calibration check is warranted.

Metals

Metals analysis basically involves two types of analytical instrumentation: inductively coupled argon plasma emission spectroscopy (ICP), and atomic absorption spectroscopy (AA).

Each ICP is calibrated prior to any analyses being performed using criteria prescribed in the CLP protocol. The calibration is then verified using standards from an independent source. The working range of the instrument is established once every quarter using a linear range verification check standard. No values are reported above this upper concentration value without dilution.

A calibration curve is established daily by analyzing a minimum of two standards, one of which is a calibration blank. The calibration is monitored throughout the day by analyzing a Continuing Calibration Blank (CCB) and a Continuing Calibration Verification standard (CCV). If the verification standard does not meet established criteria, corrective action must be performed. The procedures include examination of instrument performance and analysis information, consultation with the Supervisor and a decision path to determine if recalibration and reanalysis of samples back to the previously acceptable calibration check is warranted.

An interelement check standard is analyzed at the beginning and end of each analytical run, to verify that interelement and background correction factors have remained constant. Results outside of the established criteria trigger reanalysis of samples.

Each AA unit is calibrated prior to any analyses being conducted. A calibration curve is prepared with a minimum of a calibration blank and three standards and then verified with a standard that has been prepared from an independent source at a concentration near the middle of the calibration range. The calibration is then verified on an ongoing basis with a calibration blank and a midpoint calibration standard. If the ongoing calibration standard does not meet established acceptance criteria, corrective action must be performed. The procedures include examination of instrument performance and analysis information, consultation with the Supervisor and a decision path to determine if recalibration and reanalysis of samples back to the previously acceptable calibration check is warranted. All samples are spiked to verify the absence of matrix effects or interferences. The method of standard additions or sample dilution is used when matrix interferences are present.

Wet Chemistry

The field of conventional, non-metals analysis (wet chemistry) involves a variety of instrumental and wet chemical techniques. While calibration and standardization procedures vary depending on the type of system and analytical methodology required for a specific analysis, the general principles of calibration apply universally. Each system is calibrated prior to analyses being conducted. Calibration consists of defining the working range by use of a series of standard solutions, establishing limits of detection, and identifying potential interferences. The calibration is checked on an ongoing basis to ensure that the system remains within specifications. If the ongoing calibration check does not meet established criteria, corrective action must be performed. The procedures include examination of instrument performance and analysis information, consultation with the Supervisor and a decision path to determine if recalibration and reanalysis of samples back to the previous acceptable calibration check is warranted. Continuing calibrations are not performed for non-instrumental methods such as Total Dissolved Solids.

9. ANALYTICAL PROCEDURES

Most analyses performed by Enseco are driven by regulatory concerns. Therefore, methods used at Enseco predominantly originate from regulatory agencies. Generally the methods used are those specified by the U.S. EPA and other federal agencies, state agencies, and professional organizations, as provided in the following references:

- Current EPA (CLP) protocols for the analysis of organic and inorganic hazardous substances including chlorinated dioxins and furans.
- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act," 40 CFR, Part 136.
- "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020 (revised March, 1983 or subsequent revision).
- "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater," EPA-600/4-82-057 (July, 1982).
- "Test Methods for Evaluating Solid Waste" (SW-846), 2nd Edition (revised), Update I (1984), Update II (1985), 3rd Edition (1986), Update I (1989), Office of Solid Waste and Emergency Response, U.S. EPA.
- "Standard Methods for the Examination of Water and Wastewater," 16th Edition (1985) and 17th Edition (1989) American Public Health Association, American Water Works Association, Water Pollution Control Federation, Washington, DC (1985).
- "Official Methods of Analysis," 14th Edition, Association of Official Analytical Chemists, Arlington, VA (1984).
- "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water," U.S. EPA, Environmental Monitoring and Support Laboratory - Cincinnati (September, 1986 or subsequent revision).
- "Annual Book of ASTM Standards," Volumes 11.01 and 11.02, American Society for Testing and Materials (ASTM), Philadelphia, PA (1987).
- "Techniques of Water Resources Investigations of the United States Geological Survey (USGS), Book 5, Laboratory Analysis," USGS, Washington, DC (1979).

The choice of method is dependent on the objectives of the study in terms of qualitative certainty, quantitative sensitivity, precision and accuracy, the type of matrix to be analyzed, and the regulatory program. Each method used routinely is documented in the form of an SOP. The SOP contains detailed instructions concerning both the use and the expected performance of the method. Enseco may deviate from standard methodologies if necessary or appropriate due to the nature or composition of the sample, based on the reasonable judgment of Enseco. Any deviations will be made consistent with recognized standards of the industry and/or this QA Program Plan. Any deviations from published methodology are documented and explained in the SOP. A complete description of the contents of laboratory SOPs is given in Section 17.

Before any methods are routinely used to generate analytical data, the method is validated. Validation criteria consist of:

- Method selection by a senior staff member;
- Documentation of the method in an SOP. This includes a summary of the method, detailed description of the analytical procedure, calculations, reporting formats, safety concerns, and special remarks;
- Testing of the method to verify detection limits and linear range, establish reporting limits and precision and accuracy criteria; and
- Establishment of data acceptance criteria that must be approved by a senior staff member and the Divisional QA Director.

10. DATA REDUCTION, VALIDATION, AND REPORTING

Data Reduction and Validation

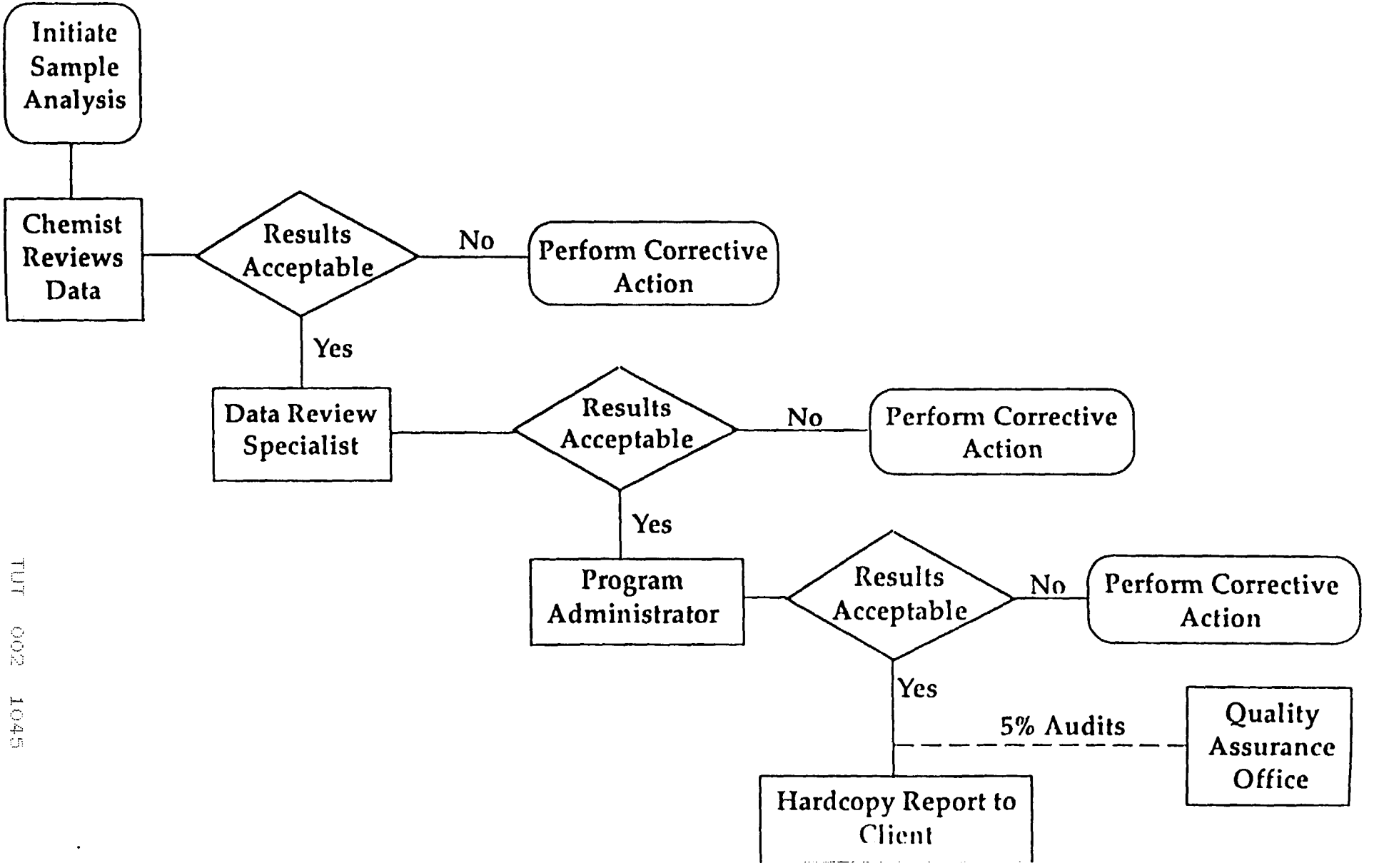
All analytical data generated within Enseco laboratories are extensively reviewed prior to report generation to assure the validity of the reported data. The data validation process consists of data generation, reduction, and three levels of documented review, as described below (also see Figure 10-1). In each stage, the review process is documented by the signature of the reviewer and the date reviewed.

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. All data are generated and reduced following protocols specified in laboratory SOPs. Each analyst reviews the quality of his or her work based on an established set of guidelines. The analyst reviews the data package to ensure that:

- Sample preparation information is correct and complete;
- Analysis information is correct and complete;
- The appropriate SOPs have been followed;
- Analytical results are correct and complete;
- QC samples are within established control limits;
- Blanks are within appropriate QC limits;
- Special sample preparation and analytical requirements have been met; and
- Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented, anomaly forms are complete; holding times are documented, etc.).

Figure 10-1

Data Validation Scheme



The data reduction and validation steps are documented, signed and dated by the analyst. This initial review step, performed by the analyst, is designated Level 1 review. The analyst then passes the data package to an independent reviewer, who performs a Level 2 review.

Level 2 review is performed by a supervisor or data review specialist whose function is to provide an independent review of the data package. This review is also conducted according to an established set of guidelines and is structured to ensure that:

- Calibration data are scientifically sound, appropriate to the method, and completely documented;
- QC samples are within established guidelines;
- Qualitative identification of sample components is correct;
- Quantitative results are correct;
- Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented; anomaly forms are complete; holding times are documented, etc.);
- The data are ready for incorporation into the final report; and
- The data package is complete and ready for data archive.

Level 2 review is structured so that all calibration data and QC sample results are reviewed and all of the analytical results from 10% of the samples are checked back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the bench sheet. The process continues until no errors are found or until the data package has been reviewed in its entirety.

An important element of Level 2 review is the documentation of any errors that have been identified and corrected during the review

process. Enseco believes that the data package submitted by the analyst for Level 2 review should be free of errors. Errors that are found are documented and transmitted to the appropriate supervisor. The cause of the errors is then addressed with additional training or clarification of procedures to ensure that quality data will be generated at the bench.

Level 2 data review is also documented and the signature of the reviewer and the date of review recorded. The reviewed data are then approved for release and a final report is prepared.

Before the report is released to the client, the data are reviewed for completeness and to ensure that the data meet the overall objectives of the project. This review is labeled Level 3 review and is typically done by the Program Administrator.

Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data of high quality are generated consistently.

In addition to the three levels of review discussed above, the Divisional QA department randomly audits 5% of all projects reported. The QA audit includes verifying that holding times have been met, calibration checks are adequate, qualitative and quantitative results are correct, documentation is complete, and QC results are complete and accurate. During the review, the QA department checks the data from 20% of the samples back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the bench sheet. The process continues until no errors are found or until the data package has been reviewed in its entirety.

Data Reporting

A variety of reporting formats, from computerized data tables, to complex reports discussing regulatory issues, to a CLP-deliverables package, are available. In general, Enseco reports contain:

General Discussion: Description of sample types, tests performed, any problems encountered and general comments are given.

Analytical Data: Data are reported by sample or by test. Pertinent information including dates sampled, received, prepared, and extracted are included on each results page. The Enseco reporting limit for each analyte is also given.

Laboratory Performance QC Information: The results (Percent Recovery and Relative Percent Difference) of the Laboratory Control Samples analyzed with the project are listed, together with the control limits. Also, the analytical results for method blanks generated during analysis of organic and metals parameters are given.

Matrix-Specific QC Information: Results of any sample duplicates, matrix spikes, matrix spike duplicates or other project-specific QC requested by the client are also reported.

Methodology: Reference for analytical methodology used is cited.

Custom Services: Special services including data interpretation, special consultation, and raw data packages (when requested) are included.

11. INTERNAL QC CHECKS

The Enseco QA/QC program monitors data quality with internal QC checks. Internal QC checks are used to answer two questions:

1) Are laboratory operations "in control," (i.e., operating within acceptable QC guidelines), during data generation?

2) What effect does the sample matrix have on the data being generated?

The first question is answered by Laboratory Performance QC. Laboratory performance QC is based on the use of a standard, control matrix to generate precision and accuracy data that are compared, on a daily basis, to control limits. This information, in conjunction with method blank data, is used to assess daily laboratory performance.

The second question is addressed with Matrix-Specific QC. Matrix-Specific QC is based on the use of an actual environmental sample for precision and accuracy determinations and commonly relies on the analysis of matrix spikes, matrix duplicates, and matrix spike duplicates. This information, supplemented with field blank results, is used to assess the effect of the matrix and field conditions on analytical data.

Laboratory Performance QC is provided as a standard part of every routine Enseco analysis. Matrix-Specific QC is available as an option to the client and should be specified based on the types of matrices to be analyzed and the Data Quality Objectives (DQOs) and regulatory requirements of the project. A complete discussion of the Enseco Internal QC Check program follows.

Laboratory Performance QC Program

Laboratory Performance QC is performed for every routine Enseco analysis to demonstrate that laboratory operations are "in control". The main elements of Laboratory Performance QC are:

- The analysis of Laboratory Control Samples, which include Duplicate Control Samples (DCS), Single Control Samples (SCS), and method blanks, and

- The use of calibration standards to assure that both qualitative identification and quantitative measurements are within control limits.

The Laboratory Control Sample program is discussed below. Please refer to Section 8 of this manual for a discussion of calibration procedures.

Laboratory Control Samples (LCS)

Laboratory Control Samples (LCS) are well-characterized, laboratory generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. Three types of LCS are routinely analyzed: Duplicate Control Samples (DCS), Single Control Samples (SCS), and method blanks. Certain LCS (DCS, SCS) are used to monitor the precision and accuracy of the analytical process, independent of matrix effects. Other LCS (method blanks) are used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data. Each of these LCS are described below.

The results of the LCS are compared to well-defined laboratory acceptance criteria to determine whether the laboratory system is "in control." Controlling lab operations with LCS (as opposed to matrix spike/matrix spike duplicate samples), offers the advantage of being able to differentiate quality problems due to laboratory procedural errors from those due to matrix effects. As a result, procedural errors can be identified and corrected by the analyst at the bench, without waiting for extensive senior level review or costly and time-consuming reanalysis of the sample.

Duplicate Control Samples (DCS)

Duplicate Control Samples (DCS) are used to monitor the precision and accuracy of the analytical system on an on-going basis. Each DCS consists of a standard, control matrix that is spiked with a group of target compounds representative of the method analytes. A DCS pair is analyzed for every 20 samples processed by the method. DCS are analyzed with environmental samples to provide evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

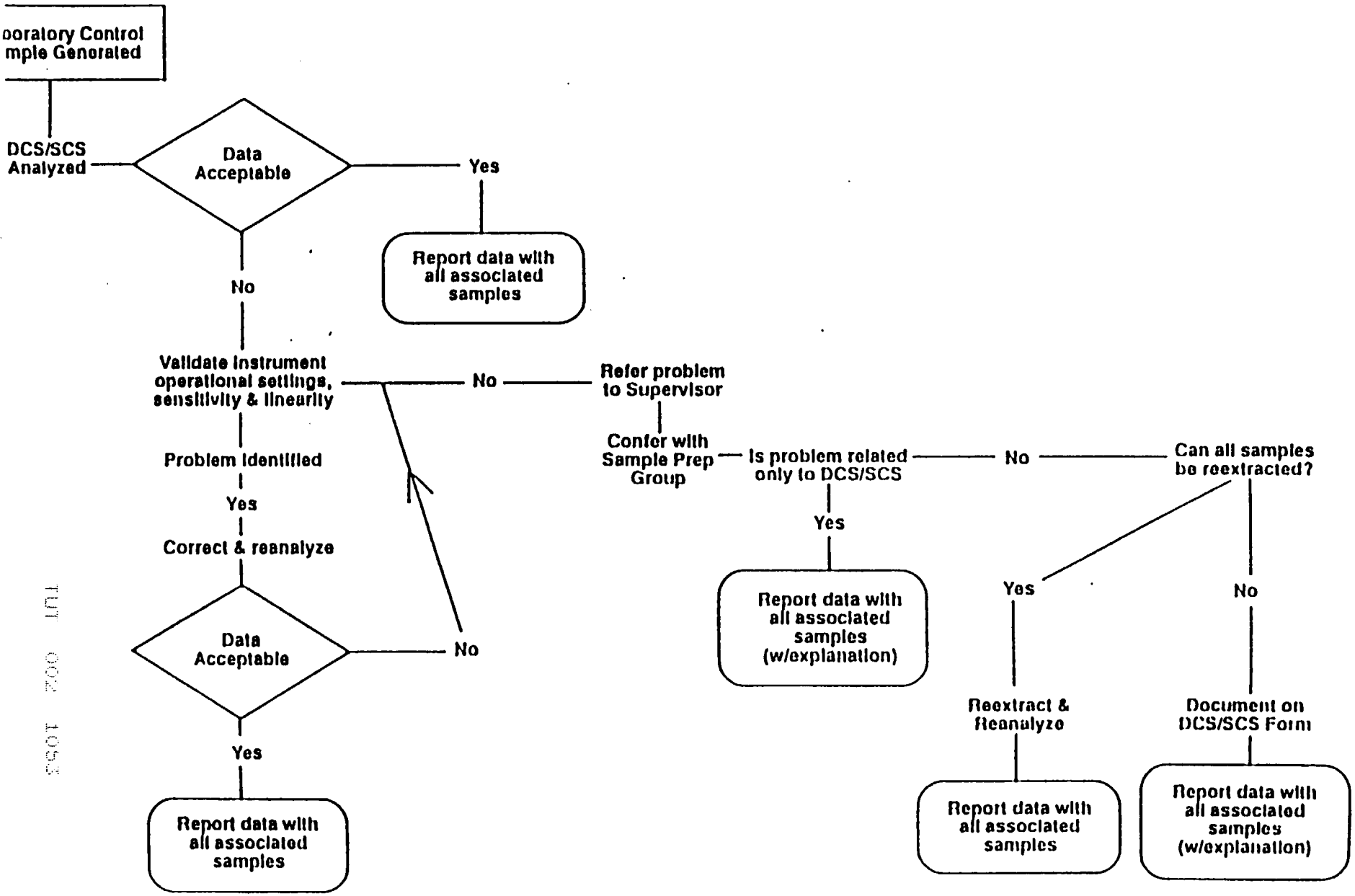
Accuracy data (average recovery of each analyte in the DCS pair) and precision data (Relative Percent Difference [RPD] between each analyte in the DCS pair) are compared to control limits that have been established for each of the analytes contained in the DCS. Initially, control limits for analytes spiked into the DCS are taken directly from the CLP program. If CLP limits are not available, Enseco historical data are used to set the control limits. The control limits are recalculated periodically, as sufficient laboratory data become available. Control limits for accuracy for each analyte are based on the historical average recovery (mean of the average recoveries of the DCS pairs) plus or minus three standard deviation units. Control limits for precision for each analyte are based on the historical RPD. Acceptable RPDs range from zero (no difference between DCS results) to the average RPD plus three standard deviation units. Analytical data that are generated with a DCS pair which falls within the established control limits are judged to be in control. Data generated with a DCS pair which falls outside of the control limits are considered suspect and corrective action must be performed. The procedure used to evaluate data from control samples is given in Figure 11-1. The procedures include examination of instrument performance and preparation and analysis information, consultation with the supervisor, and finally a decision path for determining whether reanalysis is warranted.

DCS have been established for each routine analytical method. Reagent water is used as the control matrix for the analysis of aqueous samples. The DCS compounds are spiked into reagent water and carried through the appropriate steps of the analysis. The control matrix for solids samples for organic analyses is standard Ottawa sand, an ASTM approved material for use in highway construction, due to its homogeneity. The DCS compounds are spiked into the Ottawa sand and carried through the appropriate steps of the analysis. For metal analyses, a spiked solid matrix from a commercial source is used.

As stated previously, DCS are analyzed at a frequency of no less than one DCS pair per 20 samples. The DCS program is supplemented with the SCS program to ensure that Laboratory Performance QC is available with each batch of samples processed (see following subsection).

Figure 11-1

Laboratory Performance QC Control Sample Evaluation



DCS precision and accuracy data are archived in the LIMS. In addition, the associated DCS data are reported with each set of sample results to enable the client to make a quality assessment of the data.

Single Control Samples (SCS)

As stated above, a DCS pair is analyzed with every 20 samples to measure the precision and accuracy of an analysis on an ongoing basis. However, samples are often analyzed in lots of less than 20, due to holding time or turn-around time requirements. Since it is necessary to have a measure of laboratory performance with each batch of samples processed, Enseco has instituted the SCS program.

An SCS consists of a control matrix that is spiked with surrogate compounds appropriate to the method being used. In cases where no surrogate is available, (e.g., metals or wet chemistry) a single DCS serves as the control sample. An SCS is prepared for each sample lot for which the DCS pair are not analyzed. Recovery data generated from the SCS are compared to control limits that have been established for each of the compounds being monitored. Initially, CLP control limits or Enseco historical data are used to set the control limits. Control limits are recalculated periodically as sufficient SCS data are available. Control limits for SCS components are based on the historical average recovery in the SCS plus or minus three standard deviation units.

Analytical data that are generated with an SCS which falls within the control limits are judged to be in control. Data that are generated with an SCS which falls outside of acceptance criteria are considered suspect and corrective action must be performed. The protocols for evaluating SCS are identical to those established for DCS (see Figure 11-1). SCS recovery (accuracy) data are archived in the LIMS. In addition, the associated SCS data are reported with each set of sample results to enable the client to make a quality assessment of the data.

Method Blank

Method blanks, also known as reagent, analytical, or preparation blanks, are analyzed to assess the level of background interference or contamination which exists in the analytical system and which might lead to the reporting of elevated concentration levels or false positive data.

As part of the standard Enseco QC program, a method blank is analyzed with every batch of samples processed. A method blank consists of reagents specific to the method which are carried through every aspect of the procedure, including preparation, clean-up, and analysis. The results of the method blank analysis are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples.

Ideally, the concentration of target analytes in the blank should be below the Reporting Limit for that analyte. In practice, however, some common laboratory solvents and metals are difficult to eliminate to the parts-per-billion levels commonly reported in environmental analyses. Therefore, criteria for determining blank acceptability must be based on consideration of the analytical techniques used, analytes reported, and Reporting Limits required.

For organic analyses, the concentration of target analytes in the blank must be below the Reporting Limit for that analyte in order for the blank to be considered acceptable. An exception is made for common laboratory contaminants (methylene chloride, acetone, 2-butanone, and phthalate esters) which may be present in the blank at up to 5 times the Reporting Limit and still be considered acceptable. This policy is consistent with the CLP policy and has been established in recognition of the fact that these compounds are frequently found at low levels in method blanks due to the materials used in the collection, preparation, and analysis of samples for organic parameters.

For non-routine organic analyses, other components may be established as common contaminants for that particular analysis. For example, naphthalene is frequently found in PAH-SIM analyses. If, upon thorough review of the method during validation (see Section 9) it is deemed impossible to eliminate trace amounts of analytes from the process, these analytes are likewise allowed at up to 5 times the reporting limit.

For metals and Wet Chemistry analyses, where the Reporting Limits are typically near the Instrument Detection Limit (IDL), the policy is that the concentration of the target analytes in the blank must be below two times the Reporting Limit. If the blank value for a target analyte lies below the Reporting Limit, the Reporting Limit for that analyte in the associated samples is unaffected. If the blank value lies between the Reporting Limit and two times the Reporting Limit, the Reporting Limit for that analyte in the associated samples is raised to the level found in the blank. A blank containing an analyte(s) above two times the Reporting Limit is considered unacceptable unless the lowest concentration of the analyte in the associated samples is at least ten times the blank concentration (as per CLP protocol) or the concentration of the analyte in all samples associated with the blank is below the reporting limit.

In addition, for Wet Chemistry tests, the method SOP directs how the blank is treated. Generally, a reagent blank is used both to zero the equipment and as one of the calibration standards. If a preparation step is required for the analysis, then a prep blank is also analyzed to determine the extent of contamination or background interference. The concentration found in the prep blank is subtracted from the concentration found in any associated sample prior to calculating the final result when specified by the method. Blanks have no application or significance for some Wet Chemistry parameters (e.g. pH).

If the blank does not meet acceptance criteria, the source of contamination must be investigated and appropriate corrective action must be taken and documented. Investigation includes an evaluation of the data to determine the extent and effect of the contamination on the sample results. Corrective actions may include reanalysis of the blank, and/or repreparation and reanalysis of the blank and all associated samples.

For organic and metals analyses, and selected Wet Chemistry tests, method blank results are reported with each set of sample results. Sample results are not corrected for blank contamination unless required by the analytical method or requested by the client. Occasionally, due to limited sample volume or other constraints, the laboratory reports data associated with an unacceptable blank. In these cases, the Reporting Limit for each analyte contained in the blank is raised to the level found in the blank for all sample results associated with that blank.

Matrix-Specific QC

Matrix-Specific QC is used to assess the effects of a sample matrix or field conditions on the analytical data. The main elements of Matrix-Specific QC are:

- The analysis of matrix spikes, matrix duplicates, and matrix spike duplicates;
- Monitoring the recovery of surrogate compounds from environmental samples;
- Monitoring the results of standard additions in environmental samples;
- The analysis of field blanks; and
- The determination of method detection limits in a specific matrix.

Different regulatory programs have different requirements in terms of Matrix-Specific QC. In order to ensure that the data generated meet all Data Quality Objectives, Enseco recommends that its clients include Matrix-Specific QC that fulfills the Data Quality Objectives and regulatory requirements of the project. A discussion of the different elements of Matrix-Specific QC follows.

Matrix Spikes, Matrix Duplicates, and Matrix Spike Duplicates

A Matrix Spike (MS) is an environmental sample to which known concentrations of analytes have been added. The MS is taken through the entire analytical procedure and the recovery of the analytes is calculated. Results are expressed as percent recovery. The MS is used to evaluate the effect of the sample matrix on the accuracy of the analysis.

A Matrix Duplicate (MD) is an environmental sample that is divided into two separate aliquots. The aliquots are processed separately and the results compared to determine the effects of the matrix on the precision of the analysis. Results are expressed as RPD.

A Matrix Spike Duplicate (MSD) is an environmental sample that is divided into two separate aliquots, each of which is spiked with known concentrations of analytes. The two spiked aliquots are processed separately and the results compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results are expressed as RPD and percent recovery.

Surrogate Recoveries and Standard Additions

Surrogates are organic compounds which are similar to the analytes of interest in chemical behavior, but which are not normally found in

environmental samples. Surrogates are added to samples to monitor the effect of the matrix on the accuracy of the analysis. Results are reported in terms of percent recovery.

Enseco routinely adds surrogates to samples requiring GC or GC/MS analysis and reports these surrogate recoveries to the client. The laboratory does not control its operations based on surrogate recoveries in environmental samples. As discussed earlier in this section, Enseco controls its operations based on the results of Laboratory Control Samples. The surrogate recoveries are primarily used by the laboratory to assess matrix effects. However, obvious problems with sample preparation and analysis (e.g. evaporation to dryness, leaking septum, etc.) which can lead to poor surrogate spike recoveries must be ruled out prior to attributing low surrogate recoveries to matrix effects.

Standard Additions (SA) is the practice of adding a series of known amounts of an analyte to an environmental sample. The fortified samples are then analyzed and the recovery of the analytes calculated. The practice of SA's is generally used with metal and wet chemistry to determine the effect of the sample matrix on the accuracy of the analyses.

Field Blanks

Field blanks are check samples that monitor contamination originating from the collection, transport or storage of environmental samples. One example of a field blank is an equipment blank. An equipment blank is blank water that is poured through the sample collection device to check the adequacy of the cleaning procedures for the sampling equipment. Another type of field blank is a trip blank. A trip blank is a laboratory control matrix (typically water) which is sent to the field in an appropriate sample container, remains unopened in the field, and then

is sent back to the laboratory. The purpose of the trip blank is to assess the impact of field and shipping conditions on the samples. The results from field blanks are reported to the client as samples in the same concentration units as the samples. No correction of the analytical data is done in the laboratory based on the analysis of field blanks.

Matrix-Specific Detection Limits

Method Detection Limits (MDL's) determined on a specific sample matrix are called Matrix-Specific Detection Limits. See Section 14 for a discussion of detection and reporting limits.

12. PERFORMANCE AND SYSTEM AUDITS

Enseco laboratories participate in a variety of federal and state programs, (including the U.S. EPA CLP), that subject each of the laboratories to stringent system and performance audits on a regular basis. A system audit is a review of laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff and procedures in place to generate acceptable data. A performance audit verifies the ability of the laboratory to correctly identify and quantitate compounds in blind check samples submitted by the auditing agency. The purpose of these audits is to identify those laboratories that are capable of generating scientifically sound data. Enseco is certified to perform environmental analyses under programs administered by the U.S. Department of Energy, U.S. Air Force, U.S. Navy, and over 20 states. The most current list of Enseco certifications is available upon request.

In addition to external audits conducted by certifying agencies or clients, Enseco regularly conducts the following internal audits:

- Quarterly systems audits conducted by the Divisional QA Director.
- Periodic (at least yearly) audits conducted by the Corporate QA Office.
- Special audits by the Divisional QA Director or Corporate QA Office when a problem is suspected.

Enseco laboratories also routinely analyze check samples as described below:

- Laboratory Control Samples (DCS, SCS, and method blanks) are analyzed at a frequency equal to at least 10% of the total number of samples analyzed (see Section 11).

- All Enseco laboratories participate in the analyses of EPA check samples provided under the Water Supply (WS) and Water Pollution (WP) Performance Evaluation Studies. The results of these PE samples are tabulated by the Corporate QA Office to identify performance trends within the Enseco laboratories.

- The majority of the Enseco laboratories are CLP labs and thus analyze organic and/or inorganic CLP PE samples on a quarterly basis. The results of these analyses are also tabulated and evaluated by the Corporate QA Office.

- The laboratories participate in multiple state certification programs (including New York, New Jersey and California) which require that PE samples be analyzed periodically.

- Blind check samples from an independent commercial firm are sent to the laboratories periodically by the Corporate QA Office. The frequency and type of samples sent is based on problem areas identified by evaluation of tabulated PE results.

The results of these check samples are used to identify areas where additional training is needed or clarification of procedures is required.

13. PREVENTIVE MAINTENANCE

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument. Designated laboratory personnel are trained in routine maintenance procedures for all major instrumentation. When repairs are necessary, they are performed by either trained staff or trained service engineers employed by the instrument manufacturer.

Each laboratory has detailed SOPs on file that describe preventive maintenance procedures and schedules. The laboratories also maintain detailed logbooks documenting the preventive maintenance and repairs performed on each analytical instrument.

14. SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA QUALITY AND DETERMINE REPORTING LIMITS

Data Quality Assessment

The effectiveness of a QA program is measured by the quality of data generated by the laboratory. Data quality is judged in terms of its precision, accuracy, representativeness, completeness and comparability. These terms are described as follows:

Precision is the degree to which the measurement is reproducible. Precision can be assessed by replicate measurements of DCS, reference materials, or environmental samples. Enseco routinely monitors precision by comparing the RPD between DCS measurements with control limits established at plus three standard deviations from the mean RPD of historical DCS data.

Precision is frequently determined by comparison of replicates. The standard deviation of "n" measurements of "x" is commonly used to estimate precision.

Standard deviation (s) is calculated as follows:

$$s = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (x_i - \bar{x})^2}$$

where a quantity "x" (e.g., a concentration) is measured "n" times.

The relative standard deviation, which expresses standard deviation as a percentage of the mean, is generally useful in the comparison of three or more replicates (although it may be applied in the case of n = 2).

$$RSD = 100\ (s/\bar{x})$$

where: RSD = relative standard deviation

s = standard deviation

$\bar{x}$ = mean

In the case of duplicates, the RPD between the two samples may be used to estimate precision.

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

where: RPD = relative percent difference

D_1 = first sample value

D_2 = second sample value (duplicate)

Accuracy is a determination of how close the measurement is to the true value. Accuracy can be assessed using LCS, standard reference materials, or spiked environmental samples. Unless specified otherwise in special contracts, Enseco monitors accuracy by comparing LCS results with control limits established at plus or minus three standard deviation units from the mean of historical LCS results.

The determination of the accuracy of a measurement requires a knowledge of the true or accepted value for the signal being measured. Accuracy may be calculated in terms of percent recovery as follows:

$$\text{Percent Recovery} = \frac{x}{T} \times 100$$

where: x = the observed value of measurement

T = "true" value

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Analytical data should represent the sample analyzed regardless of the heterogeneity of the original sample matrix. Enseco strives to accommodate all sample matrices. Some samples may require analysis of multiple phases to obtain representative results.

Completeness is a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under normal conditions.

To be considered complete, the data set must contain all analytical results and data specified for the project. In addition, all data are compared to project requirements to ensure that specifications were met. Any deviations are reported in the report narrative.

The percent completeness for each set of samples can be calculated as follows:

$$\text{Completeness} = \frac{\text{valid data obtained}}{\text{total data planned}} \times 100\%$$

Comparability expresses the confidence with which one data set can be compared to another data set measuring the same property. Comparability is ensured through the use of established and approved analytical methods, consistency in the basis of analysis (wet weight, volume, etc.), consistency in reporting units (ppm, ppb, etc.), and analysis of standard reference materials.

Reporting Limits

Assuring the validity of quantitative measurements at low concentrations is an extremely difficult technical problem. With regulatory action levels being pushed lower and lower, the validity of any given measurement becomes even more important. The consequences of false positive or false negative data can be significant.

A number of terms have been used, by the EPA and other technical groups, to express the lowest concentration of an analyte which can be measured. Some of these terms, their definitions, and sources are listed in Table 14-1. A graphical representation of these terms is given in Figure 14-1.

Enseco takes very seriously its responsibility to report technically defensible data. Therefore, we have established a Reporting Limit (RL) for each analyte in each method. The RL represents the value above which we believe reliable data can be routinely obtained.

These Reporting Limits were established by collecting Method Detection Limit (MDL) data for organic and wet chemistry analyses and Instrument Detection Limit (IDL) data for metals analyses from each Enseco laboratory. The MDL data were collected using the procedures described in 40 CFR 136 Appendix B. IDL data were calculated using the procedures outlined in the EPA Contract Laboratory Program (CLP) Statement of Work dated 7/88. The MDL/IDL data were then compared to various limits published in EPA methods and in the regulations. For example for

Volatile Organics, the MDL data generated in Enseco laboratories were compared to the Practical Quantitation Limits (PQLs) published in SW-846 method 8240; the PQLs contained in the July 9, 1987, Federal Register Final Rulemaking on Appendix IX; the Contract Required Quantitation Limits (CRQLs) in the CLP Method for Volatile Organics; and the MDLs in Method 624. Then a Reporting Limit for each analyte was established which considered all of this information. The RL was set at a level above which we were confident that our laboratories could detect and quantify the analyte consistently. Using this procedure, the Reporting Limits established are generally between 2 to 5 times the laboratory MDL/IDL. This range is consistent with the American Chemical Society definition for the Limit of Quantitation (LOQ). (See Table 14-1)

Enseco routinely reports results below the reporting limit as Not Detected (ND) because, by definition, the reliability of the data at that level is questionable. As an option, Enseco can report data below the reporting limit and flag the data. Reporting limits are adjusted for sample dilution.

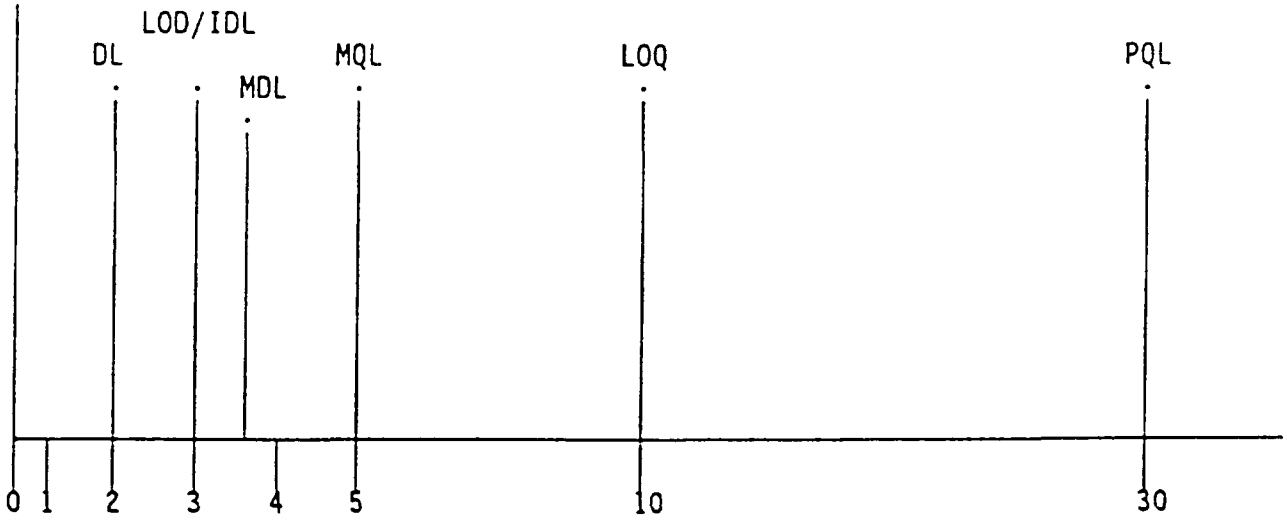
TABLE 14-1

DEFINITION OF DETECTION LIMIT TERMS

	DEFINITION	DETERMINATION	CALCULATION	SOURCE
ıtection Limit (DL)	The concentration which is distinctly detectable above, but close to a blank.	Analysis of replicate standards	Two times the standard deviation	Methods for Chemical Analysis of Water and Wastes
mit of Detection (LOD)	The lowest concentration that can be determined to be statistically different from a blank	Analysis of replicate samples	Three times the standard deviation	ACS Definition
ıthod Detection Limit MDL)	The minimum concentration of a substance that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.	Analysis of a minimum of seven replicates spiked at 1 to 5 times the expected detection limit.	The standard deviation times the Student t-value at the desired confidence level. (For seven replicates, the value is 3.14)	40 CFR 136 Definition for EPA Water Programs
ıstrument Detection ımit (IDL)	The smallest signal above background noise that an instrument can detect reliably.	Analysis of seven replicate standards on three non-consecutive days.	Three times the standard deviation	Contract Laboratory Program
ethod Quantitation Limit MQL)	The minimum concentration of a substance that can be measured and reported	Analysis of replicate samples	Five times the standard deviation	SW-846
imit of Quantitation)	The level above which quantitative results may be obtained with a specified degree of confidence	Analysis of replicate samples	Ten times the standard deviation	ACS Definition
tical Quantitation (PQL)	The lowest level that can be reliably determined within specified limits of precision and accuracy during routine laboratory operating conditions	Interlaboratory analysis of check samples	1) Ten times the MDL 2) Value where 80% of laboratories are within 20% of the true value	RCRA SDWA Programs
act Required etection Limit (CRDL)	Reporting limit specified for laboratories under contract to the EPA for	Unknown	Unknown	Contract Laboratory Program

FIGURE 14-1

Graphical Representation of Detection Limit Terms
(See Table 14-1 for Definitions)



MULTIPLIER OF STANDARD DEVIATION OF REPLICATES

NOTE: The values along the horizontal "Standard Deviation (SD)" axis are approximate values and are meant to show the relative, not absolute, relationship between the terms.

15. CORRECTIVE ACTION

When errors, deficiencies, or out-of-control situations exist, the QA program provides systematic procedures, called "corrective actions," to resolve problems and restore proper functioning to the analytical system.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the acceptable windows for precision and accuracy;
- Blanks, DCS or SCS contain contaminants above acceptable levels;
- Undesirable trends are detected in spike recoveries or RPD between duplicates;
- There are unusual changes in detection limits;
- Deficiencies are detected by the QA department during internal or external audits or from the results of performance evaluation samples; or
- Inquiries concerning data quality are received from clients.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the project records.

16. QA REPORTS TO MANAGEMENT

The reporting system is a valuable tool for measuring the overall effectiveness of the QA program. It serves as an instrument for evaluating the program design, identifying problems and trends, and planning for future needs. Divisional QA Directors submit extensive monthly reports to the Corporate QA Director, the Director of Quality Assurance and Technology, the General Manager and the President. These reports include:

- The results of internal systems audits including any corrective actions taken;
- Performance evaluation scores and commentaries;
- Results of site visits and audits by regulatory agencies and clients;
- Performance on major contracts, (including CLP);
- Problems encountered and corrective actions taken;
- Holding time violations;
- Comments and recommendations; and
- A summary of the 5% QA data audits conducted.

The Corporate QA Director regularly reports on the status of the QA Program to the President and each General Manager. These reports summarize the information gathered through the laboratory reporting system and contain a thorough review and evaluation of laboratory operations throughout Enseco.

17. LABORATORY DOCUMENTATION

Complete and accurate documentation of analytical and procedural information is an important part of the QA program. The following describes different types of documentation used in the Enseco laboratories.

SOPs

Details of analytical and QC protocols are contained in SOPs. SOPs are documents that contain detailed proprietary information on how to perform a laboratory procedure. Enseco has four categories of laboratory SOPs:

- SOPs for Performance of an Analytical Method;
- SOPs for Preparation of Standards and Reagents;
- SOPs for Equipment Operation, Calibration, and Maintenance; and
- SOPs for General Laboratory Procedures.

The formats for these SOPs are given in Appendix II.

All SOPs are approved by the QA Department before being implemented. The distribution of current SOPs and archiving of outdated ones is controlled through the QA Department.

Because of the detailed nature of SOPs, Enseco considers them to be proprietary documents. SOPs are available for review at each location.

LIMS

Enseco laboratories rely on a customized Laboratory Information Management System (LIMS) as the primary database. Client information, sample results, and QC results are all stored in the LIMS. Reports are generated directly from the database to eliminate transcription errors. A tiered security system is in place to control the ability of lab personnel to change results, and the system is designed with an audit trail that identifies when information has been changed and who changed it. The most recent data are kept on-line. Data are periodically archived on magnetic tape or optical disk.

Laboratory Bench Sheets

Laboratory bench sheets are used to document information from routine laboratory operations, including sample preparation and analysis. Bench sheets are used to ensure that the information is recorded in a complete and organized manner and that the analysis can be reconstructed, if necessary. Portions of information from the bench sheet are also stored in the LIMS. Each bench sheet is initialed and dated as information is entered.

Laboratory Notebooks

Laboratory notebooks are used to document information that cannot easily be recorded on benchsheets such as methods development information. Each page in a laboratory notebook is initialed and dated as information is entered.

Control Charts

Enseco laboratories use control charts to visually track the LCS precision and accuracy data. These control charts are used to identify trends in the analyses which may indicate a problem with the analytical procedure. When an adverse trend is detected corrective action is performed.

Anomaly Forms

Any situation which is outside of the normal scope of operations, as described in the laboratory SOPs, is documented on an Anomaly Form. Examples of anomalous situations include: formation of a precipitate in an extract; formation of an emulsion during an extraction step; or missed holding times. These situations are documented to enable a thorough review of the data to occur.

Out-of-Control situations are also documented on Anomaly Forms. An Out-of-Control situation occurs when QC data fall outside of established control limits. The documentation associated with an Out-of-Control situation is reviewed by the supervisor and the QA Department. Out-of-Control situations trigger Corrective Action. Corrective Actions taken are also documented on the Anomaly Form.

Project Files

The project file consists of a project summary file and a raw data file. The project summary file includes correspondence from the client, (letters, phone logs, contracts, project plans) copies of preliminary and final reports, chain of custody, air bills, photographs of samples, level 3 review checklists, QA review checklist when applicable and the summary

file inventory. The raw data file includes sample data, QC data, benchsheets, level 1 and level 2 review checklists, instrument logbook pages pertinent to the project and the raw data file inventory. Contracts, project plans, calibration data and QC data may be stored separately from the project record. All project records contain cross-references to this information. When a project is complete, all records are passed to the Document Custodian who inventories the file, checks for completeness, and puts the file into document archive.

APPENDIX I

MAXIMUM HOLDING TIMES AND SAMPLE COLLECTION/PRESERVATION INFORMATION

Sources: Tables A-E
Federal Register, October 26, 1984
Methods for Chemical Analyses of Water and Wastes
SW-846, 3rd Edition, Update I
State of California Leaking Underground
Fuel Tank Field Manual, May 1988

Table F:
Contract Laboratory Program Statement of
Work for Organic Analysis dated 3/90 (as amended)
Contract Laboratory Program Statement of
Work for Inorganic Analysis dated 7/88

Table G:
Federal Register, June 29, 1990

(QA Program Plan, Revision 3.4)

A. VOLATILE ORGANICS

Matrix	Container	Minimum Sample Size	Preservative	Holding Time (From Date Sampled)
Water Samples				
No Residual Chlorine Present	3 40 mL vials with Teflon lined septum caps	40 mL	HCl to pH<2, 4°C	14 days
Residual Chlorine Present	3 40 mL vials with Teflon lined septum caps	40 mL	4 drops of 10% sodium thiosulfate, HCl to pH<2, 4°C	14 days
Acrolein and Acrylonitrile	3 40 mL vials with Teflon lined septum caps	40 mL	Adjust to pH 4-5, 4°C	14 days
Soil/Sediments and Sludges	Glass jar with Teflon liner or core tube	10 g	4°C	14 days
Concentrated Waste Samples	Glass jar with Teflon liner or core tube	10 g	None	14 days

The above information applies to the following parameters and methods:

Parameter	Method
Volatile Halocarbons	601/8010 (GC)
Volatile Aromatics	602/8020 (GC)
Volatile Organics	624/8240/8260 (GC/MS), 8015 (GC)
Acrolein/Acrylonitrile	603/8030 (GC)

B. SEMIVOLATILE ORGANICS

Matrix	Container	Minimum Sample Size	Preservative	Holding Time (From Date Sampled)
Water Samples				
No Residual Chlorine Present	1 liter glass with Teflon liner	1 liter	4°C	Samples must be extracted within 7 days and analyzed within 40 days of extraction.
Residual Chlorine Present	1 liter glass with Teflon liner	1 liter	Add 3 mL 10% sodium thiosulfate per gallon, 4°C	Samples must be extracted within 7 days and analyzed within 40 days of extraction.
Soil/Sediments and Sludges	Glass jar with Teflon liner or core tube	50 g	4°C	Samples must be extracted within 14 days and analyzed within 40 days of extraction.
Concentrated Waste Samples	Glass jar with Teflon liner or core tube	50 g	None	Samples must be extracted within 14 days and analyzed within 40 days of extraction.

The above information applies to the following parameters and methods:

Parameter	Method
Phenols	604/8040 (GC)
Phthalate Esters	606/8060 (GC)
Organochlorine Pesticides/PCBs	608/8080 (GC)
Polyaromaltic Hydrocarbons	610/8310 (HPLC)
Organophosphate Pesticides	614/8140 (GC)
Phenoxy acid Herbicides	615/8150 (GC)
Semivolatile Organics	625/8270 (GC/MS)
Carbamate & Urea Pesticides	632 (HPLC)

C. OTHER ORGANICS

Parameter	Method No.	Matrix	Holding Time[a] (from Date Sampled)	Container	Preservative	Min. Sample Size
Dioxins/Furans	8280	Water	30 days extn. 45 days anal.[b]	One liter glass	4°C	1000 ml
		Soil/Waste	30 days extn. 45 days anal.[b]	core tube or glass jar	4°C	50 g
Petroleum Hydrocarbons as Gasoline	TPH-Gasoline Purge & Trap (LUFT manual)	Water	14 days	3 40 mL vials with Teflon liners	4°C, HCl to pH < 2	40 mL
		Soil/Waste	14 days	Core tube or glass jar	4°C	50 g
Petroleum Hydrocarbons as Gasoline	TPH-Gasoline Extractable (LUFT manual)	Water	14 days extn. 40 days anal.	One liter glass	4°C, HCl to pH < 2	500 mL
		Soil/Waste	14 days extn. 40 days anal.	Core tube or glass jar	4°C	50 g
Petroleum Hydrocarbons as Diesel	TPH-Diesel Extractable (LUFT manual)	Water	14 days extn. 40 days anal.	One liter glass	4°C	500 mL
		Soil/Waste	14 days extn. 40 days anal.	Core tube or glass jar	4°C	50 g
Petroleum Hydrocarbons (TPH)	TPH-IR (418.1)	Water	28 days	One liter glass	4°C, H_2SO_4 to pH < 2	1000 mL

(a) extn: extraction anal: analysis
(b) from date of collection

D. METALS

Parameter	Method No.	Matrix	Holding Time (from Date Sampled to Analysis)	Container	Preservative[a]	Min. Sample Size
Metals (ICP)	200.7/6010	Water	6 months	Poly	HNO_3 to pH < 2.0	100 ml
		Soil/Waste	6 months	core tube/glass jar	4°C	10 g
Arsenic (GF-AA)	206.2/7060	Water	6 months	Poly	HNO_3 to pH < 2.0	100 ml
		Soil/Waste	6 months	core tube/glass jar	4°C	10 g
Mercury (CV-AA)	245.1/7470	Water	28 days	Poly	HNO_3 to pH < 2.0	100 ml
		Soil/Waste	28 days	core tube/glass jar	4°C	10 g
Selenium (GF-AA)	270.2/7740	Water	6 months	Poly	HNO_3 to pH < 2.0	100 ml
		Soil/Waste	6 months	core tube/glass jar	4°C	10 g
Thallium (GF-AA)	279.2/7841	Water	6 months	Poly	HNO_3 to pH < 2.0	100 ml
		Soil/Waste	6 months	core tube/glass jar	4°C	10 g
Lead (GF-AA)	239.2/7421	Water	6 months	Poly	HNO_3 to pH < 2.0	100 ml
		Soil/Waste	6 months	core tube/glass jar	4°C	10 g
Chromium (III/VI)	220.7/218.4/ 312B/7197	Water	24 hours	Poly	4°C	100 ml
		Soil/Waste	24 hours extn. [b]	core tube/glass jar	4°C	10 g

(a) Listed preservative is for total metals. Dissolved or suspended metals require filtration prior to pH adjustment.

(b) extn: extraction

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E. WET CHEMISTRY

Parameter	Method No.	Matrix	Holding Time[a] (from Date Sampled to Analysis)	Container	Preservative	Min. Sample Size
Acidity	305.1	Water	14 days	Poly	4°C	50 ml
Alkalinity	310.1	Water	14 days	Poly	4°C	50 ml
Ammonia	350.1	Water	28 days	Glass	4°C, H_2SO_4 to pH < 2	50 ml
Biochemical Oxygen Demand	405.1	Water	48 hours	Poly	4°C	200 ml
Bromide	Dionex	Water	28 days	Poly	4°C	50 ml
Chemical Oxygen Demand	410.4	Water	28 days	Glass	4°C, H_2SO_4 to pH < 2	100 ml
Chloride	300.0	Water	28 days	Poly	4°C	50 ml
orine, idual	330.1	Water	ASAP	Poly	4°C	100 ml
iform, Total ecal	909A/ 909C	Water	6 hours	Sterile poly	4°C, $Na_2S_2O_3$	100 ml
or	110.2	Water	48 hours	Poly	4°C	100 ml

E. WET CHEMISTRY (Cont.)

Parameter	Method No.	Matrix	Holding Time[a] (from Date Sampled)	Container	Preservative	Min. Sample Size
Cyanide	335.1/ 335.2/335.3	Water	14 days	Poly	4°C, NaOH to pH > 12	250 ml
Fluoride	340.2	Water	28 days	Poly	4°C	50 ml
Gross Alpha, Beta and Radium	9310/ 9315	Water	6 months	Poly	HNO_3 to ph < 2	2000 ml
Hardness	200.7/ 314A/314B	Water	6 months	Poly	HNO_3 to pH < 2	50 ml
Iodide	Dionex	Water	28 days	Poly	4°C	50 ml
Nitrate	353.2/300.0	Water	48 hours	Poly	4°C	50 ml
Nitrite	354.1	Water	48 hours	Poly	4°C	50 ml
Nitrite plus Nitrate	353.2	Water	28 days	Glass	4°C, H_2SO_4 to pH < 2	50 ml
Odor	140.1	Water	ASAP	Glass	4°C	1000 mL

E. WET CHEMISTRY (Cont.)

Parameter	Method No.	Matrix	Holding Time[a] (from Date Sampled)	Container	Preservative	Min. Sample Size
Oil and Grease	413.1/ 413.2	Water	28 days	Glass	4°C, H_2SO_4 to pH < 2	1000 ml
Organic Carbon (TOC)	415.1	Water	28 days	Glass	4°C, H_2SO_4 to pH < 2	100 ml
Organic Halogen (TOX)	9020	Water	28 days	Glass	4°C, H_2SO_4 to pH < 2	200 ml
Orthophosphate	365.3	Water	48 hours	Poly	4°C	100 ml
pH	150.1	Water	ASAP	Poly	4°C	50 ml
Phenolics	420.1/ 420.2	Water	28 days (b)	Glass	4°C, H_2SO_4 to ph < 2	100 ml
pecific onductance	120.1	Water	28 days	Poly	4°C	50 ml
ulfate	300.0	Water	28 days	Poly	4°C	50 ml
ulfide	376.2	Water	7 days	Poly	4°C, NaOH to pH > 9 $Zn(C_2H_3O_3)2$	100 ml

E. WET CHEMISTRY (Cont.)

Parameter	Method No.	Matrix	Holding Time[a] (from Date Sampled)	Container	Preservative	Min. Sample Size
Sulfite	377.1	Water	ASAP	Poly	4°C	100 ml
Surfactants (MBAS)	425.1	Water	48 hours	Poly	4°C	100 ml
Total Dissolved Solids	160.1	Water	7 days	Poly	4°C	100 ml
Total Kjeldahl Nitrogen	351.2	Water	28 days	Glass	4°C, H_2SO_4 to pH < 2	100 ml
Total Phosphorus	365.3	Water	28 days	Glass	H_2SO_4 to pH < 2	100 ml
Total Solids	160.3	Water	7 days	Poly	4°C	100 ml
Total Suspended olids	160.2	Water	7 days	Poly	4°C	100 ml
otal Volatile olids	160.4	Water	7 days	Poly	4°C	100 ml

E. WET CHEMISTRY (Cont.)

Parameter	Method No.	Matrix	Holding Time[a] (from Date Sampled)	Container	Preservative	Min. Sample Size
Turbidity	180.1	Water	48 hours	Poly	4°C	50 ml

a) Parameters with holding times of 24 hours or less are analyzed on the day of receipt in the laboratory. Parameters with holding times between 24 and 48 hours are analyzed within one day of receipt in the laboratory.

b) The 28 day holding time comes from Table 1 of Methods for Chemical Analysis of Water and Wastes, issued March 1983. This information supercedes that contained in Method 420.1/420.2 published in 1979.

F. CLP HOLDING TIMES

Parameter	Matrix	Holding Time[a] (from Date Received)	Container	Preservative	Min. Sample Size
Volatile Organics	Water	10 days	3 40 mL vials with Teflon lined caps	4°C	40 mL
	Soil	10 days	Glass jar with Teflon liner or core tube	4°C	10 g
Extractable Organics	Water	5 days extn. 40 days anal.	1 liter glass with Teflon liner	4°C	1000 mL
	Soil	10 days extn. 40 days anal.	Glass jar with Teflon liner or core tube	4°C	50 g
Metals (other than Mercury)	Water	180 days	P,G [b]	HNO_3 to pH < 2	100 mL
	Soil	180 days	P,G	4°C	10 g
Mercury	Water	26 days	P,G	HNO_3 to pH < 2	100 mL
	Soil	26 days	P,G	4°C	10 g
Cyanide	Water	12 days	P,G	0.6 g ascorbic acid,[c] NaOH to pH >12, 4°C	100 mL
	Soil	12 days	P,G	4°C	10 g

a) Holding times calculated from date of receipt in laboratory
b) Polyethylene (P) or glass (G)
c) Only used in the presence of residual chlorine

G. TCLP HOLDING TIMES

arameter	Matrix	From: Field Collection To: TCLP Extraction	From: TCLP Extraction To: Prep Extraction	From: Prep Extraction To: Determination Analy.	Container	Preservative	Min. Sample Size
olatiles	Waste	14	NA	14	Glass	4 degrees C	4 oz
emivolatiles	Waste	14	7	40	Glass	4 degrees C	32 oz (1)
ercury	Waste	28	NA	28	Glass	4 degrees C	32 oz (1)
etals (Except Mercury)	Waste	180	NA	180	Glass	4 degrees C	32 oz (1)

(1) Smaller sample size is adequate for solid samples or individual fractions. A combined volume of 32 oz is recommended for semivolatiles and metals. A separate 4 oz container should always be used for the volatile fraction. Volatile fractions should be stored with minimal headspace.

APPENDIX II

FORMATS FOR STANDARD OPERATING PROCEDURES (SOP)

(QA Program Plan, Revision 3.1

FORMAT FOR SOP - LABORATORY, ANALYTICAL METHOD

Title (includes method number)

1. Scope and Application
   - 1.1 Analytes
   - 1.2 Detection limit (instrument and method)
   - 1.3 Applicable matrices
   - 1.4 Dynamic range
   - 1.5 Approximate analytical time (i.e., 5 minutes, 2 days)

2. Method Summary
   - 2.1 Generic description of method and chemistry behind it (i.e., extract with solvent, convert to methyl ester, analyze by electron-capture gas chromatography)

3. Comments
   - 3.1 Interferences
   - 3.2 Helpful hints

4. Safety Issues

5. Sample Collection, Preservation, Containers, and Holding Times

6. Apparatus

7. Reagents and Standards

8. Procedure (detailed step-by-step)
   - 8.1 Sample preparation
   - 8.2 Calibration
   - 8.3 Analysis

9. QA/QC Requirements

   9.1 QC samples

   9.2 Acceptance criteria (precision and accuracy, % of multi-component QC analytes which must be within windows)

   9.3 Corrective action required (reference current QC manual)

10. Calculations

11. Reporting

   11.1 Reporting units

   11.2 Reporting limits

   11.3 Significant figures and reporting values below detection limit

   11.4 LIMS data entry

12. References

   12.1 Method source

   12.2 Deviations from source method and rationale

13. Appendices (optional)

   Additional information may be placed in appendices. This may include supporting data (e.g. method validation information), tables, flow charts. etc.

FORMAT FOR SOP - LABORATORY, STANDARDS AND REAGENTS

Title

1. Reagent/Standard Name

2. Type (reagent, calibration standard, DCS, SCS, stock solution, etc.)

3. Constituents/concentration/solvent

4. Safety Issues

5. Shelf Life

6. Procedure
   - 6.1 Preparation
   - 6.2 Documentation (purchase date, open date, labeling, etc.)
   - 6.3 Verification

7. Responsibilities

8. Appendices (optional) Any additional information.

FORMAT FOR SOP - LABORATORY, EQUIPMENT OPERATION, CALIBRATION, AND MAINTENANCE

Title

1. Purpose

2. Safety Issues (applicable to the specific equipment)

3. Procedure

   3.1 Initial start-up
   3.2 Calibration and performance documentation
   3.3 Example output
   3.4 Shut-down sequence
   3.5 Maintenance and maintenance records

4. Responsibilities

5. Comments

6. Definitions

7. Appendices (optional) Any additional information.

FORMAT FOR SOP - LABORATORY, PROCEDURAL

Title

1. Purpose

2. Policies

3. Safety Issues

4. Procedure

5. Responsibilities

6. Comments

7. Definitions

8. Appendices (optional) Any additional information.