

**SECOND QUARTER SAMPLING REPORT
FEBRUARY 1991
TUTU WELLS SITE QUARTERLY SAMPLING
ST. THOMAS, U.S. VIRGIN ISLANDS**

May 13, 1991

Prepared for
Tutu Environmental Investigation Committee

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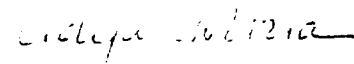
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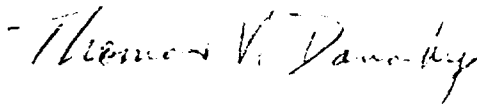
Geraghty & Miller, Inc. is pleased to have the opportunity to work for the Tutu Environmental Investigation Committee at the Tutu Wells site. If you have any questions or comments concerning this report, please contact one of the individuals listed below.

Respectfully submitted,

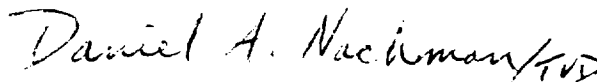
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FIGURE

1. Well Sampling Locations, Tutu Wells Site Sampling, Analysis, and Monitoring Plan, St. Thomas, USVI.

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- A. Telephone Conversation Records.
- B. Data Validation Summary Report for the Tutu Wells Site, February 1991 Quarterly Sampling, St. Thomas, USVI.

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INTRODUCTION

Geraghty & Miller, Inc. has prepared the Tutu Wells Site Second Quarter (February 1991) Sampling Report for both L'Henri, Inc. (O'Henry) and the Tutu Environmental Investigation Committee (TEIC), which is comprised of Texaco Caribbean Inc. (Texaco) and Esso U.S. Virgin Islands (Esso). This report was prepared in accordance with the Administrative Order (AO), effective March 22, 1990, and with the requirements detailed in the "Sampling, Analysis, and Monitoring Plan (SAMP) for Wells, Tutu Wells Site, St. Thomas, U.S. Virgin Islands," September 1990 (Geraghty & Miller 1990), which was approved by the U.S. Environmental Protection Agency (USEPA) in its September 21, 1990 letter to TEIC. After submission of the First Quarter Sampling Report, modifications to the analytical procedures were agreed upon by representatives of the USEPA, Geraghty & Miller, and Ceimic Corporation (the laboratory analyzing the samples) during telephone calls on January 22, January 24, and February 1, 1991. Telephone conversation logs regarding the modified analytical procedures are presented in Appendix A.

The objectives of the SAMP are to identify, quantify, and monitor the occurrence of gasoline constituents and tetrachloroethene (commonly referred to as perchloroethylene and abbreviated as PCE) and its breakdown products in particular wells in the vicinity of Route 38 within the Tutu Wells site (see Figure 1).

SAMPLING AND ANALYTICAL PROCEDURES

The second quarter sampling was conducted from February 4 through 7, 1991 by Soil Tech and Geraghty & Miller. Table 1 lists the wells and analytical parameters for the February 1991 sampling. Ms. Ana Gloria Ramos, Designated Coordinator for TEIC, was present throughout the sampling event. A representative of the Virgin Islands Department

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of Planning and Natural Resources (DPNR) was present throughout the sampling event. A representative of the USEPA Caribbean field office was present in an audit capacity on February 5, 1991.

Sampling and analysis were performed as described in the SAMP, with the exception of the few deviations outlined in the following section. In brief, the wells were sampled and analyzed for the Target Compound List (TCL) volatile organic compounds (VOCs), and specific metal parameters, under the Contract Laboratory Program (CLP). VOC analyses were performed in accordance with a modified version of USEPA Method 524.2 in order to achieve lower detection levels. Based upon data quality limitations identified during the first quarter (September 1991) data validation, metal parameters were selected for specific wells for resampling during the second quarter (February 1991) sampling event. Ceimic Corporation of Narragansett, Rhode Island, provided analytical services. The analytical modifications are described in Appendix A of the SAMP. Additionally, minor adjustments to the analytical procedures, which the USEPA approved by telephone (see Appendix A), included the following:

- o The laboratory can use USEPA Method 524.2 surrogate spike and internal standard compounds when they use this method.
- o The laboratory should analyze undiluted VOC samples first by using USEPA Method 524.2. If results are above the calibration range, samples should be analyzed using CLP methods.
- o The laboratory can use the method of standard additions (MSA) on thallium and selenium analyses before doing standard atomic adsorption/furnace analysis.

In 21 of the 22 wells sampled for the February 1991 sampling event, the samples were collected utilizing the permanent pumps and piping systems. These wells were evacuated prior to sampling by pumping the wells for a minimum of 15 minutes. One well (Gassett), which is not outfitted with a permanent pump, was evacuated using a submersible

pump powered by a portable generator. The sample collected from the Gassett well was obtained after evacuation using a Teflon bailer.

Geraghty & Miller validated the analytical data in accordance with the USEPA Standard Operating Procedures (SOP) No. HW-2, February 1990 for inorganic compounds and the USEPA SOP No. HW-6, March 1990 for organic compounds. The data validation summary is provided in Appendix B. The analytical results for the February 1991 sampling are summarized in Tables 2 and 3.

Details of the sampling and analysis procedures, data validation requirements, and reporting requirements are provided in the SAMP.

PROCEDURAL DEVIATIONS

Deviations from the scope of work and procedures as presented in the SAMP are outlined below.

1. Four Winds I was not sampled during the September 1990 and February 1991 sampling events due to an inoperable pump. VIHA I was not sampled during the February 1991 sampling event due to an inoperable pump. The well owners are not willing to repair the pumps until the wells can be used as a source of water.
2. As noted in the First Quarterly Sampling Report (Geraghty & Miller 1991), the integrity of the well sampled on the Gassett property (formerly Hartman 1) during the September 1990 sampling event was poor. An oily rag was previously reported to have been present in this well, which is located on the north side of the Gassett property. During the second quarterly sampling event (February 1991), a newer well on the western portion of the Gassett property was sampled. Due to the poor integrity, sampling conditions,

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and change in sampling locations, the September 1990 results for the Gassett well are not suitable for comparison with the February 1991 results.

3. Selected metal parameters at specific water supply wells, which were rejected during data validation of the September 1990 results, were collected and analyzed in order to supplement the first quarter sampling data. The results of this supplemental inorganic sampling are presented in Table 2.
4. During the February 1991 sampling, the Tillett well was pumped for a minimum of 15 minutes prior to sampling, as specified in the SAMP. However, due to logistical problems associated with containerizing the evacuated water, the water was pumped at a low rate and only approximately 150 gallons were removed prior to sampling. Data provided by the U.S. Geological Survey (USGS) indicate that the Tillett wells are 100 feet deep and 6 inches in diameter. Assuming a depth to water of 20 feet, three well volumes can be calculated as being equal to approximately 360 gallons.

RESULTS AND DISCUSSION

A summary of the inorganic analytical results is provided in Table 2. The volatile organic analytical results are summarized in Table 3. Table 4 presents a comparison of volatile organic results for the first two quarterly sampling events.

DATA QUALITY EXCEPTIONS

A discussion of the data validation findings is provided in Appendix B. In general, the overall quality of the data set is acceptable and meets the project data quality objectives. For some data, however, some exceptions should be noted. They are described below.

Several volatile organic samples (LaPlace, Devcon I, Devcon III [quality control (QC) samples only], Dench) received and analyzed by the laboratory contained air bubbles. The data validation SOP requires that in this instance all positive detections be assumed to be minimum values and that all non-detections be rejected based on the assumption that all VOCs in the water would diffuse into the air bubble, and consequently, escape analysis and detection. However, this procedure was only applied to the Devcon I sample. The reason for this is that the results for LaPlace and Dench were confirmed by the respective field replicates (Lavergne and Fernandez samples). Also, air bubbles were only in the Devcon III QC sample vials, only the matrix bias recovery data for the Devcon III sample would be impacted, not the actual field sample results. In reality, it is unlikely that any VOC present would stabilize in both the air and water at concentrations related to the compound vapor pressure. Additionally, the non-detection results for the Devcon I February 1991 sample, which were rejected, do correlate well with the non-detection results obtained in the September 1990 sampling event.

Holding time violations resulted in the estimation of non-detected aromatic organic compounds and the positive detecting for total 1,2-dichloroethene (1,2-DCE) and PCE in the Four Winds II sample. Additionally, the total 1,2-DCE and PCE concentrations were estimated in the Eglin II sample as the holding time for the reanalysis for these two compounds was exceeded significantly; but results correlated well to the concentrations detected in the initial analysis.

Several compound omissions in the April 1990 method detection limit (MDL) study performed in support of the USEPA Method 524.2 drinking water analyses resulted in estimation of some results. Acetone, carbon disulfide, 2-butanone, vinyl acetate, 4-methyl-2-pentanone, and 2-hexanone were estimated for all samples based on the omission from the MDL study.

Unsatisfactory instrument calibrations resulted in the rejection of some low-level analytical results. For some samples, non-detection in the Method 524.2 analyses of acetone, 2-butanone, vinyl acetate, and 2-hexanone was rejected due to poor instrument response factors and continuing calibration performance. Poor sensitivity for acetone and similar compounds is not unusual at low detection levels. Moreover, these compounds are not constituents of concern at the site.

Poor recovery values outside of the quality control limits in the laboratory fortified blanks (LFB) contributed to the rejection of some data. For some samples, bromomethane; 2-butanone; chloroethane; 1,1,2,2-tetrachloroethane; trans-1,3-dichloropropene; vinyl acetate; and vinyl chloride were rejected due to low recoveries in the respective LFB. Poor recoveries and sensitivity are not uncommon for many early eluting gases and late eluting higher molecular weight compounds, and these analytes are often measured with less accuracy and precision than other analytes. These compounds also are not constituents of concern to the site.

Many constituent concentrations are estimated due to quality control inadequacies related to holding times, method performance, calibration performance, spike recoveries, and quality control studies. Although many of the constituent concentrations are estimated, their identification and quantification were subject to strict quality control requirements, and therefore the reported values are likely to be within a reasonable range of the true values. The overall quality of the data is acceptable, and there is good reproducibility between replicate samples and the chief constituents of concern which were consistently detected.

INORGANIC ANALYTICAL RESULTS

The inorganic analytical results are provided in Table 2. Only two samples had metal parameters reported above the detection limit. The Hartman II sample had an estimated value of 30 parts per billion (ppb) of antimony. This antimony value was detected above the instrument detection limit (IDL) of 27 ppb but below the contract required detection

limit (CRDL) of 60 ppb. Therefore, this antimony value was flagged with a "B." The "J" flag was also applied to this reported value because interelement interference could not be evaluated due to the limited inorganic parameter list. The Smith sample showed an estimated selenium value of 11.7 ppb. This estimated selenium value barely exceeds the federal maximum contaminant level (MCL) of 10 ppb for public water systems.

ORGANIC ANALYTICAL RESULTS

VOCs constitute the chief analyte group of concern at the Tutu Wells site. The compounds which were consistently detected in site samples are PCE, TCE, and 1,2-DCE. All three compounds were detected in samples from the following wells: Eglin I, Eglin II, Eglin III, Four Winds II, Harvey, LaPlace, Matthias, Smith, Steele, and Tillett. One or two of these compounds were detected in samples from the Gassett, Hartman II, and Ramsey wells.

Other VOC compounds detected in samples from the site included chloromethane in the Dede sample (estimated 0.9 micrograms per liter [ug/L], vinyl chloride in the Tillett sample (7 ug/L), 1,2-dichloroethane in the Dede sample (estimated 0.6 ug/L), 1,1,2-trichloroethane in the Smith sample (5 ug/L), benzene in the Tillett sample (27 ug/L), toluene in the Gassett (44 ug/L) and Tillett samples (1 ug/L), and ethylbenzene in the Tillett sample (1 ug/L).

VOC tentatively identified compounds (TICs) were identified in 19 of the 26 site samples. Seven compounds were tentatively identified and additional unknowns were detected. Total estimated VOC TIC concentrations ranged from 1 to 299 ug/L. The presence of methylene chloride and acetone in site and blank samples, prior to rejection for calibration response factors, is attributed to laboratory contamination.

In general, the wells in which VOCs were detected are located in the central Tutu valley. With the exception of estimated trace VOCs in the Dede sample, no VOCs were detected in wells outside the central valley.

RECOMMENDATIONS

The second quarter results meet the prescribed objectives of the SAMP. Geraghty & Miller recommends that the SAMP continue to be implemented, but with a few minor changes. First, in order to minimize sample identification misspellings and variations, the sample designations listed in the legend of Figure 1 should be used for consistency. Second, Geraghty & Miller has requested that the Ceimic Corporation perform an MDL study for USEPA Method 524.2. If the results of the MDL study are beyond control limits (particularly for parameters of concern) then another laboratory with an acceptable MDL study should be selected and proposed to the USEPA before the third quarter sampling is performed. Third, if VIHA I is still inoperable, the alternative well VIHA II should be inspected and if possible, sampled for VOCs.

The proposed list of wells for the third sampling event, which is scheduled to begin on June 4, 1991, is based upon a review of the results of the first two quarterly sampling events (see Table 4). As stated in the SAMP, recommendations for removal of wells from the sampling list will be presented for USEPA approval after results have been non-detectable for constituents of concern in two successive quarterly sampling events. Table 5 presents the proposed sampling effort for the third quarterly sampling event. Based upon the results of two sampling events (September 1990 and February 1991), nine water supply wells are recommended for removal from the sampling list. The nine wells recommended for removal include Bryan, Dede, Demitri, Dench, Devcon I, Devcon III, Leonard, Rodriguez, and VIHA-III. With the exception of Dede, Dench, Devcon I, and VIHA-III, ground-water samples from these wells showed no detectable VOCs during the September 1990 (first quarter) and February 1991 (second quarter) sampling events.

The Dede sample had estimated trace levels of chloromethane (0.9 ppb) and 1, 2-dichloroethane (0.6 ppb) below the method detection limits of 1 ppb in February 1991 and no detectable VOCs in September 1990 (see Table 4). The Dench samples had an estimated chloromethane value of 1 ppb in September 1990 and no detectable VOCs in February 1991. In addition, a field replicate of Dench, labeled as Fernandez, showed no detectable VOCs in February 1991. The Devcon I samples showed non-detectable VOCs in September 1990 and February 1991, following strict data validation procedures; the February 1991 results were rejected due to the presence of air bubbles in the sample. The VIHA-III samples showed an estimated chloroform value of 3 ppb in September 1990 and no detectable VOCs in February 1991.

Due to the very low reported values (below or near the method detection limit) and because 1,2-dichloroethane, chloromethane, and chloroform are not parameters of concern at the Tutu Wells Site, the Dede, Dench and VIHA-III wells are included in the list of wells recommended for no further sampling.

There are also valid geographical and hydrogeologic reasons for removing these nine wells from further sampling efforts. They are all located outside of the central portion of the Tutu valley where VOC contamination has been documented. The Bryan well is located in the Donne valley, a completely separate drainage basin from the Tutu valley. The Demitri and Leonard wells are located upgradient and to the west of the central portion of the Tutu valley; VIHA III is similarly upgradient of this area of concern. The Rodriguez well is situated near the axes of a tributary drainage basin to the Tutu valley and hence receives significant contribution from this tributary valley. Dede, Dench, Devcon I, and Devcon II are all located at least 0.5 mile to the southeast of the area of concern, in a portion of the Turpentine River basin that receives significantly more contribution from other areas than the central Tutu valley. The absence of the VOCs of concern in these nine wells confirms the geographical and hydrogeologic reasons for removing them from future sampling.

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REFERENCES

Geraghty & Miller, Inc. 1990, Sampling, Analysis, and monitoring Plan for Wells, Tutu Wells Site, St. Thomas, U.S. Virgin Islands. Prepared for Tutu Environmental Investigation Committee, September 1990.

Geraghty & Miller, Inc. 1991, First Sampling Report, September 1990, Tutu Wells Site Quarterly Sampling, St. Thomas, U.S. Virgin Islands. Prepared for Tutu Environmental Investigation Committee, January 1991.

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TABLES

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Table 1. List of Wells and Analytical Parameters for the Second Quarterly Sampling Event February 1991, Tutu Wells Site, St. Thomas, USVI.

Well	Predetermined Analytes
Bryan	VOCs, thallium, mercury
Dede	VOCs, antimony, thallium
Dimitri	VOCs, thallium, mercury
Dench	VOCs, antimony, thallium
Devcon I	VOCs, antimony, thallium
Devcon III	VOCs, antimony, thallium
Eglin I	VOCs, thallium, mercury
Eglin II	VOCs, antimony, thallium
Eglin III	VOCs, antimony, thallium
Four Winds I ^a	VOCs
Four Winds II	VOCs, antimony, thallium
Gassett	VOCs, thallium, mercury
Hartman II	VOCs, antimony, thallium
Hartman III	VOCs, antimony, thallium
Harvey	VOCs, antimony, thallium
LaPlace	VOCs, thallium, mercury
Leonard	VOCs, antimony, thallium
Matthias	VOCs, thallium, mercury
Ramsey	VOCs, thallium, mercury
Rodriguez	VOCs, antimony, thallium
Smith	VOCs, antimony, thallium, selenium
Steele	VOCs, antimony, thallium
Tillett	VOCs, thallium, mercury
VIHA I ^b	VOCs, thallium, mercury
VIHA III	VOCs, antimony, thallium

a Four Winds I not sampled due to inoperable pump.

b VIHA I not sampled due to inoperable pump.

Table 2. Results of Quarterly Analysis for Metals in Ground-Water Samples Collected in February 1991 at the Tutu Wells Site, St. Thomas, U.S. Virgin Islands.

	Sample ID Bryan	Dede	Dench	Fernandez	Devcon I	Devcon III	Demitri	Eglin I	Eglin II	Eglin III	Eglin IV	Four Winds II	Gassett	Hartman II
Analyte	Date	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91
Antimony	NA	27 U	27 U	27 U	27 U	27 U	NA	NA	27 U	27 U	27 U	27 U	NA	30 BJ
Mercury	.2 U	NA	NA	NA	NA	NA	.2 U	.2 UJ	NA	NA	NA	NA	.2 U	NA
Selenium	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Thallium	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 UJ	1 UJ	1 UJ

Analyses were performed by Ceimic Corporation, Narragansett, Rhode Island.
 Samples were analyzed by various USEPA methods.

ug/L Analyte concentrations in micrograms per liter [parts per billion (ppb)].

B Compound is also detected in the blank.

J Result is detected below the reporting limit or is an estimated concentration.

U Compound analyzed for but not detected.

NA Not analyzed.

* Fernandez is a field replicate of Dench.

* Eglin IV is a field replicate of Eglin III.

* Lavergne is a field replicate of LaPlace.

Table 2. Results of Quarterly Analysis for Metals in Ground-Water Samples Collected in February 1991 at the Tutu Wells Site, St. Thomas, U.S. Virgin Islands.

Analyte	Sample ID	Hartman III	Harvey	LaPlace	Lavergne	Leonard	Matthias	Ramsey	Rodrigues	Smith	Steele	Tillet	VIHA III	Field Blank
	Date	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91
Antimony		27 U	27 U	NA	NA	27 U	NA	NA	27 U	27 U	27 U	NA	27 U	NA
Mercury		NA	NA	.2 BJ	.24 BJ	NA	.2 UJ	.2 U	NA	NA	NA	.2 U	NA	.2 U
Selenium		NA	NA	NA	NA	NA	NA	NA	NA	11.7 J	NA	NA	NA	NA
Thallium		1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U

Analyses were performed by Calmic Corporation, Narragansett, Rhode Island.
Samples were analyzed by various USEPA methods.

ug/L Analyte concentrations in micrograms per liter [parts per billion (ppb)].

- B Compound is also detected in the blank.
- J Result is detected below the reporting limit or is an estimated concentration.
- U Compound analyzed for but not detected.
- NA Not analyzed.
- * Fernandez is a field replicate of Dench.
- * Eglin IV is a field replicate of Eglin III.
- * Lavergne is a field replicate of LaPlace.

Table 3. Results of Quarterly Analysis for Volatile Organic Compounds in Ground-Water Samples Collected in February 1991 at the Tutu Walls Site, St. Thomas, U.S. Virgin Islands.

Analyte	Sample ID Bryan	Dede	Demitri	Dench	Fernandez	Devcon I	Devcon III	Eglin I	Eglin II	Eglin III	Eglin IV	Four Winds II	Gassett	Hartman II
Date	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91
Chloromethane	2 UJ	.9 J	2 UJ	2 UJ	2 UJ	RR	2 UJ	2 UJ	2 UJ	2 UJ	2 U	2 U	2 U	2 U
Bromomethane	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	2 UJ	2 UJ	2 U	2 U
Vinyl chloride	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	2 UJ	2 UJ	2 U	2 U
Chloroethane	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	2 UJ	2 UJ	RR	2 U
Methylene chloride	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
Acetone	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR
Carbon disulfide	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ
1,1-Dichloroethane	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
1,1-Dichloroethane	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
1,2-Dichloroethane (total)	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	60 J	72 DJ	45 J	45 J	230 D	1 U	5 J
Chloroform	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
1,2-Dichloroethane	1 U	.6 J	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
2-Butanone	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR
1,1,1-Trichloroethane	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
Carbon tetrachloride	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
Vinyl acetate	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	RR	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	RR	2 UJ
Bromodichloromethane	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
1,2-Dichloropropane	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
cis-1,3-Dichloropropene	RR	1 U	RR	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	RR	RR
Trichloroethene	1 U	1 U	1 U	1 U	1 U	RR	1 U	8	19	11	13	21	1	2
Dibromochloromethane	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
1,1,2-Trichloroethane	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
Benzene	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 UJ	1 U	1 U
trans-1,3-Dichloropropene	1 U	RR	1 U	RR	RR	RR	RR	RR	RR	RR	RR	RR	1 U	1 U
Bromoform	1 U	RR	1 U	RR	RR	RR	RR	RR	RR	RR	1 U	1 U	1 U	1 U
4-Methyl-2-pentanone	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	RR	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ
2-Hexanone	RR	2 UJ	RR	2 UJ	2 UJ	RR	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ
Tetrachloroethane	1 U	1 U	1 U	1 U	1 U	RR	1 U	26	34 DJ	35	36	90 D	1 U	9
1,1,2,2-Tetrachloroethane	1 U	RR	1 U	RR	RR	RR	RR	RR	RR	RR	1 U	1 U	1 U	1 U
Toluene	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 UJ	44 D	1 U
Chlorobenzene	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 UJ	1 U	1 U
Ethylbenzene	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 UJ	1 U	1 U
Styrene	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U
Xylenes (total)	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U
TOTAL VOCs:	0.0	1.5	0.0	0.0	0.0	0.0	0.0	95	125	91	94	341	45	16

Analyses were performed by Celmic Corporation, Narragansett, Rhode Island.
Samples were analyzed by USEPA Method 524.2.

ug/L Analyte concentrations in micrograms per liter [parts per billion (ppb)].

- D Compound identified at a secondary dilution.
- J Result is detected below the reporting limit or is an estimated concentration.
- U Compound analyzed for but not detected.
- RR Result rejected.
- * Fernandez is a field replicate of Dench.
- * Eglin IV is a field replicate of Eglin III.
- * Laverne is a field replicate of LaPlace.

Table 3. Results of Quarterly Analysis for Volatile Organic Compounds in Ground-Water Samples Collected in February 1991 at the Tutu Wells Site, St. Thomas, U.S. Virgin Islands.

Analyte	Sample ID Date	Hartman III 5-Feb-91	Harvey 5-Feb-91	LaPlace 5-Feb-91	Lavergne 5-Feb-91	Leonard 5-Feb-91	Matthias 5-Feb-91	Ramsey 5-Feb-91	Rodriguez 5-Feb-91	Smith 5-Feb-91	Steele 5-Feb-91	Tillat 5-Feb-91	VIHA III 5-Feb-91	Field Blank 5-Feb-91	Trip Blank 1 4-Feb-91
Chloromethane		2 U	200 U	2 U	2 U	2 U	25 U	2 UJ	2 UJ	2 UJ	10 U	2 U	2 UJ	2 U	2 UJ
Bromomethane		2 UJ	200 U	2 U	2 U	2 U	25 U	RR	2 U	RR	10 U	2 U	RR	2 U	RR
Vinyl chloride		2 UJ	200 U	2 U	2 U	2 U	25 U	RR	2 U	RR	10 U	7	RR	2 U	RR
Chloroethane		2 UJ	200 U	RR	RR	RR	25 U	RR	2 U	RR	10 U	2 U	RR	RR	RR
Methylene chloride		1 U	100 U	1 U	1 U	1 U	25 U	1 U	1 U	1 U	5 U	1 U	1 U	2	2
Acetone		RR	200 U	RR	RR	RR	25 U	RR	RR	RR	10 U	RR	RR	RR	RR
Carbon disulfide		1 UJ	100 U	1 UJ	1 UJ	1 UJ	12 UJ	1 UJ	1 UJ	1 UJ	5 U	1 UJ	1 UJ	1 UJ	1 UJ
1,1-Dichloroethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 U
1,1-Dichloroethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 U
1,2-Dichloroethane (total)		1 U	160	200 D	190 D	1 U	21	1 J	1 U	42 J	44	200 D	1 U	1 U	1 UJ
Chloroform		1 U	100 U	1	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 U
1,2-Dichloroethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 UJ
2-Butanone		RR	200 U	RR	RR	RR	25 U	RR	RR	RR	10 U	RR	RR	RR	RR
1,1,1-Trichloroethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 UJ
Carbon tetrachloride		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 U
Vinyl acetate		2 UJ	200 U	RR	RR	RR	25 U	2 U	RR	2 UJ	10 UJ	2 UJ	2 UJ	RR	2 UJ
Bromodichloromethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 UJ
1,2-Dichloropropane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 U
cis-1,3-Dichloropropene		1 U	100 U	RR	RR	RR	12 U	RR	RR	1 U	5 U	RR	RR	RR	1 U
Trichloroethane		1 U	60 J	32	31	1 U	7 J	1	1 U	23	12	36	1 U	1 U	1 U
Dibromochloromethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 UJ
1,1,2-Trichloroethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	5	5 U	1 U	1 U	1 U	1 U
Benzene		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	27	1 U	1 U	1 UJ
trans-1,3-Dichloropropene		RR	100 U	1 U	1 U	1 U	12 U	1 U	1 UJ	RR	5 U	1 U	1 U	1 U	RR
Bromoform		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	RR
4-Methyl-2-pentanone		2 UJ	200 U	2 UJ	2 UJ	2 UJ	25 U	2 UJ	2 UJ	2 UJ	10 U	2 UJ	2 UJ	2 UJ	2 UJ
2-Hexanone		2 UJ	200 U	2 UJ	2 UJ	2 UJ	25 U	RR	2 UJ	2 UJ	10 U	2 UJ	RR	2 UJ	2 UJ
Tetrachloroethene		1	1500	110 D	100 D	1 U	76	16	1 U	160 J	65	100 D	1 U	1 U	1 U
1,1,2,2-Tetrachloroethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 UJ	1 U	5 U	1 U	1 U	1 U	RR
Toluene		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1	1 U	1 U	1 UJ
Chlorobenzene		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 U
Ethylbenzene		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 UJ	1 U	5 U	1	1 U	1 U	1 UJ
Styrene		1 UJ	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 UJ	5 U	1 U	1 U	1 U	1 UJ
Xylenes (total)		1 UJ	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 UJ	5 U	1 U	1 U	1 U	1 UJ
TOTAL VOCs:		1	1720	343	321	0.0	104	18	0.0	230	121	372	0.0	2	2

Analyses were performed by Ceimic Corporation, Narragansett, Rhode Island.
Samples were analyzed by USEPA Method 524.2.

ug/L Analyte concentrations in micrograms per liter [parts per billion (ppb)].

- D Compound identified at a secondary dilution.
- J Result is detected below the reporting limit or is an estimated concentration.
- U Compound analyzed for but not detected.
- RR Result rejected.
- * Fernandez is a field replicate of Dench.
- * Eglin IV is a field replicate of Eglin III.
- * Laverne is a field replicate of LaPlace.

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Table 3. Results of Quarterly Analysis for Volatile Organic Compounds in Ground-Water Samples Collected in February 1991 at the Tutu Wells Site, St. Thomas, U.S. Virgin Islands.

Analyte	Sample ID	Trip	Trip	Trip
	Date	Blank 2	Blank 3	Blank 4
Chloromethane		2 UJ	1 J	1 J
Bromomethane		RR	2 U	2 U
Vinyl chloride		RR	2 U	2 U
Chloroethane		RR	2 U	2 U
Methylene chloride		3	2	2
Acetone		RR	RR	RR
Carbon disulfide		1 UJ	1 UJ	1 UJ
1,1-Dichloroethene		1 U	1 U	1 U
1,1-Dichloroethane		1 U	1 U	1 U
1,2-Dichloroethene (total)		1 UJ	1 U	1 U
Chloroform		1 U	1 U	1 U
1,2-Dichloroethane		1 UJ	1 U	1 U
2-Butanone		RR	RR	RR
1,1,1-Trichloroethane		1 UJ	1 U	1 U
Carbon tetrachloride		1 U	1 U	1 U
Vinyl acetate		2 UJ	2 UJ	2 UJ
Bromodichloromethane		1 UJ	1 U	1 U
1,2-Dichloropropane		1 U	1 U	1 U
cis-1,3-Dichloropropene		1 U	RR	RR
Trichloroethene		1 U	1 U	1 U
Dibromochloromethane		1 UJ	1 U	1 U
1,1,2-Trichloroethane		1 U	1 U	1 U
Benzene		1 UJ	1 U	1 U
trans-1,3-Dichloropropene		RR	1 U	1 U
Bromoform		RR	1 U	1 U
4-Methyl-2-pentanone		2 U	2 U	2 U
2-Hexanone		2 UJ	2 UJ	2 UJ
Tetrachloroethene		1 U	1 U	1 U
1,1,2,2-Tetrachloroethane		RR	1 U	1 U
Toluene		1 UJ	1 U	1 U
Chlorobenzene		1 U	1 U	1 U
Ethylbenzene		1 UJ	1 U	1 U
Styrene		1 UJ	1 U	1 U
Xylenes (total)		1 UJ	1 U	1 U
TOTAL VOCs:		3	3	3

Analyses were performed by Ceimic Corporation, Narragansett, Rhode Island.
 Samples were analyzed by USEPA Method 524.2.

ug/L Analyte concentrations in micrograms per liter [parts per billion (ppb)].

D Compound identified at a secondary dilution.

J Result is detected below the reporting limit or is an estimated concentration.

U Compound analyzed for but not detected.

RR Result rejected.

* Fernandez is a field replicate of Dench.

* Eglin IV is a field replicate of Eglin III.

* Levergne is a field replicate of LaPlace.

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Table 4. Comparative Results of Quarterly Analysis for Volatile Organic Compounds in Ground-Water Samples Collected in September 1990 and February 1991 at the Tutu Wells Site, St. Thomas, U.S. Virgin Islands.

Analyte	Sample ID	Bryan	Bryan	Dede	Dede	Demitri	Demitri	Dench	Dench	Fernandez	Devcon I	Devcon I	Devcon III	Devcon III	Eglin I
	Date	1-Oct-90	5-Feb-91	25-Sep-90	5-Feb-91	1-Oct-90	5-Feb-91	25-Sep-90	5-Feb-91	5-Feb-91	24-Sep-90	5-Feb-91	24-Sep-90	5-Feb-91	2-Oct-90
Chloromethane		2 U	2 UJ	2 U	.9 J	2 UJ	2 UJ	1 J	2 UJ	2 UJ	2 UJ	RR	2 U	2 UJ	RR
Bromomethane		2 U	RR	2 U	RR	2 UJ	RR	2 UJ	RR	RR	2 U	RR	2 U	RR	RR
Vinyl chloride		2 U	RR	2 U	RR	2 UJ	RR	2 UJ	RR	RR	2 U	RR	2 U	RR	RR
Chloroethane		2 U	RR	2 U	RR	2 UJ	RR	2 UJ	RR	RR	2 UJ	RR	2 U	RR	RR
Methylene chloride		2 UJ	1 U	2 UJ	1 U	2 UJ	1 U	1 UJ	1 U	1 U	1 UJ	RR	8 UJ	1 U	RR
Acetone		RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR
Carbon disulfide		1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	RR	1 UJ	1 UJ	RR
1,1-Dichloroethene		1 UJ	1 U	1 UJ	1 U	1 UJ	1 U	1 UJ	1 U	1 U	1 UJ	RR	1 UJ	1 U	RR
1,1-Dichloroethane		1 U	1 U	1 UJ	1 U	1 UJ	1 U	1 UJ	1 U	1 U	1 UJ	RR	1 UJ	1 U	RR
1,2-Dichloroethane (total)		1 U	1 U	1 UJ	1 U	1 UJ	1 U	1 UJ	1 UJ	1 UJ	1 U	RR	1 UJ	1 UJ	68 J
Chloroform		1 U	1 U	1 UJ	1 U	1 UJ	1 U	1 UJ	1 U	1 U	1 UJ	RR	1 UJ	1 U	RR
1,2-Dichloroethane		1 UJ	1 U	1 UJ	.6 J	1 UJ	1 U	1 UJ	1 UJ	1 UJ	1 UJ	RR	1 UJ	1 UJ	RR
2-Butanone		RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR
1,1,1-Trichloroethane		1 U	1 U	1 UJ	1 U	1 UJ	1 U	1 UJ	1 UJ	1 UJ	1 UJ	RR	1 UJ	1 UJ	RR
Carbon tetrachloride		1 U	1 U	1 UJ	1 U	1 UJ	1 U	1 UJ	1 U	1 U	1 UJ	RR	1 UJ	1 U	RR
Vinyl acetate		2 UJ	2 UJ	RR	2 UJ	2 UJ	2 UJ	RR	2 UJ	2 UJ	2 UJ	RR	RR	2 UJ	RR
Bromodichloromethane		1 U	1 U	1 U	1 U	1 UJ	1 U	1 UJ	1 UJ	1 UJ	1 U	RR	1 U	1 UJ	RR
1,2-Dichloropropane		1 U	1 U	1 UJ	1 U	1 UJ	1 U	1 UJ	1 U	1 U	1 UJ	RR	1 UJ	1 U	RR
cis-1,3-Dichloropropene		1 U	RR	1 U	1 U	1 UJ	RR	1 UJ	1 U	1 U	1 UJ	RR	1 U	1 U	RR
Trichloroethene		1 U	1 U	1 UJ	1 U	1 UJ	1 U	1 UJ	1 U	1 U	1 UJ	RR	1 UJ	1 U	10 J
Dibromochloromethane		1 U	1 U	1 UJ	1 U	1 UJ	1 U	1 UJ	1 UJ	1 UJ	1 UJ	RR	1 UJ	1 UJ	RR
1,1,2-Trichloroethane		1 U	1 U	1 UJ	1 U	1 UJ	1 U	1 UJ	1 U	1 U	1 UJ	RR	1 UJ	1 U	RR
Benzene		1 U	1 U	1 UJ	1 U	1 UJ	1 U	1 UJ	1 UJ	1 UJ	1 UJ	RR	1 UJ	1 UJ	RR
trans-1,3-Dichloropropene		1 U	1 U	1 UJ	RR	RR	1 U	1 UJ	RR	RR	1 UJ	RR	1 UJ	RR	RR
Bromoform		1 U	1 U	1 UJ	RR	1 UJ	1 U	1 UJ	RR	RR	1 UJ	RR	1 UJ	RR	RR
4-Methyl-2-pentanone		2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	RR	2 UJ	2 UJ	RR
2-Hexanone		RR	RR	RR	2 UJ	RR	RR	RR	2 UJ	2 UJ	RR	RR	RR	2 UJ	RR
Tetrachloroethene		1 UJ	1 U	1 UJ	1 U	1 UJ	1 U	1 UJ	1 U	1 U	1 UJ	RR	1 UJ	1 U	32 J
1,1,2,2-Tetrachloroethane		1 UJ	1 U	1 UJ	RR	1 UJ	1 U	1 UJ	RR	RR	1 UJ	RR	1 UJ	RR	RR
Toluene		1 U	1 U	1 U	1 U	1 UJ	1 U	1 UJ	1 UJ	1 UJ	1 UJ	RR	1 U	1 UJ	RR
Chlorobenzene		1 U	1 U	1 UJ	1 U	1 UJ	1 U	1 UJ	1 U	1 U	1 UJ	RR	1 UJ	1 U	RR
Ethylbenzene		1 U	1 U	1 U	1 U	1 UJ	1 U	1 UJ	1 UJ	1 UJ	1 U	RR	1 U	1 UJ	RR
Styrene		1 U	1 U	1 UJ	1 U	1 UJ	1 U	1 UJ	1 UJ	1 UJ	1 U	RR	1 UJ	1 UJ	RR
Xylenes (total)		1 U	1 U	1 U	1 U	1 UJ	1 U	1 UJ	1 UJ	1 UJ	1 U	RR	1 U	1 UJ	RR
TOTAL VOCs:		0.0	0.0	0.0	1.5	0.0	0.0	1	0.0	0.0	0.0	0.0	0.0	0.0	110

Analyses were performed by Calmic Corporation, Narragansett, Rhode Island.
Samples were analyzed by USEPA Method 524.2.

ug/L Analyte concentrations in micrograms per liter [parts per billion (ppb)].

D Compound identified at a secondary dilution.

J Result is detected below the reporting limit or is an estimated concentration.

U Compound analyzed for but not detected.

RR Result rejected.

* Fernandez is a field replicate of Dench.

* Eglin IV is a field replicate of Eglin III.

* Tutu is a field replicate of Four Winds II.

* Laverne is a field replicate of LaPlace.

* Rodrigues (PD) is a field replicate of Rodrigues (P).

* Tillet-D is a field replicate of Tillet.

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Table 4. Comparative Results of Quarterly Analysis for Volatile Organic Compounds in Ground-Water Samples Collected in September 1990 and February 1991 at the Tutu Wells Site, St. Thomas, U.S. Virgin Islands.

Analyte	Sample ID	Eglin I Date 5-Feb-91	Eglin II 27-Sep-90	Eglin II 5-Feb-91	Eglin III 27-Sep-90	Eglin III 5-Feb-91	Eglin IV 5-Feb-91	Four Winds II 26-Sep-90	Tutu * 26-Sep-90	Four Winds II 5-Feb-91	Gassett 5-Feb-91	Hartman II 26-Sep-90	Hartman II 5-Feb-91	Hartman III 26-Sep-90	Hartman III 5-Feb-91
Chloromethane		2 UJ	10 UJ	2 UJ	10 UJ	2 UJ	2 U	5 UJ	5 UJ	2 U	2 U	2 UJ	2 U	2 UJ	2 U
Bromomethane		RR	10 UJ	RR	10 UJ	RR	2 UJ	5 UJ	5 UJ	2 UJ	2 U	2 U	2 U	2 UJ	2 UJ
Vinyl chloride		RR	10 UJ	RR	10 UJ	RR	2 UJ	5 UJ	5 UJ	2 UJ	2 U	2 U	2 U	2 UJ	2 UJ
Chloroethane		RR	10 UJ	RR	10 UJ	RR	2 UJ	5 UJ	5 UJ	2 UJ	RR	2 UJ	2 U	2 UJ	2 UJ
Methylene chloride		1 U	5 UJ	1 U	10 UJ	1 U	1 U	14 UJ	10 UJ	1 U	1 U	1 UJ	1 U	3 UJ	1 U
Acetone		RR	RR	RR	22 J	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR
Carbon disulfide		1 UJ	5 UJ	1 UJ	5 UJ	1 UJ	1 UJ	2 UJ	2 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ
1,1-Dichloroethane		1 U	5 UJ	1 U	5 UJ	1 U	1 U	2 UJ	2 UJ	1 U	1 U	1 UJ	1 U	1 UJ	1 U
1,1-Dichloroethane		1 U	5 UJ	1 U	5 U	1 U	1 U	2 UJ	2 UJ	1 U	1 U	1 UJ	1 U	1 U	1 U
1,2-Dichloroethane (total)		60 J	120 J	72 DJ	52 J	45 J	45 J	75 J	61 J	230 D	1 U	5 J	5 J	2	1 U
Chloroform		1 U	5 UJ	1 U	5 U	1 U	1 U	2 UJ	2 UJ	1 U	1 U	1 UJ	1 U	1 UJ	1 U
1,2-Dichloroethane		1 UJ	5 UJ	1 UJ	5 UJ	1 UJ	1 U	2 UJ	2 UJ	1 U	1 U	1 UJ	1 U	1 UJ	1 U
2-Butanone		RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR
1,1,1-Trichloroethane		1 UJ	5 UJ	1 UJ	5 U	1 UJ	1 U	2 UJ	2 UJ	1 U	1 U	1 UJ	1 U	1 UJ	1 U
Carbon tetrachloride		1 U	5 UJ	1 U	5 U	1 U	1 U	2 UJ	2 UJ	1 U	1 U	1 UJ	1 U	1 U	1 U
Vinyl acetate		2 UJ	10 UJ	2 UJ	10 UJ	2 UJ	2 UJ	5 UJ	5 UJ	2 UJ	RR	2 UJ	2 UJ	2 UJ	2 UJ
Bromodichloromethane		1 UJ	5 UJ	1 UJ	5 UJ	1 UJ	1 U	2 UJ	2 UJ	1 U	1 U	1 U	1 U	1 U	1 U
1,2-Dichloropropane		1 U	5 UJ	1 U	5 UJ	1 U	1 U	2 UJ	2 UJ	1 U	1 U	1 UJ	1 U	1 U	1 U
cis-1,3-Dichloropropene		1 U	5 UJ	1 U	5 UJ	1 U	1 U	2 UJ	2 UJ	1 U	RR	1 UJ	RR	1 UJ	1 U
Trichloroethane		8	22 J	19	14	11	13	6 J	4 J	21	1	1 J	2	1 U	1 U
Dibromochloromethane		1 UJ	5 UJ	1 UJ	5 UJ	1 UJ	1 U	2 UJ	2 UJ	1 U	1 U	1 UJ	1 U	1 U	1 U
1,1,2-Trichloroethane		1 U	5 UJ	1 U	5 UJ	1 U	1 U	2 UJ	2 UJ	1 U	1 U	1 UJ	1 U	1 U	1 U
Benzene		1 UJ	5 UJ	1 UJ	5 UJ	1 UJ	1 U	2 UJ	2 UJ	1 UJ	1 U	1 UJ	1 U	1 U	1 U
trans-1,3-Dichloropropene		RR	5 UJ	RR	5 UJ	RR	RR	2 UJ	2 UJ	RR	1 U	1 UJ	1 U	1 UJ	RR
Bromoform		RR	5 UJ	RR	5 UJ	RR	1 U	2 UJ	2 UJ	1 U	1 U	1 UJ	1 U	1 UJ	1 U
4-Methyl-2-pentanone		2 UJ	10 UJ	2 UJ	10 UJ	2 UJ	2 UJ	5 UJ	5 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ
2-Hexanone		2 UJ	RR	2 UJ	RR	2 UJ	2 UJ	RR	RR	2 UJ	2 UJ	RR	2 UJ	RR	2 UJ
Tetrachloroethane		26	71 J	34 DJ	44 J	35	36	25 J	18 J	90 D	1 U	7 J	9	1 J	1
1,1,2,2-Tetrachloroethane		RR	5 UJ	RR	5 UJ	RR	1 U	2 UJ	2 UJ	1 U	1 U	1 UJ	1 U	1 UJ	1 U
Toluene		1 UJ	5 UJ	1 UJ	5 UJ	1 UJ	1 U	2 UJ	2 UJ	1 UJ	44 D	1 UJ	1 U	1 U	1 U
Chlorobenzene		1 U	5 UJ	1 U	5 UJ	1 U	1 U	2 UJ	2 UJ	1 UJ	1 U	1 UJ	1 U	1 U	1 U
Ethylbenzene		1 UJ	5 UJ	1 UJ	5 UJ	1 UJ	1 U	2 UJ	2 UJ	1 UJ	1 U	1 U	1 U	1 U	1 U
Styrene		1 UJ	5 UJ	1 UJ	5 UJ	1 UJ	1 UJ	2 UJ	2 UJ	1 UJ	1 U	1 U	1 U	1 UJ	1 UJ
Xylenes (total)		1 J	5 UJ	1 UJ	5 UJ	1 UJ	1 UJ	2 UJ	2 UJ	1 UJ	1 U	1 U	1 U	1 UJ	1 UJ
TOTAL VOCs:		95	213	125	132	91	94	106	83	341	45	13	16	3	1

Analyses were performed by Calmic Corporation, Narragansett, Rhode Island.
Samples were analyzed by USEPA Method 524.2.

ug/L Analyte concentrations in micrograms per liter [parts per billion (ppb)].

- D Compound identified at a secondary dilution.
- J Result is detected below the reporting limit or is an estimated concentration.
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- RR Result rejected.
- * Fernandez is a field replicate of Dench.
- * Eglin IV is a field replicate of Eglin III.
- * Tutu is a field replicate of Four Winds II.
- * Levergne is a field replicate of LaPlace.
- * Rodrigues (PD) is a field replicate of Rodrigues (P).
- * Tillet-D is a field replicate of Tillet.

Table 4. Comparative Results of Quarterly Analysis for Volatile Organic Compounds in Ground-Water Samples Collected in September 1990 and February 1991 at the Tutu Wells Site, St. Thomas, U.S. Virgin Islands.

	Sample ID	Harvey	Harvey	LaPlace	LaPlace	Lavergne	Leonard	Leonard	Matthias	Matthias	Ramsey	Ramsey	Rodrigues (P)	Rodrigues (PD)	Rodrigues
Analyte	Date	26-Sep-90	5-Feb-91	2-Oct-90	5-Feb-91	5-Feb-91	26-Sep-90	5-Feb-91	1-Oct-90	5-Feb-91	1-Oct-90	5-Feb-91	24-Sep-90	24-Sep-90	5-Feb-91
Chloromethane		100 U	200 U	RR	2 U	2 U	2 UJ	2 U	2 UJ	25 U	4 UJ	2 UJ	2 UJ	2 UJ	2 UJ
Bromomethane		100 U	200 U	RR	2 U	2 U	2 UJ	2 U	2 UJ	25 U	4 UJ	RR	2 U	2 UJ	2 U
Vinyl chloride		100 UJ	200 U	RR	2 U	2 U	2 UJ	2 U	2 UJ	25 U	4 UJ	RR	2 U	2 UJ	2 U
Chloroethane		100 U	200 U	RR	RR	RR	2 UJ	RR	2 UJ	25 U	4 UJ	RR	2 UJ	2 UJ	2 U
Methylene chloride		150 UJ	100 U	RR	1 U	1 U	2 UJ	1 U	9 UJ	25 U	17 UJ	1 U	1 UJ	2 UJ	1 U
Acetone		RR	200 U	RR	RR	RR	RR	RR	RR	25 U	RR	RR	RR	RR	RR
Carbon disulfide		50 UJ	100 U	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	12 UJ	2 UJ	1 UJ	1 UJ	1 UJ	1 UJ
1,1-Dichloroethane		50 UJ	100 U	RR	1 U	1 U	1 UJ	1 U	1 UJ	12 U	2 UJ	1 U	1 UJ	1 UJ	1 U
1,1-Dichloroethane		50 U	100 U	RR	1 U	1 U	1 UJ	1 U	1 U	12 U	2 UJ	1 U	1 UJ	1 UJ	1 U
1,2-Dichloroethane (total)		50 UJ	160	140 J	200 D	190 D	1 UJ	1 U	22	21	2 UJ	1 J	1 U	1 UJ	1 U
Chloroform		50 U	100 U	RR	1	1 U	1 UJ	1 U	1 UJ	12 U	2 UJ	1 U	1 UJ	1 UJ	1 U
1,2-Dichloroethane		50 UJ	100 U	RR	1 U	1 U	1 UJ	1 U	1 UJ	12 U	2 UJ	1 U	1 UJ	1 UJ	1 U
2-Butanone		RR	200 U	RR	RR	RR	RR	RR	RR	25 U	RR	RR	RR	RR	RR
1,1,1-Trichloroethane		50 U	100 U	RR	1 U	1 U	1 UJ	1 U	1 UJ	12 U	2 UJ	1 U	1 UJ	1 UJ	1 U
Carbon tetrachloride		50 U	100 U	RR	1 U	1 U	1 UJ	1 U	1 U	12 U	2 UJ	1 U	1 UJ	1 UJ	1 U
Vinyl acetate		100 UJ	200 U	RR	RR	RR	2 UJ	RR	2 UJ	25 U	4 UJ	2 U	2 UJ	2 UJ	RR
Bromodichloromethane		50 UJ	100 U	RR	1 U	1 U	1 UJ	1 U	1 U	12 U	2 UJ	1 U	1 U	1 UJ	1 U
1,2-Dichloropropane		50 U	100 U	RR	1 U	1 U	1 UJ	1 U	1 U	12 U	2 UJ	1 U	1 UJ	1 UJ	1 U
cis-1,3-Dichloropropene		50 UJ	100 U	RR	RR	RR	1 UJ	RR	1 UJ	12 U	2 UJ	RR	1 UJ	1 UJ	RR
Trichloroethane		50 U	60 J	24 J	32	31	1 UJ	1 U	11	7 J	2 UJ	1	1 UJ	1 UJ	1 U
Dibromochloromethane		50 U	100 U	RR	1 U	1 U	1 UJ	1 U	1 U	12 U	2 UJ	1 U	1 UJ	1 UJ	1 U
1,1,2-Trichloroethane		50 U	100 U	RR	1 U	1 U	1 UJ	1 U	1 U	12 U	2 UJ	1 U	1 UJ	1 UJ	1 U
Benzene		50 U	100 U	RR	1 U	1 U	1 UJ	1 U	1 U	12 U	2 UJ	1 U	1 UJ	1 UJ	1 U
trans-1,3-Dichloropropene		50 U	100 U	RR	1 U	1 U	1 UJ	1 U	1 UJ	12 U	2 UJ	1 U	1 UJ	1 UJ	1 UJ
Bromoform		50 UJ	100 U	RR	1 U	1 U	1 UJ	1 U	1 U	12 U	2 UJ	1 U	1 UJ	1 UJ	1 U
4-Methyl-2-pentanone		100 UJ	200 U	RR	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	25 U	4 UJ	2 UJ	2 UJ	2 UJ	2 UJ
2-Hexanone		RR	200 U	RR	2 UJ	2 UJ	RR	2 UJ	RR	25 U	RR	RR	RR	RR	2 UJ
Tetrachloroethene		890 J	1500	98 J	110 D	100 D	1 UJ	1 U	120 J	76	3 J	16	1 UJ	1 UJ	1 U
1,1,2,2-Tetrachloroethane		50 UJ	100 U	RR	1 U	1 U	1 UJ	1 U	1 UJ	12 U	2 UJ	1 U	1 UJ	1 UJ	1 UJ
Toluene		50 U	100 U	RR	1 U	1 U	1 UJ	1 U	1 U	12 U	2 UJ	1 U	1 UJ	1 UJ	1 U
Chlorobenzene		50 U	100 U	RR	1 U	1 U	1 UJ	1 U	1 U	12 U	2 UJ	1 U	1 UJ	1 UJ	1 U
Ethylbenzene		50 U	100 U	RR	1 U	1 U	1 UJ	1 U	1 U	12 U	2 UJ	1 U	1 U	1 UJ	1 UJ
Styrene		50 U	100 U	RR	1 U	1 U	1 UJ	1 U	1 UJ	12 U	2 UJ	1 U	1 U	1 UJ	1 U
Xylenes (total)		50 UJ	100 U	RR	1 U	1 U	1 UJ	1 U	1 UJ	12 U	2 UJ	1 U	1 U	1 UJ	1 U
TOTAL VOCs:		890	1720	262	343	321	0.0	0.0	153	104	3	18	0.0	0.0	0.0

Analyses were performed by Ceimic Corporation, Narragansett, Rhode Island.
Samples were analyzed by USEPA Method 524.2.

ug/L Analyte concentrations in micrograms per liter [parts per billion (ppb)].

- D Compound identified at a secondary dilution.
- J Result is detected below the reporting limit or is an estimated concentration.
- U Compound analyzed for but not detected.
- RR Result rejected.
- * Fernandez is a field replicate of Dench.
- * Eglin IV is a field replicate of Eglin III.
- * Tutu is a field replicate of Four Winds II.
- * Lavergne is a field replicate of LaPlace.
- * Rodrigues (PD) is a field replicate of Rodrigues (P).
- * Tillet.

Table 4. Comparative Results of Quarterly Analysis for Volatile Organic Compounds in Ground-Water Samples Collected in September 1990 and February 1991 at the Tutu Wells Site, St. Thomas, U.S. Virgin Islands.

	Sample ID	Smith	Smith	Steele	Steele	Tillet	Tillet-D * Tillet	VIHA I	VIHA III	VIHA III	Field Blank	H2O Blank	Trip Blank	Trip Blank	
Analyte	Date	25-Sep-90	5-Feb-91	25-Sep-90	5-Feb-91	2-Oct-90	2-Oct-90	5-Feb-91	27-Sep-90	26-Sep-90	5-Feb-91	5-Feb-91	26-Sep-90	24-Sep-90	25-Sep-90
Chloromethane		25 U	2 UJ	17 UJ	10 U	RR	RR	2 U	2 UJ	2 UJ	2 UJ	2 U	2 UJ	2 U	2 U
Bromomethane		25 U	RR	17 UJ	10 U	RR	RR	2 U	2 UJ	2 UJ	RR	2 U	2 UJ	2 U	2 U
Vinyl chloride		25 U	RR	17 UJ	10 U	17 J	12 J	7	2 UJ	2 UJ	RR	2 U	2 UJ	2 U	2 U
Chloroethane		25 U	RR	17 UJ	10 U	RR	RR	2 U	2 UJ	2 UJ	RR	RR	2 UJ	2 U	2 U
Methylene chloride		12 UJ	1 U	140 UJ	5 U	RR	RR	1 U	2 UJ	2 UJ	1 U	2	2 UJ	25 J	34 J
Acetone		25 UJ	RR	RR	10 U	RR	RR	RR	RR	RR	RR	RR	RR	12 J	42 J
Carbon disulfide		12 UJ	1 UJ	8 UJ	5 U	RR	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ
1,1-Dichloroethene		12 UJ	1 U	8 UJ	5 U	RR	RR	1 U	1 UJ	1 UJ	1 U	1 U	1 UJ	1 UJ	1 UJ
1,1-Dichloroethane		12 U	1 U	8 U	5 U	RR	RR	1 U	1 UJ	1 U	1 U	1 U	1 UJ	1 UJ	1 UJ
1,2-Dichloroethene (total)		58	42 J	120	44	280 J	270 J	200 D	6 J	1 U	1 U	1 U	1 UJ	1 UJ	1 UJ
Chloroform		12 U	1 U	8 UJ	5 U	RR	RR	1 U	1 UJ	3 J	1 U	1 U	1 UJ	1 UJ	1 UJ
1,2-Dichloroethane		12 UJ	1 U	8 UJ	5 U	RR	RR	1 U	1 UJ	1 UJ	1 U	1 U	1 UJ	1 UJ	1 UJ
2-Butanone		25 UJ	RR	RR	10 U	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR
1,1,1-Trichloroethane		12 U	1 U	8 UJ	5 U	RR	RR	1 U	1 UJ	1 UJ	1 U	1 U	1 UJ	1 UJ	1 UJ
Carbon tetrachloride		12 U	1 U	8 U	5 U	RR	RR	1 U	1 UJ	1 U	1 U	1 U	1 UJ	1 UJ	1 UJ
Vinyl acetate		25 UJ	2 UJ	17 UJ	10 UJ	RR	RR	2 UJ	2 UJ	2 UJ	2 UJ	RR	2 UJ	RR	RR
Bromodichloromethane		12 U	1 U	8 U	5 U	RR	RR	1 U	1 UJ	1 U	1 U	1 U	1 UJ	1 U	1 U
1,2-Dichloropropane		12 U	1 U	8 U	5 U	RR	RR	1 U	1 UJ	1 U	1 U	1 U	1 UJ	1 UJ	1 UJ
cis-1,3-Dichloropropene		12 U	1 U	8 UJ	5 U	RR	RR	RR	1 UJ	1 UJ	RR	RR	1 UJ	1 U	1 U
Trichloroethane		20	23	48	12	56 J	47 J	36	1 UJ	1 U	1 U	1 U	1 UJ	1 UJ	1 UJ
Dibromochloromethane		12 U	1 U	8 U	5 U	RR	RR	1 U	1 UJ	1 U	1 U	1 U	1 UJ	1 UJ	1 UJ
1,1,2-Trichloroethane		12 U	5	8 U	5 U	RR	RR	1 U	1 UJ	1 U	1 U	1 U	1 UJ	1 UJ	1 UJ
Benzene		12 U	1 U	8 U	5 U	32 J	26 J	27	1 UJ	1 U	1 U	1 U	1 UJ	1 UJ	1 UJ
trans-1,3-Dichloropropene		RR	RR	8 UJ	5 U	RR	RR	1 U	1 UJ	1 UJ	1 U	1 U	RR	1 UJ	1 UJ
Bromoform		12 UJ	1 U	8 UJ	5 U	RR	RR	1 U	1 UJ	1 UJ	1 U	1 U	1 UJ	1 UJ	1 UJ
4-Methyl-2-pentanone		25 UJ	2 UJ	17 UJ	10 U	RR	RR	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ
2-Hexanone		25 UJ	2 UJ	RR	10 U	RR	RR	2 UJ	RR	RR	RR	2 UJ	RR	RR	RR
Tetrachloroethene		330 J	160 J	130 J	65	120 J	98 J	100 D	5 J	1 UJ	1 U	1 U	1 UJ	1 UJ	1 UJ
1,1,2,2-Tetrachloroethane		12 UJ	1 U	8 UJ	5 U	RR	RR	1 U	1 UJ	1 UJ	1 U	1 U	1 UJ	1 UJ	1 UJ
Toluene		12 U	1 U	8 U	5 U	RR	RR	1	1 UJ	1 U	1 U	1 U	1 UJ	1 U	1 J
Chlorobenzene		12 U	1 U	8 U	5 U	RR	RR	1 U	1 UJ	1 U	1 U	1 U	1 UJ	1 UJ	1 UJ
Ethylbenzene		12 U	1 U	8 U	5 U	RR	RR	1	1 UJ	1 U	1 U	1 U	1 UJ	1 U	1 U
Styrene		12 U	1 UJ	8 UJ	5 U	RR	RR	1 U	1 UJ	1 UJ	1 U	1 U	1 UJ	1 UJ	1 UJ
Xylenes (total)		12 U	1 UJ	8 UJ	5 U	RR	RR	1 U	1 UJ	1 UJ	1 U	1 U	1 UJ	8	7
TOTAL VOCs:		408	230	298	121	505	453	372	11	3	0.0	2	0.0	45	84

Analyses were performed by Ceimic Corporation, Narragansett, Rhode Island.
 Samples were analyzed by USEPA Method 524.2.

ug/L Analyte concentrations in micrograms per liter [parts per billion (ppb)].

- D Compound identified at a secondary dilution.
- J Result is detected below the reporting limit or is an estimated concentration.
- U Compound analyzed for but not detected.
- RR Result rejected.
- * Fernandez is a field replicate of Dench.
- * Eglin IV is a field replicate of Eglin III.
- * Tutu is a field replicate of Four Winds II.
- * Lavergne is a field replicate of LaPlace.
- * Rodrigues (PD) is a field replicate of Rodrigues (P).
- * Tillet-D is a field replicate of Tillet.

Table 4. Comparative Results of Quarterly Analysis for Volatile Organic Compounds in Ground-Water Samples Collected in September 1990 and February 1991 at the Tutu Wells Site, St. Thomas, U.S. Virgin Islands.

Analyte	Sample ID	Trip Blank	Trip Blank	Trip Blank	Trip Blank	Trip Blank	Trip Blank	Trip Blank	Trip Blank
Date	26-Sep-90	27-Sep-90	1-Oct-90	2-Oct-90	3-Oct-90	4-Feb-91	5-Feb-91	6-Feb-91	7-Feb-91
Chloromethane	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	1 J	1 J
Bromomethane	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	RR	RR	2 U	2 U
Vinyl chloride	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	RR	RR	2 U	2 U
Chloroethane	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	RR	RR	2 U	2 U
Methylene chloride	24 J	16 J	13 J	14 J	1 UJ	2	3	2	2
Acetone	4 J	RR	6 J	7 J	RR	RR	RR	RR	RR
Carbon disulfide	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ
1,1-Dichloroethane	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
1,1-Dichloroethane	1 U	1 U	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
1,2-Dichloroethane (total)	1 U	1 U	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U
Chloroform	1 UJ	1 U	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
1,2-Dichloroethane	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U
2-Butanone	RR	RR	RR	RR	RR	RR	RR	RR	RR
1,1,1-Trichloroethane	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U
Carbon tetrachloride	1 U	1 U	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
Vinyl acetate	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ
Bromodichloromethane	1 U	1 U	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U
1,2-Dichloropropane	1 U	1 U	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
cis-1,3-Dichloropropane	1 UJ	1 U	1 UJ	1 UJ	1 UJ	1 U	1 U	RR	RR
Trichloroethene	1 U	1 U	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
Dibromochloromethane	1 U	1 U	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U
1,1,2-Trichloroethane	1 U	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
Benzene	1 U	1 U	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U
trans-1,3-Dichloropropane	1 UJ	1 UJ	RR	RR	1 UJ	RR	RR	1 U	1 U
Bromoform	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	RR	RR	1 U	1 U
4-Methyl-2-pentanone	1 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 U	2 U	2 U
2-Hexanone	RR	RR	RR	RR	RR	2 UJ	2 UJ	2 UJ	2 UJ
Tetrachloroethene	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
1,1,2,2-Tetrachloroethane	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	RR	RR	1 U	1 U
Toluene	1 U	1 U	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U
Chlorobenzene	1 U	1 U	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
Ethylbenzene	1 U	1 U	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U
Styrene	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U
Xylenes (total)	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U
TOTAL VOCs:	28	16	19	21	0.0	2	3	3	3

Analyses were performed by Ceimic Corporation, Narragansett, Rhode Island.
Samples were analyzed by USEPA Method 524.2.

ug/L Analyte concentrations in micrograms per liter [parts per billion (ppb)].

D Compound identified at a secondary dilution.

J Result is detected below the reporting limit or is an estimated concentration.

U Compound analyzed for but not detected.

RR Result rejected.

* Fernandez is a field replicate of Dench.

* Eglin IV is a field replicate of Eglin III.

* Tutu is a field replicate of Four Winds II.

* Lavergne is a field replicate of LaPlace.

* Rodrigues (PD) is a field replicate of Rodrigues (P).

* Tillet-D is a field replicate of Tillet.

GERAGHTY & MILLER, INC.

Table 5. Proposed Sampling Effort for the Third Quarterly Sampling Event (June 1991), Tutu Wells Site, St. Thomas, USVI.

Well	Proposed Analytes
Eglin I	VOCs
Eglin II	VOCs
Eglin III	VOCs
Four Winds I	VOCs
Four Winds II	VOCs
Gassett	VOCs
Hartman II	VOCs
Hartman III	VOCs
Harvey	VOCs
LaPlace	VOCs
Matthias	VOCs
Ramsey	VOCs
Smith	VOCs
Steele	VOCs
Tillett	VOCs
VIHA I or VIHA II	VOCs

VOCs Volatile organic compounds.

Analysis will be performed using modified USEPA Method 524.2.

TD:gv
#PR00801/2ndqtr.rpt

TUT 002 2093

FIGURE

TUT 002 2094



FIGURE
1

APPENDIX A

TELEPHONE CONVERSATION RECORDS

TELEPHONE CONVERSATION RECORD

Page 1 of 2

DATE: January 22, 1991

TIME: 10:00 AM

PROJECT: Tutu (PR00801)

CONFERENCE CALL PARTICIPANTS

Laura Scalise, EPA Monitoring Management Branch (MMB), (201) 906-6171

Sherrel Henry, EPA Project Manager (PM), (201) 264-8675

Dave Dickinson, Ceimic Corporation, (401) 782-8900

Inorganic Laboratory Manager, Ceimic

GC/MS Laboratory Manager, Ceimic

Juan Garcia, Data QA - Geraghty & Miller

Marcia Findlay, Project Manager - Geraghty & Miller

RE: Analytical Issues to be Solved in the Tutu Quarterly Sampling for the 2nd Quarter Event (February 1991)

1. VOC Analysis: The lab proposes to do a modified version of Methods 524.2/624, modified to achieve lower detection limits. The surrogate spike compounds used will be the 3 standards used for Contact Laboratory Program (CLP) (instead of the 2 standards used for 524.2). There is more and better historical data and QC limits for the 3 CLP standards. The only problems with this method are that a response factor of ≥ 0.05 occurs often for the ketones, bromoform and 1,1,1-TCE. The GC/MS lab manager explained that the response factors were low as a result of the concentration of the internal standard, but that the area responses were good and consistent indicating the sample data quality was good. Juan Garcia understood and agreed that this was true. Laura Scalise indicated she would need to discuss this issue with her organics laboratory supervisor.
2. Dilution and Reanalysis: Some of the wells have VOC concentrations which are high enough so that they occur outside the calibration range for the low detection levels. The lab proposes to analyze each sample using the low detection limit (Method 524.2). If the VOC concentrations are high, the lab will dilute the sample and analyze it using the higher detection levels (CLP Method). In this way, we will get accurate data for low concentration constituents and high concentration constituents.

Laura Scalise objected to re-running the analysis using the CLP method, but she will check with the organics supervisor in her laboratory regarding this.

3. Matrix Interferences on the Thallium and Selenium Analyses

The matrix spike recoveries on thallium and selenium were low because of the interference of chloride. Laura checked with the EPA inorganics lab and found this to be true. When the recoveries are poor, the method of standard additions (MSA)

should be run as a check. Ceimic pointed out that even after doing MSA, they may still not be able to get any better data. Furthermore, if they dilute to cut the chloride interference, they will probably be outside the detectable limit for thallium. Laura Scalise agreed with both points. I pointed out that we need to try to get acceptable thallium and selenium data even though these metals are probably not site contaminants and even if it is likely that we will never obtain good data. Sherrel Henry agreed with this.

The lab pointed out that there is a lot of time involved in doing the analysis, then doing MSA and wondered if they could do MSA first for thallium and selenium.

Laura Scalise said she would check with her inorganics lab supervisor and get back to us.

MF:gv
#PR00801/telconv.rec

TELEPHONE CONVERSATION RECORD

Page 1 of 1

DATE: January 24, 1991

TIME: 11:30 AM

PROJECT: Tutu (PR00801)

TO: Marcia Findlay, Geraghty & Miller

FROM: Laura Scalise, EPA Monitoring Management Branch (MMB)

TELEPHONE NO.: 201-906-6171

RE: Approval/Disapproval of Proposed Changes to Analyses for the Quarterly Sampling which were Discussed During the January 22, 1991 Conference Call

EPA Does Not Approve the Following:

1. The lab should have no problem achieving a response factor of ≥ 0.05 for bromoform and TCE; EPA will reject any analyses which does not meet this in accordance with the data validation Standard Operating Procedures (SOPs). The same holds true for the ketones even though it is commonly recognized that ≥ 0.05 is difficult to achieve for ketones.
2. The lab wanted to spike the samples at 10 parts per billion (ppb). EPA will not accept this and continues to require spiking at 1 ppb (25 nanograms in 25 milliliters).

EPA Does Approve the Following:

1. The lab can spike the samples with the 3 CLP spike compounds.
2. The lab can run each sample undiluted using method 524.2. Then, if some of the concentrations are out of the calibration range, they can re-run the diluted sample using CLP. However, the laboratory must explain in their case narrative what they did and they must justify why they did it (because the concentrations were above the calibration range). Also, they must supply all the chromatograms to the data validators.
3. The laboratory can do the method of standard additions (MSA) on the thallium and selenium analyses before doing the standard AA/furnace analysis. On any other metals for which the same problem occurs, they should do MSA afterwards.

TELEPHONE CONVERSATION RECORD

DATE: February 1, 1991

PROJECT: Tutu Quarterly Well Sampling (PR00801)

TO: Laura Scalise, EPA Management Monitoring Branch (MMB)

FROM: Marcia Findlay, Geraghty & Miller, Inc.

TELEPHONE NO. (201) 906-6171

RE: Proposed Changes to Analyses for the Quarterly Sampling Program

Marcia Findlay: We have asked Ceimic Corporation to use the Method 524 surrogate spike compounds instead of the CLP spike compounds in the interest of consistency.

Laura Scalise: That is acceptable to EPA.

MF:gv
#PR00801/030591a.

APPENDIX B

**DATA VALIDATION SUMMARY
REPORT FOR THE TUTU WELLS SITE
FEBRUARY 1991 QUARTERLY SAMPLING
ST. THOMAS, USVI**

DATA VALIDATION SUMMARY REPORT

for the

TUTU WELLS SITE

February 1991 Quarterly Sampling

USEPA REGION II

Standard Operating Procedures HW-2 and HW-6

for the

Contract Laboratory program

Organic and Inorganic

Data Review

Sample Delivery Group 910062 (Dede)

May 1991

Prepared for

Tutu Environmental Investigation Committee

Geraghty & Miller, Inc.
201 W. Passaic Street
Rochelle Park, New Jersey 07662

TUT 002 2192

GERAGHTY & MILLER, INC.

STANDARD OPERATING PROCEDURE (SOP) NO. HW-6

Revision #7

**EVALUATION OF ORGANIC DATA FOR THE CONTRACT
LABORATORY PROGRAM (CLP)**

INTRODUCTION TO DATA VALIDATION1.0 Scope

- 1.1 This procedure is applicable to organic data obtained from contractor laboratories working for the Contract Laboratory Program (CLP).
- 1.2 The data validation is based upon analytical and quality assurance requirements specified in the Statement of Work (SOW).

2.0 Responsibilities

Data reviewers will complete the following tasks as assigned by the Data Review Coordinator:

- 2.1 Data Assessment - The reviewer must answer every question on the checklist. All responses shall be in ink.
- 2.2 Data Assessment Narrative (Attachment 1) - Data reviewer is required to use these forms and must match the action in the narrative with the action taken on the Form I(s).
- 2.3 Rejection Summary Form (Attachment 2) - Fill in the total number of analytes measured by different analyses and the number of analytes rejected or flagged as estimated due to corresponding quality control criteria. Place an "X" in the boxes where analyses were not performed or criteria do not apply.
- 2.4 Organic Regional Data Assessment - Data reviewer is also required to fill out Organic Regional Data Assessment Form (Attachment 3).
- 2.5 Telephone Record Log - The data reviewer should enter the bare facts of inquiry before initiating any authorized telephone conversation with a CLP laboratory. After the case review has been completed, mail the white copy of the Telephone Record Log to the laboratory and the pink copy to SMO. File the yellow copy in the Telephone Record Log folder and attach a photocopy of the Telephone Record Log to the completed Data Assessment Narrative.
- 2.6 Forwarded Paperwork - Upon completion of the review, the following are to be forwarded to the Regional Sample Control Center (RSCC) located in the Surveillance and Monitoring Branch:
 - a. data package
 - b. completed assessment checklist
 - c. SMO Contract Compliance Screening (CCS)

Forward four (4) copies of the completed Data Assessment Narrative along with four (4) copies of the Organic Data Assessment Form: one each for the appropriate Regional DPO, the Sample Management Office (SMO), and to the last two addresses of the Data Reviewers Mailing List.

- 2.7 Filed Paperwork - Upon completion of the review, the following are to be filed within the Monitoring and Management Branch (MMB) files:

- a. Telephone Record Log (copy)
- b. Record of Communication (original)
- c. Rejection Summary Form

- 3.0 Rejection of Data - All values determined to be unacceptable on the Organic Analysis Data Sheet (Form I) must be flagged with an "R". As soon as review criteria causes data to be rejected, that data can be eliminated from any further review or consideration.
- 4.0 Acceptance Criteria - In order that the reviews be consistent among reviewers, this Standard Operating Procedure (SOP) should be used. Additional guidance can be found in the Functional Guidelines.
- 5.0 SMO Contract Compliance Screening (CCS) - This is intended to aid the reviewer in locating any problems, both corrected and uncorrected. However, the validation should be carried out even if CCS is not present. Resubmittals received from the laboratory in response to CCS must be used by the reviewer.

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: _____

LAB: _____

SITE: _____

1.0 Data Completeness and Deliverables

Yes

NO

N/A

- 1.1 Have any missing deliverables been received and added to the data package.

[]

—

✓

ACTION: Call lab for explanation/resubmittal of any missing deliverables. If lab cannot provide them, note the effort on review of the package under the "Contract Problems/Non-Compliance" section of reviewer narrative.

- 1.2 Was SMO CCS checklist included with package?

[]

—

✓

2.0 Cover Letter/Case Narrative

- 2.1 Is the Narrative or Cover Letter present?

[✓]

—

—

- 2.2 Are Case Number and/or SAS number contained in the Narrative or Cover Letter?

[✓]

—

—

3.0 Data Validation Checklist

The following checklist is divided into three parts. Part A is filled out if the data package contains any VOA analyses, Part B for any BNA analyses and Part C for Pesticides/PCBs.

Does this package contain:

VOA data?

✓

—

BNA data?

—

✓

Pesticide/PCB data:

—

✓

ACTION: Complete corresponding parts of checklist.

YES NO N/A

PART A: VOA ANALYSES1.0 Traffic Reports and Laboratory Narrative

- 1.1 Are the Traffic Report Forms present for all samples?

☒☐☐

ACTION: If no, contact lab for replacement of missing or illegible copies.

- 1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

☒☐☐

ACTION: Use professional judgement to evaluate the effect on the quality of the data.

ACTION: If any sample analyzed as a soil contains more than 50% water, all data should be flagged as estimated (J).

ACTION: If both VOA vials for a sample have air bubbles, flag all positive results "J" and all non-detects "R".

2.0 Holding Times

- 2.1 Have any VOA holding times, determined from date of collection to date of analysis, been exceeded?

☒☐☐

If unpreserved, aqueous aromatic volatiles must be analyzed within 7 days of collection and non-aromatic volatiles must be analyzed within 14 days. If preserved with hydrochloric acid and stored at 4°C, then both aromatic and non-aromatic volatiles must be analyzed within 14 days. If uncertain about preservation, contact the sampler to determine whether the samples were preserved.

A ten-day holding time for soil samples is recommended.

Table of Holding Time Violations

Sample	Sample Matrix	Preserved ?	(See Traffic Report)		Date Analyzed
			Date Sampled	Date Lab Received	
EGLIN II DL	AQ	Yes	2/5/91	2/6/91	3/22/91
FOUR WIND 2	AQ	No	2/5/91	2/6/91	2/14/91
FOUR WIND 2 DL	AQ	No	2/5/91	2/6/91	2/16/91
_____	_____	_____	_____	_____	_____

ACTION: If holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. The reviewer may determine that non-detect data are unusable ("R").

3.0 Surrogate Recovery (Form II) YES NO N/A

3.1 Are the VOA Surrogate Recovery Summaries (Form II) present for each of the following matrices:

a.	Low Water	[☒]	—	—
b.	Med Water	[☐]	—	☒
c.	Low Soil	[☐]	—	☒
d.	Med Soil	[☐]	—	☒

	YES	NO	N/A
3.2 Are all the VOA samples listed on the appropriate Surrogate Recovery Summaries for each of the following matrices:			
a. Low Water	☒	☐	☐
b. Med Water	☐	☐	☒
c. Low Soil	☐	☐	☒
d. Med Soil	☐	☐	☒
ACTION: Call lab for explanation/resubmittals. If missing deliverables are unavailable, document effect on data under "Conclusions" section of reviewer narrative.			
3.3 Were outliers marked correctly with an asterisk?	☐	☐	☒
ACTION: Circle all outliers in red.			
3.4 Was one or more VOA surrogate recovery outside of contract specifications for any sample or method blank?	☐	☒	☐
If yes, were samples re-analyzed?	☐	☐	☒
Were method blanks re-analyzed?	☐	☐	☒
ACTION: If surrogate recoveries are > 10% but all do not meet SOW specifications:			
1. Flag all positive results as estimated ("J").			
2. Flag all non-detects as estimated detection limits ("UJ").			
Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and re-analyses. Check the internal standard areas.			
3.5 Are there any transcription/calculation errors between raw data and Form II?	☒	☐	☐

YES NO N/A

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

4.0 Matrix Spikes (Form III)

4.1 Is the Matrix Spike Duplicate/Recovery Form (Form III) present?

☒	☐	☐
-------------------------------------	--------------------------	--------------------------

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Low Water

☒	☐	☐
-------------------------------------	--------------------------	--------------------------

b. Med Water

☐	☐	☒
--------------------------	--------------------------	-------------------------------------

c. Low Soil

☐	☐	☒
--------------------------	--------------------------	-------------------------------------

d. Med Soil

☐	☐	☒
--------------------------	--------------------------	-------------------------------------

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

4.3 How many VOA spike recoveries are outside QC limits?

WaterSoils

<u>0</u> out of 10	<u>N/A</u> out of 10
--------------------	----------------------

4.4 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

WaterSoils

<u>0</u> out of 5	<u>N/A</u> out of 5
-------------------	---------------------

ACTION: If MS and MSD both have less than 10% recovery for an analyte, negative results for that analyte should be rejected, and positive results should be flagged "J". The above applies only to the sample used for the MS/MSD analysis. Use professional judgement in applying this criterion to other samples in the package.

		YES	NO	N/A
5.0	<u>Blanks (Form IV)</u>			
5.1	Is the Method Blank Summary (Form IV) present?	☒	—	—
5.2	Frequency of Analysis: for the analysis of VOA TCL compounds, has a reagent/method blank been analyzed for each set of samples or every 20 samples of similar matrix (low water, med water, low soil, medium soil), whichever is more frequent?	☒	—	—
5.3	Has a VOA instrument blank been analyzed at least once every twelve hours for each GC/MS system used?	☒	—	—
	ACTION: If any method blank data are missing, call lab for explanation/resubmittal. If not available, reject all positive data ("R").			
5.4	Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.			
	Is the chromatographic performance (baseline stability) for each instrument acceptable for VOAs?	☒	—	—
	ACTION: Use professional judgement to determine the effect on the data.			
6.0	<u>Contamination</u>			
	NOTE: "Water blanks" and "distilled water blanks" are <u>not</u> used to qualify data. Do not confuse them with the other QC blanks discussed below.			
6.1	Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for VOAs? When applied as described below, the contaminant concentration in these blanks is multiplied by the sample Dilution Factor.	☒	☐	—
6.2	Do any field/trip/rinse blanks have positive VOA results (TCL and/or TIC)?	☒	☐	—
	ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)			

NOTE: Only field/rinse blanks taken the same day as the samples are used to qualify data. Trip blanks are used to qualify those samples with which they were shipped. Blanks may not be qualified because of contamination in another blank. Blanks may be qualified for surrogate, spectral, tuning or calibration QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

	Sample conc > CRQL but < 10x blank	Sample conc < CRQL & is < 10x blank value	Sample conc > CRQL value & > 10x blank value
Methylene chloride Acetone Toluene 2-butanone	Flag sample result with a 'U'; cross out 'B' flag	Reject sample result and report CRQL; cross out 'B' flag	No qualification is needed
	Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5 x blank value	Sample conc > CRQL value & > 5 blank value
Other Contaminants	Flag sample result with a 'U'; cross out 'B' flag	Reject sample result and report CRQL; cross out 'B' flag	No qualification is needed

YES NO N/A

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R" (unusable).

6.3 Are there field/rinse/equipment blanks associated with every sample?

[] — ☒

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 GC/MS Tuning and Mass Calibration (Form V)

TUT 002 2112

7.1 Are the GC/MS Tuning and Mass Calibration Forms (Form V) present for Bromofluorobenzene (BFB)?

☒ — —

- | | YES | NO | N/A |
|--|-----|----|-----|
| 7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for BFB provided for each twelve hour shift? | [✓] | — | — |
| 7.3 Has a tuning performance compound been analyzed for every twelve hours of sample analysis per instrument? | [✓] | — | — |

ACTION: If any tuning data are missing, take action specified in 3.2 above.

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available.

DATE	TIME	INSTRUMENT	SAMPLE NUMBER

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

- 7.4 Have an ion abundance criteria been met for each instrument used? [✓] — —

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If tuning calibration is in error, flag all associated sample data as unusable ("R"), However, if expanded ion criteria are met (See 1988 Functional Guidelines), the data reviewer may accept data with appropriate qualifiers.

- 7.5 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.) — [✓] —

- 7.6 Have the appropriate number of significant figures (two) been reported? (check at least two values, but if errors are found, check more values.) [✓] — —

YES NO N/A

ACTION: If large errors exist, call lab for explanation/
resubmittal, make necessary corrections and note
errors under "Conclusions".

7.7 Are the spectra of mass calibration compound acceptable?

[☒] — —

ACTION: Use professional judgement to determine whether
associated data should be accepted, qualified,
or rejected.

8.0 Target Compound List (TCL) Analytes

8.1 Are the Organic Analysis Data Sheets (Form I VOA)
present with required header information on each
page, for each of the following:

a. Samples and/or fractions as appropriate

[☒] — —

b. Matrix spikes and matrix spike duplicates

[☒] — —

c. Blanks

[☒] — —

8.2 Are the VOA Reconstructed Ion Chromatograms, the mass
spectra for the identified compounds, and the data
system printouts (Quant Reports) included in the sample
package for each of the following?

a. Samples and/or fractions as appropriate

[☒] — —

b. Matrix spikes and matrix spike duplicates (Mass
spectra not required)

[☒] — —

c. Blanks

[☒] — —

ACTION: If any data are missing, take action
specified in 3.2 above.

8.3 Are the response factors shown in the Quant Report?

[] ☒ —

8.4 Is chromatographic performance acceptable with respect to:

Baseline stability

[☒] — —

Resolution

[☒] — —

YES NO N/A

Peak shape

☒ ☐ ☐

Full-scale graph (attenuation)

☒ ☐ ☐

Other: _____

☐ ☐ ☒

ACTION: Use professional judgement to determine the acceptability of the data.

8.5 Are the lab-generated standard mass spectra of the identified VOA compounds present for each sample?

☒ ☐ ☐

ACTION: If any mass spectra are missing, take action specified in 3.2 above. If Lab does not generate their own standard spectra, make note in "Contract Problems/Non-compliance".

8.6 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?

☒ ☐ ☐

8.7 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?

☒ ☐ ☐

8.8 Do sample and standard relative ion intensities agree within 20%?

☒ ☐ ☐

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identification were made, all such data should be rejected, flagged "N" (presumptive evidence of the presence of the compound) or changed to not detected (at the calculated detection limit).

9.0 Tentatively Identified Compounds (TICs)

9.1 Are all Tentatively Identified Compound Forms (Form I, Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "J" qualifier?

☐ ☒ ☐

9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

TUT 002 2115

a. Samples and/or fractions as appropriate

YES NO N/A
[✓] — —

b. Blanks

[✓] — —

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "J" qualifier if missing and "N" qualifier to all identified TIC compounds on Form I, Part B.

9.3 Are any TCL compounds (from any fraction) listed as TIC compounds (example: 1,2-dimethylbenzene is xylene - a VOA TCL - and should not be reported as a TIC)?

— [✓] —

ACTION: Flag with "R" any TCL compound listed as a TIC.

9.4 Are all ions present in the reference mass spectrum with a relatively greater 10% also present in the sample mass spectrum?

[✓] — —

9.5 Do TIC and "best match" standard relative ion intensities agree within 20%?

[✓] — —

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate.

10.0 Compound Quantitation and Reported Detection Limits

10.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, RRF were used to calculate Form I result. Were any errors found?

✓ [] —

10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture?

[✓] — —

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and

YES NO N/A

note errors under "Conclusions".

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from other diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with data from the analysis of diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

11.0 Standard Data (GC/MS)

11.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant. Reports) present for initial and continuing calibration?

☒ ☐ ☐

ACTION: If any calibration standard data are missing, take action specified in 3.2 above.

12.0 GC/MS Initial Calibration (Form VI)

12.1 Are the Initial Calibration Forms (Form VI) present and complete for the volatile fraction?

☒ ☐ ☐

12.2 Are response factors stable for volatiles over the concentration range of the calibration (RSD < 30%)?

☐ ☒ ☐

ACTION: Circle all outliers in red.

ACTION: When RSD > 30%, non-detects may be qualified using professional judgement. Flag all positive results "J". When RSD > 90%, flag all non-detects as unusable ("R"). (Region II policy.)

12.3 Do any compounds have an average < 0.05?

☒ ☐ ☐

ACTION: Circle all outliers in red.

ACTION: If any volatile compound has an average

YES NO N/A

RRF < 0.05, flag positive results for that compound as estimated ("J"), and flag non-detects for that compound as unusable ("R").

- 12.4 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD? (Check at least two values but if errors are found, check more.)

— [✓] —

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

13.0 GC/MS Continuing Calibration (Form VII)

- 13.1 Are the Continuing Calibration Forms (Form VII) present and complete for the volatile fraction?

[✓] — —

- 13.2 Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?

[✓] — —

ACTION: List below all sample analyses that were not within twelve hours of the previous continuing calibration analysis.

ACTION: If any forms are missing or no continuing calibration standard has been analyzed within twelve hours of every sample analysis, call lab for explanation/resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable ("R").

- 13.3 Do any continuing calibration standard compounds have a RRF < 0.05?

— [✓] —

ACTION: Circle all outliers in red.

ACTION: If any volatile compound has a RRF < 0.05, flag positive results for that compound as estimated ("J"), and flag non-detects for

YES NO N/A

that compound as unusable ("R").

13.4 Do any compounds have a % difference between initial and continuing calibration RRF > 25%?

☒ ☐ ☐

ACTION: Circle all outliers in red and qualify associated sample data as outlined in the table below:

% DIFFERENCE

25-50	50-90	> 90
'J' positive results, no action for non detects	'J' positive results, 'UJ' non detects	'J' positive results, "R" non detects

13.5 Are there any transcription/calculation errors in the reporting of average response factors (RRFs) or difference (%D) between initial and continuing RRFs? (Check at least two values but if errors are found, check more.)

☐ ☒ ☐14.0 Internal Standards (Form VIII)

14.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits for each continuing calibration?

☒ ☐ ☐

ACTION: List all the outliers below.

Sample #	Internal Std	Area	Lower Limit	Upper Limit
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

(Attach additional sheets if necessary.)

ACTION: If the internal standard area count is outside the upper

YES NO N/A

lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard. If extremely low area counts are reported, or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable ("R").

14.2 Are the retention times of the internal standards within 30 seconds of the associated calibration standard?

[] ☒ —

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for VOA analysis?

☒ [] —

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

YES NO N/A

Part B: BNA Analyses1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Forms present for all samples?

[] — —

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or Lab Narrative indicate any problems with the sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

— [] —

ACTION: Use professional judgement to evaluate the effect on the quality of the data.

ACTION: If any sample analyzed as a soil contains more than 50% water, all data should be flagged as estimated (J).

2.0 Holding Times

2.1 Have any BNA holding times, determined from date of collection to date of extraction, been exceeded?

— [] —

Samples for BNA analysis, both soils and waters, must be extracted within seven days of the date of collection. Extracts must be analyzed within 40 days of the date of extraction.

Table of Holding Time Violations

(See Traffic Report)

Sample	Sample Matrix	Date Sampled	Date Lab Received	Date Extracted	Date Analyzed
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

	YES	NO	N/A
ACTION: If holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.			

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon reanalysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. The reviewer may determine that non-detect data are unusable ("R").

3.0 Surrogate Recovery (Form II)

3.1 Are the BNA Surrogate Recovery Summaries (Form II) present for each of the following matrices:

a. Low Water	☐	☐	☐
b. Med Water	☐	☐	☐
c. Low Soil	☐	☐	☐
d. Med Soil	☐	☐	☐

3.2 Are all the BNA samples listed on the appropriate Surrogate Recovery Summaries for each of the following matrices:

a. Low Water	☐	☐	☐
b. Med Water	☐	☐	☐
c. Low Soil	☐	☐	☐
d. Med Soil	☐	☐	☐

ACTION: Call lab for explanation/resubmittals. If missing deliverables are unavailable, document effect on data under "Conclusions" section of reviewer narrative.

3.3 Were outliers marked correctly with an asterisk?	☐	☐	☐
--	--------------------------	--------------------------	--------------------------

ACTION: Circle all outliers in red.

3.4 Were two or more base-neutral OR acid surrogate recoveries out of specification for any sample or method blank?

YES NO N/A

— [] —

If yes, were samples re-analyzed?

[] — —

Were method blanks re-analyzed?

[] — —

ACTION: If all BNA surrogate recoveries are > 10% but two within the base-neutral or acid fraction do not meet SOW specifications, for the affected fraction only (i.e. base-neutral OR acid compounds):

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ").

If any base-neutral OR acid surrogate has a recovery of < 10%:

1. Flag all positive results for that fraction (i.e. all acid OR base-neutral compounds) "J".
2. Flag all non-detects for that fraction "R".

Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and re-analyses. Check the internal standard area.

3.5 Are there any transcription/calculation errors between raw data and Form II.

— [] —

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

4.0 Matrix Spikes (Form III)

4.1 Is the Matrix Spike Duplicate/Recovery Form (Form III) present?

[] — —

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

	YES	NO	N/A
a. Low Water	☐	—	—
b. Med Water	☐	—	—
c. Low Soil	☐	—	—
d. Med Soil	☐	—	—

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

4.3 How many BNA spike recoveries are outside QC limits?

<u>Water</u>	<u>Soils</u>
_____ out of 22	_____ out of 22

4.4 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

<u>Water</u>	<u>Soils</u>
_____ out of 11	_____ out of 11

ACTION: If MS and MSD both have less than 10% recovery for an analyte, negative results for that analyte should be rejected, and positive results should be flagged "J". The above applies only to the sample used for MS/MSD analysis. Use professional judgement in applying this criterion to other samples.

5.0 Blanks (Form IV)

5.1 Is the Method Blank Summary (Form IV) present? ☐ — —

5.2 Frequency of Analysis: for the analysis of BNA TCL compounds, has a reagent/method blank been analyzed for each set of samples or every 20 samples of similar matrix (low water, med water, low soil, med soil), whichever is more frequent? ☐ — —

5.3 Has a BNA instrument blank been analyzed for each GC/MS system used?

YES NO N/A

[] — —

ACTION: If any method blank data are missing, call lab for explanation/resubmittal. If not available, reject all associated positive data ("R").

5.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.

Is the chromatographic performance (baseline stability) for each instrument acceptable for VOAs?

[] — —

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks" and "distilled water blanks" are validated like any other sample and not used to qualify data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for BNAs? When applied as described below, the contaminant concentration in these blanks is multiplied by the sample Dilution Factor.

— [] —

6.2 Do any field/rinse blanks have positive BNA results (TCL and/or TIC)?

— [] —

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: Only field/rinse blanks taken the same day as the samples are used to qualify data. Blanks may not be qualified because of contamination in another blank. Blanks may be qualified for surrogate, spectral, tuning or calibration QC problems.

YES NO N/A

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

	Sample conc > CRQL but < 10x blank	Sample conc < CRQL & is < 10x blank value	Sample conc > CRQL value & > 10x blank value
Common Phthalate Esters	Flag sample result with a 'U'; cross out 'B' flag	Reject sample result and report CRQL; cross out 'B' flag	No qualification is needed
	Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5 x blank value	Sample conc > CRQL value & > 5 blank value
Other Contaminants	Flag sample result with a 'U'; cross out 'B' flag	Reject sample result and report CRQL; cross out 'B' flag	No qualification is needed

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R" (unusable).

6.3 Are there field/rinse/equipment blanks associated with every sample? ☐ ☐ ☐

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank.
Exception: samples taken from a drinking water tap not have associated field blanks.

7.0 GC/MS Tuning and Mass Calibration (Form V)

7.1 Are the GC/MS Tuning and Mass Calibration Forms (Form V) present for Decafluorotriphenylphosphine (DFTPP)? ☐ ☐ ☐

7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each twelve hour shift? ☐ ☐ ☐

7.3 Has a tuning performance compound been analyzed for every twelve hours of sample analysis per instrument? ☐ ☐ ☐

YES NO N/A

ACTION: If any tuning data are missing, take action specified in 3.2 above.

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available.

DATE	TIME	INSTRUMENT	SAMPLE NUMBER

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

7.4 Have the ion abundance criteria been met for each instrument used? ☐ ☐ ☐

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If tuning calibration is in error, flag all associated sample data as unusable ("R"). However, if expanded ion criteria are met (See 1988 Functional Guidelines), the data review may accept data with appropriate qualifiers.

7.5 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more values.) ☐ ☐ ☐

7.6 Have the appropriate number of significant figures (two) been reported? (Check at least two values, but if errors are found, check more values.) ☐ ☐ ☐

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and note errors under "Conclusions".

7.7 Are the spectra of the mass calibration compound acceptable? ☐ ☐ ☐

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

YES NO N/A

8.0 Target Compound List (TCL) Analytes

8.1 Are the Organic Analysis Data Sheets (Form I BNA) present with required header information on each page, for each of the following:

- | | | | |
|--|--------------------------|--------------------------|--------------------------|
| a. Samples and/or fractions | ☐ | ☐ | ☐ |
| b. Matrix spikes and matrix spike duplicates | ☐ | ☐ | ☐ |
| c. Blanks | ☐ | ☐ | ☐ |

8.2 Are the BNA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?

- | | | | |
|---|--------------------------|--------------------------|--------------------------|
| a. Samples and/or fractions as appropriate | ☐ | ☐ | ☐ |
| b. Matrix spikes and matrix spike duplicates
(Mass spectra not required) | ☐ | ☐ | ☐ |
| c. Blanks | ☐ | ☐ | ☐ |

ACTION: If any data are missing, take action specified in 3.2 above.

8.3 Are the response factors shown in the Quant Report? ☐ ☐ ☐

8.4 Is chromatographic performance acceptable with respect to:

- | | | | |
|--------------------------------|--------------------------|--------------------------|--------------------------|
| Baseline stability | ☐ | ☐ | ☐ |
| Resolution | ☐ | ☐ | ☐ |
| Peak shape | ☐ | ☐ | ☐ |
| Full-scale graph (attenuation) | ☐ | ☐ | ☐ |
| Other: _____ | ☐ | ☐ | ☐ |

ACTION: Use professional judgement to determine the acceptability of the data.

- | | YES | NO | N/A |
|---|--------------------------|--------------------------|--------------------------|
| 8.5 Are the lab-generated standard mass spectra of the identified BNA compounds present for each sample? | ☐ | ☐ | ☐ |
| 8.6 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration? | ☐ | ☐ | ☐ |
| 8.7 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum? | ☐ | ☐ | ☐ |
| 8.8 Do sample and standard relative ion intensities agree within 20%? | ☐ | ☐ | ☐ |

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected, flagged "N" (presumptive evidence of the presence of the compound) or changed to not detected (at the calculated detection limit).

9.0 Tentatively Identified Compounds (TIC)

- | | | | |
|--|--------------------------|--------------------------|--------------------------|
| 9.1 Are all Tentatively Identified Compound Forms (Form I, Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "J" qualifier? | ☐ | ☐ | ☐ |
| 9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following: | | | |
| a. Samples and/or fractions as appropriate | ☐ | ☐ | ☐ |
| b. Blanks | ☐ | ☐ | ☐ |

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "J" qualifier if missing and "N" qualifier to all identified TIC compounds on Form I, Part B.

- | | | | |
|---|--------------------------|--------------------------|--------------------------|
| 9.3 Are any TCL compounds (from any fraction) listed as TIC compounds (example: 1,2-dimethylbenzene is xylene -- a VOA TCL -- and should not be reported as a TIC)? | ☐ | ☐ | ☐ |
|---|--------------------------|--------------------------|--------------------------|

ACTION: Flag with "R" any TCL compound listed as a TIC.

	YES	NO	N/A
9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum?	☐	—	—

9.5 Do TIC and "best match" standard relative ion intensities agree within 20%?	☐	—	—
---	--------------------------	---	---

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate.

10.0 Compound Quantitation and Reported Detection Limits

10.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found?	—	☐	—
--	---	--------------------------	---

10.2 Are the CRQLs adjusted to reflect dilutions and, for soils, sample moisture?	☐	—	—
---	--------------------------	---	---

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with data from the analysis of diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

YES NO N/A

11.0 Standards Data (GC/MS)

- 11.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant. Reports) present for initial and continuing calibration?

[] — —

12.0 GC/MS Initial Calibration (Form VI)

- 12.1 Are the Initial Calibration Forms (Form VI) present and complete for the BNA fraction?

[] — —

ACTION: If any calibration standard forms are missing, take action specified in 3.2 above.

- 12.2 Are response factors stable for BNAs over the concentration range of the calibration (RSD < 30%)?

[] — —

ACTION: Circle all outliers in red.

ACTION: When RSD > 30%, non-detects may be qualified using professional judgement. Flag all positive results "J". When RSD > 90%, flag all non-detects as unusable ("R"). (Region II policy.)

- 12.3 Do any compounds have a RRF < 0.05?

— [] —

ACTION: Circle all outliers in red.

ACTION: If any BNA compound has an average RRF < 0.05, flag positive results for that compound as estimated ("J"), and flag non-detects for that compound as unusable ("R").

- 12.4 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD? (Check at least two values but if errors are found, check more.)

— [] —

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

YES NO N/A

13.0 GC/MS Continuing Calibration (Form VII)

13.1 Are the Continuing Calibration Forms (Form VII) present and complete for the BNA fraction?

[] — —

13.2 Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?

[] — —

ACTION: List below all sample analyses that were not within twelve hours of the previous continuing calibration analysis.

ACTION: If any forms are missing or no continuing calibration standard has been analyzed within twelve hours of every sample analysis, call lab for explanation/resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable ("R").

13.3 Do any continuing calibration standard compounds have a RRF < 0.05? — [] —

ACTION: Circle all outliers in red.

ACTION: If any BNA compound has a RRF < 0.05, flag positive results for that compound as estimated ("J"), and flag non-detects for that compound as unusable ("R").

13.4 Do any compounds have a % difference between initial and continuing calibration RRF > 25%? — [] —

ACTION: Circle all outliers in red and qualify associated sample data as outlined in the table below:

% DIFFERENCE

25-50	50-90	>90
'J' positive results, no action for non detects	'J' positive results, 'UJ' non detects	'J' positive results, "R" non detects

YES NO N/A

- 13.5 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or difference (%D) between initial and continuing RRFs? (check at least two values but if errors are found, check more.)

— [] —

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

14.0 Internal Standards (Form VIII)

- 14.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits for each continuing calibration?

[] — —

ACTION: List all the outliers below.

Sample #	Internal Std	Area	Lower Limit	Upper Limit
----------	--------------	------	-------------	-------------

_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

(Attach additional sheets if necessary.)

ACTION: If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard. If extremely low area counts are reported, or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable ("R").

- 14.2 Are the retention times of the internal standards within 30 seconds of the associated calibration standard?

[] — —

YES NO N/A

ACTION: Professional judgement should be used to qualify data retention times differ by more than 30 seconds.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for BNA analysis?

[] — —

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

YES NO N/A

PART C: PESTICIDE/PCB ANALYSES**1.0 Traffic Reports and laboratory Narrative**

- 1.1 Are the Traffic Report Forms present for all samples?

[] — —

ACTION: If no, contact lab for replace of missing or illegible copies.

- 1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

— [] —

ACTION: Use professional judgement to evaluate the effect on the quality of the data.

ACTION: If any sample analyzed as a soil contains more than 50% water, all data should be flagged as estimated (J).

2.0 Holding Times

- 2.1 Have any PEST/PCB holding times, determined from date of collection to date of extraction, been exceeded?

— [] —

Samples for PEST/PCB analysis, both soils and waters, must be extracted within seven days of the date of collection. Extracts must be analyzed within 40 days of the date of extraction.

3.0 Surrogate Recovery (Form II)

- 3.1 Are the PEST/PCB Surrogate Recovery Summaries (Form II) present for each of the following matrices:

a. Low Water

[] — —

b. Med Water

[] — —

c. Low Soil

[] — —

d. Med Soil

[] — —

- 3.2 Are all the PEST/PCB samples listed on the appropriate Surrogate Recovery Summaries for each of the following matrices:

	YES	NO	N/A
a. Low Water	☐	☐	☐
b. Med Water	☐	☐	☐
c. Low Soil	☐	☐	☐
d. Med Soil	☐	☐	☐

ACTION: Call lab for explanation/resubmittals. If missing deliverables are unavailable, document effect on data under "Conclusions" section of reviewer narrative.

3.3 Were outliers marked correctly with an asterisk? ☐ ☐ ☐

ACTION: Circle all outliers in red.

3.4 Was surrogate (DBC) recovery outside of the contract specification for any sample or blank? ☐ ☐ ☐

ACTION: No qualification is done if surrogates are diluted beyond detection. If recovery is below contract limit (but above zero), flag all results for that sample "J". If recovery is zero, flag positive results "J" and non-detects "R". If recovery for the blank is zero, flag non-detects for all associated samples "R". If recovery is above contract limit, flag all positive results for that sample "J", unless in the reviewers professional judgement the high recovery is due to co-eluting interference (check the associated blank - if recovery is high there also, flag the sample data).

3.5 Are there any transcription/calculation errors between raw data and Form II? ☐ ☐ ☐

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

4.0 Matrix Spikes (Form III)

4.1 Is the Matrix Spike Duplicate/Recovery Form (Form III) present? ☐ ☐ ☐

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

	YES	NO	N/A
a. Low Water	☐	☐	☐
b. Med Water	☐	☐	☐
c. Low Soil	☐	☐	☐
d. Med Soil	☐	☐	☐

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

4.3 How many PEST/PCB spike recoveries are outside QC limits?

Water

Soils

_____ out of 12 _____ out of 12

4.4 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

Water

Soils

_____ out of 6 _____ out of 6

ACTION: If MS and MSD both have less than zero recovery for an analyte, negative results for that analyte should be rejected, and positive results should be flagged "J". The above applies only to the sample used for MS/MSD analysis. Use professional judgement in applying this criterion to other samples.

5.0 Blanks (Form IV)

5.1 Is the Method Blank Summary (Form IV) present? ☐ ☐ ☐

5.2 Frequency of Analysis: for the analysis of Pesticide TCL compounds, has a reagent/method blank been analyzed for each set of samples or every 20 samples of similar matrix (low water, med water, low soil, medium soil), whichever is more frequent? ☐ ☐ ☐

5.3 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts.

TUT 002 2137

Is the chromatographic performance (baseline stability) for each instrument acceptable for PEST/PCBs?

YES NO N/A

[] — —

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/instrument/reagent blanks have positive results for PEST/PCBs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample Dilution Factor.

— [] —

6.2 Do any field/rinse blanks have positive PEST/PCB results?

— [] —

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: Only field/rinse blanks taken the same day as the samples are used to qualify data. Blanks may not be qualified for surrogate, spectral, tuning or calibration QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL value & > 5x blank value
Flag sample result with a 'U'; cross out 'B' flag	Reject sample result and report CRQL; cross out 'B' flag	No qualification is needed

6.3 Are there field/rinse/equipment blanks associated with every sample?

YES NO N/A

[] — —

7.0 Calibration and GC Performance

7.1 Are the following Gas Chromatograms and Data System Printouts for both Primary and Confirmation (confirmation standards are not required if there are no positive results above CRQL) column present:

a. Evaluation Standard Mix A

[] — —

b. Evaluation Standard Mix B

[] — —

c. Evaluation Standard Mix C

[] — —

d. Individual Standard Mix A

[] — —

e. Individual Standard Mix B

[] — —

f. Multi-component Pesticides Toxaphene & Chlordane

[] — —

g. Aroclors 1016/1260

[] — —

h. Aroclors 1221, 1232, 1242, 1248, 1254

[] — —

ACTION: If no, take action specified in 3.2 above.

7.2 Is Form VIII Pest-1 present and complete for each GC column (primary and confirmation) and each 72 hour sequence of analyses?

[] — —

ACTION: If no, take action specified in 3.2 above.

7.3 Are there any transcription/calculation errors between raw data and Form VIII?

— [] —

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

7.4 Has the total breakdown on quantitation or confirmation column exceeded 20% for DDT?

— [] —

- for Endrin?

[] —

or if Endrin aldehyde and 4,4'-DDD co-elute and there is a peak at their retention time, has the combined DDT and Endrin breakdown exceeded 20%?

YES NO N/A

— [] —

ACTION:

- a. If DDT breakdown is greater than 20% on quantitation column beginning with the samples following the last in control standard:
1. Flag all positive DDT results "J".
 2. If DDT was not detected but DDD and/or DDE are positive, flag the DDT non-detect "R".
 3. Flag positive DDD and DDE results "JN".
 4. If DDT breakdown is > 20% on confirmation column and DDT is identified on quantitation column but not on confirmation column, use professional judgement to determine whether DDT should be reported on Form I (if reported, flag result "N").
- b. If Endrin breakdown is > 20% on quantitation column, beginning with the samples following the last in control standard:
1. Flag all positive Endrin results "J".
 2. If Endrin was not detected, but Endrin Aldehyde and/or Endrin Ketone are positive, flag the Endrin non-detect "R".
 3. Flag Endrin Ketone positive results "JN".
 4. If Endrin breakdown is > 20% on confirmation column and Endrin is identified on quantitation column but not on confirmation column, use professional judgement to determine whether Endrin should be reported on Form I (if reported, flag result "N").
- c. If the combined breakdown is used (it can only be used if the conditions in 7.4 above are met) and is > 20% on quantitation column beginning with the last in control standard, take the actions specified in 7.4 a and b above. If the combined breakdown is > 20% on confirmation column and Endrin or DDT is identified on quantitation column but not on confirmation column, use professional judgement to determine whether Endrin or DDT should be reported on Form I (if reported, flag result "N").

7.5 Is the linearity check RSD of all four calibration factors < 10% for the quantitation column?

[] — —

ACTION: If no, flag positive hits for all pesticide and PCB analytes "J" for all associated samples. Do not flag toxaphene or DDT if they are quantified from a 3-pc calibration curve.

YES NO N/A

- 7.6 Is the % difference between EVAL A and each analysis (quantitation and confirmation) DBC retention time within QC limits (2% for packed column 0.3% for capillary [I.D. < 0.32 mm], 1% for megabore [0.32 < 2 mm])?

[] — —

ACTION: DBC retention time cannot be evaluated if DBC is not detected. If it is present and has a retention time out of QC limits, then use professional judgement to determine the reliability of the analysis and flag results "R", if appropriate.

- 7.7 Was the proper analytical sequence followed for each 72 hour period of analyses (page PEST D-36 in 8/87 SOW).

[] — —

ACTION: If no, use professional judgement to determine the severity of the effect on the data and accept or reject accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the calibration was also out of limits.

8.0 Pesticide/PCB Standards Summary

- 8.1 Is Form IX present and complete for each GC column and 72 hr. sequence of analyses?

[] — —

ACTION: If no, take action specified in 3.2 above.

- 8.2 Are there any transcription/calculation errors between raw data and Form IX?

— [] —

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

- 8.3 Is DDT retention time for packed columns > 12 min (except OV-1 and OV-101 columns)?

[] — —

ACTION: If no, check that there is adequate resolution between individual components. If not, flag results for compounds that interfere with each other (co-elute) "R".

- 8.4 Do all standard retention times fall within the windows established for the first IND A and IND B analyses?

[] — —

YES NO N/A

ACTION: Beginning with the samples following the last in control standard, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and DBC is visible non-detects are valid. If peaks are present and cannot be identified through "pattern recognition" or a consistent shift in standard retention times, flag all affected compound results "R".

- 8.5 Are the continuing calibration standard calibration factors within 15% (for confirmation column) of the initial (at beginning of 72 hr sequence) calibration factors? ☐ ☐ ☐

ACTION: If no, flag all associated positive results "J". Use professional judgement to determine whether or not to flag non-detects.

9.0 Pesticide/PCB Identification

- 9.1 Is Form X complete for every sample in which a pesticide or PCB was detected? ☐ ☐ ☐

ACTION: If no, take action specified in 3.2 above.

- 9.2 Are there any transcription errors between raw data and Form X? ☐ ☐ ☐

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

- 9.3 Are retention times of sample compounds within the calculated retention time windows for both quantitation and confirmation analyses? ☐ ☐ ☐

Was GC/MS confirmation provided when required (when compound concentration is > 10 ug/ml in final extract)? ☐ ☐ ☐

ACTION: Reject ("R") all positive results (meeting quantitation column criteria, but missing confirmation by a second column or GC/MS (if appropriate). Also, reject ("R") all positive results not meeting retention time window criteria unless associated standard compounds are similarly biased (i.e. base on RRT to DBC).

YES NO N/A

- 9.4 Check chromatograms for false negatives, especially for the multiple peak components toxaphene and PCB's. Were there any false negatives?

— [] —

ACTION: If appropriate PCB standards were not analyzed, or if the lab performed no confirmation analysis, flag the appropriate data with an "R".

10.0 Compound Quantitation and Reported Detection Limits

- 10.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Were any errors found?

— [] —

NOTE: Simple peak pesticide results can be checked for rough agreement between quantitative results obtained on the two GC columns. The reviewer should use professional judgement to decide whether a much larger concentration obtained on one column versus the other indicate the presence of an interfering compound. If an interfering compound is indicated, the lower of the two values should be reported and qualified as presumptively present at an estimated quantity ("JN"). This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate that the presence of interferences has obscured the attempt at a second column confirmation.

- 10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture?

— [] —

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with data from the analysis of diluted

YES NO N/A

sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

11.0 Chromatogram Quality

11.1 Were baselines stable?

[] — —

11.2 Were any electropositive displacement (negative peaks) or unusual peaks seen?

— [] —

11.3 Were early eluting peaks (for early eluting analytes) resolved to baseline?

[] — —

ACTION: For 11.1 and 11.2, comment only. For 11.3, reject ("R") those analytes that are not sufficiently resolved.

12.0 Field Duplicates

12.1 Were any field duplicates submitted for PEST/PCB analysis?

[] — —

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

**SDG DEDE WEL (CEIMAC #910062)
TUTU WELLS SITE**

CONTAMINATED METHOD BLANKS AND ASSOCIATED SAMPLES

VBLK01	Rodrigues
VBLK02	Dede, Dench, Devcon I, Devcon III (including MS and MSD), Eglin I, Eglin II, Eglin III, Fernandez, Trip Blank (2/4/91), Trip Blank (2/5/91)
VBLK03	Smith
VBLK04	Eglin IV, Four Winds II, Hartman III
VBLK05	Matthias
VBLK08	Eglin II(DL)

**SDG DEDE WEL (CEIMAC #910062)
TUTU WELLS SITE**

CONTAMINATED TRIP BLANKS AND THEIR ASSOCIATED SAMPLES

Trip Blank (2/4/91)	Dede, Dench, Devcon I, Devcon III, Fernandez, Rodrigues
Trip Blank (2/5/91)	Eglin I, Eglin II, Eglin III, Eglin IV, Four Winds II, Hartman III, Matthias, Smith

910062/cblanks

TOTAL REVIEW
CLP DATA ASSESSMENT

Functional Guidelines for Evaluating Organics Analysis

Case No. 910062 SDG No. Dede LABORATORY Ceimic SITETutu TEIC

DATA ASSESSMENT:

The current functional guidelines (1988) for evaluating organic data have been applied.

All data are valid and acceptable except those analytes which have been qualified with a "J" (estimated), "U" (non-detects), "R" (unusable), or "JN" (presumptive evidence for the presence of the material at an estimated value). All action is detailed on the attached sheets.

Two facts should be noted by all data users. First, the "R" flag means that the associated value is unusable. In other words, due to significant QC problems the analysis is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on data tables because they cannot be relied upon, even as a last resort. The second fact to keep in mind is that no compound concentration, even if it has passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data but any value potentially contains error.

Reviewer's

Signature: Lidya Gn 2/2/10 Date: 5/1/91

Verified by: Juan Barrios / TUI Date: 5/1/91

1. HOLDING TIME:

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detects (sample quantitation limits) will be flagged as estimated, "J", or unusable, "R", if the holding times are grossly exceeded.

The following action was taken in the samples and analytes shown due to excessive holding time.

Holding time criteria was met for all samples of SDG Dede with the exception of the analyses for samples Four Winds II and the Eglin II reanalysis.

Four Winds II was received in the laboratory on February 6, 1991 in an unpreserved state although the sample chain of custody indicated that the volatile vials had been preserved with hydrochloric acid to a pH of 0.5. Information regarding the inaccuracy of the chain of custody preservation notation was relayed to the laboratory by the Geraghty & Miller project manager on February 7, 1991. The chain of custody was amended by the laboratory to reflect the unpreserved sample condition. As the sample was unpreserved, analysis within fourteen days was required for non-aromatic volatiles, whereas aromatic volatiles were to be analyzed within a 7 day period starting from sample collection. The initial analysis for sample Four Winds II was performed nine days from the sample collection date. Two days subsequent to the initial volatile analysis, the sample was reanalyzed to confirm values outside the Method 524.2 linear range for total 1,2-dichloroethene (1,2-DCE) and tetrachloroethene (PCE). Based on the dates of sample analysis with respect to the holding time criteria, all aromatic volatiles were qualified as estimated (J) in the sample Four Winds II. As the analysis for non-aromatic volatiles, including the reanalysis for total 1,2-DCE and PCE was performed within the specified 14 day holding time criteria, no other qualifications were applied to the sample data based on this criterion.

The reanalysis of sample Eglin II for 1,2-DCE and PCE values outside the linear range of Method 524.2 was performed 31 days outside of the holding time. The results for each of these compounds were qualified as estimated (J) in this sample.

2. BLANK CONTAMINATION

Quality assurance (QA) blanks, i.e., method, trip field, rinse and water blanks are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Trip blanks measure cross-contamination of samples during shipment. Field blanks measure cross-contamination of samples during field operations. If the concentration of the analyte is less than 5 times the blank contaminant level (10 times for the common contaminants), the analytes are qualified as non-detects, "U". The following analytes in the samples shown were qualified with "U" for these reasons:

A) Method blank contamination

All method blanks associated with the volatile analyses of SDG Dede were reviewed to determine laboratory contamination in the samples. Method blanks VBLK01, VBLK02, VBLK03, and VBLK04 were used in the low level Method 524.2 analysis of samples associated with this SDG. In the previously agreed to analysis of volatiles using the contract laboratory protocol (CLP) routine analytical services (RAS) methodology for those samples exceeding the linear range of Method 524.2, volatile blanks VBLK05, VBLK07, and VBLK08 were reviewed to assess laboratory contamination levels.

In method blank VBLK01, methylene chloride was detected at a concentration of 1 microgram per liter (ug/L). In the library search associated with the blank, 1,1,2-trichloro-1,2,2-trifluoroethane was identified at retention time (RT) 4.03 at an estimated level of 2 ug/L. Additionally, an unknown (tentatively identified as 2-ethyl-1-hexanol) was identified at RT 22.72 at an estimated concentration of 5 ug/L. Sample Rodrigues, associated with this method, had a similarly identified TIC at RT 22.82 at 3 ug/L. As this result did not meet the guideline with respect to blank contamination, this TIC was rejected (R) in sample well Rodrigues.

2-Ethyl-2-hexanol was tentatively identified in method blank VBLK02 at RT 22.79 at an estimated level of 4 ug/L. In samples Eglin II, Eglin III and both of the trip blanks associated with SDG Dede, 2-Ethyl-1-hexanol was identified and rejected as unusable (R) based on the validation criteria. All other samples associated with this method blank did not identify this TIC as a method blank contaminant. No TCL were detected in the analysis of VBLK02.

In the analysis of VBLK03, although no TCL analytes were detected, an unknown RT 22.70 (tentatively identified as 2-ethyl-1-hexanol) was found at an estimated

level of 9 ug/L. The TIC at 22.79 (2-ethyl-1-hexanol) in sample Smith was rejected as unusable (R) based on the 5x rule for assessing method blank contamination.

An unknown contaminant at RT 24.04 was detected in the analysis of method blank VBLK04 at a level of 3 ug/L. Using the validation guideline for non-routine laboratory contaminants, the TIC at RT 24.05 was rejected as unusable (R) in associated samples Eglin IV and Hartman III.

In the analysis of VBLK05 associated with the CLP RAS analysis of sample Matthias, methylene chloride was detected at an estimated concentration of 18 ug/L. As this level did not meet the criteria for evaluation of common lab contaminants, the sample result was changed to a non-detect analyte quantitation limit after correction for dilution. No TIC were found in the VBLK05 method blank.

In method blank VBLK08 used in the analysis of CLP RAS volatile organic compounds, methylene chloride and acetone were detected at 6 ug/L and 14 ug/L, respectively. The analysis of sample Eglin II (DL) was associated with VBLK08. As the sample levels did not meet the blank assessment criteria, both of these results were rejected in the Eglin II (D) analysis and sample results reported as non-detects at the appropriate quantitation limit. No TIC or unknowns were detected in the VBLK08 analysis.

In the analysis of VBLK07, no TCL or TIC were detected in the method blank analysis.

B) Trip blank contamination

The trip blank associated with samples collected on February 4, 1991 (Trip blank 2/4/91) was reported to contain low level contamination of methylene chloride (2 ug/L) and acetone (8 ug/L). Additionally, an unknown was detected at RT 22.79, but subsequently qualified as method blank contamination based on the analysis of VBLK02. The following samples have been qualified as non-detects for acetone and/or methylene chloride using the 10x criteria for common contaminants: Dede, Devcon I, Devcon III (MS and MSD) and Fernandez.

The trip blank associated with samples collected on February 5, 1991 (Trip blank 2/5/91) was reported to contain 3 ug/L of methylene chloride and 2 ug/L of

acetone. 2-Ethyl-1-hexanol was tentatively identified in the trip blank at RT 22.77 at an estimated concentration of 4 ug/L, but subsequently rejected on the basis of associated method blank contamination. The following samples were qualified as non-detects for methylene chloride levels based on the trip blank contamination: Eglin I, Eglin II, Eglin III and Eglin IV. The methylene chloride result in sample Matthias was previously assessed as a non-detect for methylene chloride on the basis of VBLK02.

3. MASS SPECTROMETER TUNING:

Tuning and performance criteria are established to ensure adequate mass resolution, proper identification of compounds, and to some degree, sufficient instrument sensitivity. These criteria are not sample specific. Instrument performance is determined using standard materials. Therefore, these criteria should be met in all circumstances. The tuning standard for volatile organics is bromofluorobenzene (BFB) and for semi-volatiles is decafluorotriphenyl-phosphine (DFTPP).

If the mass calibration is in error, all associated data will be classified as unusable, "R".

The mass calibration associated with volatile analyses for SDG Dede have all met the acceptance criteria to ensure adequate resolution, instrument response and proper compound identification.

4. CALIBRATION

Satisfactory instrument calibration is established to ensure that the instrument is capable of producing acceptable quantitative data. An initial calibration demonstrates that the instrument is capable of giving acceptable performance at the beginning of an experimental sequence. The continuing calibration checks document that the instrument is giving satisfactory daily performance.

A) RESPONSE FACTOR:

The response factor measures the instrument's response to specific chemical compounds. The response factor for the Target Compound List (TCL) must be ≥ 0.05 (a ratio of areas) in both the initial and continuing calibrations. A value < 0.05 indicates a serious detection and quantitation problem (poor sensitivity). Analytes detected in the sample will be qualified as estimated, "J". All non-detects for that compound will be rejected ("R").

The initial calibration for all low level drinking water analyses performed on 2/11/91 on GC/MS instrument ID MS2 resulted in response factors of < 0.05 for acetone and 2-butanone. All samples (with the exception of well sample Matthias and reanalyses for Eglin II and Four Winds II) have been qualified due to the deficient instrument response as described above. As none of the above resulted in positive hits, all associated sample data for these compounds has been qualified as unusable (R).

In the initial calibrations performed on 2/7/91 (instrument ID MS5) and 2/15/91 (instrument ID MS6) in support of CLP RAS volatile analyses, all response factors were greater than 0.05 and resulted in no data qualifications.

In the continuing calibration standard VSTD010 associated with the low level Method 524.2 analyses on 2/11/91 on instrument MS2, acetone and 2-butanone were qualified as rejected (R) in associated sample Rodrigues due to deficient response factors for these two compounds. These compounds were previously qualified as unusable (R) in all related samples due to deficient response factors observed in the initial calibration associated with low level analyses.

In the analysis of the daily standard VSTD010 injected on 2/12/91 on instrument ID MS2, the response factors for 2-hexanone, in addition to response factors for acetone and 2-butanone, were less than the 0.05 RF criteria. Sample data for Smith, associated with this standard, was qualified as unusable (R) for the noted compounds based on the standard deficiency.

In the continuing calibration standard VSTD010 analyzed on 2/13/91 on instrument ID MS2, the response factor for 2-butanone was < 0.05 . As the compound was previously assessed as unusable (R) for all low level analyses based on a deficient initial response factor, no additional qualifiers were applied to the associated samples.

In the analysis of the continuing calibration standard VSTD010 injected on 2/14/91 on instrument MS2, deficient response factors were noted for acetone and 2-butanone. Samples associated with this daily standard were already qualified and the sample results rejected (R) based on deficient initial calibration standard response factors for these compounds.

In the analysis of CLP RAS volatile analyses, the continuing calibration standard VSTD050 injected on 2/16/91 on instrument ID MS6 did not meet the minimum response factor criteria for 2-butanone. As this calibration was used solely for

the quantitation of 1,2-DCE and PCE in sample Four Winds II, no qualifications were applied to the sample data based on this criterion.

5. CALIBRATION:

A) PERCENT RELATIVE STANDARD DEVIATION (%RSD) AND PERCENT DIFFERENCE (%D):

Percent RSD is calculated from the initial calibration and is used to indicate the stability of the specific compound response factor over increasing concentration. Percent D compares the response factor of the continuing calibration check to the mean response factor (RRF) from the initial calibration. Percent D is a measure of the instrument's daily performance. Percent RSD must be <30% and %D must be <25%. A value outside of these limits indicates potential detection and quantitation errors. For these reasons, all positive results are flagged as estimated, "J" and non-detects are flagged "UJ" (if %D or RSD > 50%). If there is a gross deviation of %RSD and %D, the non-detects may be rejected ("R").

The criteria above were modified slightly to reflect the %D criteria permissible for analyses performed in accordance with Method 524.2. As per the low level method, the response factor in the continuing calibration must be within 30% of the mean value measured in the initial calibration. As such, no qualifications were made for compounds in the continuing calibration standards with %D of <30% which were used in quantitation of low level volatile analyses. All other criteria and subsequent qualifications remain unchanged with respect to %RSD and %D, including the 25%D criteria for continuing calibration verification for samples analyzed by the CLP RAS methodology.

In the initial calibration associated with low level drinking water analyses (analyzed 2/11/91 on instrument ID MS2), the %RSD for acetone and 2-hexanone were 51.5% and 31.5%, respectively. As the %RSD for 2-hexanone is less than 50% and all samples were non-detects for this compound, no data qualifiers were assessed. The acetone results remain unusable for all associated samples as a result of deficient initial response factors.

The %RSD for acetone in the initial calibrations on instruments MS5 (analyzed 2/7/91) and MS6 (analyzed 2/15/91) were each greater than 30%. Samples Matthias and Eglin II (DL) were analyzed on MS5 and the sample Four Winds II analyzed on MS6. These samples are associated with the deficient %RSD criteria on the respective instruments. No qualifers were applied to the acetone

results in any of the samples noted above due to the non-detect sample results found for this analyte.

In the analysis of the continuing calibration standard VSTD010 injected on 2/11/91 on instrument ID MS2, the %D were greater than 30% for the following:

<u>Compound</u>	<u>%D</u>
chloromethane	64.6
bromomethane	42.4
vinyl chloride	41.5
chloroethane	30.7
methylene chloride	43.2
acetone	56.0
carbon disulfide	25.8
2-butanone	91.9
carbon tetrachloride	38.3
vinyl acetate	102.4
trans-1,3-dichloropropene	78.4
benzene	35.3
2-hexanone	50.3
4-methyl-2-pentanone	39.4
1,1,2,2-tetrachloroethane	57.8
bromofluorobenzene	29.5

In sample Rodrigues, associated with this daily standard, acetone and 2-butanone were qualified previously as unusable (R) due to deficient initial response factors for these two compounds. The non-detect results for chloromethane, trans-1,3-dichloropropene and 1,1,2,2-tetrachloroethane were qualified as estimate (J) based on %D > 50% criterion for non-detected values. The vinyl acetate non-detect result was qualified as unusable due to %D greater than 90%.

The %D for 2-butanone (37.7%), trans-1,3-dichloropropene (61.1%), cis-1,3-dichloropropene (31.3%) and 4-methyl-2-pentanone (35.0%) were greater than 30% in the daily standard VSTD010 analyzed on 2/12/91 on instrument ID MS2. Sample Smith is associated with this continuing calibration standard. The non-detect result for trans-1,3-dichloropropene was qualified as estimated (J) as the %D was greater than 50%. The results for cis-1,3-dichloropropene and 4-methyl-2-pentanone remain unqualified as the sample resulted in non-detect values for these compounds and the respective %D were < 50%.

In the analysis of the continuing calibration standard VSTD010 (injected on 2/13/91 on instrument ID MS2), the %D for acetone (155%) and trans-1,3-dichloropropene (63.1%) were greater than 30%. All acetone values were assessed as unusable (R) due to a deficient initial response factor. The trans-1,3-dichloropropene non-detect results in all associated samples were qualified as estimated (UJ) as the %D recovery was in the 50-90% range. The following samples are associated with this standard: Dede, Dench, Devcon I, Devcon III (including MS and MSD), Eglin I, Eglin II, Eglin III, Fernandez, Trip blank (2/4/91) and Trip blank (2/5/91).

In the continuing calibration standard VSTD010 analyzed on 2/14/91 on instrument ID MS2, the following compounds resulted in %D greater than 30%:

<u>Compound</u>	<u>%D</u>
chloromethane	31.4
trans-1,3-dichloropropene	95.0

The trans-1,3-dichloropropene non-detect results were rejected as unusable (R) in sample Eglin IV and Four Winds II based on %D greater than 90% for this compound. The chloromethane results in these samples remain unqualified as the %D was less than 50% and the compound was not detected in either of the samples.

In the analysis of the continuing calibration standard VSTD050 injected on 2/12/91 on instrument ID MS5, the %D was greater than 25% for 1,2-dichloroethane (27.4%) and vinyl acetate (32.6%). Well sample Matthias was quantitated using this daily standard. As neither of these compounds were detected in the sample and %D is less than 50%, the non-detects remain unqualified.

The %D for chloromethane (29.8%), methylene chloride (26.4%), 1,2-dichloroethene (26.0%), carbon tetrachloride (26.7%) and vinyl acetate (63.7%) were greater than 25% in the continuing calibration standard injected on 3/22/91 on instrument MS5. The CLP RAS analysis of Eglin II for confirmation of 1,2-dichloroethene and tetrachlorethene was associated with this deficient daily standard. As such, the positive detect for 1,2-dichloroethene has been qualified as estimated (J) as per the applicable %D criterion with respect to positive detects and %D in the range of 26-50%. The non-detect for vinyl acetate has been qualified as estimated (UJ) based on %D > 50%. All other values remain qualified as previously assessed (estimated due to deficient holding time criteria)

as the CLP RAS analysis was only for 1,2-dichloroethene and tetrachlorethene result confirmation.

In the analysis of continuing calibration standard VSTD050 analyzed on 2/16/91 on instrument ID MS6, greater than 25 %D was noted for the following compounds:

<u>Compound</u>	<u>%D</u>
bromomethane	25.4
acetone	26.1
2-butanone	25.6

As none of these compounds were used in the quantitation of results associated with the CLP RAS analysis of Four Winds II, no data qualifications were applied.

It should be noted that all applicable data qualifiers that arose from deficient compound response factors, %RSD and %D criteria were applied to the Method 524.2 laboratory fortified blanks (LFB) and the respective volatile method blanks. No discussion of the added qualifiers was included in the above for these QC samples. However, sample results may be impacted and biased depending on the qualification of the LFB and the associated sample data. Refer to the more complete discussion in the System Performance and Overall Assessment.

6. SURROGATES:

All samples are spiked with surrogate compounds prior to sample preparation to evaluate overall laboratory performance and efficiency of the analytical technique. If the measured surrogate concentrations were outside contract specifications, qualifications were applied to the samples and analytes as shown below.

As specified in Method 524.2, bromofluorobenzene (BFB) and 1,2-dichlorobenzene-d4 (DCB-d4) were used for the surrogate compound spike additions in all samples analyzed by the low level drinking water method. The surrogate compounds were added at a final concentration of 1 ug/L. As noted in the quality control section dealing with establishment of laboratory precision and accuracy for the method, the mean accuracy, expressed as a percentage of the true value, should be 80-120% for most compounds and surrogates. Some analytes, particularly the early eluting gases and late eluting higher molecular weight compounds, are measured with less accuracy than other analytes. Using

the suggested guideline of 80-120% accuracy, the percent recovery was outside the recommended limits for the following samples and blanks:

<u>Sample</u>	<u>% BFB</u>	<u>%DCB-d4</u>
Dede	-	73
Devcon I	78	73
Fernandez	-	72
Four Winds II	-	73
Hartman III	-	123
Smith	-	77
Trip Blank(2/4/91)	-	124
BSTD001(LFB 2/11/91)	328	400

No sample reanalyses were performed based on the lack of firm guidance regarding surrogate recoveries in Method 524.2 analyses. As no major surrogate losses were noted, no data qualifications have been applied on the basis of surrogate recovery.

In the analysis of volatile organics by the CLP RAS volatile methodology, no deviations outside the specified contract surrogate limits were noted for any samples or associated blanks.

7. INTERNAL STANDARDS PERFORMANCE:

Internal standard (IS) performance criteria ensure that the GC/MS sensitivity and response are stable during every experimental run. The internal standard area count must not vary by more than a factor of 2 (-50% to +100%) from the associated continuing calibration standard. The retention time of the internal standard must not vary more than ± 30 seconds from the associated continuing calibration standard. If the area count is outside the (-50% to +100%) range of the associated standard, all of the positive results for compounds quantitated using that IS are qualified as estimated, "J", and all non-detects as "UJ", or "R" if there is a severe loss of sensitivity.

If an internal standard retention time varies by more than 30 seconds, the reviewer will use professional judgement to determine either partial or total rejection of the data for that sample fraction.

Fluorobenzene, added at a level of 1 ug/L, was used for the internal standard as required in low level analyses performed by Method 524.2. All samples analyzed for volatiles as per the CLP RAS protocol utilized bromochloromethane, 1,4-

difluorobenzene and chlorobenzene added at a concentration of 50 ug/L in the sample. As per the CLP RAS method, compound quantitation was performed using the response factor of the nearest internal standard.

The internal standard (IS) area counts and retention times (RT) for all samples and laboratory fortified blanks were within the quality control (QC) limits except for the following. In the analysis of low level analyses by Method 524.2, a retention time shift was noted for the internal standard midway through the analytical sequence for samples analyzed on 2/13/91 on instrument ID MS2. The shift was observed in the following samples: Dede, Dench, Devcon I, Devcon III (including MS and MSD), Eglin I and Fernandez. The analysis sequence was reviewed and it was determined that all samples injected after the sixth sample following the analysis of the continuing calibration standard displayed a consistent forward shift in internal standard retention time. As the samples shifted uniformly and are free of any interferences that may have produced such a shift, it is believed that the observed IS retention shift was caused by some instrumental change. Since the internal standard areas have remained fairly constant, there appeared to be no loss in sensitivity and response. As such, data qualifications were not applied to the samples listed above as the data quality was not impacted.

8. COMPOUND IDENTIFICATION:

A) VOLATILE AND SEMI-VOLATILE FRACTIONS:

TCL compounds are identified on the GC/MS by using the analyte's relative retention time (RRT) and by comparison to the ion spectra obtained from known standards. For the results to be a positive hit, the sample peak must be within ± 0.06 RRT units of the standard compound and have an ion spectra which has a ratio of the primary and secondary m/e intensities within 20% of that in the standard compound. For the tentatively identified compounds (TIC) the ion spectra must match accurately. In the cases where there is not an adequate ion spectrum match, the laboratory may have provided false positive identifications.

TCL compound identification by GC/MS was based on comparison of the analyte RRT and ion spectra to those obtained from known spectra. Positive hits were reviewed for all samples and the following discrepancies were noted in meeting the Relative Retention Time (RRT) criteria ± 0.06 .

In the total analysis of 1,2-dichloroethene (1,2-DCE), RRT criteria was exceeded in the Method 524.2 analyses for samples Eglin I, Eglin III, Eglin IV and Smith.

Calibration for the total 1,2-DCE analyses (cis and trans isomers) was based on the trans isomer for both the Method 524.2 and CLP RAS analyses. Quantitation of the cis isomer, if present, was based on the trans isomer response. This is the standard calibration and quantitation practice for volatile analyses using the CLP RAS methodology as the cis and trans isomers are not resolvable. Although separation and quantitation of the isomers can be achieved by Method 524.2 analysis, for the purposes of this site, 1,2-DCE was to be quantitated and reported as the total of the two isomers. As such, the exceeded RRT in the samples listed above, was potentially due to the presence of the cis-1,2-DCE isomer. As the calibration standards did not include the cis isomer, sample results were calculated using the trans isomer response factor. Based on this, the positive detects in samples Eglin I, Eglin III, Eglin IV and Smith , believed to be the result of cis isomer concentrations in the samples as tentatively identified by the Method 524.2 analyses, are considered estimated values and qualified as such (J).

9. MATRIX SPIKE/SPIKE DUPLICATE, MS/MSD:

The MS/MSD data are generated to determine the long-term precision and accuracy of the analytical method in various matrices. The MS/MID may be used in conjunction with other CC criteria for some additional qualification of the data.

In the determination of accuracy, sample Devcon III was used to determine whether the sample matrix contributed bias to the analytical results. The sample was spiked at 10 ug/L for the following analytes: 1,1-dichloroethene, trichloroethene, benzene, toluene and chlorobenzene. All matrix recoveries and relative percent difference were within the specified CLP RAS contract limits. As such, qualifications due to matrix spike bias were not applied to any sample results.

10. OTHER QC DATA OUT OF SPECIFICATION:

A) Sample Receipt

Several volatile organic samples [Devcon I, Devcon III (QC samples only) Dench] received at the laboratory contained air bubbles. In this event, the data validation SOP requires that all positive detections be assumed to be minimum values and that all non-detections be rejected (R) based on the assumption that all VOCs in the water would diffuse into the air bubble, and consequently, escape analysis and detection. This procedure was applied to the Devcon I sample only.

The results for Dench were confirmed by the field replicate sample Fernandez and, as such, no data qualifiers were applied due to high level of precision in the sample-duplicate pair. As air bubbles were received in the Devcon III QC sample vials only, this would impact matrix bias recovery data for the Devcon III sample and not the actual field sample results. As such, no data qualifications were applied to the Devcon III analysis.

B) Method Detection Limit Study

The method detection limit (MDL) study associated with Method 524.2 was performed on April 15, 1990. The MDL determination was based on the analysis of seven replicates of reagent water fortified at a concentration of 1.0 ug/L with Method 524.2 analytes. The parameter list, however, for the site consisted of TCL analytes, some of which are not in the drinking water method. As such, the following target volatile organic compounds were omitted from the MDL study: acetone, carbon disulfide, 2-butanone, vinyl acetate, 4-methyl-2-pentanone and 2-hexanone. Also, in accordance with Method 524.2, isomers of xylene (ortho, meta, and para) and 1,2-dichloroethene (cis and trans) are quantitated and calculated individually while, as in the sample data package, these isomers were reported as totals.

Due to the omission of certain target analytes from the MDL study, the laboratory has not provided evidence that the MDL criteria could be achieved for the components specified previously above. Based on this premise, all low level sample analysis results for the omitted target analytes were qualified as estimated for positive hits (J) and non-detects (UJ).

The laboratory MDL was calculated as the product of the standard deviation of seven replicate analyses and the student's t value for the 99% confidence level with six degrees of freedom ($t = 3.143$).

Section 10.3.3 of Method 524.2 states that for every analyte and surrogate the mean accuracy, expressed as a percentage of the true value, should be 80 - 120% and the RSD < 20%. In the MDL study performed in support of the February 1991 analyses for the Tutu Wells site, the following analytes did not meet the precision and accuracy criteria required prior to sample analysis:

<u>Analyte</u>	<u>Mean Accuracy, %RSD</u>	
1,1-dichloroethene	72	-
methylene chloride	148	-
1,2-dichloroethane	121	-
tetrachloroethene	76	-
trans-1,3-dichloropropene	162	-
1,1,2,2-tetrachloroethane	122	-
bromoform	-	28.0

C) Laboratory Fortified Blanks

As per the QC requirements in Method 524.2 and the project Sampling, Analysis and Monitoring Plan (SAMP), a laboratory fortified blank (LFB), to which known quantities of the target analytes are added, is required to be analyzed for every batch of low level samples processed using the method. In the analysis of well samples for the Tutu site, LFBs were prepared at a concentration of 1 ug/L with TCL analytes and processed prior to the analysis of field samples in accordance with the QC protocol. The LFBs were to be evaluated using equivalent procedural guidelines for the determination of accuracy as stated in the MDL assessment for initial demonstration of precision and accuracy. Furthermore, an assessment of laboratory capability as to achievement of MDL for every sample batch processed was to be derived from the LFB analysis. Based on these evaluations, the laboratory was to continue with sample analysis if all criteria was achieved, or alternatively, remedy deficiencies prior to sample analysis where QC was not within limits (as specified in Method 524.2 Section 10.3).

For the purposes of the Tutu Wells site volatile organic analyses, the QC criteria was modified to assess data quality for analyses performed by Method 524.2 in the following manner. Based on an EPA guidance (dated December 4, 1990) regarding additional quality assurance (QA)/QC requirements when Method 524.2 is used for aqueous sample analysis, the 80 - 120% criteria for the accuracy assessment of LFB was expanded to 50 - 150%. Based on this guidance and for purposes of validation, for any analyte for which the LFB recovery was < 50%, the associated sample data was rejected and qualified as unusable (R). For LFB analyte recoveries in the range of 50 - 79%, the associated sample data was qualified as estimated (J). Data qualifiers were not applied to any sample result for which the associated LFB analyte recovery was greater than 150%.

On this basis, the following data qualifiers were applied to sample data due to the evaluation of LFBs. In the assessment of analyte accuracy in LFB BSTD002, sample data was rejected (R) for bromomethane, vinyl chloride, chloroethane, 2-butanone, trans-1,3-dichloropropene, bromoform and 1,1,2,2-tetrachloroethane. The following compound data was qualified as estimated (J) as the LFB recovery fell in the 50-79% range: chloromethane, acetone, carbon disulfide, 1,2-dichloroethene, 1,2-dichloroethane, 1,1,1-trichloroethane, vinyl acetate, bromodichloromethane, dibromochloromethane, benzene, toluene, ethylbenzene, styrene and total xylenes. Samples Eglin I, Eglin II, Eglin III, Dede, Devcon I, Devcon III (including MS and MSD), Dench, Fernandez and both trip blanks associated with SDG Dede are associated with this LFB standard.

In the analysis of sample Smith, data qualifiers were applied based on the LFB evaluation of BSTD003. The bromomethane, vinyl chloride, chloroethane, 2-butanone and trans-1,3-dichloropropene values were less than 50% in the LFB and, as such, the sample results were rejected as unusable (R) for all samples listed above. Sample results were estimated (J) for chloromethane, carbon disulfide, vinyl acetate, styrene and total xylenes as the recoveries were between 50-79% in the associated LFB.

In LFB BSTD004, sample results for trans-1,3-dichloropropene were qualified as unusable (R) as the analyte recovery was less than 50%. Sample results for bromomethane, vinyl chloride, chloroethane, carbon disulfide, vinyl acetate, styrene and total xylenes were estimated (J) as the associated LFB recoveries were in the 50-79% range. Samples Eglin IV, Four Winds II and Hartman II are associated with LFB BSTD004.

As the recoveries of LFB standard BSTD001 were generally >120%, no qualifications were applied to sample Rodrigues as results in the LFB were biased high. Sample results for 2-butanone in the LFB were <50%. This analyte was previously rejected due to a deficient initial response factor for this compound.

11. SYSTEM PERFORMANCE AND OVERALL ASSESSMENT:

All quantitation reports, reconstructed ion chromatograms (RIC) and mass spectra for positively identified TCL volatile organic compounds were included with the respective Form I datasheets. Additionally, CLP RAS summary forms were provided as required in support of the deliverables package. Numerous translocation errors were noted on the Form II surrogate summaries and Form

VIII summaries associated with this SDG. Additionally, transcription errors were found between the sample data package and summary package with respect to the reporting of tentatively identified compounds. In one instance, the result for 1,2-dichloroethene (total) was not reported for sample Eglin III. Data corrections were provided by the reviewer for the submission of the data validation package.

Aside from the reporting errors, system performance deficiencies were noted in the Method 524.2 analyses for low level volatile organic compounds. The initial MDL study resulted in several compounds not meeting the preliminary accuracy requirements needed to establish the baseline MDL for the low level analysis. As noted previously, several target compounds were not included in the MDL study altogether. In reviewing the MDL accuracy measurements for the compounds exceeding the criteria, the recoveries for 1,1-dichloroethene, 1,2-dichloroethane, tetrachloroethene and 1,1,2,2-tetrachloroethane were within $\pm 10\%$ of the 80 - 120% recovery criteria. For the remaining two compounds outside of criteria, high recovery for methylene chloride may be attributed to background contamination which is not out of the ordinary in a laboratory environment. With respect to the trans-1,3-dichloropropene, the MDL replicates showed consistently high recoveries with very little deviation. No explanations as to the higher recoveries have been obtained. The precision requirement between replicate measurements in the MDL study ($RSD < 20\%$) was met for all compounds with exception of bromoform for which greater variability is often noted. Based on these observations and the deviations noted above regarding omitted compounds, the MDL study of April 1990 established the laboratory's initial capability to analyze samples with the accuracy and precision needed for low level analysis.

Laboratory fortified blanks (LFBs) were utilized to assess the laboratory's capability on a batch basis. In reviewing these standards against the accuracy criteria, all three of the associated LFB analyses resulted in 14, 15, and 18 analyte recoveries, respectively, outside of the 80 - 120% method criteria. In using the expanded criteria of 50 - 150%, several compounds per LFB were still observed to be outside of the criteria. As such this indicated that the laboratory had difficulty in recovering certain compounds at the levels dictated by Method 524.2 analysis for low level volatiles.

In cases of high analyte recoveries, difficulties in controlling instrument background levels and system contamination may have falsely contributed to the out of limit LFB recoveries.

In assessing the volatile data, particular attention was focused on LFB recoveries below the lower control limit (80%). Since data with associated LFB recoveries below 80% would be biased low, all affected analytes with recoveries between 50 - 79% were qualified as estimated (J). Below 50% recovery, associated sample data was rejected and qualified as unusable (R). These qualifications, primarily, affected non-site specific target compounds and, within that perspective, have had minimal effect on the data quality for the use for which it was intended.

For LFB analyte recoveries greater than 120%, sample data would, generally be, biased high. All of the associated data resulted in non-detected values at the quantitation limit. No action was taken to offset the higher analyte recoveries and data quality was not impacted.

Poor initial instrument response resulted in variable volatile calibrations and impacted daily performance for several ketones and other laboratory contaminants throughout the duration of the sample analyses. Aside from these, occasional other compound calibrations did not meet criteria in the low level method and, in some analyses, for the CLP RAS method. As no deficiency resulted in unusable data for compounds specific to the site analysis, data quality was not adversely affected due to calibration problems.

Overall, the volatile organic data for SDG Dede in support of the Tutu Well site analyses was consistent and of adequate data quality. As noted, QC issues did serve to qualify certain compounds as estimated and reject others due to common, recognized, but correctable instrument problems. Daily performance, while assessed as fair to average, with respect to the Method 524.2 analyses, needs to be improved and stabilized for data to be reported unqualified based on method and regional guidelines for full data acceptance.

12. CONTRACT PROBLEMS - NON-COMPLIANCE:

The supporting MDL study did not include six target compounds from the parameter list. Several accuracy measurements for target compounds did not meet the method and SAMP criteria for the determination of the laboratory MDL.

The laboratory fortified blanks did not meet all of the method criteria (and/or the suggested expanded criteria) as required. In accordance to the method, these

specifications must be met, or deficiencies remedied, prior to sample analysis and were not.

The aromatic volatile compounds for sample Four Winds II were analyzed outside of the holding time.

13. The Form I for Method 524.2 analyses of samples Eglin II and Four Winds II have been edited by the data reviewer to include data from CLP RAS analyses required for high levels of 1,2-dichloroethene (total) and tetrachloroethene.

STANDARD OPERATING PROCEDURE (SOP) NO. HW-2

Revision #10

**EVALUATION OF METALS DATA FOR THE CONTRACT
LABORATORY PROGRAM (CLP)**

Title: Evaluation of Inorganic Data for the
Contract Laboratory Program

Date: Feb. 1990
Number: HW-2
Revision: 10

1.0 Scope

- 1.1 This procedure is applicable to inorganic data obtained from contractor laboratories working for Hazardous Waste Site Contract Laboratory Program (CLP).
- 1.2 The data validation is based upon analytical and quality assurance requirements specified in Statement of Work (SOW 7/88).

2.0 Responsibilities - Data reviewers will complete the following tasks as assigned by the Data Review Coordinator:

2.1 For a total review:

- 2.1.1 Data Assessment - "Total Review - Inorganics" Checklist Appendix (A.1).
The reviewer must answer every question on the checklist.

- 2.1.2 Data Assessment - Data Assessment Narrative (Appendix A.2)
The answer on the checklist must match the action in the narrative (appendix A.2) and Form I's. Do not use pencil to write the narrative.

- 2.1.3 Contract Non-Compliance - SMO Report (Appendix A.3)
This report is to be completed only when a serious contract violation is encountered, or upon the request of the Data Review Manager or Deputy Project Officer (DPO). Forward 5 copies: one each for internal files, appropriate Regional DPO, Sample Management Office (SMO) and last two addresses of Mailing List for Data Reviewers (Appendix A.4). In other cases, all contract violations should be appended to end of Data Assessment Narrative (Sec. A.2.2.).

- 2.1.4 Data Summary Sheet - Summary of Inorganic Quality Control Data (Appendix A.5)
Enter in ink on Data Summary Sheet required QC values from Forms I through IX. Circle all values that require data qualification "Action".

2.1.5 CLP Data Assessment Summary Forms

2.1.5.1 Appendix A.6

Fill in the total number of analytes by different analyses and the number of analytes rejected or flagged as estimated due to corresponding quality control criteria. Place an "X" in boxes where analyses were not performed, or criteria do not apply.

2.1.5.2 Appendix A.7

Data reviewer is also required to fill out Inorganic Regional Data Assessment form (Appendix A.7) provided by EPA Headquarters. Codes listed on the form will be used to describe the Data Assessment Summary.

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-
- 2.1.6 Data Review Log: It is recommended that each data reviewer should maintain a log of reviews completed to include:
- a. date of start of case review
 - b. date of completion of case review
 - c. site
 - d. case number
 - e. contract laboratory
 - f. number of samples
 - g. matrix
 - h. hours worked
 - i. reviewer's initials
- 2.1.7 Telephone Record Log - The data reviewer should enter the bare facts of inquiry, before initiating any phone conversation with CLP laboratory. After the case review has been completed, mail white copy of Telephone Record Log to the laboratory and pink copy of SMO. File yellow copy in the Telephone Record Log folder, and attach a xerox copy of the Telephone Record Log to the completed Data Assessment Narrative (Appendix A.2).
- 2.1.8 Forwarded Paperwork
- 2.1.8.1 Upon completion of review, the following are to be forwarded to the Regional Sample Control Center (RSCC) located in the Surveillance and Monitoring Branch:
- a. data package
 - b. completed data assessment checklist (Appendix A.1, original)
 - c. SMO Contract Compliance Screening (CCS)
 - d. Data Summary Sheet (Appendix A.5) along with completed Data Assessment Narrative (Appendix A.2)
 - e. Record of Communication (copy)
 - f. CLP Re-analysis Request/Approval Record (original + 3 copies)
 - g. Appendix A.7 (original).
- 2.1.8.2 Forward 4 copies of completed Data Assessment Narrative (Appendix A.2) along with 2 copies of the Inorganic Data Assessment Form (Appendix A.7) and Telephone Record Log, if any, one each for appropriate Regional DPO, Sample Management Office (SMO), and last two addresses of Mailing List for Data Reviewers (Appendix A.4) (the Inorganic Data Assessment form does not go to the last two addresses).
- 2.1.9 Filed Paperwork - Upon completion of review, the following are to be filed within MMB files:
- a. Four copies of completed Data Assessment Narrative (Appendix A.2) each carrying Appendix A.7.
 - b. Telephone Record Log (copy)
 - c. SMO Report (copy Appendix A-3)
 - d. CLP Data Assessment Summary Form (Appendix A.6)
 - e. CLP Re-analysis Request/Approval Record (copy).

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3.0 Data Completeness

Indicate incomplete data package on the computer tracking sheet located in MMB office. Authorized contractor personnel may contact the laboratory after discovery of an incomplete data package. If a laboratory will not return phone calls or does not respond to requests, notify MMB coordinator of Region II for resolution.

4.0 Rejection Data - All values determined to be unacceptable on the Inorganic Analysis Data Sheet (Form I) must be lined over with a red pencil. As soon as any review criteria causes data to be rejected, that data can be eliminated from any further review or consideration.

5.0 Acceptance Criteria - In order that reviews be consistent among reviewers, acceptance criteria as stated in Appendix A.1 (pages 4-25) should be used. Additional guidance can be found in the National Inorganic Functional Guidelines of October 1, 1989.

6.0 SMO Contract Compliance Screening (CCS) - This is intended to aid reviewer in locating any problems, both corrected and uncorrected. However, the validation should be carried out even if CCS is not present. Resubmittals received from laboratory in response to CCS must be used by the reviewer.

7.0 Request for Re-analysis - Data reviewers must note all items of contract non-compliance within Data Assessment Narrative. If holding times and sample storage times have not been exceeded, DPO may request re-analysis if items of non-compliance are critical to data assessment. Requests are to be made on "CLP Re-analysis Request/Approval Record".

8.0 Record of Communication - Provided by the Regional Sample Control Center (RSCC) to indicate which data packages have been received and are ready to be reviewed.

9.0 Rounding off numbers - The data reviewer will follow the standard practice.

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Appendix A.1: Data Assessment - Contract
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	YES	NO	N/A
A.1.1 <u>Contract Compliance Screening Report (CCS)</u> - Present? <u>ACTION:</u> If no, contact RSCC.	☐	☐	☒
A.1.2 <u>Record of Communication (from RSCC)</u> - Present? <u>ACTION:</u> If no, request from RSCC.	☐	☐	☒
A.1.3 <u>Trip Report</u> - Present and complete? <u>ACTION:</u> If no, contact RSCC for trip report.	☐	☐	☒
A.1.4 <u>Sample Traffic Report</u> - Present or on file?	☒	☐	☐
Legible?	☒	☐	☐
<u>ACTION:</u> If no, request from Regional Sample Control Center (RSCC).			
A.1.5 <u>Cover Page</u> - Present?	☒	☐	☐
Is cover page properly filled in and signed by the lab manager or the manager's designee?	☒	☐	☐
<u>ACTION:</u> If no, prepare Telephone Record Log, and contact laboratory.			
Do numbers of samples correspond to numbers on Record of Communication?	☐	☐	☒
Do sample numbers on cover page agree with sample number on:			
(a) Traffic Report Sheet?	☐	☒	☐
(b) Form Is?	☒	☐	☐
<u>ACTION:</u> If no for any of the above, contact RSCC for clarification.			
A.1.6 <u>Form I (Final Data)</u> - Are all Form I's present and complete?	☒	☐	☐
<u>ACTION:</u> If no, prepare telephone record log and contact laboratory for submittal.			

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	YES	NO	N/A
Are correct units (ug/L for waters and mg/kg for soils) indicated on Form Is?	☒	☐	☐
Are soil sample results for each parameter corrected for percent solids?	☐	☐	☒
Are EPA sample #'s and corresponding laboratory sample ID #'s the same as the Cover Page, Form Is and in the raw data?	☒	☐	☐
Are the computation/transcription errors less than 10% of reported values?	☒	☐	☐
Are all "less than IDL" values properly coded with "U"?	☒	☐	☐
Was a brief physical description of samples given on Form Is?	☒	☐	☐
Were the result qualifiers used correctly with final data?	☒	☐	☐
<u>ACTION:</u> If no for any of the above, prepare Telephone Record Log, and contact laboratory for corrected data.			
Were any samples diluted beyond requirements of contract?	☐	☒	☐
If yes, were dilutions noted on Form Is?	☐	☐	☒
<u>ACTION:</u> If no, note under contract Problem/Non-Compliance of the "Data Assessment Narrative".			

A.1.7 **Holding Times** - (aqueous and soil samples)
(Examine sample traffic reports and digestion distillation logs.)

Mercury analysis (28 days).....exceeded?	☐	☒	☐
Cyanide distillation (14 days).....exceeded?	☐	☐	☐

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	YES	NO	N/A
Other Metals analysis (6 months)..exceeded?	—	☒	—

NOTE: Prepare a list of all samples and analytes for which holding times have been exceeded. Specify the number of days from date of collection to the date of preparation (from raw data). Attach to checklist.

ACTION: If yes, reject (red-line) values less than instrument Detection Limit (IDL) and flag as estimated (J) the values above IDL even though sample(s) was preserved properly.

A.1.8 Raw Data

A.1.8.1 Digestion Log* for flame AA/ICP (Form XIII) present?

☒ — —

Digestion Log for furnace AA Form XIII present?

☒ — —

Distillation Log for mercury Form XIII present?

☒ — —

Distillation Log for cyanides Form XIII present?

☐ — ☒

Are pH values (pH < 2 for all metals, pH > 12 for cyanide) present in Digestion Distillation Logs?

☒ — —

Percent solids calculation present for soils/sediments?

☐ — ☒

Are preparation dates present on Digestion Log?

☒ — —

A.1.8.2 Measurement read out record present? ICP

☒ — —

Flame AA

☐ — ☒

Furnace AA

☒ — —

Mercury

☒ — —

Cyanides

☐ — ☒

A.1.8.3 Are all raw data to support all sample analyses and QC operations present?

☒ — —

* Weights, dilutions, and volumes used to obtain values.

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	YES	NO	N/A
Legible?	☒	☐	☐
Properly labeled?	☒	☐	☐

ACTION: If no for any of the above, write Telephone Record Log and contact laboratory. Flag metal data as estimated if pH of sample is greater than 2. Flag cyanide data as estimated if pH sample is less than 12.

A.1.9 Data Validation and Verification

A.1.9.1 Calibration

A.1.9.1.1 Is record of at least 2 point calibration present for ICP analysis?

☒ ☐ ☐

Is record of 5 point calibration present for Hg analysis?

☒ ☐ ☐

ACTION: If no for any of the above, write in the Contract Problem/Non-Compliance section of the "Data Assessment Narrative".

A.1.9.1.2 Is record of 4 point calibration present for: Flame AA?

☐ ☐ ☒

Furnace AA?

☒ ☐ ☐

Cyanides?

☐ ☐ ☒

- NOTE:**
1. If less than 4 standards are measured in absorbance mode, then the remaining standards in concentration mode must run immediately after calibration and be within +/- 10% of true value.
 2. For all AA (except Hg) and Cyanide analyses, one calibration standard is at CRDL level. If not, write in the Contract-Problem/Non-Compliance section of the "Data Assessment Narrative".

ACTION: Flag associated data as estimated if standards

are not within +/- 10% of true values (except

YES NO N/A

TUT 002 2172

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CRDL calibration standard). Do not flag the
data as estimated in linear range indicated
by good recovery of standard.

A.1.9.1.3 Is correlation * coefficient less than 0.995 for: Mercury Analysis?	—	☒	—
Cyanide Analysis?	—	☐	☒
Atomic Absorption Analysis?	—	☒	—

ACTION: If yes, flag the associated data as estimated.

A.1.9.2 Form IIA (Initial and Continuing Calibration Verification)

A.1.9.2.1 Present and complete for every metal and cyanide?	☒	—	—
Present and complete for AA and ICP when both are used for same analyte?	☐	—	☒

ACTION: If no for any of the above, prepare
Telephone Record Log and contact
laboratory.

A.1.9.2.2 Circle all values on data summary sheet that are outside contract windows. Are all calibration standards (initial and continuing) within control limits?	Metals 90-110%	☒	—	—
	Hg 80-120%	☒	—	—
	Cyanides 85-115%	☐	—	☒

ACTION: Flag as estimated (J) all positive Data
(not flagged with a "U") analyzed between
a calibration standard with %R between
75-89% (65-79% for Hg; 70-84% for CN)
or 111-125% (121-135% for Hg; 116-130%
for CN) recovery and nearest good

* The reviewer will calculate correction coefficient.

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YES NO N/A

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YES NO N/A

calibration standard. Quality results
<IDL as estimated (UJ), if the ICV
or CCV %R is 75-89% (CN, 70-84%;
HG, 65-79%). Reject (red-line) as
unacceptable data if recovery of the
ICV or CCV is outside the range 75-125%
(CN, 70-130%; Hg, 65-135%). Qualify
five samples on either side of
verification standard out of control
limits.

Was continuing calibration performed every 10
samples or every 2 hours?

☒ ☐ ☐

ACTION: If no, flag the excess samples (eleventh
and up) data as estimated (J).

Was ICV for cyanides distilled?

☐ ☐ ☒

ACTION: If no, write in the Contract-Problem/
Non-Compliance section of the "Data
Assessment Narrative".

A.1.9.3 Form IIB (CRDL Standards for AA and ICP) -

A.1.9.3.1 Was a CRDL standard (CRA) analyzed after
initial calibration for all AA metals (except
Hg)?

☒ ☐ ☐

*Was a mid-range calib. verification standard
distilled and analyzed for cyanide analysis?

☐ ☐ ☒

Was a 2xCRDL (or 2xIDL when IDL > CRDL) analyzed
(CRI) for each ICP run?
(Note: CRI for Al, Ba, Ca, Fe, Mg, NA or K is
not required).

☒ ☐ ☐

ACTION: If no for any of the above, flag as
estimated all data falling within
the affected ranges. The affected
ranges are:

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YES NO N/A

AA Analysis - **True Value +/- CRDL

ICP Analysis - **True Value +/- 2CRDL

CN Analysis - **True Value +/- 0.5 x True Value

A.1.9.3.2 Was CRI analyzed after ICV/ICB and before the final CCV/CCB, and for every four hours of ICP run?

☒ ☐ ☐

ACTION: If no, write in Contract Problem/Non-Compliance Section of the "Data Assessment Narrative".

A.1.9.3.3 Circle all values on summary sheet that are outside acceptance windows.

Are CRA and CRI standards within control limits:
Metals 80-120 %R?

☐ ☒ ☐

Is mid-range standard within control limits:
Cyanide 80-120 %R?

☐ ☐ ☒

ACTION: Flag as estimated all data within the affected ranges if the recovery of the standard is between 50-79%; flag only positive data if the recovery is between 121-150%; reject (red line) all data if the recovery is less than 50%; reject only positive data if the recovery is greater than 150%.

A.1.9.4 Form III (Initial and Continuing Calibration Blanks)

A.1.9.4.1 Present and complete?

☒ ☐ ☐

For both AA and ICP when both are used for same analyte?

☐ ☐ ☒

**True value of CRA, CRI or mid-range standard. Substitute IDL for CRDL when IDL > CRDL.

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	YES	NO	N/A
Was an initial calibration blank analyzed?	☒	—	—
Was a continuing calibration blank analyzed after every 10 samples or every 2 hours (whichever is more frequent)?	☒	—	—

ACTION: If no prepare Telephone Record Log,
contact laboratory and write in the
contact-problems/non-compliance
section of the Data Assessment
Narrative."

A.1.9.4.2 Circle all calibration blank values on Data
Summary Sheet that are above CRDL (or 2 x IDL
when IDL > CRDL) less than or equal to Contract
Required Detection Limits (CRDL)?

☒	—	—
-------------------------------------	---	---

Are all calibration blanks less than two times
Instrument Detection Limit (when IDL > CRDL)?

☐	—	☒
--------------------------	---	-------------------------------------

ACTION: If no for any of the above, flag as
estimated (J) all positive data less
than or equal to calibration blank
values analyzed between calibration
blank with value over CRDL (or 2 x IDL)
and nearest good calibration blank.
Flag five samples on either side of
the calibration blank.

A.1.9.5 FORM III (Preparation Blank) -

(Note: The preparation blank for mercury is
the same as the calibration blank).

A.1.9.5.1 Was on prep. blank analyzed for: each 20 samples?

☒	—	—
-------------------------------------	---	---

each batch?

☒	—	—
-------------------------------------	---	---

each matrix type?

☒	—	—
-------------------------------------	---	---

both AA and ICP and when both are used for same analyte?

☐	—	☒
--------------------------	---	-------------------------------------

ACTION: If no for any of the above, flag as

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	YES	NO	N/A
estimated (J) all associated positive data < 10 x IDLs for which prep. blank was not analyzed.			
NOTE: If only one blank was analyzed for more than 20 samples, then first 20 samples analyzed do not have to be flagged as estimated (J).			
A.1.9.5.2 Is concentration of prep. blank greater than CRDL when IDL is less than or equal to CRDL?	—	☒	—
If yes, is the concentration of the sample with the least concentration analyte less than 10 times the prep. blank value?	—	☐	☒
ACTION: If yes, reject (red-line) all associated data greater than CRDL concentration but less than ten times the prep. blank value found in the raw data.			
A.1.9.5.3 Do concentrations of prep. blank fall between two times IDL when IDL is greater than CRDL?	☐	—	☒
ACTION: If no, reject (red-line) positive data that has a concentration less than 10 times the prep. blank value in the raw data.			
A.1.9.5.4 Is concentration of prep. blank below the negative CRDL?	—	☒	—
ACTION: If yes, reject (red-line) all associated data that has a concentration less than 10xCRDL.			
A.1.9.6 <u>Form IV (ICP Interference Check Sample)</u>			
A.1.9.6.1 Present and complete? (NOTE: Not required for furnace AA, flame AA, mercury, cyanide and Ca, Mg, K and Na).	☒	—	—

Was ICS analyzed at beginning and end of run

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(or at least twice every 8 hours)?

YES	NO	N/A
☒	☐	☐

ACTION: If no, flag as estimated (J) all samples for which Al, Ca, Fe, or Mg is higher than in ICS.

A.1.9.6.2 Circle all values on Data Summary Sheet that are more than + 10% of true or established mean value. Are all Interference Check Sample results inside of control limits (+20%)?

☐	☐	☒
--------------------------	--------------------------	-------------------------------------

If no, is concentration of Al, Ca, Fe or Mg lower than in ICS?

☐	☐	☒
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ACTION: If no, flag as estimated (J) those positive results for which ICS recovery is between 121-150%; flag all sample results as estimated if ICS recovery falls within 50-79%; reject (red-line) those sample results for which ICS recovery is less than 50%; if ICS recovery is above 150%; reject positive results only (not flagged with a "U").

A.1.9.7 Form V A (Spiked Sample Recovery - Pre-Digestion/Pre-Distillation) -

(Note: Not required for Ca, Mg, K, and Na (both matrices), Al, and Fe (soil only.))

A.1.9.7.1 Present and complete for: each 20 samples?

☒	☐	☐
-------------------------------------	--------------------------	--------------------------

each matrix type?

☒	☐	☐
-------------------------------------	--------------------------	--------------------------

each conc. range (i.e. low, med, high)?

☒	☐	☐
-------------------------------------	--------------------------	--------------------------

For both AA and ICP when both are used for same analyte?

☐	☐	☒
--------------------------	--------------------------	-------------------------------------

ACTION: If no for any of the above, flag as estimated (J) all positive data less than four times spiking level for

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YES NO N/A

which spiked sample was not analyzed.

NOTE: If one spiked sample was analyzed for more than 20 samples, then first 20 samples analyzed do not have to be flagged as estimated (J).

A.1.9.7.2 Was field blank used for spiked sample?

— ☒ —

ACTION: If yes, flag all positive data less than 4x spike added as estimated (J) for which field blank was used as spike sample.

NOTE: Matrix spike analysis should be performed on a field blank when it is the only aqueous sample in SDG.

A.1.9.7.3 Circle all values on Data Summary Sheet that are outside control limits (75% to 125%). Are all recoveries within control limits?

[] ☒ —

If no, is sample concentration greater than or equal to four times spike concentration?

[] ☒ —

ACTION: If yes, disregard spike recoveries for analytes whose concentrations are greater than or equal to four times spike added. If no, circle those analytes on Form V for which sample concentration is less than four times the spike concentration.

Are results outside the control limits (75-125%) flagged with "N" on Form Is and Form VA?

☒ — —

ACTION: If no, write in the Contract-Problem/Non-Compliance section of "Data Assessment Narrative."

A.1.9.7.4 Aqueous

Are any spike recoveries:

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Narrative."

A.1.9.7.4 Aqueous

Are any spike recoveries:

	YES	NO	N/A
(a) less than 30%?	—	☒	—
(b) between 30-74%?	☒	☐	—
(c) between 126-150%?	—	☒	—
(d) greater than 150%?	—	☒	—

ACTION: If less than 30%, reject all associated aqueous data; if between 30-74%, flag all associated aqueous data as estimated (J); if between 126-150%, flag as estimated (J) all associated aqueous data not flagged with a "U"; if greater than 150%, reject (red-line) all associated aqueous data not flagged with a "U".

NOTE: If pre-digestion spike result is rejectable due to coefficient of correlation of MSA, analytical spike recovery, or duplicate injections criteria, disregard spike recovery on Form V. Flag the associated data as estimated (J).

A.1.9.7.5 Soil/Sediment

Are any spike recoveries:

(a) less than 10%?	—	☐	☒
(b) less than 10-74%?	—	☐	☒
(c) between 126-200%?	—	☐	☒
(d) greater than 200%?	—	☐	☒

ACTION: If less than 10%, reject all associated data; if between 10-74%, flag all associated data as estimated; if between

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	YES	NO	N/A
A.1.9.8 <u>Form VI (Lab Duplicates)</u>			
A.1.9.8.1 Present and complete for:			
each 20 samples?	☒	—	—
each matrix type?	☒	—	—
each concentration range (i.e. low, med, high)?	☒	—	—
both AA and ICP when both are used for same analyte?	☐	—	☒

ACTION: If no for any of the above, flag as estimated (J) all data > CRDL* for which duplicate sample was not analyzed.

NOTE:

1. If one duplicate sample was analyzed for more than 20 samples, then first 20 samples do not have to be flagged as estimated.
2. If percent solids for soil sample and its duplicate differ by more than 1%, prepare a Form VI for each duplicate pair, report concentrations in ug/L on wet weight basis and calculate RPD or Difference for each analyte.

A.1.9.8.2 Was field blank used for duplicate analysis?

— ☒ —

ACTION: If yes, flag all data > CRDL* as estimated (J) for which field blank was used as duplicate.

NOTE: Duplicate analysis should be performed on a field blank when it is the only aqueous sample in SDG.

A.1.9.8.3 Are all values within control limits (RPD 20% or difference < +/- CRDL)?

☐ ☒ —

* Substitute IDL for CRDL when IDL > CRDL.

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If no, are all results outside the control limits
flagged with an * on Form Is and VI?

YES	NO	N/A
☒	☐	☐

ACTION: If no, write in the Contract-Problems/Non-
Compliance section of "Data Assessment
Narrative".

NOTE: 1. RPD is not calculable for an analyte of
the sample - duplicate pair when both
values are less than IDL.
2. If lab duplicate result is rejectable
due to coefficient of correlation of MSA,
analytical spike recovery, or duplicate
injections criteria, do not apply precision
criteria.

A.1.9.8.4 Is any value for sample duplicate pair less than CRDL*
and other value greater than or equal to 10 x *CRDL?

☐	☒	☐
--------------------------	-------------------------------------	--------------------------

ACTION: If yes, flag the associated data as
estimated (J).

A.1.9.8.5 Aqueous

Circle all values on Data Summary Sheet that are:
RPD > 50%, or
Difference > = +/- CRDL*

Is any RPD greater than 50% where sample and duplicate
are both greater than or equal to 5 times *CRDL?

☐	☒	☐
--------------------------	-------------------------------------	--------------------------

Is any difference between sample and duplicate
greater than *CRDL where sample and/or duplicate
is less than 5 times *CRDL?

☐	☒	☐
--------------------------	-------------------------------------	--------------------------

ACTION: If yes, flag the associated data as estimated.

Substitute IDL for CRDL when IDL > CRDL.

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	YES	NO	N/A
A.1.9.8.6 <u>Soil/Sediment</u> Circle all values on Data Summary Sheet that are: RPD > 100%, or Difference > 2 x CRDL*			

Is any RPD (where sample and duplicate are both
greater than or equal to 5 times *CRDL):

> 100%?

— ☐ ☒

Is any ** difference between sample and
duplicate (where sample and/or duplicate is
less than 5 x *CRDL):

> 2 x *CRDL?

— ☐ ☒

ACTION: If yes, flag the associated data as estimated.

A.1.9.9 Field Duplicates

A.1.9.9.1 Were field duplicates analyzed?

☒ — —

ACTION: If yes, prepare a Form VI for each
aqueous field duplicate pair. Prepare
a Form VI for each soil duplicate pair,
if percent solids for sample and its
duplicate differ by more than 1%;
report concentrations of soils in ug/L
on wet weight basis and calculate RPDs
or Difference for each analyte.

NOTE: 1. Do not calculate RPD when both values
are less than IDL.
2. Flag all associated data only for
field duplicate pair.

A.1.9.9.2 Is any value for sample duplicate pair less than
*CRDL and other value greater than or equal to
10 x *CRDL?

— ☒ —

* Substitute IDL for CRDL when IDL > CRDL.

** Use absolute values of sample and duplicate to calculate difference.

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YES NO N/A

ACTION: If yes, flag the associated data as estimated.

A.1.9.9.3 Aqueous

Circle all values on Form VI for field duplicates
that are:

RPD > 50%, or
Difference > +/- CRDL*

Is any RPD greater than 50% where sample and duplicate
are both greater than or equal to 5 times *CRDL?

___ [___] ☒

Is any **difference between sample and duplicate
greater than *CRDL where sample and/or duplicate
is less than 5 times *CRDL?

___ [___] ☒

ACTION: If yes, flag the associated data as estimated.

A.1.9.9.4 Soil/Sediment

Circle all values on Form VI for field duplicates
that are:

RPD > 100%, or
Difference > 2 x CRDL*

Is any RPD (where sample and duplicate are both
greater than 5 times *CRDL):
> 100%?

___ [___] ☒

Is any **difference between sample and duplicate
(where sample and/or duplicate is less than
5 x *CRDL):

> 2 x *CRDL?

___ [___] ☒

A.1.9.10 Form VII (Laboratory Control Sample) (Note: LCS -
not required for aqueous Hg and cyanide analyses).

* Substitute IDL for CRDL when IDL > CRDL.

** Use absolute values of sample and duplicate to calculate difference.

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	YES	NO	N/A
A.1.9.10.1 Was one LCS prepared and analyzed for:			
every 20 water samples?	☒	☐	☐
every 20 solid samples?	☒	☐	☐
both AA and ICP when both are used for same analyte?	☐	☐	☒

ACTION: If no for any of the above, prepare Telephone Record Log and contract laboratory for submittal of results of LCS. Flag as estimated (J) all data for which LCS was not analyzed.

NOTE: If only one LCS was analyzed for more than 20 samples, then first 20 samples close to LCS do not have to be flagged as estimated.

A.1.9.10.2 Aqueous LCS

Circle all LCS values outside control limits (80-120% - except aqueous Ag and Sb).

Is any LCS recovery: less than 50%?	☐	☒	☐
between 50% and 79%?	☐	☒	☐
between 121% and 150%?	☐	☒	☐
greater than 150%?	☐	☒	☐

ACTION: Less than 50%, reject (red-line) all data; between 50% and 79%, flag all associated data as estimated (J); between 121% and 150%, flag all positive (not flagged with a "U") results as estimated; greater than 150%, reject all positive results.

A.1.9.10.3 Solid LCS

NOTE: 1. If "Found" value of LCS is rejectable due to duplicate injections or analytical

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YES NO N/A

spike recovery criteria, regardless of
LCS recovery, flag the associated data
as estimated (J).

2. If IDL of an analyte is equal to or
greater true value of LCS, disregard
the "Action" below even though LCS is
out of control limits.

Is LCS "Found" value higher than the control limits
on Form VII?

— ☐ ☒

ACTION: If yes, qualify all associated positive
data as estimated.

Is LCS "Found" value lower than the control limits
on Form VII?

— ☐ ☒

ACTION: If yes, qualify all associated data as
estimated.

A.1.9.11 Form IX (ICP Serial Dilution) -

NOTE: Serial dilution analysis is required
only for initial concentrations equal
to or greater than 10 x IDL.

A.1.9.11.1 Was Serial Dilution analysis performed for:

each 20 samples?

☒ — —

each matrix type?

☒ — —

each concentration range (i.e. low, med.)?

☒ — —

ACTION: If no for any of the above, flag all
positive data greater than or equal to
10 x IDLs as estimated (J) for which
Serial Dilution Analysis was not performed,
and summarize the deficiency on the DPO report.

A.1.9.11.2 Was field blank(s) used for Serial Dilution Analysis?

— ☒ —

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YES NO N/A

ACTION: If yes, flag all associated data $\geq 10 \times$ IDL as estimated (J).

NOTE: Serial dilution analysis should be performed on a field blank when it is the only aqueous sample in SDG.

A.1.9.11.3 Are results outside control limit flagged with an "E" on Form Is and Form IX when initial concentration on Form IX is equal to 50 times IDL or greater?

☐ ☐ ☒

ACTION: If no, write in the contract-problem/non-compliance section of the "Data Assessment Narrative".

A.1.9.11.4 Circle all values on Data Summary Sheet that are outside control limit for initial concentrations equal to or greater than $10 \times$ IDLs only. Are any % difference values:

$> 10\%$?

☐ ☐ ☒

$\geq 100\%$?

☐ ☐ ☒

ACTION: Flag as estimated (J) all associated equal to or greater than $10 \times$ IDLs for which difference is greater than 10% but less than 100%. Reject (red-line) all associated sample results equal to or greater than $10 \times$ IDLs for which RPD is greater than or equal to 100%.

A.1.9.12 Furnace Atomic Absorption (AA) OC Analysis

A.1.9.12.1 Are duplicate injections present in furnace raw data (except during full Method of Standard Addition) for each sample analyzed by GFAA?

☒ ☐ ☐

ACTION: If no, reject the data on Form Is for which duplicate injections were not performed.

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	YES	NO	N/A
A.1.9.12.2 Do the duplicate injection readings agree within 20% Relative Standard Deviation (RSD) or Coefficient of Variation (CV) for concentration greater than CRDL?	☒	☐	☐
Was a dilution analyzed for sample with post digestion spike recovery less than 40%?	☐	☐	☒
A.1.9.12.3 Is *post digestion spike recovery less than 10% or greater than 150% for any result?	☐	☒	☐
<u>ACTION:</u> If yes, reject (red-line) the affected data if recovery			
<u>NOTE:</u> Reject the data only if the affected sample was not subsequently analyzed by Method of Standard Addition.			
A.1.9.13 <u>Form VII (Method of Standard Addition Results)</u>			
A.1.9.13.1 Present?	☒	☐	☐
If no, is any Form I result coded with "S" or a "+"?	☐	☐	☒
<u>ACTION:</u> If yes, write request on Telephone record Log and contact laboratory for submittal of Form VIII.			
A.1.9.13.2 Is coefficient of correlation for MSA less than 0.990 for any sample?	☒	☐	☐
<u>ACTION:</u> If yes, reject (red-line) affected data.			
A.1.9.13.3 Was **MSA required for any sample but not performed?	☐	☒	☐

* Post digestion spike is not required on the pre-digestion spiked sample when predigestion spike recovery is within control limits of 75-125% or when SR > 4 x SA.

** MSA is not required on LCS and prep. blank.

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	YES	NO	N/A
Is coefficient of correlation for MSA less than 0.995?	—	☐	☒

Are MSA calculations outside the linear range of the calibration curve generated at the beginning of the analytical run?

—	☒	—
---	-------------------------------------	---

ACTION: If yes for any of the above, flag all the associated data as estimated (J).

A.1.9.13.4 Was proper quantitation procedure followed correctly as outlined in the SOW on page E-16 through E-17?

☒	—	—
-------------------------------------	---	---

ACTION: If no, note exception under contract problem/non-compliance of data assessment narrative, or prepare a separate list.

A.1.9.14 Dissolved/Total or Inorganic/Total Analytes

A.1.9.14.1 Were any analyses performed for dissolved as well as total analytes on the same sample(s)?

—	☒	—
---	-------------------------------------	---

Were any analyses performed for inorganic as well as total (organic + inorganic) analytes on the same sample(s)?

—	☒	—
---	-------------------------------------	---

NOTE:

1. If yes, prepare a list comparing differences between all dissolved (or inorganic) and total analytes. Compute the differences as a percent of the total analyte only when dissolved concentration is greater than CRDL as well as total concentration.
2. Apply the following questions only if inorganic (or dissolved) results are (i) above CRDL, and (ii) greater than total constituents.
3. At least one preparation blank, ICS, and LCS should be analyzed in each analytical run.

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	YES	NO	N/A
A.1.9.14.2 Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 10%?	—	☐	☒
A.1.9.14.3 Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 50%?	—	☐	☒

ACTION: If more than 10%, flag both dissolved (or inorganic) and total values as estimated (J); if more than 50%, reject (red-line) the data for both values.

A.1.9.15 Form I to IX

A.1.9.15.1 Are all the Form I through Form IX labeled with:

Laboratory name?	☒	—	—
Case/SAS number?	☒	—	—
EPA sample No.?	☒	—	—
SDG No.?	☒	—	—
Contract No.?	☒	—	—
Correct units?	☒	—	—
Matrix?	☒	—	—

ACTION: If no for any of the above, note under contract problem/non-compliance section of the "Data Assessment Narrative".

A.1.9.15.2 Do any computation/transcription errors exceed 10% of reported values on Form I-IX for:
(NOTE: Check all forms against raw data.)

(a) all analytes analyzed by ICP?	—	☒	—
(b) all analytes analyzed by GFAA?		☒	—

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	YES	NO	N/A
(c) all analytes analyzed by AA Flame?	—	[]	✓
(d) Mercury?	—	✓	—
(e) Cyanide?	—	[]	✓

ACTION: If yes, prepare Telephone Log, contact laboratory for corrected data and correct errors with red pencil and initial.

A.1.9.16 Form I (Field Blank)

Circle all field blank values on Data Summary Sheet that are greater than CRDL, 2 x IDL when IDL > CRDL.

Do concentrations of field blank(s) fall below CRDL (or 2 x IDL when IDL > CRDL) for all parameters of associated aqueous and soil samples?

[] — ✓

If no, was field blank value already rejected due to other QC criteria?

[] — ✓

ACTION: If no, reject (except field blank results) all associated positive sample data less than or equal to five times the field blank value.

A.1.9.17 Form X, XI, XII (Verification of Instrumental Parameters)

A.1.9.17.1 Is verification report present for:

Instrument Detection Limits (quarterly)?

✓ [] — —

ICP Interelement Correction Factors (annually)?

✓ [] — —

ICP Linear Ranges (quarterly)?

✓ [] — —

ACTION: If no, contact DPO of the lab.

A.1.9.17.2 Form X (Instrument Detection Limits) - (Note: IDL is not required for Cyanide.)

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	YES	NO	N/A
Are IDLs present for: all the analytes?	☒	☐	☐
all the instruments used?	☒	☐	☐
For both AA and ICP when both are used for same analyte?	☐	☐	☒
<u>ACTION:</u> If no for any of the above, prepare Telephone Record Log and contact laboratory.			
Is IDL greater than CRDL for any analyte?	☐	☒	☐
If yes, is the concentration on Form I of the sample analyzed on the instrument whose IDL exceeds CRDL, greater than 5 x IDL?	☐	☐	☒
<u>ACTION:</u> If no, flag as estimated all values less than five times IDL of the instrument whose IDL exceeds CRDL.			

A.1.9.17.3 Form XI (Linear Ranges)

Was any sample result higher than high linear range of ICP?	☐	☒	☐
Was any sample result higher than the highest calibration standard for non-ICP parameters?	☐	☒	☐
If yes for any of the above, was the sample diluted to obtain the result on Form I?	☐	☐	☒
<u>ACTION:</u> If no, flag the result reported on Form I as estimated (J).			

A.1.9.18 Percent Solids of Sediments

Is soil content in sediment(s) less than 50%?	☐	☐	☒
<u>ACTION:</u> If yes, qualify as estimated all data not previously rejected or flagged due to other QC criteria.			

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DUPLICATES

EPA SAMPLE NO.

Lab Name: CEIMACContract: GERAGHTY & MILLEREglin III (D)Lab Code: CEIMACCase No.: G 2M

SAS No.: _____

SDG No.: DEDE WELMatrix (soil/water): WATERLevel (low/med): LOW% Solids for Sample: 0.0% Solids for Duplicate: 0.0Concentration Units (ug/L or mg/kg dry weight): ug/L

Analyte	Control Limit	Sample (S)	C	Duplicate (D)	C	RPD	Q	M
Aluminum								
* Antimony		27.0	U	27.0	U			P
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
* Thallium		1.0	U	1.0	U			F
Vanadium								
Zinc								
Cyanide								

FORM VI - IN

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DUPLICATES

EPA SAMPLE NO.

Lab Name: CEIMACContract: GERASHTV 2DENCH (D)Lab Code: CEIMACCase No.: G&MSAS No.: MILNERSDG No.: DEDE WELMatrix (soil/water): WATERLevel (low/med): LOW% Solids for Sample: 0.0% Solids for Duplicate: 0.0Concentration Units (ug/L or mg/kg dry weight): ug/L

Analyte	Control Limit	Sample (S)	C	Duplicate (D)	C	RPD	Q	M
Aluminum								
* Antimony		27.0	U	27.0	U			P
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
* Thallium		1.0	U	1.0	U			F
Vanadium								
Zinc								
Cyanide								

FORM VI - IN

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Case #: G & M	Site: Tutu Teic (St.Thomas)	Matrix: Soil
SDG #: Dede Wel	Lab: Ceimic	Water X
Contractor: Geraghty & Miller	Reviewer: Lidya Gulizia	Other

A.2.1 The case description and exceptions, if any, are noted below with reason(s) for rejection or qualification as estimated value(s) J.

This evaluation was prepared assessing the laboratory data package supporting the inorganic analyses of drinking water samples in Sample Delivery Group (SDG) Dede Wel. These samples were collected at the Tutu Wells Site in St. Thomas, U.S. Virgin Islands on February 4 and 5, 1991. They were received at Ceimic Corporation February 6 for quantitative analysis of antimony (Sb), mercury (Hg), selenium (Se) and/or thallium (Tl) as specified on the sample chain of custody.

All holding time criteria were met for SDG Dede Wel inorganic analyses.

Sample preservation to a pH of ≤ 2 for the metals analyses was verified at the laboratory prior to analysis and documented in the appropriate preparation/digestion logbook.

With respect to data reporting, while laboratory sample identifications (ID) for SDG Dede Well were consistent throughout the data package, several client sample names were incorrectly referenced resulting in numerous misspelled ID(s) in the sample data package. Most notably, the Eglin well samples I - IV were referred to as 'Elgin'. Additionally, Form XIII-IN graphite furnace analysis preparation log, the spike and duplicate suffixes were omitted from the sample identification although they appeared correctly on the Form XIV-IN analysis log and raw data. In general, data validation was based on technical judgement and general knowledge of the site by the reviewer.

All calibrations for analysis of antimony by inductively coupled plasma (ICP) emission spectrometer, mercury by cold vapor (CV) flameless atomic absorption, and selenium and thallium by graphite furnace atomic absorption (GFAA) were

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A.2.1 (continuation)

performed daily as per the 2/88 inorganic statement of work (SOW) protocol and for every instrument set-up as required. All subsequent initial and continuing calibration verification samples (ICV and CCV) were within criteria with respect to percent recovery (%R), control limits, and correlation coefficient.

In the analysis of blanks, negative values above the absolute value of the instrument detection limit (IDL) were reported in the analysis of selenium for the Smith Well sample (analytical sequence initiating on 3/15/91 at 13:28). As the analysis of selenium for this sample was calculated by the Method of Standard Additions (MSA), the negative blank values will have had minimal impact on the quality of final selenium results for the Smith Well sample.

In the thallium analysis for all field samples in this SDG (analytical sequence on beginning 3/13/91 at 08:46), a negative initial blank value was reported at the level of the absolute value of the IDL. No action or further evaluation is necessary as this blank met the minimum criteria and is viewed as an isolated occurrence in the analytical sequence. Quantitating the associated preparation blank (PBW) and laboratory control sample (LCS) to the sequence, initiating on 3/15/91 at 09:11, the blank result was above the analyte IDL. As thallium was undetected in these quality control (QC) samples, no change in result qualification was necessary and they remain as originally reported.

The ICP interference check solutions (ICS) were analyzed as per SOW protocol and the antimony results were reported on Form IV-IN. Since antimony is not included in the ICS solutions per EPA protocol, percent recoveries were not calculated for the initial and final analyses. Additionally, as the requested analyses did not provide information regarding the inherent sample levels for the common antimony interferents (aluminum, calcium, iron, magnesium and manganese), no additional judgements can be made for any sample with antimony results above the IDL (absolute value).

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A.2.1 (continuation)

In SDG Dede Wel, no sample was assessed for correctness of interelement correction as no antimony was detected above the IDL in any field or QC sample. As indicated on the cover page, no interelement corrections were applied due, in all probability, to the sole request for antimony analysis by ICP.

Contract Required Detection Limit (CRDL) QC standards were prepared in accordance with the CLP protocol for all of the inorganic target analytes and analyzed in the proper order in each of the analytical sequences as required. The accepted control limits for CRDL metals analyses are 80 - 120% recovery of the true value. In the CRDL analyses provided in support of this SDG, the CRDL standard recoveries were within the control limits for the antimony, selenium and both thallium analytical sequences.

In the assessment of the mercury CRDL standard, the laboratory reported a negative value below the IDL for the observed report and, consequently, calculated a negative recovery for this QC sample. Since calculations were based on regression analysis and the derived best fit curve does not always necessarily pass through the origin, a negative sample result was confirmed for the CRDL standard. However, as an observable peak was detected for the standard analysis, an estimated recovery of 63% was calculated based on a direct correlation between the peak height of the lowest mercury calibration standard (0.5 ug/L) and the CRDL standard by the data reviewer. As the approximated mercury recovery was outside of the CRDL acceptance criteria and fell within the range of 50 - 79% recovery, all data results within the mercury affected range of the true value $\pm$ the CRDL are to be qualified as estimated (J). If applied as noted and using the true value of the mercury CRDL standard of 0.2 ug/L, all sample data falling within the range of 0 ug/L through 0.4 ug/L was to be qualified as estimated. As such, well samples Eglin I and Matthias, mercury analyses were associated with the deficient CRDL standard and have sample results within the affected range. The mercury results for both of these samples have been qualified as estimated (J).

Aqueous laboratory control samples (LCS) were prepared and analyzed as per the CLP protocol to assess laboratory performance in the analysis of ICP and

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GFAA analytes. All LCS met the performance criteria requirements in the antimony, selenium and thallium analyses. As per inorganic SOW guidelines, LCS are not required to assess performance of mercury analyses and were, therefore, not analyzed.

Precision and accuracy was determined for the requested analytical parameters as per the following breakdown. Smith Well sample was used to assess selenium, the Matthias Well sample for mercury, and antimony and thallium precision and accuracy data was assessed in the Devcon III Well sample.

Matrix spike percent recoveries were within the required acceptance criteria for antimony and thallium only. All samples analyzed for selenium and mercury were assessed the appropriate qualifier code (N) to denote exceeded spiked sample analyte recoveries. As per the functional guidelines, the positive detect of selenium in the Smith Well sample was qualified as estimated (J) since the recovery for the spike fell in the range of 30 - 74%. Data qualifiers were not added to any mercury results in accordance with the same guideline as no positive detects above the IDL were reported.

In assessing precision, the relative percent difference (%RPD) was not calculated for antimony (Devcon III), mercury (Matthias) and thallium (Devcon III) due to non-detect results for each of the sample - duplicate pairs. With respect to the analysis of selenium in the Smith Well sample - duplicate pair, precision criteria was not applied due to the rejected lab duplicate result on the basis of the coefficient of correlation of the MSA analysis. As such, the asterisk flag (*) denoting a duplicate analysis not within control limits was removed from the selenium analysis of Smith Well (see Form I-IN).

In the analysis of thallium, all sample results were reported from the mean of duplicate injections which had met contract guidelines for analysis, as applicable. All samples, post-digestion analytical spikes, associated standards and QC samples with calculated values above the CRDL met the %RSD as required upon initial analysis. No samples required reanalysis.

STANDARD OPERATING PROCEDURE

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Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.1: Data Assessment - Contract
Compliance (Total Review - Inorganics)

Date: Feb. 1990
Number: HW-2
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A.2.1 (continuation)

The thallium post-digestion analytical spikes for the following samples recovered outside of the percent spike recovery criteria of 85 - 115% and were flagged with the qualifier code (W) denoting the acceptance window exceedance: Dede, Dench, Devcon I, Devcon III, Eglin I, Fernandez, Four Winds II, Hartman III, Rodriquez and Smith wells. As these samples displayed absorbances less than 50 percent of the post-digestate spike absorbance and are less than the IDL, the thallium results for all of the above were qualified as estimated (UJ).

In the analysis of selenium in the Smith Well sample, the calculated result was obtained by MSA at spike levels of 20 ppb, 40 ppb and 60 ppb. Additionally, a pre-digestion matrix spike and duplicate were analyzed concurrently with the field sample analysis. As reported and verified in the raw data, the MSA correlation coefficient was greater than 0.995 for both the sample and the matrix spike. The Smith Well duplicate sample was analyzed two times with each respective MSA resulting in a deficient correlation coefficient. As the unacceptable MSA result is for the duplicate sample only, the selenium field sample result remains unqualified.

The ICP serial dilution for antimony was performed using the Devcon III Well sample. Analyte concentrations should be minimally a magnitude of fifty times above the IDL to evaluate the effect of any significant physical or chemical interferences due to the sample matrix. As a non-detect result was reported for this sample, no data qualifications have been assessed based on the evaluation of this criterion.

Data accuracy were examined for method anomalies, transcription and/or reduction errors, and for results calculated outside of the appropriate linear ranges per analyte. One thallium continuing calibration blank (CCB) value was corrected to reflect the average for the replicate injections. No changes were made to correct sample names and they remain as originally reported since no major difficulties were encountered by the reviewer during the data evaluation.

Overall precision was evaluated using field duplicate samples. In SDG Dede Wel, two field duplicate pairs were collected and analyzed for antimony and

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Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.1: Data Assessment - Contract
Compliance (Total Review - Inorganics)

Date: Feb. 1990
Number: HW-2
Revision: 10

A.2.1 (continuation)

thallium. The field duplicate pairs consisted of Eglin III (replicated as Eglin IV) and Dench Well (replicate denoted as Fernandez). %RPD was not calculated for either field duplicate - sample pairs as all values were below the respective analyte IDLs. As a qualitative assessment, a comparison indicated good precision in the field and laboratory processes.

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Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.1: Data Assessment - Contract
Compliance (Total Review - Inorganics)

Date: Feb. 1990
Number: HW-2
Revision: 10

A.2.2 Contract Problems/Non Compliance:

In the analysis of selenium by Method of Standard Additions (MSA), the spiking concentrations for analysis were 0 ppb, 20 ppb, 40 ppb and 60 ppb. As per the modifications made to the Sampling, Analysis and Monitoring Plan (SAMP), the selenium standard additions were to be fortified at 0 ppb, 10 ppb, 20 ppb, and 40 ppb.

Based on the modified SAMP, all thallium analyses were to be performed using MSA and were not.

No other major deviations, contractual or compliance issues were found in the review of SDG Dede Wel for inorganics.

MMB Reviewer: _____
Signature

Date: _____

Contractor Reviewer: Lidia G. Lina
Signature

Date: 5/10/91

Verifier by: Juan B. Garcia

Date: 5/10/91

DATA VALIDATION SUMMARY REPORT

for the

TUTU WELLS SITE

February 1991 Quarterly Sampling

USEPA REGION II

Standard Operating Procedures HW-2 and HW-6

for the

Contract Laboratory Program

Organic and Inorganic

Data Review

Sample Delivery Group 910070 (Hartman)

May 1991

Prepared for

Tutu Environmental Investigation Committee

**Geraghty & Miller, Inc.
201 W. Passaic Street
Rochelle Park, New Jersey 07662**

TUT 002 2202

GERAGHTY & MILLER, INC.

STANDARD OPERATING PROCEDURE (SOP) NO. HW-6

Revision #7

**EVALUATION OF ORGANIC DATA FOR THE CONTRACT
LABORATORY PROGRAM (CLP)**

INTRODUCTION TO DATA VALIDATION

1.0 Scope

- 1.1 This procedure is applicable to organic data obtained from contractor laboratories working for the Contract Laboratory Program (CLP).
- 1.2 The data validation is based upon analytical and quality assurance requirements specified in the Statement of Work (SOW).

2.0 Responsibilities

Data reviewers will complete the following tasks as assigned by the Data Review Coordinator:

- 2.1 Data Assessment - The reviewer must answer every question on the checklist. All responses shall be in ink.
- 2.2 Data Assessment Narrative (Attachment 1) - Data reviewer is required to use these forms and must match the action in the narrative with the action taken on the Form I(s).
- 2.3 Rejection Summary Form (Attachment 2) - Fill in the total number of analytes measured by different analyses and the number of analytes rejected or flagged as estimated due to corresponding quality control criteria. Place an "X" in the boxes where analyses were not performed or criteria do not apply.
- 2.4 Organic Regional Data Assessment - Data reviewer is also required to fill out Organic Regional Data Assessment Form (Attachment 3).
- 2.5 Telephone Record Log - The data reviewer should enter the bare facts of inquiry before initiating any authorized telephone conversation with a CLP laboratory. After the case review has been completed, mail the white copy of the Telephone Record Log to the laboratory and the pink copy to SMO. File the yellow copy in the Telephone Record Log folder and attach a photocopy of the Telephone Record Log to the completed Data Assessment Narrative.
- 2.6 Forwarded Paperwork - Upon completion of the review, the following are to be forwarded to the Regional Sample Control Center (RSCC) located in the Surveillance and Monitoring Branch:
 - a. data package
 - b. completed assessment checklist
 - c. SMO Contract Compliance Screening (CCS)

Forward four (4) copies of the completed Data Assessment Narrative along with four (4) copies of the Organic Data Assessment Form: one each for the appropriate Regional DPO, the Sample Management Office (SMO), and to the last two addresses of the Data Reviewers Mailing List.

- 2.7 Filed Paperwork - Upon completion of the review, the following are to be filed within the Monitoring and Management Branch (MMB) files:

- a. Telephone Record Log (copy)
- b. Record of Communication (original)
- c. Rejection Summary Form

- 3.0 Rejection of Data - All values determined to be unacceptable on the Organic Analysis Data Sheet (Form I) must be flagged with an "R". As soon as review criteria causes data to be rejected, that data can be eliminated from any further review or consideration.
- 4.0 Acceptance Criteria - In order that the reviews be consistent among reviewers, this Standard Operating Procedure (SOP) should be used. Additional guidance can be found in the Functional Guidelines.
- 5.0 SMO Contract Compliance Screening (CCS) - This is intended to aid the reviewer in locating any problems, both corrected and uncorrected. However, the validation should be carried out even if CCS is not present. Resubmittals received from the laboratory in response to CCS must be used by the reviewer.

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER: _____

LAB: _____

SITE: _____

1.0 Data Completeness and Deliverables

- 1.1 Have any missing deliverables been received and added to the data package.

Yes	NO	N/A
☒	☐	☐

ACTION: Call lab for explanation/resubmittal of any missing deliverables. If lab cannot provide them, note the effort on review of the package under the "Contract Problems/Non-Compliance" section of reviewer narrative.

- 1.2 Was SMO CCS checklist included with package?

☐	☐	☒
--------------------------	--------------------------	-------------------------------------

2.0 Cover Letter/Case Narrative

- 2.1 Is the Narrative or Cover Letter present?

☒	☐	☐
-------------------------------------	--------------------------	--------------------------

- 2.2 Are Case Number and/or SAS number contained in the Narrative or Cover Letter?

☒	☐	☐
-------------------------------------	--------------------------	--------------------------

3.0 Data Validation Checklist

The following checklist is divided into three parts. Part A is filled out if the data package contains any VOA analyses, Part B for any BNA analyses and Part C for Pesticides/PCBs.

Does this package contain:

VOA data?

☒	☐
-------------------------------------	--------------------------

BNA data?

☐	☒
--------------------------	-------------------------------------

Pesticide/PCB data:

☐	☒
--------------------------	-------------------------------------

ACTION: Complete corresponding parts of checklist.

YES NO N/A

PART A: VOA ANALYSES1.0 Traffic Reports and Laboratory Narrative

- 1.1 Are the Traffic Report Forms present for all samples?
- ☒
- ☐
- ☐

ACTION: If no, contact lab for replacement of missing or illegible copies.

- 1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?
- ☒
- ☐
- ☐

ACTION: Use professional judgement to evaluate the effect on the quality of the data.

ACTION: If any sample analyzed as a soil contains more than 50% water, all data should be flagged as estimated (J).

ACTION: If both VOA vials for a sample have air bubbles, flag all positive results "J" and all non-detects "R".

2.0 Holding Times

- 2.1 Have any VOA holding times, determined from date of collection to date of analysis, been exceeded?
- ☐
- ☒
- ☐

If unpreserved, aqueous aromatic volatiles must be analyzed within 7 days of collection and non-aromatic volatiles must be analyzed within 14 days. If preserved with hydrochloric acid and stored at 4°C, then both aromatic and non-aromatic volatiles must be analyzed within 14 days. If uncertain about preservation, contact the sampler to determine whether the samples were preserved.

A ten-day holding time for soil samples is recommended.

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Table of Holding Time Violations

Sample	Sample Matrix	Preserved ?	(See Traffic Report)		Date Analyzed
			Date Sampled	Date Lab Received	
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

ACTION: If holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. The reviewer may determine that non-detect data are unusable ("R").

3.0 Surrogate Recovery (Form II) YES NO N/A

3.1 Are the VOA Surrogate Recovery Summaries (Form II) present for each of the following matrices:

a.	Low Water	☒	—	—
b.	Med Water	☐	—	☒
c.	Low Soil	☐	—	☒
d.	Med Soil	☐	—	☒

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	YES	NO	N/A
3.2 Are all the VOA samples listed on the appropriate Surrogate Recovery Summaries for each of the following matrices:			
a. Low Water	☒	—	—
b. Med Water	☐	—	☒
c. Low Soil	☐	—	☒
d. Med Soil	☐	—	☒
ACTION: Call lab for explanation/resubmittals. If missing deliverables are unavailable, document effect on data under "Conclusions" section of reviewer narrative.			
3.3 Were outliers marked correctly with an asterisk?	☐	—	☒
ACTION: Circle all outliers in red.			
3.4 Was one or more VOA surrogate recovery outside of contract specifications for any sample or method blank?	—	☒	—
If yes, were samples re-analyzed?	☐	—	☒
Were method blanks re-analyzed?	☐	—	☒
ACTION: If surrogate recoveries are > 10% but all do not meet SOW specifications:			
1. Flag all positive results as estimated ("J").			
2. Flag all non-detects as estimated detection limits ("UJ").			
Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and re-analyses. Check the internal standard areas.			
3.5 Are there an transcription/calculation errors between raw data and Form II?	☒	☐	—

		YES	NO	N/A
ACTION:	If large errors exist, call lab for explanation/ resubmittal, make any necessary corrections and note errors under "Conclusions".			

4.0 Matrix Spikes (Form III)

4.1 Is the Matrix Spike Duplicate/Recovery Form (Form III) present?

[✓]	—	—
-----	---	---

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Low Water

[✓]	—	—
-----	---	---

b. Med Water

[]	—	✓
-----	---	---

c. Low Soil

[]	—	✓
-----	---	---

d. Med Soil

[]	—	✓
-----	---	---

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

4.3 How many VOA spike recoveries are outside QC limits?

WaterSoils2 out of 10 N/A out of 10

4.4 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

WaterSoils3 out of 5 N/A out of 5

ACTION: If MS and MSD both have less than 10% recovery for an analyte, negative results for that analyte should be rejected, and positive results should be flagged "J". The above applies only to the sample used for the MS/MSD analysis. Use professional judgement in applying this criterion to other samples in the package.

	YES	NO	N/A
5.0 <u>Blanks (Form IV)</u>			
5.1 Is the Method Blank Summary (Form IV) present?	☒	☐	☐
5.2 Frequency of Analysis: for the analysis of VOA TCL compounds, has a reagent/method blank been analyzed for each set of samples or every 20 samples of similar matrix (low water, med water, low soil, medium soil), whichever is more frequent?	☒	☐	☐
5.3 Has a VOA instrument blank been analyzed at least once every twelve hours for each GC/MS system used?	☒	☐	☐
ACTION: If any method blank data are missing, call lab for explanation/resubmittal. If not available, reject all positive data ("R").			
5.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.			
Is the chromatographic performance (baseline stability) for each instrument acceptable for VOAs?	☒	☐	☐
ACTION: Use professional judgement to determine the effect on the data.			
6.0 <u>Contamination</u>			
NOTE: "Water blanks" and "distilled water blanks" are <u>not</u> used to qualify data. Do not confuse them with the other QC blanks discussed below.			
6.1 Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for VOAs? When applied as described below, the contaminant concentration in these blanks is multiplied by the sample Dilution Factor.	☒	☐	☐
6.2 Do any field/trip/rinse blanks have positive VOA results (TCL and/or TIC)?	☒	☐	☐
ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)			

NOTE: Only field/rinse blanks taken the same day as the samples are used to qualify data. Trip blanks are used to qualify those samples with which they were shipped. Blanks may not be qualified because of contamination in another blank. Blanks may be qualified for surrogate, spectral, tuning or calibration QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

	Sample conc > CRQL but < 10x blank	Sample conc < CRQL & is < 10x blank value	Sample conc > CRQL value & > 10x blank value
Methylene chloride Acetone Toluene 2-butanone	Flag sample result with a 'U'; cross out 'B' flag	Reject sample result and report CRQL; cross out 'B' flag	No qualification is needed
	Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5 x blank value	Sample conc > CRQL value & > 5 blank value
Other Contaminants	Flag sample result with a 'U'; cross out 'B' flag	Reject sample result and report CRQL; cross out 'B' flag	No qualification is needed

YES NO N/A

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R" (unusable).

6.3 Are there field/rinse/equipment blanks associated with every sample?

☐ ☒ ☐

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 GC/MS Tuning and Mass Calibration (Form V)

7.1 Are the GC/MS Tuning and Mass Calibration Forms (Form V) present for Bromofluorobenzene (BFB)?

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☒ ☐ ☐

- 7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for BFB provided for each twelve hour shift? YES NO N/A
[✓] — —
- 7.3 Has a tuning performance compound been analyzed for every twelve hours of sample analysis per instrument? YES NO N/A
[✓] — —

ACTION: If any tuning data are missing, take action specified in 3.2 above.

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available.

DATE	TIME	INSTRUMENT	SAMPLE NUMBER

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

- 7.4 Have an ion abundance criteria been met for each instrument used? YES NO N/A
[✓] — —

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If tuning calibration is in error, flag all associated sample data as unusable ("R"). However, if expanded ion criteria are met (See 1988 Functional Guidelines), the data reviewer may accept data with appropriate qualifiers.

- 7.5 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.) YES NO N/A
— [✓] —
- 7.6 Have the appropriate number of significant figures (two) been reported? (check at least two values, but if errors are found, check more values.) YES NO N/A
[✓] — —

YES NO N/A

ACTION: If large errors exist, call lab for explanation/
resubmittal, make necessary corrections and note
errors under "Conclusions".

7.7 Are the spectra of mass calibration compound acceptable?

[☒] — —

ACTION: Use professional judgement to determine whether
associated data should be accepted, qualified,
or rejected.

8.0 Target Compound List (TCL) Analytes

8.1 Are the Organic Analysis Data Sheets (Form I VOA)
present with required header information on each
page, for each of the following:

a. Samples and/or fractions as appropriate

[☒] — —

b. Matrix spikes and matrix spike duplicates

[☒] — —

c. Blanks

[☒] — —

8.2 Are the VOA Reconstructed Ion Chromatograms, the mass
spectra for the identified compounds, and the data
system printouts (Quant Reports) included in the sample
package for each of the following?

a. Samples and/or fractions as appropriate

[] ☒ —

b. Matrix spikes and matrix spike duplicates (Mass
spectra not required)

[☒] — —

c. Blanks

[☒] — —

ACTION: If any data are missing, take action
specified in 3.2 above.

8.3 Are the response factors shown in the Quant Report?

[] ☒ —

8.4 Is chromatographic performance acceptable with respect to:

Baseline stability

[☒] — —

Resolution

[☒] — —

YES NO N/A

Peak shape

☒

—

—

Full-scale graph (attenuation)

☒

—

—

Other: _____

☐

—

☒

ACTION: Use professional judgement to determine the acceptability of the data.

8.5 Are the lab-generated standard mass spectra of the identified VOA compounds present for each sample?

☒

—

—

ACTION: If any mass spectra are missing, take action specified in 3.2 above. If Lab does not generate their own standard spectra, make note in "Contract Problems/Non-compliance".

8.6 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?

☐☒

—

8.7 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?

☒

—

—

8.8 Do sample and standard relative ion intensities agree within 20%?

☒

—

—

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identification were made, all such data should be rejected, flagged "N" (presumptive evidence of the presence of the compound) or changed to not detected (at the calculated detection limit).

9.0 Tentatively Identified Compounds (TICs)

9.1 Are all Tentatively Identified Compound Forms (Form I, Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "J" qualifier?

☒

—

—

9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

a. Samples and/or fractions as appropriate

YES NO N/A
[✓] — —

b. Blanks

[✓] — —

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "J" qualifier if missing and "N" qualifier to all identified TIC compounds on Form I, Part B.

9.3 Are any TCL compounds (from any fraction) listed as TIC compounds (example: 1,2-dimethylbenzene is xylene - a VOA TCL - and should not be reported as a TIC)?

— [✓] —

ACTION: Flag with "R" any TCL compound listed as a TIC.

9.4 Are all ions present in the reference mass spectrum with a relatively greater 10% also present in the sample mass spectrum?

[✓] — —

9.5 Do TIC and "best match" standard relative ion intensities agree within 20%?

[✓] — —

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate.

10.0 Compound Quantitation and Reported Detection Limits

10.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, RRF were used to calculate Form I result. Were any errors found?

— [✓] —

10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture?

[✓] — —

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and

YES NO N/A

note errors under "Conclusions".

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from other diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with data from the analysis of diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

11.0 Standard Data (GC/MS)

11.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant. Reports) present for initial and continuing calibration?

☒ ☐ ☐

ACTION: If any calibration standard data are missing, take action specified in 3.2 above.

12.0 GC/MS Initial Calibration (Form VI)

12.1 Are the Initial Calibration Forms (Form VI) present and complete for the volatile fraction?

☒ ☐ ☐

12.2 Are response factors stable for volatiles over the concentration range of the calibration (RSD <30%)?

☐ ☒ ☐

ACTION: Circle all outliers in red.

ACTION: When RSD >30%, non-detects may be qualified using professional judgement. Flag all positive results "J". When RSD >90%, flag all non-detects as unusable ("R"). (Region II policy.)

12.3 Do any compounds have an average < 0.05?

☒ ☐ ☐

ACTION: Circle all outliers in red.

ACTION: If any volatile compound has an average

YES NO N/A

RRF < 0.05, flag positive results for that compound as estimated ("J"), and flag non-detects for that compound as unusable ("R").

- 12.4 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD? (Check at least two values but if errors are found, check more.)

— [✓] —

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

13.0 GC/MS Continuing Calibration (Form VII)

- 13.1 Are the Continuing Calibration Forms (Form VII) present and complete for the volatile fraction?

[✓] — —

- 13.2 Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?

[✓] — —

ACTION: List below all sample analyses that were not within twelve hours of the previous continuing calibration analysis.

ACTION: If any forms are missing or no continuing calibration standard has been analyzed within twelve hours of every sample analysis, call lab for explanation/resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable ("R").

- 13.3 Do any continuing calibration standard compounds have a RRF < 0.05?

✓ [] —

ACTION: Circle all outliers in red.

ACTION: If any volatile compound has a RRF < 0.05, flag positive results for that compound as estimated ("J"), and flag non-detects for

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YES NO N/A

that compound as unusable ("R").

13.4 Do any compounds have a % difference between initial and continuing calibration RRF > 25%?

☒ ☐ ☐

ACTION: Circle all outliers in red and qualify associated sample data as outlined in the table below:

% DIFFERENCE

25-50	50-90	> 90
'J' positive results, no action for non detects	'J' positive results, 'UJ' non detects	'J' positive results, "R" non detects

13.5 Are there any transcription/calculation errors in the reporting of average response factors (RRFs) or difference (%D) between initial and continuing RRFs? (Check at least two values but if errors are found, check more.)

☒ ☐ ☐

14.0 Internal Standards (Form VIII)

14.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits for each continuing calibration?

☒ ☐ ☐

ACTION: List all the outliers below.

Sample #	Internal Std	Area	Lower Limit	Upper Limit
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

(Attach additional sheets if necessary.)

ACTION: If the internal standard area count is outside the upper c

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YES NO N/A

lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard. If extremely low area counts are reported, or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable ("R").

- 14.2 Are the retention times of the internal standards within 30 seconds of the associated calibration standard?

[✓] — —

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

15.0 Field Duplicates

- 15.1 Were any field duplicates submitted for VOA analysis?

[✓] — —

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

YES NO N/A

Part B: BNA Analyses1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Forms present for all samples?

[] — —

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or Lab Narrative indicate any problems with the sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

— [] —

ACTION: Use professional judgement to evaluate the effect on the quality of the data.

ACTION: If any sample analyzed as a soil contains more than 50% water, all data should be flagged as estimated (J).

2.0 Holding Times

2.1 Have any BNA holding times, determined from date of collection to date of extraction, been exceeded?

— [] —

Samples for BNA analysis, both soils and waters, must be extracted within seven days of the date of collection. Extracts must be analyzed within 40 days of the date of extraction.

Table of Holding Time Violations

(See Traffic Report)

Sample	Sample Matrix	Date Sampled	Date Lab Received	Date Extracted	Date Analyzed
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

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	YES	NO	N/A
ACTION: If holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.			

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon reanalysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. The reviewer may determine that non-detect data are unusable ("R").

3.0 Surrogate Recovery (Form II)

3.1 Are the BNA Surrogate Recovery Summaries (Form II) present for each of the following matrices:

a. Low Water	☐	___	___
b. Med Water	☐	___	___
c. Low Soil	☐	___	___
d. Med Soil	☐	___	___

3.2 Are all the BNA samples listed on the appropriate Surrogate Recovery Summaries for each of the following matrices:

a. Low Water	☐	___	___
b. Med Water	☐	___	___
c. Low Soil	☐	___	___
d. Med Soil	☐	___	___

ACTION: Call lab for explanation/resubmittals. If missing deliverables are unavailable, document effect on data under "Conclusions" section of reviewer narrative.

3.3 Were outliers marked correctly with an asterisk?	☐	___	___
--	--------------------------	-----	-----

ACTION: Circle all outliers in red.

	YES	NO	N/A
3.4 Were two or more base-neutral <u>OR</u> acid surrogate recoveries out of specification for any sample or method blank?	—	[]	—

If yes, were samples re-analyzed?	[]	—	—
-----------------------------------	-----	---	---

Were method blanks re-analyzed?	[]	—	—
---------------------------------	-----	---	---

ACTION: If all BNA surrogate recoveries are > 10% but two within the base-neutral or acid fraction do not meet SOW specifications, for the affected fraction only (i.e. base-neutral OR acid compounds):

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ").

If any base-neutral OR acid surrogate has a recovery of < 10%:

1. Flag all positive results for that fraction (i.e. all acid OR base-neutral compounds) "J".
2. Flag all non-detects for that fraction "R".

Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and re-analyses. Check the internal standard area.

3.5 Are there any transcription/calculation errors between raw data and Form II.	—	[]	—
--	---	-----	---

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

4.0 Matrix Spikes (Form III)

4.1 Is the Matrix Spike Duplicate/Recovery Form (Form III) present?	[]	—	—
---	-----	---	---

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

	YES	NO	N/A
a. Low Water	☐	☐	☐
b. Med Water	☐	☐	☐
c. Low Soil	☐	☐	☐
d. Med Soil	☐	☐	☐

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

4.3 How many BNA spike recoveries are outside QC limits?

Water

Soils

_____ out of 22 _____ out of 22

4.4 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

Water

Soils

_____ out of 11 _____ out of 11

ACTION: If MS and MSD both have less than 10% recovery for an analyte, negative results for that analyte should be rejected, and positive results should be flagged "J". The above applies only to the sample used for MS/MSD analysis. Use professional judgement in applying this criterion to other samples.

5.0 Blanks (Form IV)

5.1 Is the Method Blank Summary (Form IV) present?

☐ ☐ ☐

5.2 Frequency of Analysis: for the analysis of BNA TCL compounds, has a reagent/method blank been analyzed for each set of samples or every 20 samples of similar matrix (low water, med water, low soil, med soil), whichever is more frequent?

☐ ☐ ☐

5.3 Has a BNA instrument blank been analyzed for each GC/MS system used?

YES NO N/A

[] — —

ACTION: If any method blank data are missing, call lab for explanation/resubmittal. If not available, reject all associated positive data ("R").

5.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.

Is the chromatographic performance (baseline stability) for each instrument acceptable for VOAs?

[] — —

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks" and "distilled water blanks" are validated like any other sample and not used to qualify data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/instrument/reagent blanks have positive results (TCL and/or TIC) for BNAs? When applied as described below, the contaminant concentration in these blanks is multiplied by the sample Dilution Factor.

— [] —

6.2 Do any field/rinse blanks have positive BNA results (TCL and/or TIC)?

— [] —

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: Only field/rinse blanks taken the same day as the samples are used to qualify data. Blanks may not be qualified because of contamination in another blank. Blanks may be qualified for surrogate, spectral, tuning or calibration QC problems.

YES NO N/A

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

	Sample conc > CRQL but < 10x blank	Sample conc < CRQL & is < 10x blank value	Sample conc > CRQL value & > 10x blank value
Common Phthalate Esters	Flag sample result with a 'U'; cross out 'B' flag	Reject sample result and report CRQL; cross out 'B' flag	No qualification is needed
	Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5 x blank value	Sample conc > CRQL value & > 5 blank value
Other Contaminants	Flag sample result with a 'U'; cross out 'B' flag	Reject sample result and report CRQL; cross out 'B' flag	No qualification is needed

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R" (unusable).

6.3 Are there field/rinse/equipment blanks associated with every sample? ☐ ☐ ☐

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank.
Exception: samples taken from a drinking water tap not have associated field blanks.

7.0 GC/MS Tuning and Mass Calibration (Form V)

7.1 Are the GC/MS Tuning and Mass Calibration Forms (Form V) present for Decafluorotriphenylphosphine (DFTPP)? ☐ ☐ ☐

7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each twelve hour shift? ☐ ☐ ☐

7.3 Has a tuning performance compound been analyzed for every twelve hours of sample analysis per instrument? ☐ ☐ ☐

YES NO N/A

ACTION: If any tuning data are missing, take action specified in 3.2 above.

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available.

DATE	TIME	INSTRUMENT	SAMPLE NUMBER

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

7.4 Have the ion abundance criteria been met for each instrument used? ☐ ☐ ☐

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If tuning calibration is in error, flag all associated sample data as unusable ("R"). However, if expanded ion criteria are met (See 1988 Functional Guidelines), the data review may accept data with appropriate qualifiers.

7.5 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more values.) ☐ ☐ ☐

7.6 Have the appropriate number of significant figures (two) been reported? (Check at least two values, but if errors are found, check more values.) ☐ ☐ ☐

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and note errors under "Conclusions".

7.7 Are the spectra of the mass calibration compound acceptable? ☐ ☐ ☐

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

	YES	NO	N/A
8.0 <u>Target Compound List (TCL) Analytes</u>			
8.1 Are the Organic Analysis Data Sheets (Form I BNA) present with required header information on each page, for each of the following:			
a. Samples and/or fractions	[]	—	—
b. Matrix spikes and matrix spike duplicates	[]	—	—
c. Blanks	[]	—	—
8.2 Are the BNA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			
a. Samples and/or fractions as appropriate	[]	—	—
b. Matrix spikes and matrix spike duplicates (Mass spectra not required)	[]	—	—
c. Blanks	[]	—	—
ACTION: If any data are missing, take action specified in 3.2 above.			
8.3 Are the response factors shown in the Quant Report?	[]	—	—
8.4 Is chromatographic performance acceptable with respect to:			
Baseline stability	[]	—	—
Resolution	[]	—	—
Peak shape	[]	—	—
Full-scale graph (attenuation)	[]	—	—
Other: _____	[]	—	—

ACTION: Use professional judgement to determine the acceptability of the data.

- | | YES | NO | N/A |
|---|--------------------------|--------------------------|--------------------------|
| 8.5 Are the lab-generated standard mass spectra of the identified BNA compounds present for each sample? | ☐ | ☐ | ☐ |
| 8.6 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration? | ☐ | ☐ | ☐ |
| 8.7 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum? | ☐ | ☐ | ☐ |
| 8.8 Do sample and standard relative ion intensities agree within 20%? | ☐ | ☐ | ☐ |

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected, flagged "N" (presumptive evidence of the presence of the compound) or changed to not detected (at the calculated detection limit).

9.0 Tentatively Identified Compounds (TIC)

- | | | | |
|--|--------------------------|--------------------------|--------------------------|
| 9.1 Are all Tentatively Identified Compound Forms (Form I, Part B) present; and do listed TICs include scan number or retention time, estimated concentration and "J" qualifier? | ☐ | ☐ | ☐ |
| 9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following: | | | |
| a. Samples and/or fractions as appropriate | ☐ | ☐ | ☐ |
| b. Blanks | ☐ | ☐ | ☐ |

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "J" qualifier if missing and "N" qualifier to all identified TIC compounds on Form I, Part B.

- | | | | |
|---|--------------------------|--------------------------|--------------------------|
| 9.3 Are any TCL compounds (from any fraction) listed as TIC compounds (example: 1,2-dimethylbenzene is xylene -- a VOA TCL -- and should not be reported as a TIC)? | ☐ | ☐ | ☐ |
|---|--------------------------|--------------------------|--------------------------|

ACTION: Flag with "R" any TCL compound listed as a TIC

- | | YES | NO | N/A |
|--|--------------------------|--------------------------|--------------------------|
| 9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum? | ☐ | ☐ | ☐ |
| 9.5 Do TIC and "best match" standard relative ion intensities agree within 20%? | ☐ | ☐ | ☐ |

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate.

10.0 Compound Quantitation and Reported Detection Limits

- | | | | |
|--|--------------------------|--------------------------|--------------------------|
| 10.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found? | ☐ | ☐ | ☐ |
| 10.2 Are the CRQLs adjusted to reflect dilutions and, for soils, sample moisture? | ☐ | ☐ | ☐ |

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with data from the analysis of diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

	YES	NO	N/A
11.0 Standards Data (GC/MS)			
11.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant. Reports) present for initial and continuing calibration?	☐	☐	☐
12.0 GC/MS Initial Calibration (Form VI)			
12.1 Are the Initial Calibration Forms (Form VI) present and complete for the BNA fraction?	☐	☐	☐
ACTION: If any calibration standard forms are missing, take action specified in 3.2 above.			
12.2 Are response factors stable for BNAs over the concentration range of the calibration (RSD <30%)?	☐	☐	☐
ACTION: Circle all outliers in red.			
ACTION: When RSD >30%, non-detects may be qualified using professional judgement. Flag all positive results "J". When RSD >90%, flag all non-detects as unusable ("R"). (Region II policy.)			
12.3 Do any compounds have a RRF < 0.05?	☐	☐	☐
ACTION: Circle all outliers in red.			
ACTION: If any BNA compound has an average RRF < 0.05, flag positive results for that compound as estimated ("J"), and flag non-detects for that compound as unusable ("R").			
12.4 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or %RSD? (Check at least two values but if errors are found, check more.)	☐	☐	☐
ACTION: Circle errors in red.			
ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".			

13.0 GC/MS Continuing Calibration (Form VII)

YES NO N/A

13.1 Are the Continuing Calibration Forms (Form VII) present and complete for the BNA fraction?

[] — —

13.2 Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument?

[] — —

ACTION: List below all sample analyses that were not within twelve hours of the previous continuing calibration analysis.

ACTION: If any forms are missing or no continuing calibration standard has been analyzed within twelve hours of every sample analysis, call lab for explanation/resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable ("R").

13.3 Do any continuing calibration standard compounds have a RRF < 0.05? — [] —

ACTION: Circle all outliers in red.

ACTION: If any BNA compound has a RRF < 0.05, flag positive results for that compound as estimated ("J"), and flag non-detects for that compound as unusable ("R").

13.4 Do any compounds have a % difference between initial and continuing calibration RRF > 25%? — [] —

ACTION: Circle all outliers in red and qualify associated sample data as outlined in the table below:

% DIFFERENCE

25-50	50-90	> 90
'J' positive results, no action for non detects	'J' positive results, 'UJ' non detects	'J' positive results, "R" non detects

YES NO N/A

13.5 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or difference (%D) between initial and continuing RRFs? (check at least two values but if errors are found, check more.)

— [] —

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

14.0 Internal Standards (Form VIII)

14.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits for each continuing calibration?

[] — —

ACTION: List all the outliers below.

Sample #	Internal Std	Area	Lower Limit	Upper Limit
----------	--------------	------	-------------	-------------

_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

(Attach additional sheets if necessary.)

ACTION: If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard. If extremely low area counts are reported, or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable ("R").

14.2 Are the retention times of the internal standards within 30 seconds of the associated calibration standard?

[] — —

ACTION: Professional judgement should be used to qualify data retention times differ by more than 30 seconds.

YES NO N/A

15.0 Field Duplicates

15.1 Were any field duplicates submitted for BNA analysis?

[] — —

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

YES NO N/A

PART C: PESTICIDE/PCB ANALYSES1.0 Traffic Reports and laboratory Narrative

1.1 Are the Traffic Report Forms present for all samples?

[] — —

ACTION: If no, contact lab for replace of missing or illegible copies.

1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data?

— [] —

ACTION: Use professional judgement to evaluate the effect on the quality of the data.

ACTION: If any sample analyzed as a soil contains more than 50% water, all data should be flagged as estimated (J).

2.0 Holding Times

2.1 Have any PEST/PCB holding times, determined from date of collection to date of extraction, been exceeded?

— [] —

Samples for PEST/PCB analysis, both soils and waters, must be extracted within seven days of the date of collection. Extracts must be analyzed within 40 days of the date of extraction.

3.0 Surrogate Recovery (Form II)

3.1 Are the PEST/PCB Surrogate Recovery Summaries (Form II) present for each of the following matrices:

a. Low Water

[] — —

b. Med Water

[] — —

c. Low Soil

[] — —

d. Med Soil

[] — —

3.2 Are all the PEST/PCB samples listed on the appropriate Surrogate Recovery Summaries for each of the following matrices:

	YES	NO	N/A
a. Low Water	☐	☐	☐
b. Med Water	☐	☐	☐
c. Low Soil	☐	☐	☐
d. Med Soil	☐	☐	☐

ACTION: Call lab for explanation/resubmittals. If missing deliverables are unavailable, document effect on data under "Conclusions" section of reviewer narrative.

3.3 Were outliers marked correctly with an asterisk? ☐ ☐ ☐

ACTION: Circle all outliers in red.

3.4 Was surrogate (DBC) recovery outside of the contract specification for any sample or blank? ☐ ☐ ☐

ACTION: No qualification is done if surrogates are diluted beyond detection. If recovery is below contract limit (but above zero), flag all results for that sample "J". If recovery is zero, flag positive results "J" and non-detects "R". If recovery for the blank is zero, flag non-detects for all associated samples "R". If recovery is above contract limit, flag all positive results for that sample "J", unless in the reviewers professional judgement the high recovery is due to co-eluting interference (check the associated blank - if recovery is high there also, flag the sample data).

3.5 Are there any transcription/calculation errors between raw data and Form II? ☐ ☐ ☐

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

4.0 Matrix Spikes (Form III)

4.1 Is the Matrix Spike Duplicate/Recovery Form (Form III) present? ☐ ☐ ☐

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

	YES	NO	N/A
a. Low Water	☐	☐	☐
b. Med Water	☐	☐	☐
c. Low Soil	☐	☐	☐
d. Med Soil	☐	☐	☐

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

4.3 How many PEST/PCB spike recoveries are outside QC limits?

<u>Water</u>	<u>Soils</u>
_____ out of 12	_____ out of 12

4.4 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

<u>Water</u>	<u>Soils</u>
_____ out of 6	_____ out of 6

ACTION: If MS and MSD both have less than zero recovery for an analyte, negative results for that analyte should be rejected, and positive results should be flagged "J". The above applies only to the sample used for MS/MSD analysis. Use professional judgement in applying this criterion to other samples.

5.0 Blanks (Form IV)

5.1 Is the Method Blank Summary (Form IV) present? ☐ ☐ ☐

5.2 Frequency of Analysis: for the analysis of Pesticide TCL compounds, has a reagent/method blank been analyzed for each set of samples or every 20 samples of similar matrix (low water, med water, low soil, medium soil), whichever is more frequent? ☐ ☐ ☐

5.3 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable for PEST/PCBs?

YES NO N/A

[] — —

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/instrument/reagent blanks have positive results for PEST/PCBs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample Dilution Factor.

— [] —

6.2 Do any field/rinse blanks have positive PEST/PCB results?

— [] —

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: Only field/rinse blanks taken the same day as the samples are used to qualify data. Blanks may not be qualified for surrogate, spectral, tuning or calibration QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL value & > 5x blank value
Flag sample result with a 'U'; cross out 'B' flag	Reject sample result and report CRQL; cross out 'B' flag	No qualification is needed

	YES	NO	N/A
6.3 Are there field/rinse/equipment blanks associated with every sample?	☐	☐	☐

7.0 Calibration and GC Performance

7.1 Are the following Gas Chromatograms and Data System Printouts for both Primary and Confirmation (confirmation standards are not required if there are no positive results above CRQL) column present:

a. Evaluation Standard Mix A	☐	☐	☐
b. Evaluation Standard Mix B	☐	☐	☐
c. Evaluation Standard Mix C	☐	☐	☐
d. Individual Standard Mix A	☐	☐	☐
e. Individual Standard Mix B	☐	☐	☐
f. Multi-component Pesticides Toxaphene & Chlordane	☐	☐	☐
g. Aroclors 1016/1260	☐	☐	☐
h. Aroclors 1221, 1232, 1242, 1248, 1254	☐	☐	☐

ACTION: If no, take action specified in 3.2 above.

7.2 Is Form VIII Pest-1 present and complete for each GC column (primary and confirmation) and each 72 hour sequence of analyses?	☐	☐	☐
---	--------------------------	--------------------------	--------------------------

ACTION: If no, take action specified in 3.2 above.

7.3 Are there any transcription/calculation errors between raw data and Form VIII?	☐	☐	☐
--	--------------------------	--------------------------	--------------------------

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

7.4 Has the total breakdown on quantitation or confirmation column exceeded 20% for DDT?	☐	☐	☐
--	--------------------------	--------------------------	--------------------------

- for Endrin?

☐ ☐

or if Endrin aldehyde and 4,4'-DDD co-elute and there is a peak at their retention time, has the combined DDT and Endrin breakdown exceeded 20%?

YES NO N/A

— [] —

ACTION:

- a. If DDT breakdown is greater than 20% on quantitation column beginning with the samples following the last in control standard:
 1. Flag all positive DDT results "J".
 2. If DDT was not detected but DDD and/or DDE are positive, flag the DDT non-detect "R".
 3. Flag positive DDD and DDE results "JN".
 4. If DDT breakdown is > 20% on confirmation column and DDT is identified on quantitation column but not on confirmation column, use professional judgement to determine whether DDT should be reported on Form I (if reported, flag result "N").
- b. If Endrin breakdown is > 20% on quantitation column, beginning with the samples following the last in control standard:
 1. Flag all positive Endrin results "J".
 2. If Endrin was not detected, but Endrin Aldehyde and/or Endrin Ketone are positive, flag the Endrin non-detect "R".
 3. Flag Endrin Ketone positive results "JN".
 4. If Endrin breakdown is > 20% on confirmation column and Endrin is identified on quantitation column but not on confirmation column, use professional judgement to determine whether Endrin should be reported on Form I (if reported, flag result "N").
- c. If the combined breakdown is used (it can only be used if the conditions in 7.4 above are met) and is > 20% on quantitation column beginning with the last in control standard, take the actions specified in 7.4 a and b above. If the combined breakdown is > 20% on confirmation column and Endrin or DDT is identified on quantitation column but not on confirmation column, use professional judgement to determine whether Endrin or DDT should be reported on Form I (if reported, flag result "N").

7.5 Is the linearity check RSD of all four calibration factors < 10% for the quantitation column?

[] — —

ACTION: If no, flag positive hits for all pesticide and PCB analytes "J" for all associated samples. Do not flag toxaphene or DDT if they are quantified from a 3-point calibration curve.

- | | YES | NO | N/A |
|---|--------------------------|--------------------------|--------------------------|
| 7.6 Is the % difference between EVAL A and each analysis (quantitation and confirmation) DBC retention time within QC limits (2% for packed column 0.3% for capillary [I.D. < 0.32 mm], 1% for megabore [0.32 < 2 mm])? | ☐ | ☐ | ☐ |

ACTION: DBC retention time cannot be evaluated if DBC is not detected. If it is present and has a retention time out of QC limits, then use professional judgement to determine the reliability of the analysis and flag results "R", if appropriate.

- | | | | |
|---|--------------------------|--------------------------|--------------------------|
| 7.7 Was the proper analytical sequence followed for each 72 hour period of analyses (page PEST D-36 in 8/87 SOW). | ☐ | ☐ | ☐ |
|---|--------------------------|--------------------------|--------------------------|

ACTION: If no, use professional judgement to determine the severity of the effect on the data and accept or reject accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the calibration was also out of limits.

8.0 Pesticide/PCB Standards Summary

- | | | | |
|---|--------------------------|--------------------------|--------------------------|
| 8.1 Is Form IX present and complete for each GC column and 72 hr. sequence of analyses? | ☐ | ☐ | ☐ |
|---|--------------------------|--------------------------|--------------------------|

ACTION: If no, take action specified in 3.2 above.

- | | | | |
|--|--------------------------|--------------------------|--------------------------|
| 8.2 Are there any transcription/calculation errors between raw data and Form IX? | ☐ | ☐ | ☐ |
|--|--------------------------|--------------------------|--------------------------|

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

- | | | | |
|---|--------------------------|--------------------------|--------------------------|
| 8.3 Is DDT retention time for packed columns > 12 min (except OV-1 and OV-101 columns)? | ☐ | ☐ | ☐ |
|---|--------------------------|--------------------------|--------------------------|

ACTION: If no, check that there is adequate resolution between individual components. If not, flag results for compounds that interfere with each other (co-elute) "R".

- | | | | |
|---|--------------------------|--------------------------|--------------------------|
| 8.4 Do all standard retention times fall within the windows established for the first IND A and IND B analyses? | ☐ | ☐ | ☐ |
|---|--------------------------|--------------------------|--------------------------|

YES NO N/A

ACTION: Beginning with the samples following the last in control standard, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and DBC is visible non-detects are valid. If peaks are present and cannot be identified through "pattern recognition" or a consistent shift in standard retention times, flag all affected compound results "R".

- 8.5 Are the continuing calibration standard calibration factors within 15% (for confirmation column) of the initial (at beginning of 72 hr sequence) calibration factors? ☐ ☐ ☐

ACTION: If no, flag all associated positive results "J". Use professional judgement to determine whether or not to flag non-detects.

9.0 Pesticide/PCB Identification

- 9.1 Is Form X complete for every sample in which a pesticide or PCB was detected? ☐ ☐ ☐

ACTION: If no, take action specified in 3.2 above.

- 9.2 Are there any transcription errors between raw data and Form X? ☐ ☐ ☐

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

- 9.3 Are retention times of sample compounds within the calculated retention time windows for both quantitation and confirmation analyses? ☐ ☐ ☐

Was GC/MS confirmation provided when required (when compound concentration is > 10 ug/ml in final extract)? ☐ ☐ ☐

ACTION: Reject ("R") all positive results (meeting quantitation column criteria, but missing confirmation by a second column or GC/MS (if appropriate). Also, reject ("R") all positive results not meeting retention time window criteria unless associated standard compounds are similarly biased (i.e. base on RRT to DBC).

- 9.4 Check chromatograms for false negatives, especially for the multiple peak components toxaphene and PCB's. Were there any false negatives?

YES NO N/A

— [] —

ACTION: If appropriate PCB standards were not analyzed, or if the lab performed no confirmation analysis, flag the appropriate data with an "R".

10.0 Compound Quantitation and Reported Detection Limits

- 10.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Were any errors found?

— [] —

NOTE: Simple peak pesticide results can be checked for rough agreement between quantitative results obtained on the two GC columns. The reviewer should use professional judgement to decide whether a much larger concentration obtained on one column versus the other indicate the presence of an interfering compound. If an interfering compound is indicated, the lower of the two values should be reported and qualified as presumptively present at an estimated quantity ("JN"). This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate that the presence of interferences has obscured the attempt at a second column confirmation.

- 10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, sample moisture?

— [] —

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors under "Conclusions".

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with data from the analysis of diluted

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YES NO N/A

sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

11.0 Chromatogram Quality

- 11.1 Were baselines stable? ☐ — —
- 11.2 Were any electropositive displacement (negative peaks) or unusual peaks seen? — ☐ —
- 11.3 Were early eluting peaks (for early eluting analytes) resolved to baseline? ☐ — —

ACTION: For 11.1 and 11.2, comment only. For 11.3, reject ("R") those analytes that are not sufficiently resolved.

12.0 Field Duplicates

- 12.1 Were any field duplicates submitted for PEST/PCB analysis? ☐ — —

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

SDG HARTMA (CEIMAC #910070)
TUTU WELLS SITE

CONTAMINATED VOLATILE METHOD BLANKS AND ASSOCIATED SAMPLES

VBLK02	Bryan, Demitri, Ramsey, VIHA III
VBLK03	Hartman II, Tillett, Trip Blank (2/6/91), Trip Blank (2/7/91), Hartman II (MSD)
VBLK04	Field Blank, Gassett, LaPlace, Lavergne, Leonard, Hartman II (MS)
VBLK05	Gassett (DL), LaPlace (DL) Lavergne (DL)
VBLK06	Harvey

SDG HARTMA (CEIMAC #910070)
TUTU WELLS SITE

CONTAMINATED TRIP BLANKS AND ASSOCIATED SAMPLES

Trip Blank (2/6/91)	Steele, Ramsey, Hartman II Harvey, Tillett, Hartman II (MS) Hartman II (MSD)
Trip Blank (2/7/91)	Leonard, VIHA III, Bryan, LaPlace, Lavergne, Demitri, Gassett, Field Blank

SDG HARTMA (CEIMAC #910070)
TUTU WELLS SITE

CONTAMINATED FIELD BLANKS AND ASSOCIATED SAMPLES

Field Blank (2/7/91)	Leonard, VIHA III, Bryan, LaPlace, Lavergne, Demitri, Gassett
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TOTAL REVIEW

CLP DATA ASSESSMENT

Functional Guidelines for Evaluating Organics Analysis

Case No. 910070 SDG No. Hartman LABORATORY Ceimic SITE Tutu TEIC

DATA ASSESSMENT:

The current functional guidelines (1988) for evaluating organic data have been applied.

All data are valid and acceptable except those analytes which have been qualified with a "J" (estimated, "U" (non-detects), "R" (unusable), or "JN" (presumptive evidence for the presence of the material at an estimated value). All action is detailed on the attached sheets.

Two facts should be noted by all data users. First, the "R" flag means that the associated value is unusable. In other words, due to significant QC problems the analysis is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on data tables because they cannot be relied upon, even as a last resort. The second fact to keep in mind is that no compound concentration, even if it has passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data but any value potentially contains error.

Reviewer's

Signature: Lidia Gilman Date: 5/10/91

Verified by: Juan Garcia/TVD Date: 5/10/91

1. HOLDING TIME:

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detects (sample quantitation limits) will be flagged as estimated, "J", or unusable, "R", if the holding times are grossly exceeded.

The following action was taken in the samples and analytes shown due to excessive holding time.

All samples including diluted sample reanalyses were analyzed within the prescribed holding time for volatile organics. No qualification of data has been assessed due to this criterion.

2. BLANK CONTAMINATION

Quality assurance (QA) blanks, i.e., method, trip field, rinse and water blanks are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Trip blanks measure cross-contamination of samples during shipment. Field blanks measure cross-contamination of samples during field operations. If the concentration of the analyte is less than 5 times the blank contaminant level (10 times for the common contaminants), the analytes are qualified as non-detects, "U". The following analytes in the samples shown were qualified with "U" for these reasons:

A) Method blank contamination

All method blanks associated with the volatile analyses of SDG Hartman were reviewed to determine laboratory contamination in the samples. Method blanks VBLK02, VBLK03, and VBLK04 were used in the low level Method 524.2 analysis of samples associated with this SDG. In the previously agreed to analysis of volatiles using the contract laboratory protocol (CLP) routine analytical services (RAS) methodology for those samples exceeding the linear range of Method 524.2, volatile blanks VBLK05, VBLK07, VBLK06, and VBLK08 were reviewed to assess laboratory contamination levels.

In the analysis of method blank VBLK02, although no target compound list (TCL) organic compounds were detected, one tentatively identified compound

(TIC) at retention time (RT) 22.79 was reported at an estimated concentration of 9 micrograms per liter (ug/L). This same TIC was detected in the following samples associated with VBLK02: Bryan, Demitri, Ramsey and VIHA III. Since the TIC concentration was less than five times (5x) the associated blank level in all of the samples listed above, the sample TIC was qualified as unusable (R) as per Region II validation guidelines.

In method blank VBLK03, one TIC was detected at RT 24.04 at an estimated level of 3 ug/L. In the review of associated samples, this same contaminant was detected in Hartman II, Trip blank (2/6/91) and Trip blank (2/7/91). As the sample TIC concentrations were less than 5x the blank contamination level, the TIC concentrations were rejected and qualified as unusable (R). No target compounds were detected in VBLK03.

Method blank VBLK04 was reported to contain a TIC at RT 22.64 at an estimated concentration of 3 ug/L. This same unknown was detected in the following samples and qualified as unusable (R) using the 5x contamination guideline: Field blank, LaPlace, Lavergne and Léonard. As the TIC level was equal to five times the blank level in sample Gassett, presumptive evidence of this TIC remains reported for this sample.

In the analysis of method blank VBLK05, acetone and methylene chloride were detected at 10 ug/L and an estimated level of 3 ug/L, respectively. No other TCL or tentatively identified organic compounds were found. As the only samples associated with this blank were for sample reanalyses for high levels of analytes other than these contaminants, no qualification of data was necessary for acetone and methylene chloride levels in the samples Gassett (DL), LaPlace (DL) and Lavergne (DL).

Method blank VBLK06 was reported to contain estimated levels of acetone (9 ug/L) and methylene chloride (3 ug/L). Sample Harvey is associated with this blank and was found to contain these same analytes at low levels. Following the validation guidelines, both contaminants were qualified as non-detects (U) in this sample after correction for sample dilution using the 10x review criteria for common contaminants.

Sample data associated with method blanks VBLK07 and VBLK08 were not qualified based on blank contamination as both these blanks were free of any TCL and/or TIC contamination.

In reviewing the Tillett (DL) sample analysis by the CLP RAS protocol, a 200 ppb of acetone result was reported by the laboratory. Although no corresponding method blank contamination was associated with the sample analysis (see results for VBLK07), the sample result has been summarily rejected (R) by the data reviewer since the low level volatile analysis by Method 524.2 for sample Tillett did not confirm the presence of acetone in the sample. Acetone results were subsequently rejected in all low level analyses due to a deficient relative response factor (RRF 0.027) in the GC/MS initial calibration. Nevertheless, the level of acetone detected in the RAS volatile analysis for Tillett would, in all probability, have been detected at least to a minimal degree in the low level analysis. Consequently, the acetone found in the Tillett RAS analysis was attributed to probable contamination during the analytical process and the sample result rejected.

- B) Field or rinse blank contamination ("water blanks" or "distilled water blanks" are validated like any other sample)

A field blank was associated with samples collected on February 7, 1991 (samples Leonard, VIHA III, Bryan, LaPlace, Lavergne, Demitri, and Gassett). Methylene chloride was detected at a level of 2 ug/L in addition to the following estimated levels of TIC:

Unknown	RT 5.58	3 ug/L
C6H14 isomer	RT 6.42	97 ug/L
C6H12 isomer	RT 7.57	100 ug/L
Unknown	RT 22.62	12 ug/L*

* Rejected based on VBLK04 blank contamination level

In sample Gassett, the TICs at RT 6.42 and 7.57 have been qualified as unusable (R) based on the 5x rule for blank contaminants. With respect to the methylene chloride contamination, all associated samples were qualified as non-detects (U) using the 10x criteria based on the field blank and associated trip blank (Trip blank 2/7/91) contaminant levels. No other qualifications were made based on the field blank assessment.

- C) Trip blank contamination

The trip blank associated with samples collected on February 6, 1991 (Trip blank 2/6/91) was reported to contain low level contamination of chloromethane

(estimated 1 ug/L), methylene chloride (2 ug/L), and acetone (4 ug/L). Additionally, an unknown was detected at RT 24.04, but subsequently qualified as method blank contamination based on the analysis of VBLK03. The following samples have been qualified as non-detects for acetone and/or methylene chloride using the 10x criteria for common contaminants: Ramsey, Tillett, Hartman II (MS and MSD, only).

The trip blank associated with samples collected on February 7, 1991 (Trip blank 2/7/91) was reported to contain similar contaminants and levels as detected in Trip blank (2/6/91). The following samples were qualified as non-detects for methylene chloride and/or acetone contamination levels based on the trip blank: Leonard, VIHA III, Bryan, LaPlace, Lavergne, Demitri and Gassett. No qualification of the field blank was made based on the associated trip blank data.

3. MASS SPECTROMETER TUNING:

Tuning and performance criteria are established to ensure adequate mass resolution, proper identification of compounds, and to some degree, sufficient instrument sensitivity. These criteria are not sample specific. Instrument performance is determined using standard materials. Therefore, these criteria should be met in all circumstances. The tuning standard for volatile organics is bromofluorobenzene (BFB) and for semi-volatiles is decafluorotriphenylphosphine (DFTPP).

If the mass calibration is in error, all associated data will be classified as unusable, "R".

The mass calibrations associated with volatile analyses for SDG Hartman have all met the acceptance criteria to ensure adequate resolution, instrument response and proper compound identification.

4. CALIBRATION

Satisfactory instrument calibration is established to ensure that the instrument is capable of producing acceptable quantitative data. An initial calibration demonstrates that the instrument is capable of giving acceptable performance at the beginning of an experimental sequence. The continuing calibration checks document that the instrument is giving satisfactory daily performance.

A) RESPONSE FACTOR:

The response factor measures the instrument's response to specific chemical compounds. The response factor for the Target Compound List (TCL) must be ≥ 0.05 (a ratio of areas) in both the initial and continuing calibrations. A value < 0.05 indicates a serious detection and quantitation problem (poor sensitivity). Analytes detected in the sample will be qualified as estimated, "J". All non-detects for that compound will be rejected ("R").

The initial calibration for all drinking water analyses performed on 2/11/91 on GC/MS instrument ID MS2 resulted in response factors of < 0.05 for acetone and 2-butanone. All samples (with the exception of well samples Harvey, Steele and diluted reanalyses for Gassett, LaPlace and Lavergne) have been qualified due to the deficient instrument response as described above. As none of the above resulted in positive hits, all associated sample data for these compounds has been qualified as unusable (R).

For the initial calibration performed on 2/7/91 on instrument ID MS5, all response factors were greater than 0.05 and resulted in no data qualifications.

In the continuing calibrations associated with the low level Method 524.2 analyses, two response factors were below the control limit (0.05) in each respective continuing calibration standard (VSTD010). Namely, acetone and 2-butanone were each below 0.05 on instrument ID MS2 injected on 2/12/91, 2/14/91 and 2/20/91. In addition to these two compounds, 2-hexanone also displayed an unacceptable response factor in the continuing calibration standard analyzed on 2/12/91. Acetone and 2-butanone results were qualified as unusable (R) previously based on deficient initial calibration response factors. Qualifications have been applied to 2-hexanone results for the following samples: Ramsey, VIHA III, Bryan and Demitri. All of the above data for this compound was rejected following the validation guidelines.

There were no deficient response factors for any of the continuing calibrations associated with the CLP RAS analysis of volatile samples for instrument ID MS5 for SDG Hartman.

5. CALIBRATION:

A) PERCENT RELATIVE STANDARD DEVIATION (%RSD) AND PERCENT DIFFERENCE (%D):

Percent RSD is calculated from the initial calibration and is used to indicate the stability of the specific compound response factor over increasing concentration. Percent D compares the response factor of the continuing calibration check to the mean response factor (RRF) from the initial calibration. Percent D is a measure of the instrument's daily performance. Percent RSD must be $<30\%$ and %D must be $<25\%$. A value outside of these limits indicates potential detection and quantitation errors. For these reasons, all positive results are flagged as estimated, "J" and non-detects are flagged "UJ" (if %D or RSD $> 50\%$). If there is a gross deviation of %RSD and %D, the non-detects may be rejected ("R").

The criteria above were modified slightly to reflect the %D criteria permissible for analyses performed in accordance with Method 524.2. As per the low level method, the response factor in the continuing calibration must be within 30% of the mean value measured in the initial calibration. As such, no qualifications were made for compounds in the continuing calibration standards with %D of $<30\%$ which were used in quantitation of low level volatile analyses. All other criteria and subsequent qualifications remain unchanged with respect to %RSD and %D, including the 25%D criteria for continuing calibration verification for samples analyzed by the CLP RAS methodology.

In the initial calibration associated with low level drinking water analyses (analyzed 2/11/91 on MS2), the %RSD for acetone and 2-hexanone were 51.2% and 31.2%, respectively. As the %RSD for 2-hexanone is less than 50% and all samples were non-detects for this compound, no data qualifiers were assessed. The acetone concentrations remain unusable for all associated samples as a result of deficient response factors.

The %RSD for acetone in the initial calibration on instrument ID MS5 (injected on 2/7/91) was greater than 30%. Samples Harvey and Steele are associated with this calibration (excluding sample dilutions for Tillett, Gassett, LaPlace and Lavergne for high concentrations of other analytes) and resulted in no qualifications to these compounds based on non-detected values for this analyte.

The %Ds for 2-butanone and 4-methyl-2-pentanone were $>30\%$ in the continuing calibration (VSTD010) injected on 2/12/91 on instrument ID MS2. The 2-butanone was qualified as unusable previously due to a deficient initial response factor. No qualifications were assessed for 4-methyl-2-pentanone for any related samples since %D was $<50\%$ and all samples were non-detects for this compound. Samples Ramsey, VIHA III, Bryan and Demitri are associated with this calibration.

In the continuing calibration VSTD010 analyzed on 2/14/91 on instrument ID MS2, chloromethane was reported with a %D of 31.4%. Samples Hartman II, Hartman II (MSD), and Tillett remain unqualified for this compound based on non-detected values. The estimated qualifiers for chloromethane (qualified due to an estimated value below the quantitation limit) remain as reported due to the associated trip blank contamination and the applicable %D criterion.

In the analysis of the continuing calibration standard VSTD010 injected on 2/20/91 on instrument MS2, the %D were greater than 30% for the following:

<u>Compound</u>	<u>%D</u>
chloromethane	49.6
vinyl chloride	44.2
chloroethane	34.7
acetone	40.7
carbon disulfide	37.3

Samples Hartman II (MS), Gassett, Leonard, LaPlace, Lavergne and the Field Blank were associated with this daily standard. As none of these samples had positive results for any of the listed compounds, no data qualifiers were applied as per the <50% RF criterion stipulated for non-detects.

In the continuing calibration analyses supporting the CLP RAS volatile analyses, the daily standard VSTD050 injected on 2/12/91 on instrument ID MS5 reported %D greater than 25% for 1,2-dichloroethane (27.4%) and vinyl acetate (32.6%). Well sample Harvey was the quantitated using this continuing calibration standard. As neither of these compounds were detected in the sample and %D is less than 50%, the non-detects remain unqualified.

The %D for vinyl acetate was 56.8% in the continuing calibration standard VSTD050 injected on 2/15/91 on instrument ID MS5. As this standard applies to the Tillett sample dilution for total 1,2-dichloroethene and tetrachloroethene, the high %D has no impact on the Tillett sample vinyl acetate result and the non-detect remains unqualified.

In the analysis of continuing calibration standard VSTD050 injected on 2/16/91 on instrument ID MS5, the following compounds were calculated to have >25 %D:

Chloromethane	46.2%
Bromomethane	26.4%
Carbon Tetrachloride	27.1%
Vinyl Acetate	54.4%

In associated well sample Steele, the non-detect vinyl acetate result was qualified as estimated (UJ) as the %D was greater than 50%. For all other compounds listed above, no qualification was made to Steele sample data using the applicable %D criterion.

In the continuing calibration standard VSTD050 injected on 2/21/91 on instrument ID MS5, %D was greater than 25% for acetone (28.6%), vinyl acetate (39.8%), and trans-1,3-dichloropropene (28.9%). As none of these response factors for the affected compounds were used in the quantitation of the results for associated sample data (dilutions of Gassett, LaPlace, and Lavergne), no data qualifications were applied.

It should be noted that all applicable data qualifiers that arose from deficient compound response factors, %RSD and %D criteria were applied to the Method 524.2 laboratory fortified blanks (LFB) and the respective volatile method blanks. No discussion of the added qualifiers was included in the above for these QC samples. However, sample results may be impacted and biased depending on the qualification of the LFB and the associated sample data. Refer to the more complete discussion in the System Performance and Overall Assessment.

6. SURROGATES:

All samples are spiked with surrogate compounds prior to sample preparation to evaluate overall laboratory performance and efficiency of the analytical technique. If the measured surrogate concentrations were outside contract specifications, qualifications were applied to the samples and analytes as shown below.

As specified in Method 524.2, bromofluorobenzene (BFB) and 1,2-dichlorobenzene-d4 (DCB-d4) were used for the surrogate compound spike additions in all samples analyzed by the low level drinking water method. The surrogate compounds were added at a final concentration of 1 ug/L. As noted in the quality control section dealing with establishment of laboratory precision and accuracy for the method, the mean accuracy, expressed as a percentage of the true value, should be 80-120% for most compounds and surrogates. Some

analytes, particularly the early eluting gases and late eluting higher molecular weight compounds, are measured with less accuracy than other analytes. Using the suggested guideline of 80-120% accuracy, the percent recovery was outside the recommended limits for the following samples and blanks:

<u>Sample</u>	<u>% BFB</u>	<u>%DCB-d4</u>
Ramsey	-	75
Trip Blank 01(2/6/91)	-	78
VIHA 3	-	78
VBLK02	79	76
VBLK03	-	74

No sample reanalyses were performed based on the lack of firm guidance regarding surrogate recoveries in Method 524.2 analyses. As no major surrogate losses were noted, no data qualifications have been applied on the basis of surrogate recovery.

In the analysis of volatile organics by the CLP RAS volatile methodology, no deviations outside the specified contract surrogate limits were noted for any samples or associated blanks.

7. INTERNAL STANDARDS PERFORMANCE:

Internal standard (IS) performance criteria ensure that the GC/MS sensitivity and response are stable during every experimental run. The internal standard area count must not vary by more than a factor of 2 (-50% to +100%) from the associated continuing calibration standard. the retention time of the internal standard must not vary more than ± 30 seconds from the associated continuing calibration standard. If the area count is outside the (-50% to +100%) range of the associated standard, all of the positive results for compounds quantitated using that IS are qualified as estimated, "J", and all non-detects as "UJ", or "R" if there is a severe loss of sensitivity.

If an internal standard retention time varies by more than 30 seconds, the reviewer will use professional judgement to determine either partial or total rejection of the data for that sample fraction.

The internal standard (IS) area counts and retention times (RT) for all samples and laboratory fortified blanks were within the quality control (QC) limits. Fluorobenzene, added at a level of 1 ug/L, was used for the internal standard as

required in low level analyses performed by Method 524.2. All samples analyzed for volatiles as per the CLP RAS protocol utilized bromochloromethane, 1,4-difluorobenzene and chlorobenzene added at a concentration of 50 ug/L in the sample. As per the CLP RAS method, compound quantitation was performed using the response factor of the nearest internal standard.

8. COMPOUND IDENTIFICATION:

A) VOLATILE AND SEMI-VOLATILE FRACTIONS:

TCL compounds are identified on the GC/MS by using the analyte's relative retention time (RRT) and by comparison to the ion spectra obtained from known standards. For the results to be a positive hit, the sample peak must be within ± 0.06 RRT units of the standard compound and have an ion spectra which has a ratio of the primary and secondary m/e intensities within 20% of that in the standard compound. For the tentatively identified compounds (TIC) the ion spectra must match accurately. In the cases where there is not an adequate ion spectrum match, the laboratory may have provided false positive identifications.

TCL compound identification by GC/MS was based on comparison of the analyte RRT and ion spectra to those obtained from known spectra. Positive hits were reviewed for all samples and the following discrepancies were noted in meeting the Relative Retention Time (RRT) criteria of ± 0.06 .

In the total analysis of 1,2-dichloroethene (1,2-DCE) RRT criteria was exceeded in the Method 524.2 analyses for samples Hartman II, LaPlace, Lavergne, Ramsey and Tillett. Calibration for the total 1,2-DCE analyses (cis and trans isomers) was based on the trans isomer for both the Method 524.2 and CLP RAS analyses. Quantitation of the cis isomer, if present, was based on the trans isomer response. This is the standard calibration and quantitation practice for volatile analyses using the CLP RAS methodology as the cis and trans isomers are not resolvable. Although separation and quantitation of the isomers can be individually achieved by Method 524.2 analysis, for the purposes of this site, 1,2-DCE was to be quantitated and reported as the total of the two isomers. As such, the exceeded RRT, in the samples listed above, was potentially due to the presence of the cis-1,2-DCE isomer. As the calibration standards did not include the cis isomer, sample results were calculated using the trans isomer response factor. Based on this, the positive detects in samples Hartman II and Ramsey, believed to be the result of the cis isomer concentrations in the samples as

tentatively and identified by the Method 524.2 analyses, are considered estimated values are qualified as such (J).

9. MATRIX SPIKE/SPIKE DUPLICATE, MS/MSD:

The MS/MSD data are generated to determine the long-term precision and accuracy of the analytical method in various matrices. The MS/MSD may be used in conjunction with other QC criteria for some additional qualification of the data.

In the determination of accuracy, sample Hartman II was used to determine whether the sample matrix contributed bias to the analytical results. The sample was spiked at a level of 10 ug/L for the following analytes: 1,1-dichloroethene, trichloroethene, benzene, toluene and chlorobenzene. Two out of the five analyte recoveries (trichloroethene and toluene) for the sample matrix spike duplicate (MSD) recovered outside of the upper control limit. In general, MSD recoveries were all found to be at, or in excess of, the CLP RAS upper control limits for volatile spike criteria. In contrast, the Hartman II matrix spike sample recovered at the mean of the control limits established for the accuracy assessment. Due to the higher recoveries noted for the MSD sample, the relative percent difference (%RPD) was out of criteria for three analytes (trichloroethene, toluene and chlorobenzene).

As the higher recoveries were noted only for the MSD sample, bias due to sample matrix was determined to have little to no effect on the final sample results for Hartman II and, as a result, data qualifications were not applied.

10. OTHER QC DATA OUT OF SPECIFICATION:

A) Sample Receipt

A volatile analysis container for sample LaPlace was received the laboratory with headspace. As per a combination of the chain of custody and laboratory information provided and evaluated by the reviewer, it was determined that only one of the three vials for the sample was received with air bubbles. As a result of headspace in the vials, volatile results would typically be biased low and data qualifiers added accordingly. As the laboratory was not able to confirm which of the three vials were used for the sample initial analysis and reanalyses, Region II guidelines dictate that the sample results be qualified in this event. As sample

LaPlace was replicated in the field (sample Lavergne) and excellent precision was shown for the sample-duplicate pair, it is unnecessary to qualify the LaPlace data based on the possibility of headspace in the vial.

B) Method Detection Limit Study

The method detection limit (MDL) study associated with Method 524.2 was performed on April 15, 1990. The MDL determination was based on the analysis of seven replicates of reagent water fortified at a concentration of 1.0 ug/L with Method 524.2 analytes. The parameter list, however, for the site consisted of TCL analytes, some of which are not in the drinking water method. As such, the following target volatile organic compounds were omitted from the MDL study: acetone, carbon disulfide, 2-butanone, vinyl acetate, 4-methyl-2-pentanone and 2-hexanone. Also, in accordance with Method 524.2, isomers of xylene (ortho, meta, and para) and 1,2-dichloroethene (cis and trans) are quantitated and calculated individually while as in the sample data package, these isomers were reported as totals.

Due to the omission of certain target analytes from the MDL study, the laboratory has not provided evidence that the MDL criteria could be achieved for the components specified previously above. Based on this premise, all low level sample analysis results for the omitted target analytes were qualified as estimated for positive hits (J) and non-detects (UJ).

The laboratory MDL was calculated as the product of the standard deviation of seven replicate analyses and the student's t value for the 99% confidence level with six degrees of freedom ($t = 3.143$).

Section 10.3.3 of Method 524.2 states that for every analyte and surrogate the mean accuracy, expressed as a percentage of the true value, should be 80 - 120% and the RSD < 20%. In the MDL study performed in support of the February 1991 analyses for the Tutu Wells site, the following analytes did not meet the precision and accuracy criteria required prior to sample analysis:

<u>Analyte</u>	<u>Mean Accuracy, %</u>	<u>%RSD</u>
1,1-dichloroethene	72	-
Methylene chloride	148	-
1,2-dichloroethane	121	-
tetrachloroethene	76	-
trans-1,3-dichloropropene	162	-
1,1,2,2-tetrachloroethane	122	-
bromoform	-	28.0

C) Laboratory Fortified Blanks

As per the QC requirements in Method 524.2 and the project Sampling, Analysis and Monitoring Plan (SAMP), a laboratory fortified blank (LFB), to which known quantities of the target analytes are added, is required to be analyzed for every batch of low level samples processed using the method. In the analysis of well samples for the Tutu site, LFBs were prepared at a concentration of 1 ug/L with TCL analytes and processed prior to the analysis of field samples in accordance with the QC protocol. The LFBs were to be evaluated using equivalent procedural guidelines for the determination of accuracy as stated in the MDL assessment for initial demonstration of precision and accuracy. Furthermore, an assessment of laboratory capability as to achievement of MDL for every sample batch processed was to be derived from the LFB analysis. Based on these evaluations, the laboratory was to continue with sample analysis if all criteria was achieved, or alternatively, remedy deficiencies prior to sample analysis where QC was not within limits (as specified in Method 524.2 Section 10.3).

For the purposes of the Tutu Wells site volatile organic analyses, the QC criteria was modified to assess data quality for analyses performed by Method 524.2 in the following manner. Based on an EPA guidance (dated December 4, 1990) regarding additional quality assurance (QA)/QC requirements when Method 524.2 is used for aqueous sample analysis, the 80 - 120% criteria for the accuracy assessment of LFB was expanded to 50 - 150%. Based on this guidance and for purposes of validation, for any analyte for which the LFB recovery was < 50%, the associated sample data was rejected and qualified as unusable (R). For LFB analyte recoveries in the range of 50 - 79%, the associated sample data was qualified as estimated (J). Data qualifiers were not applied to any sample result for which the associated LFB analyte recovery was greater than 150%.

On this basis, the following data qualifiers were applied to sample data due to the evaluation of LFBs. In the assessment of analyte accuracy in the LFB identified as VSTD001-2, sample data were rejected (R) for bromomethane, vinyl chloride, chloroethane, 2-butanone and cis-1,3-dichloropropene. Chloromethane values were qualified as estimated (J) as the LFB recovery fell in the 50 - 79% range. Sample data for analyses of Ramsey, VIHA III, Bryan and Demitri were associated with VSTD001-2.

In the analysis of sample Hartman II (including MS and MSD samples), Tillett and both trip blank analyses associated with SDG Hartman, data qualifiers were applied based on the LFB evaluation of VSTD001-3. The cis-1,3-dichloropropene recovery was less than 50% in the LFB and, as such, the sample results were unusable (R) for all of the samples listed above. All other analyte recoveries were greater or equal to 80%.

In the LFB VSTD00-4 sample, results for chloroethane, 2-butanone, vinyl acetate and cis-1,3-dichloropropene were qualified as unusable (R) in samples Hartman II (MS), Field Blank, Gassett, Leonard, LaPlace and Lavergne as the associated LFB recovery was < 50%. All other LFB recoveries were $\geq$ 80% in VSTD001-4 and no additional qualifiers were applied on that basis.

Please note that for many of the analytes qualified based on the LFB assessment, previous data qualifications were present due to the MDL study analyte omissions and/or deficient instrument system performance. In all cases, a data qualification resulting in a rejected value (R) for any assessment criteria not satisfied superceded all other qualifiers previously assessed.

11. SYSTEM PERFORMANCE AND OVERALL ASSESSMENT:

All quantitation reports, reconstructed ion chromatograms (RIC) and mass spectra for positively identified TCL volatile organic compounds were included with the respective sample Form I data sheets with the exception of the following. For the CLP RAS analysis of sample Gassett, the quantitation report was omitted from the supporting sample data package. Additionally, in the raw data provided for the field blank analysis, the supporting library search spectra for TIC at RT 22.62 was omitted and substituted with library search spectra from sample Leonard for a similarly identified TIC. The result was verified from the mass ion abundance data provided in the raw data for the field blank and found to be correctly calculated and reported.

In addition to the above, several data transcription errors were noted between the sample data package and the summary package.

Aside from the transcription errors, system performance deficiencies were noted in the Method 524.2 analyses for low level volatile organic compounds. The initial MDL study resulted in several compounds not meeting the preliminary accuracy requirements needed to establish the baseline MDL for the low level analysis. As noted previously, several target compounds were not included in the MDL study altogether. In reviewing the MDL accuracy measurements for the compounds exceeding the criteria, the recoveries for 1,1-dichloroethene, 1,2-dichloroethane, tetrachloroethene and 1,1,2,2-tetrachloroethane were within $\pm 10\%$ of the 80 - 120% recovery criteria. For the remaining two compounds outside of criteria, high recovery for methylene chloride may be attributed to background contamination which is not out of the ordinary in a laboratory environment. With respect to the trans-1,3-dichloropropene, the MDL replicates showed consistently high recoveries with very little deviation. No explanations as to the higher recoveries have been obtained. The precision requirement between replicate measurements in the MDL study ($RSD < 20\%$) was met for all compounds with exception of bromoform for which greater variability is often noted. Based on these observations and the deviations noted above regarding omitted compounds, the MDL study of April 1990 established the laboratory's initial capability to analyze samples with the accuracy and precision needed for low level analysis.

Laboratory fortified blanks (LFBs) were utilized to assess the laboratory's capability on a batch basis. In reviewing these standards against the accuracy criteria, all three of the associated LFB analyses resulted in 14, 15, and 18 analyte recoveries, respectively, outside of the 80 - 120% method criteria. In using the expanded criteria of 50 - 150%, several compounds per LFB were still observed to be outside of the criteria. As such this indicated that the laboratory had difficulty in recovering certain compounds at the levels dictated by Method 524.2 analysis for low level volatiles.

In cases of high analyte recoveries, difficulties in controlling instrument background levels and system contamination may have falsely contributed to the out of limit LFB recoveries.

In assessing the volatile data, particular attention was focused on LFB recoveries below the lower control limit (80%). Since data with associated LFB recoveries below 80% would be biased low, all affected analytes with recoveries between

50 - 79% were qualified as estimated (J). Below 50% recovery, associated sample data was rejected and qualified as unusable (R). These qualifications, primarily, affected non-site specific target compounds and, within that perspective, have had minimal effect on the data quality for the use for which it was intended.

For LFB analyte recoveries greater than 120%, sample data would, generally be, biased high. All of the associated data resulted in non-detected values at the quantitation limit. No action was taken to offset the higher analyte recoveries and data quality was not impacted.

Poor initial instrument response resulted in variable volatile calibrations and impacted daily performance for several ketones and other laboratory contaminants throughout the duration of the sample analyses. Aside from these, occasional other compound calibrations did not meet criteria in the low level method and, in some analyses, for the CLP RAS method. As no deficiency resulted in unusable data for compounds specific to the site analysis, data quality was not adversely affected due to calibration problems.

Overall, the volatile organic data for SDG Hartman in support of the Tutu Well site analyses was consistent and of adequate data quality. As noted, QC issues did serve to qualify certain compounds as estimated and reject others due to common, recognized, but correctable instrument problems. Daily performance, while assessed as fair to average, with respect to the Method 524.2 analyses, needs to be improved and stabilized for data to be reported unqualified based on method and regional guidelines for full data acceptance.

12. CONTRACT PROBLEMS - NON-COMPLIANCE:

The supporting MDL study did not include six target compounds from the parameter list. Several accuracy measurements for target compounds did not meet the method and SAMP criteria for the determination of the laboratory MDL.

The laboratory fortified blanks did not meet all of the method criteria (and/or the suggested expanded criteria) as required. In accordance to the method, these specifications must be met, or deficiencies remedied, prior to sample analysis and were not.

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13. The Form I for Method 524.2 analyses of samples Gassett, LaPlace, Lavergne, and Tillett have been edited by the data reviewer to include data from CLP RAS analyses required for high levels of 1,2-dichloroethene (total), tetrachloroethene and/or toluene.

STANDARD OPERATING PROCEDURE (SOP) NO. HW-2

Revision #10

**EVALUATION OF METALS DATA FOR THE CONTRACT
LABORATORY PROGRAM (CLP)**

Title: Evaluation of Inorganic Data for the
Contract Laboratory Program

Date: Feb. 1990
Number: HW-2
Revision: 10

1.0 Scope

- 1.1 This procedure is applicable to inorganic data obtained from contractor laboratories working for Hazardous Waste Site Contract Laboratory Program (CLP).
- 1.2 The data validation is based upon analytical and quality assurance requirements specified in Statement of Work (SOW 7/88).

2.0 Responsibilities - Data reviewers will complete the following tasks as assigned by the Data Review Coordinator:

2.1 For a total review:

- 2.1.1 Data Assessment - "Total Review - Inorganics" Checklist Appendix (A.1).
The reviewer must answer every question on the checklist.

- 2.1.2 Data Assessment - Data Assessment Narrative (Appendix A.2)
The answer on the checklist must match the action in the narrative (appendix A.2) and Form I's. Do not use pencil to write the narrative.

- 2.1.3 Contract Non-Compliance - SMO Report (Appendix A.3)
This report is to be completed only when a serious contract violation is encountered, or upon the request of the Data Review Manager or Deputy Project Officer (DPO). Forward 5 copies: one each for internal files, appropriate Regional DPO, Sample Management Office (SMO) and last two addresses of Mailing List for Data Reviewers (Appendix A.4). In other cases, all contract violations should be appended to end of Data Assessment Narrative (Sec. A.2.2.).

- 2.1.4 Data Summary Sheet - Summary of Inorganic Quality Control Data (Appendix A.5)
Enter in ink on Data Summary Sheet required QC values from Forms I through IX. Circle all values that require data qualification "Action".

2.1.5 CLP Data Assessment Summary Forms

- 2.1.5.1 Appendix A.6
Fill in the total number of analytes by different analyses and the number of analytes rejected or flagged as estimated due to corresponding quality control criteria. Place an "X" in boxes where analyses were not performed, or criteria do not apply.

- 2.1.5.2 Appendix A.7
Data reviewer is also required to fill out Inorganic Regional Data Assessment form (Appendix A.7) provided by EPA Headquarters. Codes listed on the form will be used to describe the Data Assessment Summary.

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- 2.1.6 Data Review Log: It is recommended that each data reviewer should maintain a log of reviews completed to include:
- a. date of start of case review
 - b. date of completion of case review
 - c. site
 - d. case number
 - e. contract laboratory
 - f. number of samples
 - g. matrix
 - h. hours worked
 - i. reviewer's initials

- 2.1.7 Telephone Record Log - The data reviewer should enter the bare facts of inquiry, before initiating any phone conversation with CLP laboratory. After the case review has been completed, mail white copy of Telephone Record Log to the laboratory and pink copy of SMO. File yellow copy in the Telephone Record Log folder, and attach a xerox copy of the Telephone Record Log to the completed Data Assessment Narrative (Appendix A.2).

2.1.8 Forwarded Paperwork

- 2.1.8.1 Upon completion of review, the following are to be forwarded to the Regional Sample Control Center (RSCC) located in the Surveillance and Monitoring Branch:
- a. data package
 - b. completed data assessment checklist (Appendix A.1, original)
 - c. SMO Contract Compliance Screening (CCS)
 - d. Data Summary Sheet (Appendix A.5) along with completed Data Assessment Narrative (Appendix A.2)
 - e. Record of Communication (copy)
 - f. CLP Re-analysis Request/Approval Record (original + 3 copies)
 - g. Appendix A.7 (original).
- 2.1.8.2 Forward 4 copies of completed Data Assessment Narrative (Appendix A.2) along with 2 copies of the Inorganic Data Assessment Form (Appendix A.7) and Telephone Record Log, if any; one each for appropriate Regional DPO, Sample Management Office (SMO), and last two addresses of Mailing List for Data Reviewers (Appendix A.4) (the Inorganic Data Assessment form does not go to the last two addresses).
- 2.1.9 Filed Paperwork - Upon completion of review, the following are to be filed within MMB files:
- a. Four copies of completed Data Assessment Narrative (Appendix A.2) each carrying Appendix A.7.
 - b. Telephone Record Log (copy)
 - c. SMO Report (copy Appendix A-3)
 - d. CLP Data Assessment Summary Form (Appendix A.6)
 - e. CLP Re-analysis Request/Approval Record (copy).

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3.0 Data Completeness

Indicate incomplete data package on the computer tracking sheet located in MMB office. Authorized contractor personnel may contact the laboratory after discovery of an incomplete data package. If a laboratory will not return phone calls or does not respond to requests, notify MMB coordinator of Region II for resolution.

4.0 Rejection Data - All values determined to be unacceptable on the Inorganic Analysis Data Sheet (Form I) must be lined over with a red pencil. As soon as any review criteria causes data to be rejected, that data can be eliminated from any further review or consideration.

5.0 Acceptance Criteria - In order that reviews be consistent among reviewers, acceptance criteria as stated in Appendix A.1 (pages 4-25) should be used. Additional guidance can be found in the National Inorganic Functional Guidelines of October 1, 1989.

6.0 SMO Contract Compliance Screening (CCS) - This is intended to aid reviewer in locating any problems, both corrected and uncorrected. However, the validation should be carried out even if CCS is not present. Resubmittals received from laboratory in response to CCS must be used by the reviewer.

7.0 Request for Re-analysis - Data reviewers must note all items of contract non-compliance within Data Assessment Narrative. If holding times and sample storage times have not been exceeded, DPO may request re-analysis if items of non-compliance are critical to data assessment. Requests are to be made on "CLP Re-analysis Request/Approval Record".

8.0 Record of Communication - Provided by the Regional Sample Control Center (RSCC) to indicate which data packages have been received and are ready to be reviewed.

9.0 Rounding off numbers - The data reviewer will follow the standard practice.

Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.1: Data Assessment - Contract
Compliance (Total Review - Inorganics)

Date: Feb. 1991
Number: HW-2
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	YES	NO	N/A
A.1.1 <u>Contract Compliance Screening Report (CCS)</u> - Present?	☐	☐	☒
<u>ACTION:</u> If no, contact RSCC.			
A.1.2 <u>Record of Communication (from RSCC)</u> - Present?	☐	☐	☒
<u>ACTION:</u> If no, request from RSCC.			
A.1.3 <u>Trip Report</u> - Present and complete?	☐	☐	☒
<u>ACTION:</u> If no, contact RSCC for trip report.			
A.1.4 <u>Sample Traffic Report</u> - Present or on file?	☒	☐	☐
Legible?	☒	☐	☐
<u>ACTION:</u> If no, request from Regional Sample Control Center (RSCC).			
A.1.5 <u>Cover Page</u> - Present?	☒	☐	☐
Is cover page properly filled in and signed by the lab manager or the manager's designee?	☒	☐	☐
<u>ACTION:</u> If no, prepare Telephone Record Log, and contact laboratory.			
Do numbers of samples correspond to numbers on Record of Communication?	☐	☐	☒
Do sample numbers on cover page agree with sample number on:			
(a) Traffic Report Sheet?	☐	☒	☐
(b) Form Is?	☒	☐	☐
<u>ACTION:</u> If no for any of the above, contact RSCC for clarification.			
A.1.6 <u>Form I (Final Data)</u> - Are all Form I's present and complete?	☒	☐	☐
<u>ACTION:</u> If no, prepare telephone record log and contact laboratory for submittal.			

Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.1: Data Assessment - Contract
Compliance (Total Review - Inorganics)

Date: Feb. 1996
Number: HW-2
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	YES	NO	N/A
Are correct units (ug/L for waters and mg/kg for soils) indicated on Form Is?	☒	☐	☐
Are soil sample results for each parameter corrected for percent solids?	☐	☐	☒
Are EPA sample #'s and corresponding laboratory sample ID #'s the same as the Cover Page, Form Is and in the raw data?	☒	☐	☐
Are the computation/transcription errors less than 10% of reported values?	☒	☐	☐
Are all "less than IDL" values properly coded with "U"?	☒	☐	☐
Was a brief physical description of samples given on Form Is?	☒	☐	☐
Were the result qualifiers used correctly with final data?	☒	☐	☐
<u>ACTION:</u> If no for any of the above, prepare Telephone Record Log, and contact laboratory for corrected data.			
Were any samples diluted beyond requirements of contract?	☐	☒	☐
If yes, were dilutions noted on Form Is?	☐	☐	☒
<u>ACTION:</u> If no, note under contract Problem/Non-Compliance of the "Data Assessment Narrative".			
A.1.7 <u>Holding Times</u> - (aqueous and soil samples) (Examine sample traffic reports and digestion distillation logs.)			
Mercury analysis (28 days).....exceeded?	☐	☒	☐
Cyanide distillation (14 days)....exceeded?	☐	☐	☒

Title: Evaluation of Metals Data for the
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	YES	NO	N/A
Other Metals analysis (6 months)..exceeded?	—	☒	—

NOTE: Prepare a list of all samples and analytes for which holding times have been exceeded. Specify the number of days from date of collection to the date of preparation (from raw data). Attach to checklist.

ACTION: If yes, reject (red-line) values less than instrument Detection Limit (IDL) and flag as estimated (J) the values above IDL even though sample(s) was preserved properly.

A.1.8 Raw Data

A.1.8.1 Digestion Log* for flame AA/ICP (Form XIII) present?

☒ — —

Digestion Log for furnace AA Form XIII present?

☒ — —

Distillation Log for mercury Form XIII present?

☒ — —

Distillation Log for cyanides Form XIII present?

☐ — ☒

Are pH values (pH < 2 for all metals, pH > 12 for cyanide) present in Digestion Distillation Logs?

☒ — —

Percent solids calculation present for soils/sediments?

☐ — ☒

Are preparation dates present on Digestion Log?

☒ — —

A.1.8.2 Measurement read out record present? ICP

☒ — —

Flame AA

☐ — ☒

Furnace AA

☒ — —

Mercury

☒ — —

Cyanides

☐ — ☒

A.1.8.3 Are all raw data to support all sample analyses and QC operations present?

☒ — —

* Weights, dilutions, and volumes used to obtain values.

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Title: Evaluation of Metals Data for the
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Appendix A.1: Data Assessment - Contract
Compliance (Total Review - Inorganics)

Date: Feb. 1990
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	YES	NO	N/A
Legible?	☒	☐	☐
Properly labeled?	☒	☐	☐

ACTION: If no for any of the above, write Telephone Record Log and contact laboratory. Flag metal data as estimated if pH of sample is greater than 2. Flag cyanide data as estimated if pH sample is less than 12.

A.1.9 Data Validation and Verification

A.1.9.1 Calibration

A.1.9.1.1 Is record of at least 2 point calibration present for ICP analysis?

☒ ☐ ☐

Is record of 5 point calibration present for Hg analysis?

☒ ☐ ☐

ACTION: If no for any of the above, write in the Contract Problem/Non-Compliance section of the "Data Assessment Narrative".

A.1.9.1.2 Is record of 4 point calibration present for: Flame AA?

☐ ☐ ☒

Furnace AA?

☒ ☐ ☐

Cyanides?

☐ ☐ ☒

- NOTE:**
1. If less than 4 standards are measured in absorbance mode, then the remaining standards in concentration mode must run immediately after calibration and be within +/- 10% of true value.
 2. For all AA (except Hg) and Cyanide analyses, one calibration standard is at CRDL level. If not, write in the Contract-Problem/Non-Compliance section of the "Data Assessment Narrative".

ACTION: Flag associated data as estimated if standards

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are not within +/- 10% of true values (except

YES NO N/A

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CRDL calibration standard). Do not flag the
data as estimated in linear range indicated
by good recovery of standard.

A.1.9.1.3 Is correlation * coefficient less than 0.995 for: Mercury Analysis? ☐ ☒ ☐

Cyanide Analysis? ☐ ☐ ☒

Atomic Absorption Analysis? ☐ ☒ ☐

ACTION: If yes, flag the associated data as estimated.

A.1.9.2 Form IIA (Initial and Continuing Calibration Verification)

A.1.9.2.1 Present and complete for every metal and cyanide? ☒ ☐ ☐

Present and complete for AA and ICP when both
are used for same analyte? ☐ ☐ ☒

ACTION: If no for any of the above, prepare
Telephone Record Log and contact
laboratory.

A.1.9.2.2 Circle all values on data summary sheet that
are outside contract windows. Are all
calibration standards (initial and continuing)
within control limits?

Metals 90-110% ☒ ☐ ☐

Hg 80-120% ☒ ☐ ☐

Cyanides 85-115% ☐ ☐ ☒

ACTION: Flag as estimated (J) all positive Data
(not flagged with a "U") analyzed between
a calibration standard with %R between
75-89% (65-79% for Hg; 70-84% for CN)
or 111-125% (121-135% for Hg; 116-130%
for CN) recovery and nearest good

* The reviewer will calculate correction coefficient.

YES NO N/A

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YES NO N/A

calibration standard. Quality results
<IDL as estimated (UJ), if the ICV
or CCV %R is 75-89% (CN, 70-84%;
HG, 65-79%). Reject (red-line) as
unacceptable data if recovery of the
ICV or CCV is outside the range 75-125%
(CN, 70-130%; Hg, 65-135%). Qualify
five samples on either side of
verification standard out of control
limits.

Was continuing calibration performed every 10
samples or every 2 hours?

☒ ☐ ☐

ACTION: If no, flag the excess samples (eleventh
and up) data as estimated (J).

Was ICV for cyanides distilled?

☐ ☐ ☒

ACTION: If no, write in the Contract-Problem/
Non-Compliance section of the "Data
Assessment Narrative".

A.1.9.3 Form IIB (CRDL Standards for AA and ICP) -

A.1.9.3.1 Was a CRDL standard (CRA) analyzed after
initial calibration for all AA metals (except
Hg)?

☒ ☐ ☐

*Was a mid-range calib. verification standard
distilled and analyzed for cyanide analysis?

☐ ☐ ☒

Was a 2xCRDL (or 2xIDL when IDL > CRDL) analyzed
(CRI) for each ICP run?
(Note: CRI for Al, Ba, Ca, Fe, Mg, NA or K is
not required).

☒ ☐ ☐

ACTION: If no for any of the above, flag as
estimated all data falling within
the affected ranges. The affected
ranges are:

Title: Evaluation of Metals Data for the
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YES NO N/A

AA Analysis - **True Value +/- CRDL

ICP Analysis - **True Value +/- 2CRDL

CN Analysis - **True Value +/- 0.5 x True Value

A.1.9.3.2 Was CRI analyzed after ICV/ICB and before the
final CCV/CCB, and for every four hours of ICP
run?

☒ ☐ ☐

ACTION: If no, write in Contract Problem/Non-
Compliance Section of the "Data
Assessment Narrative".

A.1.9.3.3 Circle all values on summary sheet that are
outside acceptance windows.

Are CRA and CRI standards within control limits:
Metals 80-120 %R?

☒ ☐ ☐

Is mid-range standard within control limits:
Cyanide 80-120 %R?

☐ ☐ ☒

ACTION: Flag as estimated all data within the
affected ranges if the recovery of the
standard is between 50-79%; flag only
positive data if the recovery is between
121-150%; reject (red line) all data if
the recovery is less than 50%; reject
only positive data if the recovery is
greater than 150%.

A.1.9.4 Form III (Initial and Continuing Calibration Blanks)

A.1.9.4.1 Present and complete?

☒ ☐ ☐

For both AA and ICP when both are used for same
analyte?

☐ ☐ ☒

**True value of CRA, CRI or mid-range standard. Substitute IDL
for CRDL when IDL > CRDL.

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	YES	NO	N/A
Was an initial calibration blank analyzed?	☒	☐	☐
Was a continuing calibration blank analyzed after every 10 samples or every 2 hours (whichever is more frequent)?	☒	☐	☐

ACTION: If no prepare Telephone Record Log,
contact laboratory and write in the
contact-problems/non-compliance
section of the Data Assessment
Narrative."

A.1.9.4.2 Circle all calibration blank values on Data
Summary Sheet that are above CRDL (or 2 x IDL
when IDL > CRDL) less than or equal to Contract
Required Detection Limits (CRDL)?

☒	☐	☐
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Are all calibration blanks less than two times
Instrument Detection Limit (when IDL > CRDL)?

☐	☐	☒
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ACTION: If no for any of the above, flag as
estimated (J) all positive data less
than or equal to calibration blank
values analyzed between calibration
blank with value over CRDL (or 2 x IDL)
and nearest good calibration blank.
Flag five samples on either side of
the calibration blank.

A.1.9.5 FORM III (Preparation Blank) -

(Note: The preparation blank for mercury is
the same as the calibration blank).

A.1.9.5.1 Was on prep. blank analyzed for: each 20 samples?

☒	☐	☐
-------------------------------------	--------------------------	--------------------------

each batch?

☒	☐	☐
-------------------------------------	--------------------------	--------------------------

each matrix type?

☒	☐	☐
-------------------------------------	--------------------------	--------------------------

both AA and ICP and when both are used for same analyte?

☐	☐	☒
--------------------------	--------------------------	-------------------------------------

ACTION: If no for any of the above, flag as

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YES NO N/A

estimated (J) all associated positive
data < 10 x IDLs for which prep. blank
was not analyzed.

NOTE: If only one blank was analyzed for more
than 20 samples, then first 20 samples
analyzed do not have to be flagged as
estimated (J).

A.1.9.5.2 Is concentration of prep. blank greater than CRDL
when IDL is less than or equal to CRDL?

— ☒ —

If yes, is the concentration of the sample with
the least concentration analyte less than 10
times the prep. blank value?

— ☐ ☒

ACTION: If yes, reject (red-line) all associated
data greater than CRDL concentration
but less than ten times the prep. blank
value found in the raw data.

A.1.9.5.3 Do concentrations of prep. blank fall between two
times IDL when IDL is greater than CRDL?

☐ — ☒

ACTION: If no, reject (red-line) positive data
that has a concentration less than 10 times
the prep. blank value in the raw data.

A.1.9.5.4 Is concentration of prep. blank below the negative
CRDL?

— ☒ —

ACTION: If yes, reject (red-line) all associated
data that has a concentration less than
10xCRDL.

A.1.9.6 Form IV (ICP Interference Check Sample)

A.1.9.6.1 Present and complete?

☒ — —

(NOTE: Not required for furnace AA, flame AA,
mercury, cyanide and Ca, Mg, K and Na).

Was ICS analyzed at beginning and end of run

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(or at least twice every 8 hours)?

YES	NO	N/A
☒	☐	☐

ACTION: If no, flag as estimated (J) all samples for which Al, Ca, Fe, or Mg is higher than in ICS.

A.1.9.6.2 Circle all values on Data Summary Sheet that are more than + 10% of true or established mean value. Are all Interference Check Sample results inside of control limits (+20%)?

☐	☐	☒
--------------------------	--------------------------	-------------------------------------

If no, is concentration of Al, Ca, Fe or Mg lower than in ICS?

☐	☐	☒
--------------------------	--------------------------	-------------------------------------

ACTION: If no, flag as estimated (J) those positive results for which ICS recovery is between 121-150%; flag all sample results as estimated if ICS recovery falls within 50-79%; reject (red-line) those sample results for which ICS recovery is less than 50%; if ICS recovery is above 150%; reject positive results only (not flagged with a "U").

A.1.9.7 Form V A (Spiked Sample Recovery - Pre-Digestion/Pre-Distillation) -

(Note: Not required for Ca, Mg, K, and Na (both matrices), Al, and Fe (soil only.))

A.1.9.7.1 Present and complete for: each 20 samples?

☐	☒	☐
--------------------------	-------------------------------------	--------------------------

each matrix type?

☐	☒	☐
--------------------------	-------------------------------------	--------------------------

each conc. range (i.e. low, med, high)?

☐	☒	☐
--------------------------	-------------------------------------	--------------------------

For both AA and ICP when both are used for same analyte?

☐	☐	☒
--------------------------	--------------------------	-------------------------------------

ACTION: If no for any of the above, flag as estimated (J) all positive data less than four times spiking level for

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YES NO N/A

which spiked sample was not analyzed.

NOTE: If one spiked sample was analyzed for more than 20 samples, then first 20 samples analyzed do not have to be flagged as estimated (J).

A.1.9.7.2 Was field blank used for spiked sample?

— ☒ —

ACTION: If yes, flag all positive data less than 4x spike added as estimated (J) for which field blank was used as spike sample.

NOTE: Matrix spike analysis should be performed on a field blank when it is the only aqueous sample in SDG.

A.1.9.7.3 Circle all values on Data Summary Sheet that are outside control limits (75% to 125%). Are all recoveries within control limits?

☒ — —

If no, is sample concentration greater than or equal to four times spike concentration?

☐ — ☒

ACTION: If yes, disregard spike recoveries for analytes whose concentrations are greater than or equal to four times spike added. If no, circle those analytes on Form V for which sample concentration is less than four times the spike concentration.

Are results outside the control limits (75-125%) flagged with "N" on Form Is and Form VA?

☐ — ☒

ACTION: If no, write in the Contract-Problem/Non-Compliance section of "Data Assessment Narrative."

A.1.9.7.4 **Aqueous**
Are any spike recoveries:

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Narrative."

A.1.9.7.4 Aqueous

Are any spike recoveries:

	YES	NO	N/A
(a) less than 30%?	—	☒	—
(b) between 30-74%?	—	☒	—
(c) between 126-150%?	—	☒	—
(d) greater than 150%?	—	☒	—

ACTION: If less than 30%, reject all associated aqueous data; if between 30-74%, flag all associated aqueous data as estimated (J); if between 126-150%, flag as estimated (J) all associated aqueous data not flagged with a "U"; if greater than 150%, reject (red-line) all associated aqueous data not flagged with a "U".

NOTE: If pre-digestion spike result is rejectable due to coefficient of correlation of MSA, analytical spike recovery, or duplicate injections criteria, disregard spike recovery on Form V. Flag the associated data as estimated (J).

A.1.9.7.5 Soil/Sediment

Are any spike recoveries:

(a) less than 10%?	—	☐	☒
(b) less than 10-74%?	—	☐	☒
(c) between 126-200%?	—	☐	☒
(d) greater than 200%?	—	☐	☒

ACTION: If less than 10%, reject all associated data; if between 10-74%, flag all associated data as estimated; if between

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	YES	NO	N/A
A.1.9.8 <u>Form VI (Lab Duplicates)</u>			
A.1.9.8.1 Present and complete for:			
each 20 samples?	☐	☒	☐
each matrix type?	☐	☒	☐
each concentration range (i.e. low, med, high)?	☐	☒	☐
both AA and ICP when both are used for same analyte?	☐	☐	☒
<u>ACTION:</u> If no for any of the above, flag as estimated (J) all data > CRDL* for which duplicate sample was not analyzed.			
<u>NOTE:</u> 1. If one duplicate sample was analyzed for more than 20 samples, then first 20 samples do not have to be flagged as estimated. 2. If percent solids for soil sample and its duplicate differ by more than 1%, prepare a Form VI for each duplicate pair, report concentrations in ug/L on wet weight basis and calculate RPD or Difference for each analyte.			
A.1.9.8.2 Was field blank used for duplicate analysis?	☐	☒	☐
<u>ACTION:</u> If yes, flag all data > CRDL* as estimated (J) for which field blank was used as duplicate.			
<u>NOTE:</u> Duplicate analysis should be performed on a field blank when it is the only aqueous sample in SDG.			
A.1.9.8.3 Are all values within control limits (RPD 20% or difference < +/- CRDL)?	☒	☐	☐

* Substitute IDL for CRDL when IDL > CRDL.

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If no, are all results outside the control limits
flagged with an * on Form Is and VI?

YES NO N/A

[] [] [✓]

ACTION: If no, write in the Contract-Problems/Non-
Compliance section of "Data Assessment
Narrative".

NOTE: 1. RPD is not calculable for an analyte of
the sample - duplicate pair when both
values are less than IDL.
2. If lab duplicate result is rejectable
due to coefficient of correlation of MSA,
analytical spike recovery, or duplicate
injections criteria, do not apply precision
criteria.

A.1.9.8.4 Is any value for sample duplicate pair less than CRDL*
and other value greater than or equal to 10 x *CRDL?

[] [✓] []

ACTION: If yes, flag the associated data as
estimated (J).

A.1.9.8.5 Aqueous

Circle all values on Data Summary Sheet that are:

RPD > 50%, or

Difference > = +/- CRDL*

Is any RPD greater than 50% where sample and duplicate
are both greater than or equal to 5 times *CRDL?

[] [✓] []

Is any difference between sample and duplicate
greater than *CRDL where sample and/or duplicate
is less than 5 times *CRDL?

[] [✓] []

ACTION: If yes, flag the associated data as estimated.

* Substitute IDL for CRDL when IDL > CRDL.

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YES NO N/A

A.1.9.8.6 Soil/Sediment

Circle all values on Data Summary Sheet that are:

RPD > 100%, or
Difference > 2 x CRDL*

Is any RPD (where sample and duplicate are both
greater than or equal to 5 times *CRDL):

> 100%?

— [] ☒

Is any ** difference between sample and
duplicate (where sample and/or duplicate is
less than 5 x *CRDL):

> 2 x *CRDL?

— [] ☒

ACTION: If yes, flag the associated data as estimated.

A.1.9.9 Field Duplicates

A.1.9.9.1 Were field duplicates analyzed?

☒ — —

ACTION: If yes, prepare a Form VI for each
aqueous field duplicate pair. Prepare
a Form VI for each soil duplicate pair,
if percent solids for sample and its
duplicate differ by more than 1%;
report concentrations of soils in ug/L
on wet weight basis and calculate RPDs
or Difference for each analyte.

NOTE: 1. Do not calculate RPD when both values
are less than IDL.
2. Flag all associated data only for
field duplicate pair.

A.1.9.9.2 Is any value for sample duplicate pair less than
*CRDL and other value greater than or equal to
10 x *CRDL?

— ☒ —

* Substitute IDL for CRDL when IDL > CRDL.

** Use absolute values of sample and duplicate to calculate difference.

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YES NO N/A

ACTION: If yes, flag the associated data as estimated.

A.1.9.9.3 Aqueous

Circle all values on Form VI for field duplicates
that are:

RPD > 50%, or
Difference > +/- CRDL*

Is any RPD greater than 50% where sample and duplicate
are both greater than or equal to 5 times *CRDL?

— [] ☒

Is any **difference between sample and duplicate
greater than *CRDL where sample and/or duplicate
is less than 5 times *CRDL?

— ☒ —

ACTION: If yes, flag the associated data as estimated.

A.1.9.9.4 Soil/Sediment

Circle all values on Form VI for field duplicates
that are:

RPD > 100%, or
Difference > 2 x CRDL*

Is any RPD (where sample and duplicate are both
greater than 5 times *CRDL):
> 100%?

— [] ☒

Is any **difference between sample and duplicate
(where sample and/or duplicate is less than
5 x *CRDL):

> 2 x *CRDL?

— [] ☒

A.1.9.10 Form VII (Laboratory Control Sample) (Note: LCS -
not required for aqueous Hg and cyanide analyses).

* Substitute IDL for CRDL when IDL > CRDL.

** Use absolute values of sample and duplicate to calculate difference.

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	YES	NO	N/A
A.1.9.10.1 Was one LCS prepared and analyzed for:			
every 20 water samples?	☒	☐	☐
every 20 solid samples?	☐	☐	☒
both AA and ICP when both are used for same analyte?	☐	☐	☒

ACTION: If no for any of the above, prepare Telephone Record Log and contract laboratory for submittal of results of LCS. Flag as estimated (J) all data for which LCS was not analyzed.

NOTE: If only one LCS was analyzed for more than 20 samples, then first 20 samples close to LCS do not have to be flagged as estimated.

A.1.9.10.2 Aqueous LCS

Circle all LCS values outside control limits (80-120% - except aqueous Ag and Sb).

Is any LCS recovery: less than 50%?	☐	☒	☐
between 50% and 79%?	☐	☒	☐
between 121% and 150%?	☐	☒	☐
greater than 150%?	☐	☒	☐

ACTION: Less than 50%, reject (red-line) all data; between 50% and 79%, flag all associated data as estimated (J); between 121% and 150%, flag all positive (not flagged with a "U") results as estimated; greater than 150%, reject all positive results.

A.1.9.10.3 Solid LCS

NOTE: 1. If "Found" value of LCS is rejectable due to duplicate injections or analytical

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YES NO N/A

spike recovery criteria, regardless of
LCS recovery, flag the associated data
as estimated (J).

2. If IDL of an analyte is equal to or
greater true value of LCS, disregard
the "Action" below even though LCS is
out of control limits.

Is LCS "Found" value higher than the control limits
on Form VII?

— ☐ ☒

ACTION: If yes, qualify all associated positive
data as estimated.

Is LCS "Found" value lower than the control limits
on Form VII?

— ☐ ☒

ACTION: If yes, qualify all associated data as
estimated.

A.1.9.11 Form IX (ICP Serial Dilution) -

NOTE: Serial dilution analysis is required
only for initial concentrations equal
to or greater than 10 x IDL.

A.1.9.11.1 Was Serial Dilution analysis performed for:

each 20 samples?

☒ — —

each matrix type?

☒ — —

each concentration range (i.e. low, med.)?

☒ — —

ACTION: If no for any of the above, flag all
positive data greater than or equal to
10 x IDLs as estimated (J) for which
Serial Dilution Analysis was not performed,
and summarize the deficiency on the DPO report.

A.1.9.11.2 Was field blank(s) used for Serial Dilution Analysis?

— ☒ —

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YES NO N/A

ACTION: If yes, flag all associated data $\geq 10 \times$ IDL as estimated (J).

NOTE: Serial dilution analysis should be performed on a field blank when it is the only aqueous sample in SDG.

A.1.9.11.3 Are results outside control limit flagged with an "E" on Form Is and Form IX when initial concentration on Form IX is equal to 50 times IDL or greater?

[] — ✓

ACTION: If no, write in the contract-problem/non-compliance section of the "Data Assessment Narrative".

A.1.9.11.4 Circle all values on Data Summary Sheet that are outside control limit for initial concentrations equal to or greater than $10 \times$ IDLs only. Are any % difference values:

$> 10\%$

— [] ✓

$\geq 100\%$

— [] ✓

ACTION: Flag as estimated (J) all associated equal to or greater than $10 \times$ IDLs for which difference is greater than 10% but less than 100%. Reject (red-line) all associated sample results equal to or greater than $10 \times$ IDLs for which RPD is greater than or equal to 100%.

A.1.9.12 Furnace Atomic Absorbtion (AA) OC Analysis

A.1.9.12.1 Are duplicate injections present in furnace raw data (except during full Method of Standard Addition) for each sample analyzed by GFAA?

✓ [] — —

ACTION: If no, reject the data on Form Is for which duplicate injections were not performed.

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	YES	NO	N/A
A.1.9.12.2 Do the duplicate injection readings agree within 20% Relative Standard Deviation (RSD) or Coefficient of Variation (CV) for concentration greater than CRDL?	☒	☐	☐
Was a dilution analyzed for sample with post digestion spike recovery less than 40%?	☐	☐	☒
A.1.9.12.3 Is *post digestion spike recovery less than 10% or greater than 150% for any result?	☐	☒	☐
<u>ACTION:</u> If yes, reject (red-line) the affected data if recovery			
<u>NOTE:</u> Reject the data only if the affected sample was not subsequently analyzed by Method of Standard Addition.			
A.1.9.13 <u>Form VII (Method of Standard Addition Results)</u>			
A.1.9.13.1 Present?	☒	☐	☐
If no, is any Form I result coded with "S" or a "+"?	☐	☒	☐
<u>ACTION:</u> If yes, write request on Telephone record Log and contact laboratory for submittal of Form VIII.			
A.1.9.13.2 Is coefficient of correlation for MSA less than 0.990 for any sample?	☐	☐	☒
<u>ACTION:</u> If yes, reject (red-line) affected data.			
A.1.9.13.3 Was **MSA required for any sample but not performed?	☐	☒	☐

* Post digestion spike is not required on the pre-digestion spiked sample when predigestion spike recovery is within control limits of 75-125% or when $SR > 4 \times SA$.

** MSA is not required on LCS and prep. blank.

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YES NO N/A

Is coefficient of correlation for MSA less than
0.995?

— ☐ ☒

Are MSA calculations outside the linear range of the
calibration curve generated at the beginning of
the analytical run?

— ☐ ☒

ACTION: If yes for any of the above, flag all
the associated data as estimated (J).

A.1.9.13.4 Was proper quantitation procedure followed correctly
as outlined in the SOW on page E-16 through E-17?

☐ — ☒

ACTION: If no, note exception under contract
problem/non-compliance of data assessment
narrative, or prepare a separate list.

A.1.9.14 Dissolved/Total or Inorganic/Total Analytes

A.1.9.14.1 Were any analyses performed for dissolved as well
as total analytes on the same sample(s)?

— ☒ —

Were any analyses performed for inorganic as
well as total (organic + inorganic) analytes
on the same sample(s)?

— ☒ —

NOTE:

1. If yes, prepare a list comparing differences between all dissolved (or inorganic) and total analytes. Compute the differences as a percent of the total analyte only when dissolved concentration is greater than CRDL as well as total concentration.
2. Apply the following questions only if inorganic (or dissolved) results are (i) above CRDL, and (ii) greater than total constituents.
3. At least one preparation blank, ICS, and LCS should be analyzed in each analytical run.

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	YES	NO	N/A
A.1.9.14.2 Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 10%?	—	☐	☒
A.1.9.14.3 Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 50%?	—	☐	☒

ACTION: If more than 10%, flag both dissolved (or inorganic) and total values as estimated (J); if more than 50%, reject (red-line) the data for both values.

A.1.9.15 Form I to IX

A.1.9.15.1 Are all the Form I through Form IX labeled with:

Laboratory name?	☒	—	—
Case/SAS number?	☒	—	—
EPA sample No.?	☒	—	—
SDG No.?	☒	—	—
Contract No.?	☒	—	—
Correct units?	☒	—	—
Matrix?	☒	—	—

ACTION: If no for any of the above, note under contract problem/non-compliance section of the "Data Assessment Narrative".

A.1.9.15.2 Do any computation/transcription errors exceed 10% of reported values on Form I-IX for:
(NOTE: Check all forms against raw data.)

(a) all analytes analyzed by ICP?	—	☒	—
(b) all analytes analyzed by GFAA?	—	☒	—

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	YES	NO	N/A
(c) all analytes analyzed by AA Flame?	—	[]	✓
(d) Mercury?	—	✓	—
(e) Cyanide?	—	[]	✓

ACTION: If yes, prepare Telephone Log, contact laboratory for corrected data and correct errors with red pencil and initial.

A.1.9.16 Form I (Field Blank)

Circle all field blank values on Data Summary Sheet that are greater than CRDL, 2 x IDL when IDL > CRDL.

Do concentrations of field blank(s) fall below CRDL (or 2 x IDL when IDL > CRDL) for all parameters of associated aqueous and soil samples?

[✓] — —

If no, was field blank value already rejected due to other QC criteria?

[] — ✓

ACTION: If no, reject (except field blank results) all associated positive sample data less than or equal to five times the field blank value.

A.1.9.17 Form X, XI, XII (Verification of Instrumental Parameters)

A.1.9.17.1 Is verification report present for:

Instrument Detection Limits (quarterly)?

[✓] — —

ICP Interelement Correction Factors (annually)?

[✓] — —

ICP Linear Ranges (quarterly)?

[✓] — —

ACTION: If no, contact DPO of the lab.

— A.1.9.17.2 Form X (Instrument Detection Limits) - (Note: IDL is not required for Cyanide.)

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	YES	NO	N/A
Are IDLs present for: all the analytes?	☒	☐	☐
all the instruments used?	☒	☐	☐
For both AA and ICP when both are used for same analyte?	☐	☐	☒
<u>ACTION:</u> If no for any of the above, prepare Telephone Record Log and contact laboratory.			
Is IDL greater than CRDL for any analyte?	☐	☒	☐
If yes, is the concentration on Form I of the sample analyzed on the instrument whose IDL exceeds CRDL, greater than 5 x IDL?	☐	☐	☒
<u>ACTION:</u> If no, flag as estimated all values less than five times IDL of the instrument whose IDL exceeds CRDL.			

A.1.9.17.3 Form XI (Linear Ranges)

Was any sample result higher than high linear range of ICP?	☐	☒	☐
Was any sample result higher than the highest calibration standard for non-ICP parameters?	☐	☒	☐
If yes for any of the above, was the sample diluted to obtain the result on Form I?	☐	☐	☒
<u>ACTION:</u> If no, flag the result reported on Form I as estimated (J).			

A.1.9.18 Percent Solids of Sediments

Is soil content in sediment(s) less than 50%?	☐	☐	☒
<u>ACTION:</u> If yes, qualify as estimated all data not previously rejected or flagged due to other QC criteria.			

U.S. EPA - CLP

6
DUPLICATES

EPA SAMPLE NO.

Lab Name: CEIMACContract: GERAGHTY & MINERLaplace (D)Lab Code: CEIMACCase No.: G & M

SAS No.: _____

SDG No.: HARTMANMatrix (soil/water): WATERLevel (low/med): LOW% Solids for Sample: 0.0% Solids for Duplicate: 0.0Concentration Units (ug/L or mg/kg dry weight): ug/L

Analyte	Control Limit	Sample (S)	C	Duplicate (D)	C	RPD	Q	M
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury	CRDL	0.20	B	0.24	B	18.2		CV
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium	N/A	1.0	U	1.0	U			J F
Vanadium								
Zinc								
Cyanide								

CRDL - Contract Required Detection Limit.

N/A - Not applicable.

FORM VI - IN

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Case #: G & M	Site: Tutu Teic(St. Thomas)	Matrix: Soil
SDG #: Hartman	Lab: Ceimic	Water X
Contractor: Geraghty & Miller	Reviewer: Lidya Gulizia	Other

A.2.1 The case description and exceptions, if any, are noted below with reason(s) for rejection or qualification as estimated values(s) J.

This evaluation is prepared assessing the laboratory data package supporting the inorganic analyses of drinking water samples in Sample Delivery Group (SDG) Hartman. These samples and associated field blank were collected at the Tutu Wells Site in St. Thomas, U.S. Virgin Islands on February 6 and 7, 1991. They were received at Ceimic Corporation February 8 for quantitative analysis of antimony (Sb), mercury (Hg), and/or thallium (Tl) as specified on the sample chain of custody.

All holding time criteria were met for SDG Hartman analyses.

Sample preservation to a pH of ≤ 2 for the metals analyses was verified at the laboratory prior to analysis and documented in the appropriate preparation/digestion logbook.

The field blank associated with SDG Hartman was not analyzed for antimony due to omission on the chain of custody record.

With respect to data reporting, while laboratory sample identifications (ID) for SDG Hartman were consistent throughout the data package, several client sample names were incorrectly referenced resulting in numerous misspelled ID(s) in the sample data package. In particular, sample points Bryan, Demitri, Lavergne and Ramsey were misspelled in several sections of the raw data and summary forms. Data validation was based on technical judgement and general knowledge of the site by the data reviewer.

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A.2.1 (continuation)

All calibrations for analysis of antimony by inductively coupled plasma (ICP) emission spectrometer, mercury by cold vapor (CV) atomic absorption and thallium by graphite furnace atomic absorption (GFAA) were performed daily as per the 2/88 inorganic statement of work (SOW) protocol and for every instrument set-up as required. All subsequent initial and continuing calibration verification (ICV and CCV) were within criteria with respect to percent recovery (%R) control limits and correlation coefficient.

In the thallium analysis, two initial calibration blanks (analytical sequences initiating on 3/13/91 at 08:46 and 3/13/91 at 16:07) resulted in negative values. As the blank values were either equal to or within 20% of the absolute value of the IDL and thallium was undetected in the samples at any level, no action or data qualification is necessary. No data qualification will be made for thallium analytical sequence on 3/15/91 at 09:11 for which the ICB and continuing calibration blank (CCB) were greater than, but less than two times (2x) the IDL. The slight variability of the thallium blanks is considered to have little, if any, impact on the thallium sample data results.

Contract Required Detection Limit (CRDL) QC standards were prepared in accordance with the CLP protocol for all of the inorganic target analytes and analyzed in the proper order in each of the analytical sequences as required. The accepted control limits for CRDL metals analyses are 80 - 120% recovery of the true value. In the CRDL analyses provided in support of this SDG, all CRDL standards for antimony, mercury and each of the thallium standards recovered within the criteria stated above. No data qualifiers were applied to sample results based on this criterion.

The ICP interference check solutions (ICS) were analyzed as per SOW protocol and antimony results were reported on Form IV-IN. Since antimony is not included in the ICS solutions as per EPA protocol, percent recoveries were not calculated for this analyte. Additionally, as the requested analyses and did not provide information regarding samples levels for the common antimony interferents (aluminum, calcium, iron, magnesium and manganese), no additional judgements can be made. In SDG Hartman, the Hartman 2 Well sample is the

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A.2.1 (continuation)

only sample to which the evaluation as a false positive could apply (antimony result of 30.0B ug/L) and, as such, the antimony result in this sample has been qualified as an estimated positive hit (J).

Aqueous laboratory control samples (LCS) were prepared and analyzed as per the CLP protocol to assess laboratory performance in the analysis of ICP and GFAA analytes. In the analysis of thallium, percent recovery criteria was met and no data qualifications have been made.

Hartman 2 Well sample was utilized for assessment of precision between laboratory duplicates in the analysis of thallium and antimony. As per protocol, precision for both of these analytes was not evaluated with respect to an applicable control limit since all values were below the respective IDL. No laboratory duplicates were analyzed in support of the mercury analyses. Since no mercury results were greater than the CRDL, data qualifications (J) were not applied.

Hartman 2 Well sample was used to evaluate accuracy based on pre-digest spike additions for antimony and thallium. No qualification of spike recovery (%R) was necessary as both analytes recovered within the required criteria (75-125%). Assessment of accuracy with respect to the mercury analyses cannot be made as no associated sample matrix spike was prepared in support of mercury for SDG Hartman. As per validation guidelines, all positive data less than four times the spiking level has been qualified as estimated (J) for which spiked samples were not analyzed. In SDG Hartman, LaPlace Well and Lavergne Well samples have been qualified as estimated based on the above.

In the analysis for thallium by GFAA, all sample results were reported from the mean of duplicate injections which had met contract guidelines for analysis, as applicable. All samples, post-digestion analytical spikes, associated standards and quality control (QC) samples with calculated values above the analyte CRDL met the %RSD criteria as required upon initial analysis. No samples required reanalysis.

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STANDARD OPERATING PROCEDURE

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Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.1: Data Assessment - Contract
Compliance (Total Review - Inorganics)

Date: Feb. 1990
Number: HW-2
Revision: 10

A.2.1 (continuation)

For all samples of this SDG requiring thallium analysis with the exception of the field blank sample, the post-digestion thallium spikes recovered outside the spike recovery criteria of 85-115 percent recovery. As all samples, excluding the field blank, displayed absorbances less than 50 percent of the post digestate spike absorbance and are less than the IDL, results for all samples excluding the field blank are qualified as estimated (UJ) for thallium.

The ICP serial dilution was performed using the Hartman 2 Well sample. As the antimony concentration should be minimally a magnitude of fifty times above the IDL to evaluate the effect of any significant physical or chemical interferences due to the sample matrix, no data qualifications have been assessed based on the evaluation of this criterion.

Data accuracy were examined for method anomalies, transcription and/or reduction errors, and for results calculated outside of the appropriate linear ranges per analyte. No discrepancies other than sample identification errors were found. Sample identifications were left as reported by the laboratory.

Overall precision was evaluated using field duplicate samples. In SDG Hartman, the LaPlace Well sample was duplicated and identified as Lavergne. Each of the field duplicate pair were analyzed for mercury and thallium only. In the analysis of mercury, field replicate precision was evaluated by comparing the absolute value of the difference between the field duplicate pair to the CRDL for mercury. As the criteria for relative percent difference was met, no qualification of mercury data was made. As both values in the analysis of thallium in the duplicate field pair were less than the IDL, relative percent difference (%RPD) was not calculated.

STANDARD OPERATING PROCEDURE

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Title: Evaluation of Metals Data for the
Contract Laboratory Program
Appendix A.1: Data Assessment - Contract
Compliance (Total Review - Inorganics)

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Number: HW-2
Revision: 10

A.2.2 Contract Problems/Non Compliance:

The field blank sample did not have a request for antimony analysis specified on the sample traffic report (chain of custody record), although associated well samples had this analyte targeted for analysis.

A mercury matrix spike and laboratory duplicate were not prepared and analyzed in support of mercury analyses in SDG Hartman.

As per modifications made to the Sampling, Analysis and Monitoring Plan, all thallium analyses were to be performed using the Method of Standard Additions (MSA) and were not.

MMB Reviewer: _____
Signature

Date: _____

Contractor Reviewer: Lidia Galizia
Signature

Date: 5/10/91

Verifier by: Juan Garcia / TUD

Date: 5/10/91

Table 2. Results of Quarterly Analysis for Metals in Ground-Water Samples Collected in February 1991 at the Tutu Wells Site, St. Thomas, U.S. Virgin Islands.

	Sample ID	Bryan	Dede	Dench	Fernandez	Devcon I	Devcon III	Demitri	Eglin I	Eglin II	Eglin III	Eglin IV	Four Winds II	Gassett	Hartman II
Analyte	Date	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5 Feb 91
Antimony		NA	27 U	27 U	27 U	27 U	27 U	NA	NA	27 U	27 U	27 U	27 U	NA	30 B.J
Mercury		.2 U	NA	NA	NA	NA	NA	.2 U	.2 U.J	NA	NA	NA	NA	.2 U	NA
Selenium		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Thallium		1 U.J	1 U.J	1 U.J	1 U.J	1 U.J	1 U.J	1 U.J	1 U.J	1 U	1 U	1 U	1 U.J	1 U.J	1 U.J

Analyses were performed by Ceimic Corporation, Narragansett, Rhode Island.
 Samples were analyzed by various USEPA methods.

ug/L Analyte concentrations in micrograms per liter [parts per billion (ppb)].

- B Compound is also detected in the blank.
- J Result is detected below the reporting limit or is an estimated concentration.
- U Compound analyzed for but not detected.
- NA Not analyzed.
- * Fernandez is a field replicate of Dench.
- * Eglin IV is a field replicate of Eglin III.
- * Lavergne is a field replicate of LaPlace.

Table 2. Results of Quarterly Analysis for Metals in Ground-Water Samples Collected in February 1991 at the Tutu Wells Site, St. Thomas, U.S. Virgin Islands.

	Sample ID Hartman III	Harvey	LaPlace	Lavergne	Leonard	Matthias	Ramsey	Rodriguez	Smith	Steele	Tillet	VIHA III	Field Blank
Analyte	Date	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91
Antimony	27 U	27 U	NA	NA	27 U	NA	NA	27 U	27 U	27 U	NA	27 U	NA
Mercury	NA	NA	.2 BJ	.24 BJ	NA	.2 UJ	.2 U	NA	NA	NA	.2 U	NA	.2 U
Selenium	NA	NA	NA	NA	NA	NA	NA	NA	11.7 J	NA	NA	NA	NA
Thallium	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U

Analyses were performed by Ceimic Corporation, Narragansett, Rhode Island.
 Samples were analyzed by various USEPA methods.

ug/L Analyte concentrations in micrograms per liter [parts per billion (ppb)].

- B Compound is also detected in the blank.
- J Result is detected below the reporting limit or is an estimated concentration.
- U Compound analyzed for but not detected.
- NA Not analyzed.
- * Fernandez is a field replicate of Dench.
- * Eglin IV is a field replicate of Eglin III.
- * Lavergne is a field replicate of LaPlace.

Table 3. Results of Quarterly Analysis for Volatile Organic Compounds in Ground-Water Samples Collected in February 1991 at the Tutu Wells Site, St. Thomas, U.S. Virgin Islands.

Analyte	Sample ID Bryan	Dede	Demitri	Dench	Fernandez	Devcon I	Devcon III	Eglin I	Eglin II	Eglin III	Eglin IV	Four Winds II	Gassett	Hartman II
Date	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5-Feb-91	5 Feb 91
Chloromethane	2 UJ	.9 J	2 UJ	2 UJ	2 UJ	RR	2 UJ	2 UJ	2 UJ	2 UJ	2 U	2 U	2 U	2 U
Bromomethane	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	2 UJ	2 UJ	2 U	2 U
Vinyl chloride	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	2 UJ	2 UJ	2 U	2 U
Chloroethane	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	2 UJ	2 UJ	RR	2 U
Methylene chloride	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
Acetone	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR
Carbon disulfide	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ
1,1-Dichloroethane	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
1,1-Dichloroethane	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
1,2-Dichloroethane (total)	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	60 J	72 DJ	45 J	45 J	230 U	1 U	5 J
Chloroform	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
1,2-Dichloroethane	1 U	.6 J	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
2-Butanone	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR	RR
1,1,1-Trichloroethane	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
Carbon tetrachloride	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
Vinyl acetate	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	RR	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	RR	2 UJ
Bromodichloromethane	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
1,2-Dichloropropene	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
cis-1,3-Dichloropropene	RR	1 U	RR	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	RR	RR
Trichloroethane	1 U	1 U	1 U	1 U	1 U	RR	1 U	8	19	11	13	21	1	2
Dibromochloromethane	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U	1 U	1 U
1,1,2-Trichloroethane	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
Benzene	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 UJ	1 U	1 U
trans-1,3-Dichloropropene	1 U	RR	1 U	RR	RR	RR	RR	RR	RR	RR	RR	RR	1 U	1 U
Bromoform	1 U	RR	1 U	RR	RR	RR	RR	RR	RR	RR	RR	1 U	1 U	1 U
4-Methyl-2-pentanone	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	RR	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ
2-Hexanone	RR	2 UJ	RR	2 UJ	2 UJ	RR	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ	2 UJ
Tetrachloroethane	1 U	1 U	1 U	1 U	1 U	RR	1 U	26	34 DJ	35	36	90 D	1 U	9
1,1,2,2-Tetrachloroethane	1 U	RR	1 U	RR	RR	RR	RR	RR	RR	RR	1 U	1 U	1 U	1 U
Toluene	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 UJ	44 D	1 U
Chlorobenzene	1 U	1 U	1 U	1 U	1 U	RR	1 U	1 U	1 U	1 U	1 U	1 UJ	1 U	1 U
Ethylbenzene	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 UJ	1 U	1 U
Styrene	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U
Xylenes (total)	1 U	1 U	1 U	1 UJ	1 UJ	RR	1 UJ	1 J	1 UJ	1 UJ	1 UJ	1 UJ	1 U	1 U
TOTAL VOCs:	0.0	1.5	0.0	0.0	0.0	0.0	0.0	95	125	91	94	341	45	16

Analyses were performed by Ceimic Corporation, Narragansett, Rhode Island.
 Samples were analyzed by USEPA Method 524.2.

ug/L Analyte concentrations in micrograms per liter [parts per billion (ppb)].

- D Compound identified at a secondary dilution.
- J Result is detected below the reporting limit or is an estimated concentration.
- U Compound analyzed for but not detected.
- RR Result rejected.
- * Fernandez is a field replicate of Dench.
- * Eglin IV is a field replicate of Eglin I.
- * Laverne is a field replicate of Dench.

Table 3. Results of Quarterly Analysis for Volatile Organic Compounds in Ground-Water Samples Collected in February 1991 at the Tutu Wells Site, St. Thomas, U.S. Virgin Islands.

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Analyte	Sample ID Date	Hartman 5-Feb-91	Harvey 5-Feb-91	LaPlace 5-Feb-91	Lavergne 5-Feb-91	Leonard 5-Feb-91	Matthias 5-Feb-91	Ramsey 5-Feb-91	Rodriguez 5-Feb-91	Smith 5-Feb-91	Steele 5-Feb-91	Tillot 5-Feb-91	VIHA III 5-Feb-91	Field Blank 5-Feb-91	Trip Blank 1 4-Feb-91
Chloromethane		2 U	200 U	2 U	2 U	2 U	25 U	2 UJ	2 UJ	2 UJ	10 U	2 U	2 UJ	2 U	2 UJ
Bromomethane		2 UJ	200 U	2 U	2 U	2 U	25 U	RR	2 U	RR	10 U	2 U	RR	2 U	RR
Vinyl chloride		2 UJ	200 U	2 U	2 U	2 U	25 U	RR	2 U	RR	10 U	7	RR	2 U	RR
Chloroethane		2 UJ	200 U	RR	RR	RR	25 U	RR	2 U	RR	10 U	2 U	RR	RR	RR
Methylene chloride		1 U	100 U	1 U	1 U	1 U	25 U	1 U	1 U	1 U	5 U	1 U	1 U	2	2
Acetone		RR	200 U	RR	RR	RR	25 U	RR	RR	RR	10 U	RR	RR	RR	RR
Carbon disulfide		1 UJ	100 U	1 UJ	1 UJ	1 UJ	12 UJ	1 UJ	1 UJ	1 UJ	5 U	1 UJ	1 UJ	1 UJ	1 UJ
1,1-Dichloroethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 U
1,1-Dichloroethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 U
1,2-Dichloroethane (total)		1 U	160	200 D	190 D	1 U	21	1 J	1 U	42 J	44	200 D	1 U	1 U	1 UJ
Chloroform		1 U	100 U	1	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 U
1,2-Dichloroethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 UJ
2-Butanone		RR	200 U	RR	RR	RR	25 U	RR	RR	RR	10 U	RR	RR	RR	RR
1,1,1-Trichloroethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 UJ
Carbon tetrachloride		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 U
Vinyl acetate		2 UJ	200 U	RR	RR	RR	25 U	2 U	RR	2 UJ	10 UJ	2 UJ	2 UJ	RR	2 UJ
Bromodichloromethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 UJ
1,2-Dichloropropane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 U
cis-1,3-Dichloropropene		1 U	100 U	RR	RR	RR	12 U	RR	RR	1 U	5 U	RR	RR	RR	1 U
Trichloroethene		1 U	60 J	32	31	1 U	7 J	1	1 U	23	12	36	1 U	1 U	1 U
Dibromochloromethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 UJ
1,1,2-Trichloroethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	5	5 U	1 U	1 U	1 U	1 U
Benzene		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	27	1 U	1 U	1 UJ
trans-1,3-Dichloropropene		RR	100 U	1 U	1 U	1 U	12 U	1 U	1 UJ	RR	5 U	1 U	1 U	1 U	RR
Bromoform		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	RR
4-Methyl-2-pentanone		2 UJ	200 U	2 UJ	2 UJ	2 UJ	25 U	2 UJ	2 UJ	2 UJ	10 U	2 UJ	2 UJ	2 UJ	2 UJ
2-Hexanone		2 UJ	200 U	2 UJ	2 UJ	2 UJ	25 U	RR	2 UJ	2 UJ	10 U	2 UJ	RR	2 UJ	2 UJ
Tetrachloroethane		1	1500	110 D	100 D	1 U	76	16	1 U	160 J	65	100 D	1 U	1 U	1 U
1,1,2,2-Tetrachloroethane		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 UJ	1 U	5 U	1 U	1 U	1 U	RR
Toluene		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1	1 U	1 U	1 UJ
Chlorobenzene		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 U	5 U	1 U	1 U	1 U	1 U
Ethylbenzene		1 U	100 U	1 U	1 U	1 U	12 U	1 U	1 UJ	1 U	5 U	1	1 U	1 U	1 UJ
Styrene		1 UJ	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 UJ	5 U	1 U	1 U	1 U	1 UJ
Xylenes (total)		1 UJ	100 U	1 U	1 U	1 U	12 U	1 U	1 U	1 UJ	5 U	1 U	1 U	1 U	1 UJ
TOTAL VOCs:		1	1720	343	321	0.0	104	18	0.0	230	121	372	0.0	2	2

TUT 002 2301

Analyses were performed by Celmic Corporation, Narragansett, Rhode Island.
Samples were analyzed by USEPA Method 524.2.

ug/L Analyte concentrations in micrograms per liter [parts per billion (ppb)].

- D Compound identified at a secondary dilution.
- J Result is detected below the reporting limit or is an estimated concentration.
- U Compound analyzed for but not detected.
- RR Result rejected.
- * Fernandez is a field replicate of Danch.
- * Eglin IV is a field replicate of Eglin III.
- * Lavergne is a field replicate of LaPlace.

Table 3. Results of Quarterly Analysis for Volatile Organic Compounds in Ground-Water Samples Collected in February 1991 at the Tutu Wells Site, St. Thomas, U.S. Virgin Islands.

Analyte	Sample ID	Trip	Trip	Trip
	Blank 2	Blank 3	Blank 4	
Date	5-Feb-91	6-Feb-91	7-Feb-91	
Chloromethane	2 UJ	1 J	1 J	
Bromomethane	RR	2 U	2 U	
Vinyl chloride	RR	2 U	2 U	
Chloroethane	RR	2 U	2 U	
Methylene chloride	3	2	2	
Acetone	RR	RR	RR	
Carbon disulfide	1 UJ	1 UJ	1 UJ	
1,1-Dichloroethene	1 U	1 U	1 U	
1,1-Dichloroethane	1 U	1 U	1 U	
1,2-Dichloroethene (total)	1 UJ	1 U	1 U	
Chloroform	1 U	1 U	1 U	
1,2-Dichloroethane	1 UJ	1 U	1 U	
2-Butanone	RR	RR	RR	
1,1,1-Trichloroethane	1 UJ	1 U	1 U	
Carbon tetrachloride	1 U	1 U	1 U	
Vinyl acetate	2 UJ	2 UJ	2 UJ	
Bromodichloromethane	1 UJ	1 U	1 U	
1,2-Dichloropropane	1 U	1 U	1 U	
cis-1,3-Dichloropropene	1 U	RR	RR	
Trichloroethene	1 U	1 U	1 U	
Dibromochloromethane	1 UJ	1 U	1 U	
1,1,2-Trichloroethane	1 U	1 U	1 U	
Benzene	1 UJ	1 U	1 U	
trans-1,3-Dichloropropene	RR	1 U	1 U	
Bromoform	RR	1 U	1 U	
4-Methyl-2-pentanone	2 U	2 U	2 U	
2-Hexanone	2 UJ	2 UJ	2 UJ	
Tetrachloroethane	1 U	1 U	1 U	
1,1,2,2-Tetrachloroethane	RR	1 U	1 U	
Toluene	1 UJ	1 U	1 U	
Chlorobenzene	1 U	1 U	1 U	
Ethylbenzene	1 UJ	1 U	1 U	
Styrene	1 UJ	1 U	1 U	
Xylenes (total)	1 UJ	1 U	1 U	
TOTAL VOCs:	3	3	3	

TUT 002 2302

Analyses were performed by Ceimic Corporation, Narragansett, Rhode Island.
 Samples were analyzed by USEPA Method 524.2.

ug/L Analyte concentrations in micrograms per liter [parts per billion (ppb)].

- D Compound identified at a secondary dilution.
 J Result is detected below the reporting limit or is an estimated concentration.
 U Compound analyzed for but not detected.
 RR Result rejected.
 * Fernandez is a field replicate of Dench.
 * Eglin IV is a field replicate of Eglin III.
 * Lavergne is a field replicate of LePlace.