64579

SOIL OVERSIGHT AND SAMPLING
SOIL AND GROUNDWATER INVESTIGATION

U.S. EPA CONTRACT NO. 68-W9-0002

REVISED
BROSSMAN SHORT FORM
FOR THE

TUTU WELLFIELD SITE
ST. THOMAS, U.S. VIRGIN ISLANDS

WORK ASSIGNMENT NO: C02048
DOCUMENT NO: TESV-C02048-QA-CLXF

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1.	PROJECT:	Remedial Investigation of Groundwater and Soil Tutu Wellfield Site U.S. Virgin Islands
2.	PROJECT REQUESTED BY:	USEPA Region II USEPA Contract No.: 68-W9-0002 Site Number: 2P1D Work Assignment No.: C02048
3.	DATE OF REQUEST:	May 19, 1992
4.	DATE OF PROJECT INITIATION:	June 2, 1992
5.	EPA WORK ASSIGNMENT MANAGER:	Caroline Kwan
6.	EPA QUALITY ASSURANCE OFFICER:	Laura Scalise

7. PROJECT DESCRIPTION

Volatile organic contamination of the drinking water aquifer by petroleum hydrocarbons and chlorinated compounds has been documented at the Tutu Wellfield Site on St. Thomas, U.S. Virgin Islands. Several facilities in the area use and/or store some form of the contaminants of concern. To date, three potentially responsible parties have been identified: Esso Standard Oil SA., Texaco Caribbean, Inc., and L'Henry, Inc., which operates the O'Henry dry cleaning facility. Esso and Texaco have joined together to form the Tutu Environmental Investigation Committee (TEIC). Pursuant to an Administrative Order on Consent issued by EPA, TEIC and L'Henry are initiating a remedial investigation to determine the extent of contamination in groundwater and subsurface soil.

CDM FEDERAL PROGRAMS CORPORATION (CDM FPC) TES V personnel will provide enforcement support for EPA through document evaluation and review, oversight of field activities, and confirmatory sampling and analysis at the Tutu Wellfield site.

A. Objective and Scope:

CDM FPC will provide field investigation oversight and split sample analyses in accordance with established EPA procedures and the Scope of Work incorporated in the Administrative Order. The oversight team will observe and document all procedures to ensure that the TEIC contractor, Geraghty and Miller (G&M), adheres to established Region II protocol and the EPA approved G&M March 1992 Work Plan and site Field Operations Plan.

TES V personnel will accept split-samples from the PRP of approximately 20% of their subsurface soil and groundwater samples. For drummed sludge and and surface soil samples, EPA has requested TES V personnel to accept splits of 100% of the PRP samples. All split samples will be analyzed through EPA's Contract Laboratory Program (CLP) for the same constituents as the PRP samples (see section 7D).

CDM FPC will provide sample containers for split samples and coordinate the packaging and shipping of all samples to the CLP laboratories.

B. Data Usage:

The CLP split-sample data will be used to confirm the accuracy of the PRP's analytical data.

C. Sampling Locations:

Based on Geraghty and Miller's March 1992 Work Plan, G&M will collect a minimum of one subsurface soil sample from each of 30 soil borings, at the site. Seventeen of the borings will be converted to monitoring wells. A map of G&M's proposed boring and well locations is shown in Figure 1. Two rounds of groundwater samples will be collected from the 17 wells. As determined in a recent technical meeting with EPA and the PRP on May 5, 1992, surface soil and drummed solid waste samples will be collected from the area near the Curriculum Building. Background surface soil samples will be collected near outcrop areas upgradient of the Curriculum Building.

CDM FPC plans to accept splits of six subsurface soil samples, two solid waste samples, two onsite surface soil samples, three background surface soil samples and eight groundwater samples.

D. Analytical Parameters and Their Rationale:

The principal contaminants of concern in site groundwater are petroleum hydrocarbons, including benzene, toluene, ethylbenzene and xylenes (BTEX), and chlorinated volatile organic compounds.

Subsurface soil samples selected by G&M for laboratory analysis will be analyzed for Target Compound List (TCL) volatile organic compounds (VOCs) plus 1,2-dibromoethane, n-propylbenzene, and methyl-tertiary-butyl-ether (MBTE); TCL base-neutral/acid extractables (BNAs); total petroleum hydrocarbons (TPH); and Target Analyte List (TAL) metals and cyanide. Geraghty and Miller will field screen all subsurface soil samples for BTEX, dichloroethylene (DCE), trichloroethylene (TCE) and tetrachloroethylene (PCE) gas using a portable gas chromatograph.

Groundwater samples collected by G&M will be analyzed for the same constituents as for subsurface soil listed above. Geraghty and Miller will also collect filtered groundwater samples for dissolved TAL metals analysis. Onsite surface soil samples will be analyzed for EPA's TCL VOCs and BNAs and for TAL metals and cyanide. Background surface soil samples will be analyzed for TAL metals only. Solid waste samples from drums will be analyzed for TCL VOCs only.

Per EPA direction, split samples will be analyzed for the same constituents as the PRP, except that splits of filtered groundwater will not be accepted. Analysis for the TCL organic compounds plus 1,2-dibromoethane, n-propylbenzene, and MBTE will be in accordance with the EPA CLP Statement of Work (SOW) for organics, document number OLM01.8 (or current revision). TAL analytes will be analyzed in accordance with the EPA CLP SOW for inorganics, document number ILM01.0 (or current revision). TPH analysis

will be performed in accordance with method 418.1 as stated in Methods for Chemical Analysis of Water and Wastes (MCAWW), EPA-600/4-79-020, revised March 1983.

E. Analytes:

See Table 1.

F. QC Sample Analysis:

See Table 2.

Duplicates

Environmental duplicates will not be collected because the samples accepted per this Brossman Short Form are split samples of the PRP samples.

Trip Blanks

A. A trip blank will be prepared by the CDM FPC TES V team at the start of each day on which aqueous samples will be collected for the analysis of TCL volatiles. Trip blanks are used to determine whether on site atmospheric contaminants are seeping into the sample vials, or if any cross-contamination of samples is occurring during shipment or storage of samples containers. A trip blank consists of demonstrated analyte-free water (based on TCL analysis results below Contract Required Detection Limits) sealed in 40 ml septum vials with no headspace (including bubbles) in the vials. Trip blanks will be preserved as described in section 11.D.3 with HCl to a pH <2. Trip blanks will be kept in close proximity to the samples being collected and maintained at a temperature of 4 degrees celcius. One trip blank will be included with each daily shipment to CLP laboratories that contains aqueous samples collected for TCL volatile organic analysis. Trip blanks will be analyzed for volatile organic compounds only. Control of all trip blanks related to split samples will be the responsibility of the CDM FPC TES V field personnel.

   Certification of blank water quality received in the field will be kept on site and/or will be requested from the supplier and will be filed in the CDM FPC TES V project files once field work is completed. Further definition of what constitutes "demonstrated analyte-free water" may be found in the EPA Region II CERCLA Quality Assurance Manual, Revision 1 (October 1989), Section 10-A.2 (pages 59-60).

Field Blanks

   CDM FPC will accept a limited number of random splits of G&M's field blank samples. Field blanks, also known as "rinse blanks" or "equipment rinsate blanks" are used to assess the effectiveness of equipment decontamination. One field blank will be accepted for each equipment type and will be analyzed for the same constituents as the environmental sample splits. Field blanks will be preserved per the requirements for aqueous samples analyzed for these parameters of interest. Field blanks will be collected prior to use of the

decontaminated equipment for sampling. The frequency for field blanks is one per "decon" event, not to exceed one per day, for each equipment type and for each sample matrix. Field blanks are generated by pouring demonstrated analyte-free water over the decontaminated sampling piece of equipment. Field blanks will be collected in a manner that will minimize potential contamination from the ambient air. It is permissible to use the same aliquot of water on all equipment associated with a particular sample matrix for analysis of semi-volatile organics, and inorganic analytes. However, a separate field rinse blank must be collected for each piece of equipment associated with a particular sample media that will be analyzed for volatile organic compounds.

Matrix Spike/Matrix Spike Duplicates

Matrix spike samples are collected to demonstrate the precision of laboratory analysis. It should be noted that ground water and surface water constitute two distinct matrices. An aqueous matrix spike sample requires the collection of a triple volume in order to perform matrix spike and matrix spike duplicate (MS/MSD) analysis for organics.

According to EPA's Region II CERCLA QA Manual, a minimum of one matrix spike sample per matrix must be submitted to the laboratory for every sample delivery group (SDG). An SDG is defined as one of the following:

1. All samples of a RAS or SAS case if the sample number is less than 20 (including environmental duplicates, but excluding QC blanks) and if sampling is completed in less than 14 calendar days.

2. Each group of 20 samples within a RAS or SAS case (including environmental duplicates, but excluding QC blank) if the number is greater than 20.

3. Each 14 calendar day period within a RAS case if the number of samples collected is less than 20. Each 7 calendar day period within a SAS case if the number of samples collected is less than 20.

8. SCHEDULE OF TASKS AND PRODUCTS:

The field work associated with the sampling activities is scheduled to begin the week of June 2, 1992, and is estimated to take approximately 18 weeks. Split samples will be collected starting in mid-July when the shallow wells and borings are being drilled.

9. PROJECT ORGANIZATION:

The following is a list of key project personnel and their corresponding responsibilities:

Sampling Operations	Andrew Scano
Sampling QC	Pam Phillip
CLP Coordinator	Vasu Desikan
Laboratory Analysis	Contract Laboratory Program

Laboratory QC	Contract Laboratory Program
Data Processing Activities	Andrew Scano
Data Processing QC	Jeniffer Oxford
Data Quality Review	TES V data validation staff
Internal Systems Audit	TES V QA staff
Technical Systems Audit	TES V QA staff
Overall QA	Rosemary Ellersick
Health & Safety Coordinator	Jeanne Litwin
Overall Project Coordination	Sally Odland

10. DATA QUALITY REQUIREMENTS AND ASSESSMENTS:

Data quality will be assessed in terms of accuracy, precision, sensitivity, representativeness, comparability and completeness within each data set and comparison of the split sample data with the PRP's data.

The accuracy and precision requirements for the TCL to be performed through Routine Analytical Services (RAS) and Special Analytical Services (SAS) and TAL analyses to be performed through RAS are specified in the EPA CLP Statement of Work for Organics (SOW OLM01.8, or current version) and the EPA CLP Statement of Work for Inorganics (SOW OLM01.0, or current version). In addition to the analysis for volatile organic parameters on the TCL list, analysis will be performed for n-propylbenzene, 1,2-dichloroethane, and MTBE. The method for these organic compounds will be SOW OLM01.8, or current version, and performed through SAS. Historical precision and accuracy requirements will be observed for TPH analyses, to be performed through SAS by the CLP as specified in MCAWW method 418.1 for TPH.

Sensitivities required for the CLP analysis of QA samples will be method detection limits from the same cited Statements of Work and the SAS request for TPH.

A. Data Representativeness:

Representativeness expresses the degree to which the sample portrays the population characteristics of process/environmental conditions at a given location and point in time.

Samples from the six soil borings and four monitoring wells (two rounds of sampling yielding 8 groundwater samples) shown in Figure 1 will be split between Geraghty and Miller and the TES V sampling team, or will be collected by G&M and accepted by the TES V sampling team. The samples will be split by equally dividing the contents into two sets of sample containers. Soil samples for analysis of VOCs will be collocated rather than homogenized and divided. Samples will then be sent to different laboratories for analysis.

B. Data Comparability:

All data will be presented in the units specified in the Contract Laboratory Program's Information For Bidders (IFBs). Split sample data will be compared to the results reported by Geraghty and Miller.

C. Data Completeness:

Completeness is the percentage of all measurements made whose results are judged to be valid. Completeness will be ensured by accepting an adequate percentage of split samples to accomplish project objectives.

11. SAMPLING PROCEDURES:

Prior to sampling, and between sampling points, all field equipment will be decontaminated by G&M using the following EPA approved method:

- o Wash and scrub with low phosphate detergent
- o Potable water rinse
- o Rinse with 10% HNO_3* (1% for carbon steel implements)
- o Potable water rinse+
- o Methanol rinse*
- o Hexane rinse*
- o Thorough Deionized demonstrated analyte free water rinse (at least five times the volume of solvent used)
- o Air dry
- o Wrap in aluminum foil

* Acid will be Ultrapure grade; solvent will be pesticide grade or better.
+ Tap water from any municipal water treatment system may be used. The use of untreated potable water supply is not acceptable.

During the sampling activity, the TES V team will note the following in the field logbook:

- o Date, time and weather
- o Identity of the notetaker
- o Field personnel/affiliations
- o Level of personal protective clothing/gear
- o Schedule for the day
- o Equipment used (record ID numbers) and calibration information
- o Sample collection equipment and procedures
- o Decontamination procedures for the equipment
- o Sample container and demonstrated analyte-free water shipment lot numbers (as received in the field)
- o Sample description, depth and location (from two reference points)
- o Any unusual discoloration or evidence of contamination
- o Field parameter measurements (such as temp., pH, and specific cond.)
- o Calculations
- o Any preservation requirements
- o Sample volumes and containers used
- o Sample analysis to be performed
- o Sample matrix
- o Sample identification number used by the contractor for their sample and by the TES team for the split sample.
- o CLP RAS or SAS sample number
- o Overnight courier airbill number
- o Deviations from the PRP's approved field operations plan, this Brossman or other problems
- o Descriptions of any photographs taken

o Notes of conversations with project coordinators.

A. Subsurface Soil Sampling

1. Geraghty and Miller will collect subsurface soil samples during soil boring installation in accordance with their March 1992 Work Plan and Field Operations Plan.

2. Split samples for VOA, and TPH soil aliquots will consist of collocated grab samples.

3. Soil samples for all remaining analyses will be homogenized in a stainless steel bowl before filling the containers.

4. All soil samples and splits samples will be carefully logged, packed in coolers with bagged ice sufficient to cool the media to 4°C, and sealed for shipment according to the procedures described in section 12.

B. Surface Soil Sampling

1. Geraghty and Miller will collect two surface soil samples from the area around the Curriculum Building and three background samples from areas upgradient of the Curriculum Building in accordance with their March 1992 Work Plan and Field Operations Plan. Background samples will be analyzed for TAL metals only.

2. Split samples for VOA, and TPH soil aliquots will consist of collocated grab samples.

3. Soil samples for all remaining analyses will be homogenized in a stainless steel bowl before filling the containers.

4. All soil samples and split samples will be carefully logged, packed in coolers with bagged ice sufficient to cool the media to 4°C, and sealed for shipment according to the procedures described in section 12.

C. Solid Waste Sampling

1. Geraghty and Miller will collect two solid waste samples from drums stored at the Curriculum Building in accordance with their March 1992 Work Plan and Field Operations Plan. Samples will be analyzed for TCL VOCs only. Since the samples may contain high concentrations of organic compounds, they will be analyzed by the CLP method for organic analysis, multi-media, high concentration (SOW for Revision 9/88, including Revision 4/89).

2. Split samples will consist of collocated grab samples.

3. All solid waste samples and split samples will be carefully logged, packed in coolers with bagged ice sufficient to cool the media to 4°C, and sealed for shipment according to the procedures described in section 12.

D. Groundwater Sampling

1. A minimum of two weeks after well development, Geraghty and Miller will collect groundwater samples from newly installed monitoring wells in accordance with their approved March 1992 Work Plan and Field Operations Plan.

2. Split samples for VOCs (TCL) plus 1,2-dibromoethane, n-propylbenzene, and MTBE; TCL BNAs; TAL metals plus cyanide; and TPH will be provided by the alternate filling of G&M and CDM FPC sample bottles such that representative samples are collected. VOA bottles will be filled first, followed by BNAs, TPH and inorganics.

3. The pH of the VOA sample aliquots will be adjusted to <2 by the dropwise addition of 1:1 HCl to the VOA vials prior to sample collection. The amount of preservative required at each well will be determined by adding HCl to a separate aliquot of equal volume and testing with pH paper. All vials will be checked to ensure zero headspace.

4. The pH of the TPH samples will be adjusted to <2 by adding H_2SO_4. The H_2SO_4 will be added a small amount at a time, mixed into the sample by tipping, and tested with pH paper.

5. The pH of the metals sample aliquot will be adjusted to <2 by adding HNO_3. The HNO_3 will be added a small amount at a time, mixed into the sample by tipping the bottle, and tested with pH paper.

6. The pH of the cyanide sample aliquot will be adjusted to >12 by adding NaOH. The NaOH will be added a small amount at a time, mixed into the sample by tipping the bottle, and tested with pH paper.

7. All samples and split samples will be carefully logged, packed in coolers with bagged ice sufficient to cool the media to $4^{o}C$, and sealed for shipment according to the procedures described in Section 12.

12. SAMPLE CUSTODY PROCEDURES:

A. Traffic Reports:

A CLP Sample Traffic Report is a four page carbonless form used for RAS samples. A preprinted identification number will be assigned in the field by the TES V team for each RAS sample collected and will be affixed by the TES V team to each container of the sample. An organic traffic report (Figure 2) will be completed for samples receiving organic analyses (VOC and BNA analysis). An inorganic traffic report (Figure 3) will be completed for RAS samples receiving inorganic analyses (metals plus cyanide). A SAS Packing List (Figure 4) will be completed for all SAS samples (TPH; MTBE, n-propylbenze and 1,2-dibromethane; and solid waste TCL VOC analysis). The information that must be completed on the traffic reports and SAS packing lists is detailed in the User's Guide to the Contract Laboratory Program (CLP User's Guide), EPA/540/P-91/002, January 1991, and the Samplers Guide to the Contract Laboratory Program (Samplers Guide), EPA/540/P-90/006, December 1990. Copies of these guides will be on-site. Field QC blanks and matrix spike designated sample splits will be

noted on these forms.

B. Chain of Custody Record:

To maintain a record of sample collection and transfer for every sample processed, a "Chain-of-Custody Record" will be completed for each cooler shipment. In Region II, the EPA Environmental Services Division form should be used. Figure 5 is an example of this chain-of-custody record which must be secured to the inside of the shipping cooler along with the CLP traffic reports (for samples submitted for RAS analyses) or SAS packing lists. A copy of the custody record is retained for the FPC TES V files. Shipping coolers will be secured with nylon fiber strapping tape or its equivalent and custody seals (see Figure 6) will be placed across cooler openings so that the cooler may not be opened without breaking the seals. A mailing label addressed to the laboratory will also be attached to the cooler and protected by clear tape as back-up to the courier airbill so that coolers may not need to be opened by courier staff in the event that the airbill is separated from the cooler.

The PRP contractor will be required to sign CDM FPC's custody form as the sample collector. Each time the samples are transferred to another person, signatures of the persons relinquishing and receiving them, as well as the time and date of transfer will be completed in the appropriate spaces on the chain-of-custody record. The PRP contractor will relinquish the sample to the TES V sampler (CDM FPC) who will be the second sampler.

Samples will be shipped from the field directly to the laboratories designated to perform the analyses via Federal Express or an equivalent overnight courier. Samples will be sent to the laboratory from the field within 24 hours of sample collection. All courier receipts and/or paperwork associated with shipment of the samples will serve as a custody record for the samples while they are in transit from the field to the laboratory. Custody seals should remain intact during this transfer.

When the samples are delivered to the laboratory, signatures of the labortory personnel receiving them and courier personnel relinquishing them will be completed in the appropriate spaces on the chain-of-custody record. This will complete sample transfer.

It will be the CLP laboratory's responsibility to maintain internal log books and records that provide a custody record throughout sample preparation and analysis. To track field samples through data handling, CDM FPC will maintain photocopies of all traffic reports, SAS packing lists and chain-of-custody records.

When responsible for sample custody, field personnel should keep in mind the definition of sample custody according to the EPA Office of Enforcement and Compliance Monitoring National Enforcement Investigations Center (NEIC) Policies and Procedures (May 1986) that states that a sample is under custody if:

1. it is in one's possession, or
2. it is in one's view, after being in one's possession, or
3. it is locked up after being in one's possession, or

4. it is in a designated secure area.

C. Sample Labels and Tags:

One numbered adhesive RAS label from the strip (Figure 4) or a hand written label for SAS samples (and the additional MS/MSD bottles filled for aqueous RAS organic extractables) is affixed to each container making up the sample. To protect the label from water and solvent damage, each label is covered with clear waterproof tape.

For samples requiring decontamination, the self-adhesive sample labels must be completely covered with clear mylar tape prior to sampling. The sample labels permanently identify each sample collected and link each sample component throughout the analytical process.

In addition to the preprinted sample identification number, each split sample container for CLP analysis will be identified with a sample tag (Figure 6). These sample tags are retained by the CLP laboratories as physical evidence of sample receipt and must therefore be signed by the sampler, which in the case of split samples is the TES V oversight contractor (CDM FPC) sampler accepting the split sample. The sample tag will contain an abbreviated summary of the field logbook entry for the sample. The information that must be entered on the sample tag is detailed in the CLP User's Guide and listed below.

- o Site spill number (do not provide the site name or address)
- o CLP case number or SAS number
- o RAS of SAS sample number
- o Date and time of collection
- o Station number/location
- o Sampler signature
- o Sample type (composite or grab)
- o Sample matrix
- o Preservative added and type
- o Type of analysis required.

D. Field Notebooks:

A final element in the documentation process will be the completion of a formal field notebook by TES V personnel. The field notebooks will be maintained by the TES V team and will be stored in the TES V document control systems. All entries will be made in accordance with the TES V requirements for maintaining field notebooks as detailed in the Camp Dresser and McKee Inc. (CDM) Site Investigation Procedures Manual, SOP #5621004. A copy of this SOP will be available on-site. The information that will be recorded in the field notebooks for each sample collected by the Contractor is described in Section 12, Sampling Procedures.

E. Sample Bottles:

CDM FPC will purchase pre-cleaned sample containers which meet EPA specifications. These bottles are pre-cleaned in accordance with OSWER

Directive number 9240.0-05 and QC tested according to prescribed procedures to ensure that no contamination exists that might affect sample data. QC screening procedures include container cleaning by lot group; each lot group is assigned a unique identifying number, which is permanently affixed to each container. Clean, empty bottles are shipped to users in protective cardboard cartons.

F. Sample Handling, Packaging, and Shipping:

When sent by common carrier, the packaging, labeling and shipping of hazardous wastes and substances is regulated by the U.S. Department of Transportation (DOT) under CFR 49. Samples obtained at uncontrolled hazardous waste sites are classified according to pollutant concentration. "Low Level" samples are generally dilute and are usually collected from areas surrounding a spill or dump site (i.e., offsite samples from soils, rivers, lakes, etc.). "Medium Level" samples are generally collected on-site, in areas of moderate dilution by normal environmental processes.

It is anticipated that contaminant concentrations in all samples collected from the Tutu site will range between low level and medium level. All samples will be packaged and shipped according to the following procedures detailed in the current (January 1991) CLP User's Guide and Samplers Guide (see previous references).

Samples will be kept on ice to maintain a temperature of $4o^{C}$. The samples will be shipped within 24 hours of collection via an overnight carrier.

Each sample label will be covered with clear waterproof tape to protect it from water and solvent attack. A custody seal (Figure 6) is to be placed over the lid of all sample bottles and also the shipping cooler, before shipping it to the laboratory. This will ensure that the samples are not opened prior to their arrival at the laboratory.

G. Sample Shipment Coordination:

To enable the Sample Management Office (SMO) to track the shipment of samples from the field to the laboratory and ensure timely laboratory receipt of samples, the TES V team will notify the SMO (phone number: (703) 684-5678) immediately following all sample shipments, and provide the following information:

- o Sampler name and phone number
- o Case number and RAS or SAS number
- o Site name/code
- o Exact number(s) and matrix(ces) and concentrations of samples shipped
- o batch numbers (dioxin only)
- o Laboratory(ies) samples were shipped to
- o Carrier and airbill number(s) for the shipment
- o Method (e.g., overnight, two-day)
- o Date of shipment
- o Any irregularities or anticipated problems with the samples, including special handling instructions, or deviations from established sampling procedures.
- o Suspected hazards associated with the samples or site.

- o Status of the sampling project (e.g., final shipment, update of future shipping schedule).

H. Sample Trip Reports:

A sample trip report is required to be completed for each case number and must contain all the information as shown below. All blanks and duplicates must be clearly indicated.

- o Site number
- o Sampling date
- o EPA case number and RAS or SAS number
- o Site location/name
- o Sample description
- o Names, addresses of laboratories receiving samples and sample types going to those laboratories.
- o Sample dispatch data (e.g., Federal Express airbill number(s))
- o Names, organization, affiliation and duties of onsite sampling personnel
- o Additional comments (samples types, totals, blanks, etc.)
- o Name of preparer and date of report
- o Approval signature and date.

This trip report will be sent directly to the Regional Sample Control Center (RSCC) within 10 days of final sample shipment with a copy to the TES V CLP coordinator.

13. CALIBRATION PROCEDURES AND PREVENTATIVE MAINTENANCE:

A. Field Equipment:

Each piece of TES V equipment used for measuring, monitoring, analytical purposes is calibrated and maintained periodically to ensure accuracy within specified limits. Calibration and maintenance procedures and the frequency at which these procedures should be applied are detailed in the CDM Field Equipment Operation, Maintenance, and Calibration Manual (FEOMC Manual). A copy of this document will be onsite.

TES V field equipment undergoes calibration and maintenance by qualified personnel before and after every field activity. If an instrument is to be in the field for longer than four weeks, it is returned to the Region II equipment room to undergo full calibration and maintenance. Where applicable, instrument calibration is field checked.

The following equipment will be used by the TES V team at the Tutu site:

Instrument	SOP Reference
HNu Photoionization Detector (PID)	FEOMC Manual, Section 1.0

A field equipment status report is kept for each piece of field equipment and kept in a Region II Log Book. The Log Book is stored at the CDM FPC Regional office. These reports contain the following information:

- o Date of calibration and date of last maintenance
- o Initials of person performing the calibration and/or maintenance
- o Accuracy prior to and following calibration and/or maintenance
- o Notations on equipment failures.

B. Laboratory Equipment:

Calibration procedures and preventative maintenance for laboratory equipment is the responsibility of the CLP lab selected to analyze the samples.

14. DOCUMENTATION, DATA REDUCTION, AND REPORTING:

A. Documentation:

This QAPP and the Geraghty and Miller Investigation Work Plan will be available to CDM FPC personnel in the field.

Each sample submitted for analysis will be properly documented to ensure timely, correct, and complete analysis for all parameters requested and to support the use of analytical data in potential enforcement actions. The following documentation will be submitted:

- o Organic and inorganic sample traffic reports
- o Chain of Custody Records
- o Field logbooks
- o Sampling labels
- o SAS packing list (where appropriate).

B. Data Reduction and Reporting

The CLP will be responsible for preparing the analytical data packages for organic analysis and providing this information to the SMO. SMO will then send the package to the RSCC for data validation.

Data reduction will consist of summarizing the data collected during the field work. The data will be used to prepare a report summarizing the analytical results and notes taken during the field work.

15. DATA VALIDATION:

Analytical data will be validated by the TES V team by the following EPA Region II standard operating procedures:

1. SOP Number HW-2: Inorganic Data Validation, Revision 11, January 1992.

2. SOP Number HW-6: Organic Data Validation, Revision 8, January 1992.

16. SYSTEM AUDIT:

A field technical and/or internal system audit may be conducted by the CDM

FPC QA staff during the course of this assignment. If so, each audit will be documented in a report discussing any observed deficiencies for each phase of work. If needed, a Corrective Action Request will be completed and sent with the audit report. Upon receipt of a satisfactory response, an Audit Completion Notice will be issued. If no deficiencies are noted, the original audit report will serve as the Audit Completion Notice. Reports are sent to the audit file with copies to the audited entity, CDM FPC management and EPA. System audits may also be performed or arranged by EPA.

Laboratory performance audits are the responsibility of the CLP and EPA.

17. CORRECTIVE ACTION:

If a nonconformance or deficiency is identified during a systems audit, and has not been corrected during the audit, corrective action will be initiated by the TES V Regional QA Coordinator (RQAC), as appropriate. The TES V Quality Assurance Director (QAD) will be responsible for filing a report of the audit along with a Corrective Action Request Form if any nonconformances were noted during the audit. The TES V QAD will also be responsible for ensuring that corrective action is taken and will forward the report to the audited entity, CDM FPC TES V management and EPA.

The CDM FPC TES V team will be responsible for reporting all suspected technical nonconformances. Any staff member who discovers or suspects a nonconformance in an approved document shall inform the TES V work assignment manager (WAM), who is responsible for determining if a corrective action is required. The WAM shall ensure that no additional work, which is dependent on the nonconforming activity, is performed until the nonconformance report is corrected.

Oversight personnel should maintain a logbook in which observations of poor practices or discrepancies from the governing plans on the part of the PRP contractor should be noted. The EPA WAM should be notified of such findings by phone.

18. REPORTS:

The EPA will be provided with all the data gathered during this field activity. During field activities, CDM FPC will provide weekly written or verbal reports to EPA detailing PRP activities and any observed discrepancies from the approved sampling plans. An overall field summary report will be provided to EPA at the end of each period of field work. Laboratory results from split samples will be evaluated and presented to EPA in the form of a Letter Report, detailing QA/QC measures taken during sampling/field activities.

Any report containing measurements will contain a QA section which includes data quality objectives and parameters, the results of any audits peformed by the FPC TES V QA staff and/or EPA, and any deviations from the EPA approved guidance documents (such as this Brossman Short Form). Such a report will undergo QA review by the RQAC or her designee after it has received technical review.

TABLE 1
Page 1 of 3

PARAMETER TABLE FOR TES V SPLIT SAMPLES
SUBSURFACE AND SURFACE SOIL INVESTIGATION
TUTU WELLFIELD SITE
ST. THOMAS, U.S. VIRGIN ISLANDS

ANALYSIS	EST'D NO OF SAMPLES*	MATRIX	ANALYTICAL METHOD	HOLDING TIME PRESERVATIVE VTSR **	SAMPLE CONTAINER
VOCs	8	soil	Ref. 1	Cool to 4°C 10 days	2x40-ml glass vials/ Teflon septa
MTBE, n-Propyl-benzene + 1,2-Dibro-moethane	6	soil	Ref. 1	Cool to 4°C 10 days	2x40-ml glass vials/ Teflon septa
BNAs	8	soil	Ref. 1	Cool to 4°C 10 days to extraction/ 40 days to analysis	1x8oz wide-mouth glass jar
Metals	5	soil	Ref. 2	Cool to 4°C 6 months Hg-26 days	1x8oz wide-mouth glass jar
Metals + Cyanide	6	soil	Ref. 2	Cool to 4°C 6 months Hg-26 days Cn-12 days	1x8oz wide mouth glass jar
TPH	6	soil	418.1(3)	Cool to 4°C *** 26-days	1x8oz wide-mouth glass jar

* - Based on splitting 20% (6) of the PRP subsurface soil samples and 100% (5) of the PRP surface soil samples. The 2 onsite surface soils will be analyzed for TCL VOCs, TCL BNAs and TAL metals only; the 3 background surface soil samples will be analyzed for TAL metals only.
** - From verified time of sample receipt
*** - The extraction of soil for TPH analysis is to be performed according to EPA/Army Corps of Engineers approved Oil and Grease Procedure for Sediment Samples given in Attachment I. After extraction proceed to Step 7.6 in Method 418.1 for TPH analysis

TABLE 1
Page 2 of 3

PARAMETER TABLE FOR TES V SPLIT SAMPLES
GROUNDWATER INVESTIGATION
TUTU WELLFIELD SITE
ST. THOMAS, U.S. VIRGIN ISLANDS

ANALYSIS	ESTIMATED NO. OF SAMPLES*	MATRIX	ANALYTICAL METHOD	PRESERVATION HOLDING TIMES VTSR **	SAMPLE CONTAINERS
VOCs	8	aqueous	Ref. 1	Cool to 4°C 1:1 HCL to pH<2 (a) 10 days	2x40ml glass vials/ Teflon septa
MTBE, n-Propyl-benzene + 1,2-Dibro-moethane	8	aqueous	Ref. 1	Cool to 4°C 1:1 HCL to pH<2 (a) 10 days	2x40ml glass vials/ Teflon septa
BNAs	8	aqueous	Ref. 1	Cool to 4°C 5 days to extraction/ 40 days to analysis	4x1-liter amber jars
Metals	8	aqueous	Ref. 2	Cool to 4°C HNO3 to pH<2 6 months Hg - 26 days	1x1-liter poly bottle
Cyanide	8	aqueous	Ref. 2	Cool to 4°C NaOH to pH>12 12 days	1x1-liter poly bottle
TPH	8	aqueous	418.1(3)	Cool to 4oC 1:1 H_2SO_4 to pH<2 26 days	1x1-liter amber jar

* - Based on splitting 20% of the PRP samples
** - From verified time of sample receipt

(a) If sample acidification causes effervescence, the sample will not be acidified but will be cooled to approximately 4°C. In this event, the holding time is 7 days from the time of sampling.

TABLE 1
Page 3 of 3

PARAMETER TABLE FOR TES V SPLIT SAMPLES
SOLID WASTE INVESTIGATION
TUTU WELLFIELD SITE
ST. THOMAS, U.S. VIRGIN ISLANDS

ANALYSIS	ESTIMATED NO. OF SAMPLES*	MATRIX	ANALYTICAL METHOD	PRESERVATION HOLDING TIMES VTSR **	SAMPLE CONTAINERS
VOCs	2	solid waste	Ref. 4	Cool to 4°C 10 days	2x4oz wide-mouth glass jars with teflon-lined septa cap

* - Based on splitting 100% of the PRP samples
** - From verified time of sample receipt

References

1. "Statement of Work for Organic Analysis, Multi-Concentration." USEPA Contract Laboratory Program, OLM01.8 (or current revision).

2. "Statement of Work for Inorganic Analysis, Multi-Concentration." USEPA Contract Laboratory Program, ILM01.0 (or current revision).

3. Methods for Chemical Analysis of Water and Wastes: EPA-600/4-79-020, revised March, 1983.

4. "Statement of Work for Organic Analysis, Multi-Media, High-Concentration." Revised 9/88, including Revision 4/89.

TABLE 2

QA/QC SAMPLE SUMMARY TABLE
SOIL AND GROUNDWATER INVESTIGATIONS
TUTU WELLFIELD, ST. THOMAS, U.S. VIRGIN ISLANDS

Matrix	Parameter	Sample Type				
		Investigative	Matrix spike	Field blank	Trip blank	Total
Aqueous	VOCs	8	2**	2	4	16
Aqueous	MTBE, n-Propylbenze, + 1,2-dibromoethane	8	2**	2	4	16
Aqueous	Extractables (BNAs)	8	2**	2	-	12
Aqueous	Inorganics (Metals + Cyanide)	8	2	2	-	12
Aqueous	TPH	8	2+	-	-	10
Soil	VOCs	8	*	3	-	11
Soil	MTBE, n-Propylbenze, + 1,2-dibromoethane	6	*	2	-	8
Soil	BNA Extractables	8	*	3	-	11
Soil	Inorganics (metals)	5	*	1	-	6
Soil	Inorganics (metals + cyanide)	6	*	2	-	8
Soil	TPH	6	*	-	-	6
Solid Waste	VOCs	2	*	-	-	2

NOTES FOR TABLE 2

1) Based on accepting splits of 20% of the PRP's subsurface samples and 100% of the PRP's surface soil samples. Two randomly selected field blanks will be split with the PRP for subsurface soils and one for surface soils.

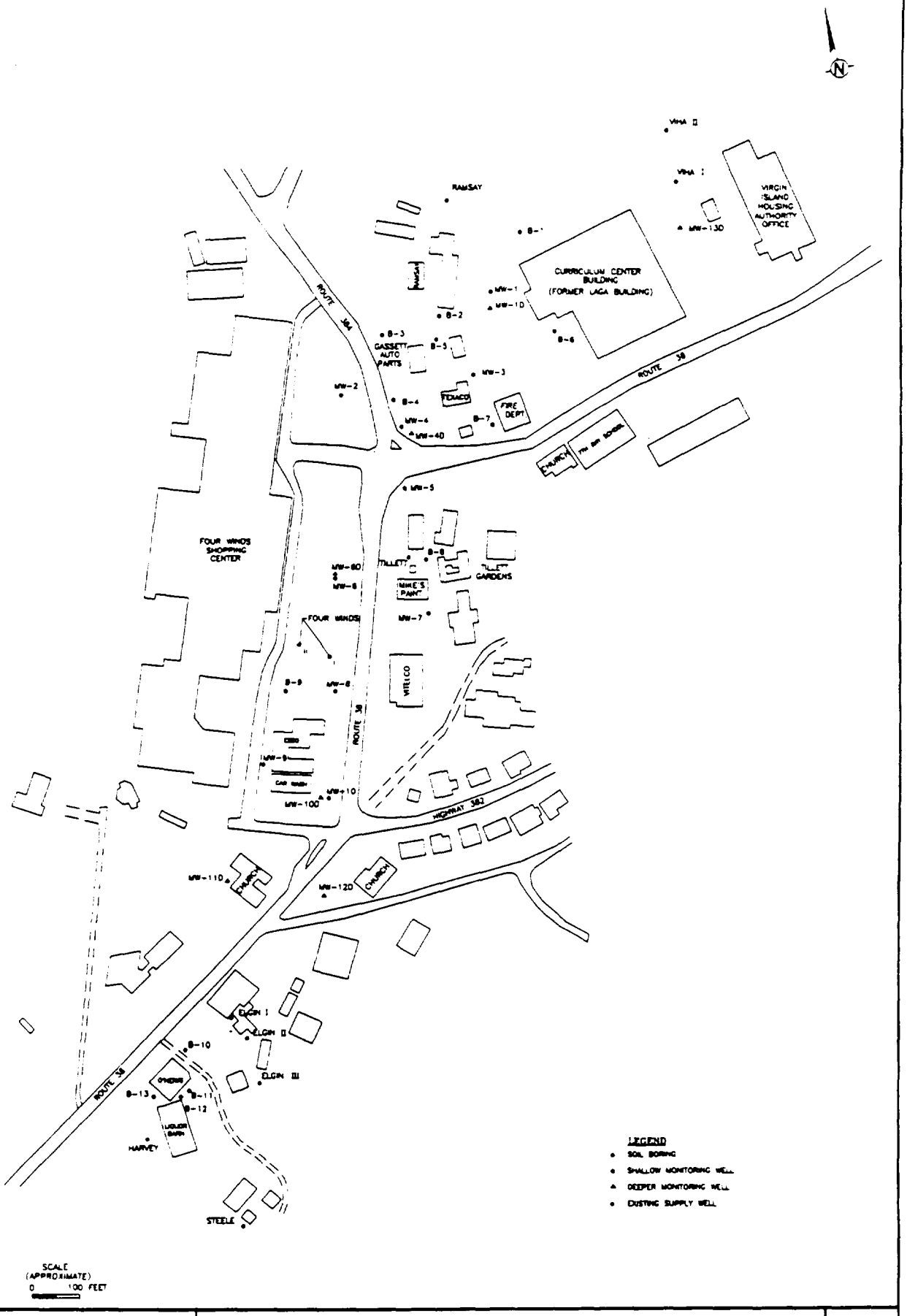
2) Assumes two groundwater sampling events with split samples accepted on 2 days for each event.

* The lab will analyze a matrix spike and matrix spike duplicate from existing soil or sediment sample volume.

** Triple volume must be collected (volume includes investigative sample).

+ Because the entire sample is consumed in analysis, a laboratory duplicate will be collected for every sample delivery group for aqueous TPH analyses and labeled as a lab duplicate.

DWG DATE 08APR92 PROJECT NO PR0130' FILE NO - DRAWING TU-2D CHECKED MOFFATT APPROVED DANAHY DRA LON



GERAGHTY & MILLER, INC.
Environmental Services

SITE PLAN

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

FIGURE 1

TUT 004 0308

EPA

United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Organic Traffic Report
(For CLP Use Only)

Case Number: 10001
SAS No. (If applicable):

1. Type of Activity (Check one)
ENF NPLD RA SI STSI
ER O&M RD ST Other (Specify)
ESI PA X RI/FS STPA

2. Region Number: I
Sampling Co.: XYZ Co.
Sampler (Name): Joe Sampler

4. Date Shipped: 11-4-88
Airbill Number: 1234567890
Carrier: Fed Ex

Non-Superfund Program

Site Name

City, State

Site Spill ID: 03

3. Ship To:
CLP Lab
100 Main St.
Tiny Town, ME 01234
Attn M. Spec

Triple volume required for matrix spike/duplicate aqueous sample.

Ship medium and high concentration samples in paint cans.

See reverse for additional instructions.

5. Sample Description (Enter in Column A)
1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) (Specify)

CLP Sample Number (From labels)	(A) Sample Description (From Box 5)	(B) Concentration L=low M=med H=High	(C) RAS Analysis VOA	BNA	Pest/PCB	(D) Special Handling	(E) Station Location	(F) Date/Time of Sample Collection	(G) Corresponding CLP Inorganic Sample Number
AB123	2	L	X	X	X		MW-1	11-3/0800	MAZ221
AB124	2	L	X	X	X		MW-2	11-3/0830	MAZ22
AB125	2	L	X	X	X		MW-3	11-3/0845	MAZ223
AB126	2	L	X	X	X		MW-4	11-3/0900	MAZ224
AB127	2	L	X	X	X		MW-5	11-3/1000	MAZ225
AB128	2	L	X	X	X		MW-6	11-3/1030	MAZ226
AB129	2	L	X	X	X		MW-7	11-3/1100	MAZ227
AB130	2	L	X	X	X		MW-8	11-3/1130	MAZ228
AB131	2	L	X	X	X		MW-9	11-3/1400	MAZ229
AB132	2	L	X	X	X		MW-10	11-3/1500	MAZ230
AB133	2	L	X	X	X		MW-11	11-3/1530	MAZ231
AB134	2	L	X	X	X		MW-12	11-3/1600	MAZ232
AB135	2	L	X	X	X	MS/MSD	MW-13	11-3/1700	MAZ233
AB136	2	L	X	X	X		MW-14	11-4/0900	MAZ234
AB137	2	L	X	X	X		MW-15	11-4/1000	MAZ235
AB138	2	L	X	X	X		MW-16	11-4/1100	MAZ236
—	SHIPPING					COMPLETE	—		
	No more to ship under this case no.								

EPA Form 9109-2 (9-88) Replaces EPA Form 2075-7, which may be used. Blue - SMO Copy Pink - Region Copy White - Lab Copy for Return to SMO Yellow - Lab Copy

FIGURE 1

TLT 006 0309

United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

EPA

Inorganic Traffic Report
(For CLP Use Only)

Case Number: 10101

SAS No. (If applicable):

1 Type of Activity (Check one)
- [ ] ENF
- [ ] ER
- [ ] ESI
- [ ] NPLD
- [ ] O&M
- [ ] PA
- [ ] RA
- [ ] RD
- [ ] RIFS
- [X] SI
- [ ] ST
- [ ] STPA
- [ ] STSI
- [ ] Other (Specify)

Non-Superfund Program

Site Name

City, State

Site Spill ID: 05

2 Region Number: X

Sampling Co: Acme Co.

Sampler (Name): Joan Sampler

3 Ship To: Analytical Lab, 100 Center Ave, Anytown, CA 94568, Attn A. Metal

4 Date Shipped: 11-4-88

Airbill Number: 0987654321

Carrier: Fed Ex

Double volume required for matrix spike/duplicate aqueous sample

Ship medium and high concentration samples in paint cans

See reverse for additional instructions

5. Sample Description (Enter in Column A)
1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) (Specify)

CLP Sample Number (From labels)	(A) Sample Description (From Box 5)	(B) Concentration L=low M=med H=high	(C) RAS Analysis: Total Metals	(C) RAS Analysis: Cyanide	(D) Special Handling	(E) Station Location	(F) Date/Time of Sample Collection	(G) Corresponding Organic Sample Number
MJZ 900	1	L	X	X		LOC-1	11-4/0700	JA 321
MJZ 901	1	L	X	X		LOC-2	11-4/0730	JA 322
MJZ 902	1	L	X	X		LOC-3	11-4/0800	JA 323
MJZ 903	1	L	X	X		LOC-4	11-4/0830	JA 324
MJZ 904	1	L	X	X		LOC-5	11-4/0900	JA 325
MJZ 905	1	L	X	X		LOC-6	11-4/0930	JA 326
MJZ 906	1	L	X	X		LOC-7	11-4/0945	JA 327
MJZ 907	1	L	X	X		LOC-8	11-4/1000	JA 328
MJZ 908	1	L	X	X		LOC-9	11-4/1030	JA 329
MJZ 909	1	L	X	X		LOC-10	11-4/1100	JA 330
MJZ 910	1	L	X	X		LOC-11	11-4/1130	JA 331
MJZ 911	1	L	X	X		LOC-12	11-4/1200	JA 332
MJZ 912	1	L	X	X		LOC-13	11-4/1215	JA 333
MJZ 913	1	L	X	X		LOC-14	11-4/1245	JA 334
MJZ 914	1	L	X	X		LOC-15	11-4/1300	JA 335
MJZ 915	1	L	X	X		LOC-16	11-4/1330	JA 336
MJZ 916	1	L	X	X		LOC-17	11-4/1400	JA 337
MJZ 917	1	L	X	X		LOC-18	11-4/1430	JA 338
MJZ 918	1	L	X	X		LOC-19	11-4/1500	JA 339
MJZ 920	1	L	X	X	MS/dup	LOC-20	11-4/1530	JA 340

EPA Form 9110-1 (8-88) Replaces EPA Form 2075-6, which may be used. Green - SMO Copy Pink - Region Copy White - Lab Copy for Return to SMO Yellow - Lab Copy

TUT 006 0210

FIGURE 1

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number
1000 - A

SPECIAL ANALYTICAL SERVICE
PACKING LIST

Sampling Office:	Sampling Date(s):	Ship To:	For Lab Use Only
Region I	11/2 - 11/4/88	SAS LAB 100 Main Street Anytown, CO 98765	Date Samples Rec'd:
Sampling Contact:	Date Shipped:		
Joe Sampler (name)	11/4/88		Received By:
617/555-1234 (phone)	Site Name/Code: #01	Attn: Jim Smith	

	Sample Numbers	Sample Description i.e., Analysis, Matrix, Concentration		Sample Condition on Receipt at Lab
1.	1000A-01	Low Conc. Water - 2,4-D; 2,4,5-TP		
2.	1000A-02	"	"	
3.	1000A-03	"	"	
4.	1000A-04	"	"	
5.	1000A-05	"	"	
6.	1000A-06	"	"	
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				

For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

CHAIN OF CUSTODY RECORD

ENVIRONMENTAL PROTECTION AGENCY – REGION II
Environmental Services Division
EDISON, NEW JERSEY 08817

page 1 of 2 chain of custody for cooler #1

Name of User and Address: ICF Technology, Region 2 — RAS # 12013

Sample Number	Number of Containers	Description of Samples	
MBZ 222	2	TCL inorganics (Tot. metals) + CN⁻, water, low conc.	GW-LIN-002
MBZ 223	1	" " Dissolved metals, " " "	GW-LIN-002
MBY 474	2	" " Total metals + CN⁻, " " "	GW-104A-002
MBY 475	1	" " dissolved metals " " "	GW-104A-002
MBY 478	2	" " Total metals + CN⁻, " " "	GW-104B-002
MBY 475	1	" " dissolved metals " " "	GW-104B-002
MBY 208	2	" " Total metals + CN⁻ " " "	GW-307S-002
MBY 209	1	" " dissolved metals " " "	GW-307S-002
MBY 482	2	" " Total metals + CN⁻ " " "	GW-205B-002
MBY 483	1	" " dissolved metals " " "	GW-205B-002
MBZ 206	2	" " TOTAL METALS & CN⁻ " " "	GW-307D 002
MBZ 207	1	" " DISSOLVED METALS " " "	GW-307D 002
MBY 476	2	" " TOTAL METALS & CN⁻ " " "	GW-105 002
MBY 477	1	" " DISSOLVED METALS " " "	GW-105 002
MBZ 226	2	" " TOTAL METALS & CN⁻ " " "	GW-106 302
MBZ 227	1	" " DISSOLVED METALS " " "	GW-106 302

Note: All dissolved metal samples have been filtered

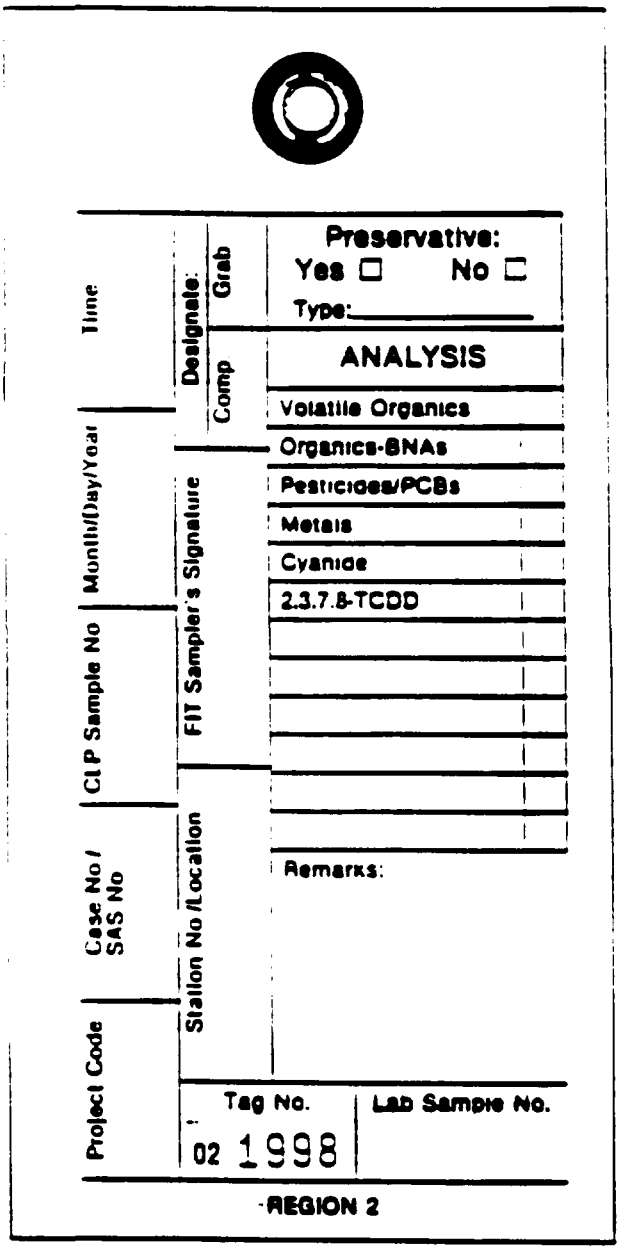
Person Assuming Responsibility for Samples: Robert J. Mehin — Time: 1350 — Date: 5/28/

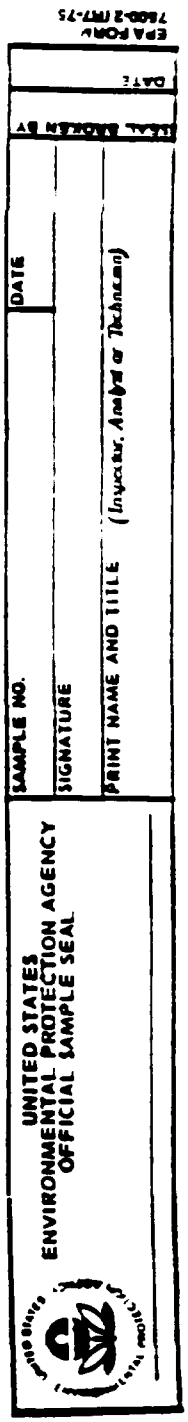
Sample Number	Relinquished By:	Received By:	Time	Date	Reason for Change of Custody
all listed above + page 2	Robert J. Mehin	Federal Express Airbill # 2227200076	1400	5/28/87	Sent to ENSECO RMAL for analysis
Sample Number	Relinquished By:	Received By:	Time	Date	Reason for Change of Custody
Sample Number	Relinquished By:	Received By:	Time	Date	Reason for Change of Custody
Sample Number	Relinquished By:	Received By:	Time	Date	Reason for Change of Custody

FIGURE 5

Page No.

TUT 006 0312





TUT 006 0313

Figure 6

Sample Tag/Custody Seal

Attachment I
EPA/Army Corps of Engineers
Oil and Grease Procedure for Sediment Samples

Procedure for Sediment Samples (SID)

The procedure for determination of oil and grease in sediment samples is similar to that used to quantify oil and grease in water samples. The sample is extracted with Freon and the extractable material is quantified. As indicated in Figure 3-39, a moist sediment sample must be used as a dried sample will yield low results. Because of the operational definition of the oil and grease procedure and the lack of precision associated with the test, it is recommended that conditions of sampling, sample pretreatment, and analysis be standardized to ensure comparability of the final data.[3]

Method 1: Freon Extraction[1]

Apparatus

Extraction apparatus, Soxhlet

Vacuum pump or other source of vacuum

Extraction thimble, paper

Infrared spectrophotometer or balance

Reagents

Either conc. hydrochloric acid, HCl, or conc. sulfuric acid, H_2SO_4.

Magnesium sulfate monohydrate: prepare $MgSO_4 \cdot H_2O$ by drying a thin layer of $MgSO_4 \cdot 7H_2O$ overnight in an oven at 103°C.

Freon (1,1,2-trichloro-1,2,2-trifluoroethane), boiling point 47°C. The solvent should leave no measurable residue on evaporation. Redistill if necessary.

Grease-free cotton: extract nonabsorbent cotton with Freon.

Known oil reference standard: accurately weigh about 0.05 g of known oil directly into a 100-ml volumetric flask. Add 80 ml Freon and dissolve the oil. If, as in the case of a heavy fuel oil, all the oil does not go into solution, let stand overnight. Filter through a Whatman No. 40 filter paper into a second 100-ml volumetric flask and dilute to volume with Freon.

Unknown oil reference standard (10 µl = 7.69 mg oil): pipet 15 ml n-hexadecane, 15 ml isooctane, and 10 ml benzene into a 50-ml glass-stoppered bottle. Assume the specific gravity of this mixture to be 0.769 and maintain the integrity of the mixture by keeping the bottle stoppered except when withdrawing aliquots.

Procedure

Weigh a 20.0-g sample of moist sediment (SID) in a 150-ml

beaker. (The solids content of the sample should be known in advance or determined on a separate sample aliquot.) Acidify the sample with conc. sulfuric or conc. hydrochloric acid to pH 2.

Add 25 g $MgSO_4 \cdot H_2O$ to the acidified sediment sample. Stir to make a uniformly smooth paste that is spread on the beaker wall. Allow to stand 15 to 30 min until solidified. Remove the solids and grind in a porcelain mortar. The use of a desiccated, uniformly ground sample improves the efficiency of the extraction process.

Add the ground sample to a paper extraction thimble. The beaker and mortar should be wiped with a small piece of filter paper that has been soaked in Freon. Add the filter paper to the paper thimble. Fill the thimble with glass wool or small glass beads. Extract the prepared sample with Freon in a Soxhlet apparatus at a rate of 20 cycles/hr for - hr. If the final extract is turbid, filter the sample through grease-free cotton into a clean flask. Rinse the initial sample container and the cotton with Freon and add the washing to the filtered sample. Determine the oil and grease concentration of the extract by either infrared spectrophotometry (a) or gravimetry (b). The infrared method would be preferred because it is generally more precise; particularly at low oil and grease concentrations.

a. Infrared spectrophotometry.[1,2] Quantitatively transfer the sediment extract to a convenient size volumetric flask and dilute to volume with Freon.

   Prepare calibration standards using either the known oil reference standard or the unknown oil reference standard. (A known oil is defined as the only grease and/or oil component in the samples being analyzed. An unknown oil is defined as the grease and/or oil component(s) in the sample being analyzed for which standard preparations are not available.) Transfer required amounts of the appropriate reference material into 100-ml volumetric flasks using microliter pipettes. Dilute to volume with Freon.

   The most appropriate pathlength for the quartz cells to be used in the spectrophotometric determination is determined by the expected sample concentration. The following information is presented as a guide for selecting cell length:

Pathlength, cm	Expected Range, mg
1	4 - 40
5	0.5 - 8
10	0.1 - 4

Based on observed ranges of oil and grease in sediments, it may be necessary to dilute the sample extracts to the working ranges indicated above.

Scan the standards and samples from 3200 to 2700 cm^{-1} using a recording infrared spectrophotometer. Freon should be used in the reference beam of a dual beam instrument or to zero a single beam instrument. The absorbance of the 2930-cm^{-1} peak should be used to construct a standard curve.

b. Gravimetry.[1] The gravimetric determination of oil and grease does not require dilution of the samples. However, the procedure is considered less precise than the infrared determination because the method is subject to positive sulfur interference and greater uncertainty at low oil and grease concentrations.

To implement the method, quantitatively transfer the sediment extract to a tared distilling flask. Rinse the extract container with Freon and add to the distilling flask. Distill the Freon from the extraction flasks using a water bath at 70°C. After the solvent has been evaporated, place the flask on a warm steam bath for 15 min and draw air through the flask by means of an applied vacuum for the final 1 min. Cool in a desiccator for 30 min and weigh. The gain in weight is due to oil and grease if the solvent is free of residue.

Calculations

Select the appropriate method and calculate the oil and grease concentration based on the method of quantification that was used.

a. Infrared spectrophotometry. When colorimetry is used, prepare a standard curve by plotting measured absorbance vs. oil and grease concentration of the standards. Compare the absorbance of the Freon extract to the standard curve to determine the oil and grease concentration.

Calculate the oil and grease concentration O + G of the original water sample as follows:

$$O + G \text{ mg/kg (wet weight)} = \frac{(X)(V)(1000)}{g}$$

$$O + G \text{ mg/kg (dry weight)} = \frac{(X)(V)(1000)}{(g)(\% S)}$$

where

X = concentration of oil and grease in the Freon extract, mg/ℓ

V = volume of Freon extract, ℓ

g = wet weight of sediment extracted, g

% S = percent solids in the sediment sample (expressed as a decimal fraction)

b. Gravimetry. When the amount of oil and grease is determined by weighing the material extracted, the oil and grease concentration of the sediment sample is calculated as follows:

$$O + G \text{ mg/kg (wet weight)} = \frac{(A - B - C)(1000)}{(g)}$$

$$O + G \text{ mg/kg (dry weight)} = \frac{(A - B - C)(1000)}{(g)(\% S)}$$

where

A = weight of tared flask and oil and grease residue, mg

B = weight of tared flask, mg

C = calculated residue based on Freon flask, mg

g = wet weight of sediment extracted, g

% S = percent solids in the sediment sample (expressed as a decimal fraction)

References

1. American Public Health Association. Standard Methods for the Examination of Water and Wastewater Including Bottom Sediments and Sludges. 14th Edition. APHA; New York, New York. 1193 p. (1976).

2. Environmental Protection Agency. "Manual of Methods for Chemical Analysis of Water and Wastes." Methods Development and Quality Assurance Research Laboratory, National Environmental Research Center; Cincinnati, Ohio. 298 p. (1974).

3. Disalvo, L., Guard, H., Hirsch, N., and Ng, J. "Assessment and Significance of Sediment-Associated Oil and Grease in Aquatic Environments." Naval Biosciences Laboratory, Naval Supply Center; Oakland, California. Technical Report D-77-26; U. S. Army Engineer Waterways Experiment Station, CE; Vicksburg, Mississippi. 145 p. (1977).

PETROLEUM HYDROCARBONS, TOTAL RECOVERABLE

Method 418.1 (Spectrophotometric, Infrared)

STORET NO. 45501

1. Scope and Application
   - 1.1 This method is for the measurement of fluorocarbon-113 extractable petroleum hydrocarbons from surface and saline waters, industrial and domestic wastes.
   - 1.2 The method is applicable to measurement of light fuels, although loss of about half of any gasoline present during the extraction manipulations can be expected.
   - 1.3 The method is sensitive to levels of 1 mg/l and less, and may be extended to ambient monitoring.
2. Summary of Method
   - 2.1 The sample is acidified to a low pH (< 2) and serially extracted with fluorocarbon-113 in a separatory funnel. Interferences are removed with silica gel adsorbant. Infrared analysis of the extract is performed by direct comparison with standards.
3. Definitions
   - 3.1 As in the case of Oil and Grease, the parameter of Petroleum Hydrocarbons is defined by the method. The measurement may be subject to interferences and the results should be evaluated accordingly.
   - 3.2 Oil and Grease is a measure of biodegradable animal greases and vegetable oils along with the relative non-biodegradable mineral oils. Petroleum hydrocarbons is the measure of only the mineral oils. Maximum information may be obtained using both methods to measure and characterize oil and grease of all sources.
4. Sampling and Storage
   - 4.1 A representative sample of 1 liter volume should be collected in a glass bottle. Because losses of grease will occur on sampling equipment, the collection of a composite sample is impractical. The entire sample is consumed by this test; no other analyses may be performed using aliquots of the sample.
   - 4.2 A delay between sampling and analysis of greater than 4 hours requires sample preservation by the addition of 5 ml HCl (6.1). A delay of greater than 48 hours also requires refrigeration for sample preservation.
5. Apparatus
   - 5.1 Separatory funnel, 2000 ml, with Teflon stopcock.
   - 5.2 Filter paper, Whatman No. 40, 11 cm.
   - 5.3 Infrared spectrophotometer, scanning or fixed wavelength, for measurement around 2950 cm^{-1}.
   - 5.4 Cells, 10 mm, 50 mm, and 100 mm pathlength, sodium chloride or infrared grade glass.
   - 5.5 Magnetic stirrer, with Teflon coated stirring bars.
6. Reagents
   - 6.1 Hydrochloric acid, 1:1. Mix equal volumes of conc HCl and distilled water.

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6.2 Fluorocarbon-113,(1,1,2-trichloro-1,2,2-trifluroethane), b.p. 48°C.

6.3 Sodium sulfate, anhydrous crystal.

6.4 Silica gel, 60–200 mesh, Davidson Grade 950 or equivalent. Should contain 1–2% water as defined by residue test at 130°C. Adjust by overnight equilibration if needed.

6.5 Calibration mixtures:

6.5.1 Reference oil: Pipet 15.0 ml n-hexadecane, 15.0 ml isooctane, and 10.0 ml chlorobenzene into a 50 ml glass stoppered bottle. Maintain the integrity of the mixture by keeping stoppered except when withdrawing aliquots.

6.5.2 Stock standard: Pipet 1.0 ml reference oil (6.5.1) into a tared 200 ml volumetric flask and immediately stopper. Weigh and dilute to volume with fluorocarbon-113.

6.5.3 Working standards: Pipet appropriate volumes of stock standard (6.5.2) into 100 ml volumetric flasks according to the cell pathlength to be used. Dilute to volume with fluorocarbon-113. Calculate concentration of standards from the stock standard.

7. Procedure

7.1 Mark the sample bottle at the water meniscus for later determination of sample volume. If the sample was not acidified at time of collection, add 5 ml hydrochloric acid (6.1) to the sample bottle. After mixing the sample, check the pH by touching pH-sensitive paper to the cap to insure that the pH is 2 or lower. Add more acid if necessary.

7.2 Pour the sample into a separatory funnel.

7.3 Add 30 ml fluorocarbon-113 (6.2) to the sample bottle and rotate the bottle to rinse the sides. Transfer the solvent into the separatory funnel. Extract by shaking vigorously for 2 minutes. Allow the layers to separate.

7.4 Filter the solvent layer through a funnel containing solvent-moistened filter paper into a 100 ml volumetric flask.

NOTE 1: An emulsion that fails to dissipate can be broken by pouring about 1 g sodium sulfate (6.3) into the filter paper cone and slowly draining the emulsion through the salt. Additional 1 g portions can be added to the cone as required.

7.5 Repeat (7.3 and 7.4) twice more with 30 ml portions of fresh solvent, combining all solvent into the volumetric flask.

7.6 Rinse the tip of the separatory funnel, filter paper, and the funnel with a total of 5–10 ml solvent and collect the rinsings in the flask. Dilute the extract to 100 ml. If the extract is known to contain greater than 100 mg of non-hydrocarbon organic material, pipet an appropriate portion of the sample to a 100 ml volumetric and dilute to volume.

7.7 Discard about 5–10 ml solution from the volumetric flask. Add 3 g silica gel (6.4) and a stirring bar; stopper the volumetric flask, and stir the solution for a minimum of 5 min on a magnetic stirrer.

7.8 Select appropriate working standards and cell pathlength according to the following table of approximate working ranges:

Pathlength	Range
10 mm	2–40 mg
50 mm	0.4–8 mg
100 mm	0.1–4 mg

Calibrate the instrument for the appropriate cells using a series of working standards (6.5.3). It is not necessary to add silica gel to the standards. Determine absorbance directly for each solution at the absorbance maximum at about 2930 cm^{-1}. Prepare a calibration plot of absorbance vs. mg petroleum hydrocarbons per 100 ml solution.

7.9 After the silica gel has settled in the sample extract, fill a clean cell with solution and determine the absorbance of the extract. If the absorbance exceeds 0.8 prepare an appropriate dilution.

NOTE 2: The possibility that the absorptive capacity of the silica gel has been exceeded can be tested at this point by adding another 3.0 g silica gel to the extract and repeating the treatment and determination.

7.10 Determine the concentration of petroleum hydrocarbons in the extract by comparing the response against the calibration plot.

8. Calculations

8.1 Calculate the petroleum hydrocarbons in the sample using the formula:

$$\text{mg/l Petroleum Hydrocarbons} = \frac{R \times D}{V}$$

where:

R = mg of Petroleum Hydrocarbons as determined from the calibration plot (7.10).
D = extract dilution factor, if made.
V = volume of sample, in litres.

9. Precision and Accuracy

9.1 Precision and accuracy data are not available at this time.