CEMP Memorandum No. 200-1-1	Department of the Army U.S. Army Corps of Engineers Washington, DC 20314-1000	EM 200-1-1 1 Jul 94
	Environmental Quality VALIDATION OF ANALYTICAL CHEMISTRY	
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DEPARTMENT OF THE ARMY
U.S. Army Corps of Engineers
Washington, DC 20314-1000

CEMP-RT
CECW-E

Manual
No. 200-1-1

1 July 1994

Environmental Quality
VALIDATION OF ANALYTICAL CHEMISTRY LABORATORIES

Table of Contents

Appendices | Page

Figures

Tables

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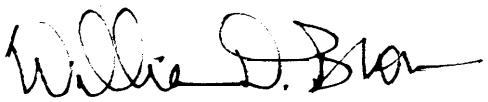
VALIDATION OF ANALYTICAL CHEMISTRY LABORATORIES

1. Purpose. This manual implements the USACE policy and requirements on validation of analytical chemistry laboratories as prescribed in the USACE Engineer Regulation 1110-1-263. This manual provides detailed procedures, guidance, and criteria for validation of commercial and USACE Division analytical chemistry laboratories. Laboratory validation is required to ensure that analytical chemistry laboratories meet the minimum requirements of the USACE quality assurance/quality control program that facilitates the generation of chemical data of known and acceptable quality.

2. Applicability. This manual applies to HQUSACE/OCE elements, major subordinate commands, districts, laboratories, and separate field operating activities having responsibility for in-house or contracted projects involving chemical measurements of waste and/or environmental samples. This includes, but is not limited to, execution of the following programs: Defense Environmental Restoration Program; Base Realignment and Closure; Installation Environmental Compliance; Military Construction; Superfund; Civil Works; and Department of Energy.

FOR THE COMMANDER:



WILLIAM D. BROWN
Colonel, Corps of Engineers
Chief of Staff

CHAPTER 1

INTRODUCTION

1-1. Purpose. This manual implements policy and provides guidance, procedures, and criteria for the validation of commercial and U.S. Army Corps of Engineers (USACE) division analytical chemistry laboratories. Laboratory validation is required to ensure that analytical chemistry laboratories meet the USACE Chemical Data Quality Management (CDQM) requirements as prescribed in the USACE Engineer Regulation (ER) 1110-1-263 for generation of chemical data of sufficient quality to meet intended usages within the project.

1-2. Applicability.

a. This manual applies to HQUSACE/OCE elements, major subordinate commands, districts, laboratories, and separate field operating activities (FOA) having responsibility for in-house or contracted projects involving chemical measurements of waste and/or environmental samples. This includes, but is not limited to, execution of the following programs: Defense Environmental Restoration Program (DERP); Base Realignment and Closure (BRAC); Installation Environmental Compliance; Military Construction; Superfund; Civil Works; and Department of Energy (DOE).

b. This manual and its prescribed laboratory validation process also apply to the validation of USACE division laboratories with minor modifications. USACE division laboratories and commercial laboratories, which perform the QA function, shall be evaluated under more stringent criteria than commercial primary project laboratories.

1.3 References.

a. ER 1110-1-263, Chemical Data Quality Management for Hazardous Waste Remedial Activities.

b. “Hazardous, Toxic & Radioactive Waste (HTRW) - Policy Guidance on Validation of Commercial Analytical Chemistry Laboratories”, CEMP-RT memorandum, (See Appendix A.)

1-4. Overview.

a. The purpose of laboratory validation is to ensure that analytical chemistry laboratories meet the minimum requirements of the USACE quality assurance/quality control (QA/QC) program that facilitates the generation of chemical data of known and

acceptable quality. Objectives of commercial laboratory validation are: to communicate USACE QA/QC requirements; to verify that commercial laboratories are performing specified analytical methods with no unacceptable deviations; and to verify these laboratories meet USACE QA/QC requirements prior to sample analysis. In general, all commercial laboratories that support USACE HTRW response activities shall obtain a USACE laboratory validation prior to field studies or sample analyses and shall maintain the validated status throughout the response activities. Appendix B is an introduction to laboratory validation procedures for commercial laboratories that express interest in USACE laboratory validation but have not been tasked to execute chemical analysis in support of USACE HTRW response activities.

b. The USACE laboratory validation process consists of three major sequential steps: (1) review of general qualifications, (2) analysis of performance evaluation (PE) samples, and (3) on-site laboratory inspection. The validation provides a parameter, method, and matrix-specific approval. The period of validation is 18 months. For each new contract/ project/task order (hereafter referred to as the contract or project) awarded to a commercial laboratory after its initial validation, a project-specific evaluation of the laboratory's capability and past performance is still required. A simplified flow diagram is shown in Figure 1-1 to show the major events in a laboratory validation process.

c. Abbreviations, acronyms, formulas, symbols, numbers, and terms used in this manual are defined in Appendix M.

1-5. Responsibilities. The USACE HTRW Mandatory Center of Expertise (HTRW MCX) located at the Missouri River Division in Omaha, Nebraska is tasked by HQUSACE with the operation and management responsibilities for this centralized laboratory validation program. A Laboratory Validation Committee (hereafter referred to as the Committee), composed of staff members from the Chemistry Branch of the HTRW MCX, is generally responsible for all aspects of the USACE HTRW laboratory validation program. One of the Committee members is designated as the Laboratory Validation Coordinator (hereafter referred to as the Coordinator) who is the point-of-contact for the Committee and is responsible for coordination and execution of the daily activities of the laboratory validation process. The Committee will meet as needed and is primarily responsible for proposing policy and making ultimate decisions with regard to laboratory-specific validation status. Besides the Committee, a number of other parties, including government agencies and private contractors, are involved in the USACE HTRW laboratory validation process.

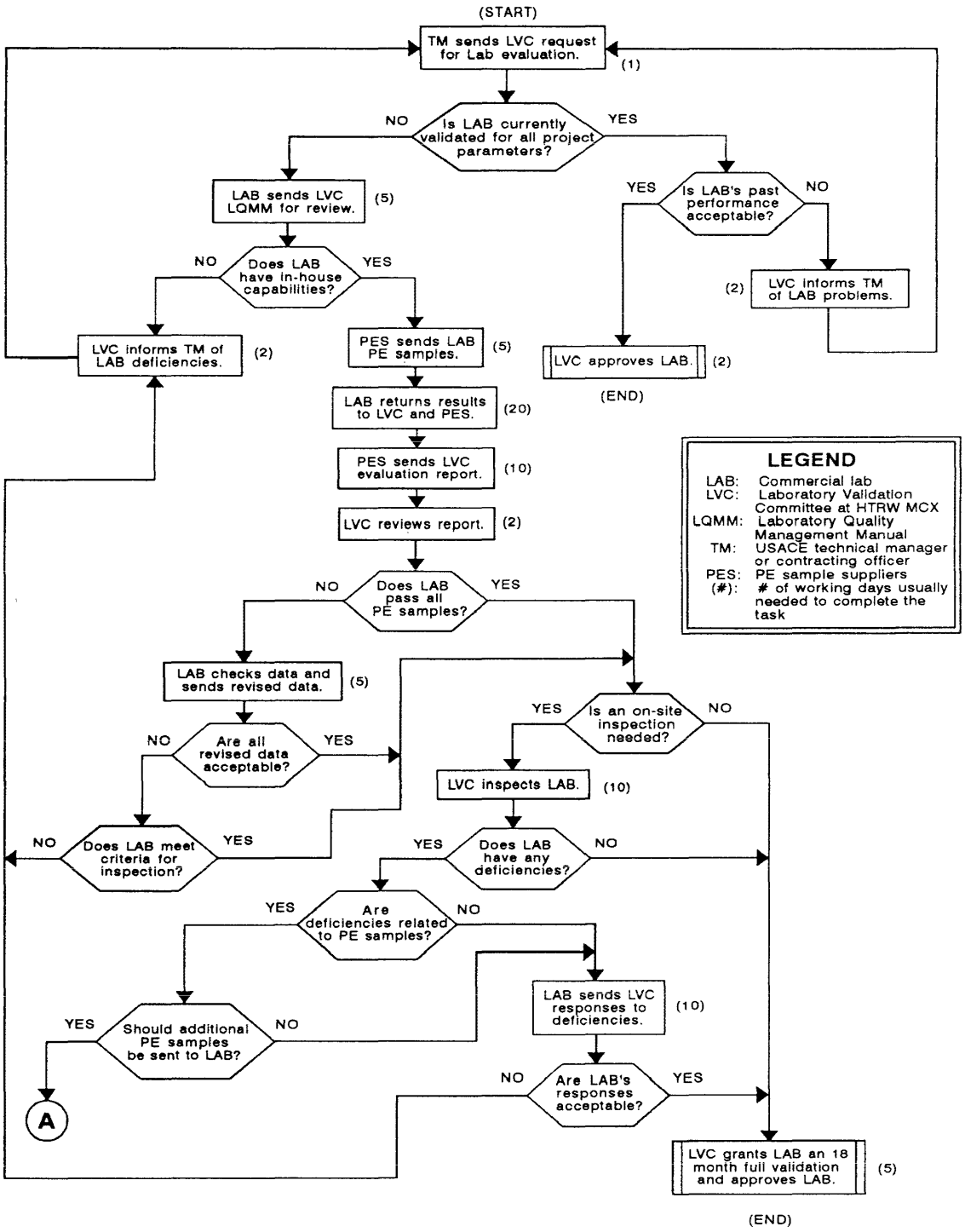


Figure 1-1. Flow Diagram of Commercial Laboratory Validation Procedures

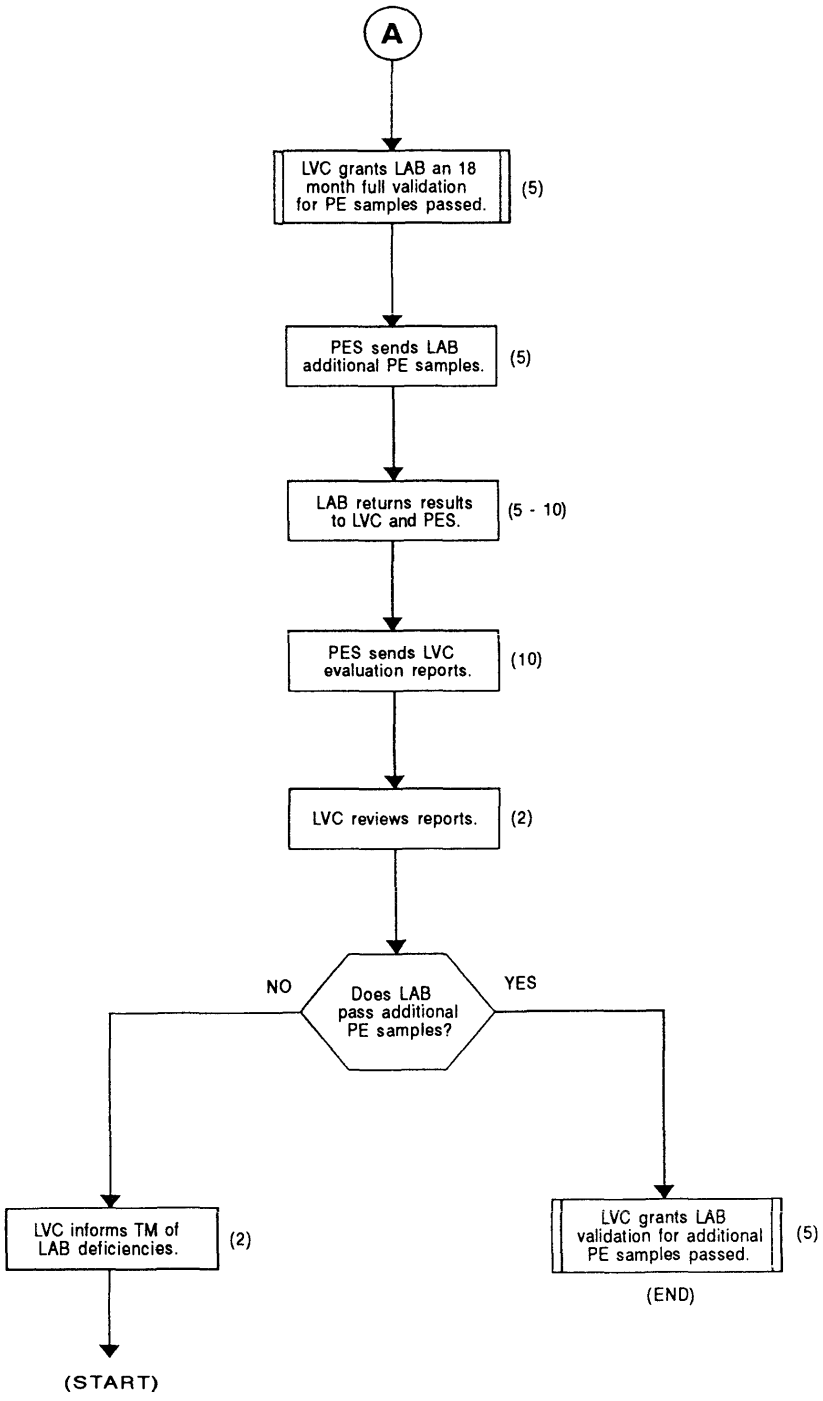


Figure 1-1. Flow Diagram of Commercial Laboratory Validation Procedures (continued)

Details on the responsibilities of all involved parties are addressed below.

a. HQUSACE:

(1) Actively performs oversight for the USACE HTRW laboratory validation program.

(2) Administers approval authority for the policies and procedures of the USACE HTRW laboratory validation program.

b. The Committee, HTRW MCX:

(1) Is responsible for all aspects of the USACE HTRW laboratory validation program including planning, programming, execution, budget, and management.

(2) Coordinates laboratory validation activities and provides liaison with various government agencies and private sector parties on laboratory validation issues. Ensures all laboratory evaluations and/or validations are successfully completed in a timely manner.

(3) Identifies the PE samples and analytical methods required for each laboratory validation. Assures that PE sample suppliers are provided with proper information to prepare and ship PE samples.

(4) Monitors the performance of PE sample suppliers through review of their most recent analytical results of any proficiency testing programs and all QA/QC data associated with the verification of PE samples on a quarterly basis. Also conducts on-site audits of PE sample suppliers on a regular basis.

(5) Reviews the qualification documents of commercial laboratories, evaluates PE sample results, and conducts or delegates on-site laboratory inspections.

(6) Trains USACE personnel to perform on-site laboratory inspections. Monitors the inspector's performance to ensure that consistent inspection approach and results of high quality are carried out within the USACE HTRW laboratory validation program.

(7) Decides the pass/fail status for each step of the laboratory validation process, additional work required for completion of laboratory validation, or the appropriate time to terminate a laboratory validation process or to revoke an active validation status.

(8) Prepares and distributes laboratory inspection and evaluation reports.

(9) Establishes and maintains a performance database for PE sample results from commercial laboratories. Statistically evaluates PE sample results to adjust or update the acceptance limits for PE sample analysis.

(10) Provides technical assistance to USACE Technical Managers/Contracting Officer Representatives (TM/CORs) to resolve problematic issues on laboratory validation and performance.

(11) Upon request, provides technical assistance to USACE TM/CORs in selection of contract laboratories prior to nomination for validation to support USACE HTRW response activities.

(12) Provides liaison with various government agencies and private sector parties on national laboratory "accreditation" programs. Revises the USACE HTRW laboratory validation program as needed to meet Federal and/or State regulatory requirements.

c. PE Sample Suppliers (including Waterways Experiment Station and Missouri River Division Laboratory):

(1) Prepare or purchase PE samples of high quality. Verify the PE samples prior to use. Maintain proper in-house documentation on PE sample preparation and verification per the U.S. Environmental Protection Agency (USEPA) and the USACE guidance. Arrange for multiple laboratory analyses of PE samples and statistically evaluate PE sample results to establish initial acceptance limits.

(2) Supply PE samples to candidate laboratories with overnight express delivery services. Ensure all PE samples are packed and shipped according to the USEPA, USACE, and the Department of Transportation (DOT) regulations and guidelines. Maintain a full chain-of-custody for each shipment of PE samples. Generate and send a sample-specific instruction letter for PE sample analysis with each PE sample shipment. Notify the Committee of any problems with PE sample preparation, verification, and shipment immediately.

(3) Provide technical assistance in resolving problems with PE sample analysis to the Committee and commercial laboratories. Keep the Committee informed of any major problems or issues on PE sample analysis.

(4) Evaluate PE sample results based on statistically established confidence limits for precision and accuracy.

Prepare and send written evaluation reports on PE sample results to the Committee within the required time frame. Provide the Committee with verbal reports on PE sample results, if a quick answer is needed.

(5) Ensure the availability and readiness of multiple sets of PE samples of different constituents and/or concentrations. Avoid sending same PE samples to same laboratory twice, including affiliated laboratories belonging to same parent corporation when possible.

(6) Actively participate in proficiency testing programs of State, Federal, and/or private firms. Provide the Committee with most recent proficiency testing results on a quarterly basis.

D. USACE TM/CORs:

(1) Submit a fully completed format of “Request for Evaluation of Commercial Laboratory” or an equivalent for each laboratory-project case to the Committee in a timely manner.

(2) Inform the Committee of any major changes in project requirements related to chemical analyses in a timely manner.

(3) Notify the Committee immediately to terminate validation efforts if a commercial laboratory undergoing the validation process is replaced by another commercial laboratory.

(4) Provide funding, if appropriate, for laboratory validation.

(5) Inform the Committee of any performance problems with sample analysis.

e. Prime Contractors (including Architect Engineering Firms, Construction Contractors, and Government Agencies):

(1) Select a subcontract laboratory and notify the USACE TM/COR early.

(2) Provide a subcontract laboratory a copy of the final Chemical Data Acquisition Plan (CDAP) for information prior to laboratory inspection. If a CDAP is not available prior to the inspection, as a minimum, provide a copy of the Scope of Services.

f. Analytical Chemistry Laboratories (including Commercial and Government Laboratories):

(1) Respond to the Committee's requirements within the required time frame.

(2) Follow instructions to analyze and report PE sample results.

(3) Inform the Committee immediately of any major changes on the laboratory's facility, instruments, or key technical staff during the laboratory's 18-month validation period.

1-6. Expenses and Funding.

In general, "billable" items related to specific laboratory validations include: travel and per diem for on-site inspection plus time and labor spent on review of documents, inspection of laboratory, and preparation of inspection report and on preparation, testing, and shipment of PE samples. Depending on the program, customer billable items are funded on a yearly program basis or project specifically. Mixed funding for a particular validation is used if appropriate. Verbal communication with the USACE TM/COR will cover the topic of funding for a particular request. For projects under programs or missions without yearly program funds available at the HTRW MCX, the USACE TM/CORs who request the validation shall be responsible for the expense of laboratory validation that is approximately $2,500 per laboratory validated. The cost of laboratory validation may be adjusted as needed, based on updated expenses.

1-7. Effective Date and Amendments.

a. This manual is effective upon approval by the HQUSACE and shall remain in effect until superseded or terminated.

b. These procedures may be modified, revised, or amended upon approval by the HQUSACE.

c. This manual and any future revisions or amendments shall be distributed by the HQUSACE.

CHAPTER 2

PROCEDURES FOR COMMERCIAL LABORATORIES

Section I. Validation Procedures

2-1. Initiation Procedures. A laboratory validation will be initiated after a commercial laboratory successfully bids a contract to support USACE HTRW response activities. A written request from a USACE TM/COR to the Coordinator initiates the laboratory validation process. A request format as shown in Figure 2-1 or a memorandum with all information contained in Figure 2-1 may be submitted to the Coordinator by mail or facsimile, as follows:

U.S. Army Corps of Engineers
ATTN: CEMRD-ED-EC (Laboratory Validation Coordinator)
HTRW Mandatory Center of Expertise
Missouri River Division
12565 West Center Road
Omaha, Nebraska 68144-3869

Voice: (402) 221-7494
FAX : (402) 221-7403

2-2. Implementation Procedures.

a. Upon receiving the laboratory evaluation request, the Coordinator will immediately check the laboratory's current validation status. If the laboratory is currently validated by the USACE for all project-required analytical parameters and has no performance problems noted, the Coordinator will notify the USACE TM/COR in writing of the Committee's approval within ten working days. If the laboratory is not currently validated by the USACE for all project-required analytical parameters, the Coordinator will immediately notify the USACE TM/COR by phone and initiate the laboratory validation process.

b. The laboratory validation process may take up to 12 weeks; therefore, the primary contractor and/or the USACE TM/COR should plan the project schedule to allow adequate time for laboratory validation and the USACE TM/COR should submit a request for evaluation to the Coordinator as early as possible. The Committee shall also make a concerted effort to ensure that the validation process is completed within the time frame required by the project. Unless projects require specialized chemical analyses or a quick turnaround of large number of samples, normally a minimal number of commercial laboratories should be used for each contract and be requested for validation.

TO: CEMRD-ED-EC FROM:_____ DATE:____/_____/____

SUBJECT: REQUEST FOR EVALUATION OF COMMERCIAL LABORATORY

Project Name:_____
Location:_____ State:_____
Contract No:_____ Type: POL TANK REMOVAL:___HTRW:___
Program: SF:___ FUDS :___ IRP:___ AF(ACC):_____ OTHER:_____
Phase: PA/SI:____RI/SI:____ RD:____RA:____ RFA :___ RFI:____ CMS:___

Approximate Sampling Dates:_____
Project-Specific Sample Turnaround Time:_____

USACE Technical Manager:_____
Address:_____
Phone:_____ FAX:_____

A-E/Contractor:_____ State:_____
Lab Name:_____
Address:_____
POC:_____
Phone:_____ FAX:_____

Required analytical parameters, methods, and approximate number of samples to be taken for above project.

PARAMETERS & METHODS	No. of LIQUID SAMPLES	No. of SOLID SAMPLES

State or other laboratory certifications that will be required for this project:_____

Note: If the laboratory is planning to subcontract any samples to another laboratory or location, all of these laboratories shall be evaluated separately. This format should be sent for verification of laboratory status regardless of expiration date on the list of validated laboratories.

Figure 2-1 Laboratory Evaluation Request Format

c. Although three major sequential steps are involved in the laboratory validation process, the actual steps required for each laboratory, as determined by the Committee, may be different, based on the following guidelines:

(1) For commercial laboratories that have never been validated under the USACE HTRW Program: A full, three-step laboratory validation process conducted by the Committee representatives is required.

(2) For commercial laboratories that have expired laboratory validation under the USACE HTRW Program: When the next contract is awarded to support USACE HTRW response activities, a revalidation will be required. After considering the use of the laboratory and the laboratory's previous performance, the Committee will determine which of the three steps will apply to the revalidation process.

(3) For commercial laboratories that are currently validated under the USACE HTRW Program: When the laboratory obtains a new contract(s) to support USACE HTRW response activities during its validation period, the capability and past performance on USACE HTRW projects shall be verified by the Committee. If different analytes and/or matrices are involved in the new contract(s), the laboratory must pass additional PE samples for those different analytes and/or matrices. If past performance has been satisfactory, the USACE TM/COR will be notified that no further actions are required and the laboratory is validated for all parameters of the new contract(s); otherwise, a full laboratory validation might be required as determined by the Committee on a case-by-case basis.

(4) For commercial laboratories whose validations might expire while the laboratories are working on ongoing projects: A revalidation will be required if a USACE TM/COR expects that an ongoing project will extend more than six months beyond the validation expiration date. The Committee will determine which validation steps are required for the revalidation process on a case-by-case basis. If the completion of an ongoing project is anticipated within six months after the expiration date, no actions are required.

(5) For on-site mobile laboratories: The same procedures used for validation/revalidation of an off-site "fixed" commercial laboratory will apply to an on-site mobile laboratory. However, no PE samples will be sent to a mobile laboratory until the mobile laboratory is mobilized and settled down at the project site. Due to the timing of PE sample analysis and the quick turnaround nature of mobile laboratory, the laboratory

inspection for an on-site mobile laboratory can be coordinated with project schedule. The validation status of an on-site mobile laboratory terminates if the laboratory moves to a new location prior to the validation expiration date. After an on-site mobile laboratory is mobilized to a new location, another full laboratory validation is required. No laboratory validation is required for an on-site mobile laboratory that only performs field screening analysis, i.e., Level II data quality.

(6) For commercial laboratories to be used for underground storage tank removal projects:

(a) For projects involving removal of tanks, both underground storage tanks (USTs) and aboveground storage tanks (ASTs), that have been used only for storage of petroleum, oils, or lubricants (POL), there are two alternatives to the validation process. These two alternatives apply only to predesign sampling of UST organic phase contents and soil sampling during removal. They do not apply to investigations required by groundwater contamination or extensive soil contamination.

Alternative 1: State certified laboratories may be used without USACE validation, if the state considers its certification to be applicable to UST removal. When this alternative is selected, a document in the project file must identify the individual responsible for coordination with the state.

Alternative 2: The HTRW MCX will conduct an abbreviated laboratory validation process if a USACE TM/COR submits a request for evaluation of commercial laboratory. The laboratory must submit its qualification documents including laboratory quality management manual (LQMM) and standard operating procedures (SOP) for the required analyses to the Coordinator for review. If the laboratory has been recently validated for the project-specific analytical parameters and has no performance problems with USACE projects, the laboratory may be exempted from PE sample analysis. However, if performance problems with the commercial laboratories are noted, a full laboratory validation by the Committee representatives will be performed.

(b) If alternative 2 is selected, an on-site inspection by the Committee representatives for POL UST/AST removal projects is generally exempted. The USACE division laboratory that serves as the project QA laboratory, the geographic district, and/or FOA are encouraged to perform inspection per the protocols addressed in this manual. If inspections are not conducted by the Committee representatives, the inspectors must be trained and certified by the Committee prior to on-site inspections.

The Committee shall be kept fully informed of these inspections and will be free to send representative(s) to the inspections at any time. The same inspection approach and checklists as described in this manual shall also be used by the "non-Committee representative" inspectors.

(c) A commercial laboratory validated for POL UST/AST removal projects may not be used to support other HTRW projects unless a full laboratory validation is performed by the Committee representatives. A full laboratory validation will be required for a UST/AST site investigation if leaking tanks cause groundwater contamination or severe soil contamination. For projects involving removal of non-POL tanks that have contained HTRW substances or wastes, a full laboratory validation conducted by the Committee is required.

2-3. Implementation Procedure Steps. A full laboratory validation involves three major sequential steps conducted by the Committee representatives. Ordinarily, each step in the sequence is completed before the subsequent step is initiated.

a. Step 1: Review of Qualification Documents.

(1) The Coordinator will inform a commercial laboratory by phone or mail of the upcoming laboratory validation and request for review copies of the laboratory's qualification documents, including generic LQMM and other appropriate documents such as SOPs, laboratory certificates, etc. The laboratory shall submit the required documents within five working days of the request. If the laboratory does not have a LQMM, USACE will not pay for the preparation of this document. The submittals should provide appropriate information (including personnel, facilities, instrumentation, SOPs, QA/QC policies, etc.) for the Committee to evaluate and assess the laboratory's technical capabilities on the project-required chemical analyses.

(2) Upon receiving the qualification documents, one of the committee members will be designated to compare the laboratory's in-house technical capabilities with the project requirements. Within two working days, the designee will verbally convey the results of this comparison to the Coordinator. If the comparison identifies deficiencies, the Coordinator or designee shall: immediately contact the laboratory to verify the deficiencies; coordinate any follow-up actions; and verbally notify the USACE TM/COR of the problems. If deficiencies are verified, the Coordinator or designee shall present the findings to the Committee and recommend termination of the validation. Upon approval by the Committee, the Coordinator shall immediately issue a follow-up letter to notify the USACE TM/COR and the

commercial laboratory of the problems, the Committee's decision of termination of the validation process, and the need for selection of another laboratory. If it appears that the capabilities of the laboratory are adequate to meet the project requirements, the Coordinator shall immediately mail the following documents to the laboratory for information and action, and step 2 will be initiated.

- Information for Commercial Analytical Chemistry Laboratories Undergoing Validation by the U.S. Army Corps of Engineers (Appendix C),
- Guidelines for Analyzing and Reporting Performance Evaluation Samples from the U.S. Army Corps of Engineers (Appendix D), and
- Preliminary Questionnaire for the U.S. Army Corps of Engineers Validation Program for Analytical Laboratories (Appendix E).

(3) The laboratory shall complete and return a copy of the completed preliminary questionnaire within ten working days from the date of receipt.

b. Step 2: Analysis of PE Samples.

(1) The Coordinator will arrange to have PE samples sent to the laboratory for analysis. Project-specific PE samples are mandatory and must be passed. In addition to project-specific PE samples, the laboratory may volunteer for validation of additional parameters by requesting non-project-specific PE samples. The cost for the first set of project-specific PE samples will be covered by the USACE HTRW program management funds. However, for any additional sets or any non-project-specific PE samples, the laboratory will be responsible for the expense of PE samples which ranges from $100 to $300 per method, per matrix, and per shipment. Appendix F shows the fee schedule, which is subject to annual review and adjustment without notice to reflect currency value fluctuations or changes in program administration costs, for PE samples available from the USACE. A commercial laboratory is not reimbursed for costs involved in the analysis of the PE samples.

(2) If a nonstandard analytical method or a modified standard analytical method is required, the laboratory shall submit its in-house SOP and method validation data (including method detection limits, precision, accuracy, QC limits, chromatograms, etc.) to the Coordinator for review and approval. PE samples for a nonstandard or a modified standard method will

only be sent after the Committee has reviewed and approved the method. PE samples for validation of a mobile laboratory shall only be sent after the laboratory is mobilized to the project site and all instruments are calibrated. The Committee may request instrument calibration data for review prior to shipping PE samples to a mobile laboratory.

(3) Analysis of PE Samples.

(a) In general, the PE samples are method- and matrix-specific. A commercial laboratory may not subcontract PE samples to another laboratory. A commercial laboratory must use project-required analytical methods for analyses of all project-specific PE samples unless otherwise instructed by the Coordinator. The sources of analytical methods usually required for USACE HTRW projects, and therefore for the PE sample analysis, in a preferential order are as follows:

- Test Methods for Evaluating Solid Waste, SW-846 (Third Edition, Revision 0, September 1986; Revision 1, July 1992; or the most recently promulgated revisions.)

- Statements of Work for Organics Analysis, Inorganics Analysis, and Dioxin Analysis, (USEPA Contract Laboratory Program, Document Number OLM02.0, ILM03.0, DFLMO1.0, and the most recent revisions.)

- Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020 (Revised March 1983 or the most recently promulgated revisions.)

- Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88/039 (December 1988 or the most recently promulgated revisions.)

- Other standard and published methods of the most recent versions from USEPA, American Society for Testing and Materials (ASTM), American Public Health Association, American Water Works Association, Water Pollution Control Federation, United States Geological Survey (USGS), National Institute for Occupational Safety and Health (NIOSH), Department of Energy (DOE), etc.

(b) The parameters and commonly required methods for PE sample analyses are listed in Appendix F. Any changes or modifications in analytical methods for PE samples must be preapproved by the Committee. Use of nonstandard or modified standard analytical methods without a proapproval from the Committee may result in failure of PE sample analysis.

(c) PE samples will be prepared and sent out from reliable suppliers by overnight express delivery. All PE samples shall be preserved and shipped according to USACE, USEPA, and DOT regulations and guidelines. Full chain-of-custody shall be maintained for each shipment of PE samples. The analytical laboratory of Waterways Experiment Station (WES) in Vicksburg, Mississippi, and the Missouri River Division Laboratory (MRDL) in Omaha, Nebraska, are currently two of the major USACE PE sample suppliers. Guidance for PE sample suppliers including WES, MRDL, and commercial vendors on PE sample preparation, handling, and validation are described in Appendix G. The general guidelines for PE sample analysis and reporting by a commercial laboratory are described in Appendix D. Special sample-specific instructions for PE sample analysis will be provided by PE sample suppliers on the chain-of-custody document enclosed in each PE sample shipment. Any questions on PE sample analyses should be directed to the Coordinator. A commercial laboratory shall also conduct all method-specific QC analyses which include but are not limited to method blank, replicate, matrix spike, matrix spike duplicate, and surrogate spike. If the amount of material constituting the PE samples is not enough for all QC analyses, the QC analyses shall be performed on spiked reagent water.

(4) Reports of PE Sample Results.

(a) A commercial laboratory shall report the concentrations of all target analytes listed in the required analytical methods, including estimated values and the quantitation limits for target analytes not detected. The quantitation limit of each analyte must meet or be less than those specified in the method for the particular matrix. Except for petroleum hydrocarbons PE samples, all soil/sediment PE sample analyses shall be reported on a dry-weight basis along with percent moisture. For petroleum hydrocarbons PE samples, the results shall be reported on an “as-received” basis (i.e., no correction should be made for moisture content). Neither should any data be corrected for spike recoveries nor for any contamination found in trip blank or laboratory’s method blank.

(b) All method-specific QC data associated with the PE sample analysis, including method blank, replicate analysis, spike recovery, etc., shall be reported. Written reports of all PE sample analyses are to be received by the PE sample suppliers within 20 working days after receipt of the samples. For projects requiring quick turnaround for field sample analyses, the turnaround times for the PE samples may be reduced. For example, due to the often short lead-time and the quick turnaround nature of most UST removal projects, the turnaround

time for PE sample analysis needed for UST removal projects will range from five to ten working days depending on the number of parameters required. Failure to analyze the PE samples correctly and within the required time frame may result in termination of the validation process. An additional copy of all PE sample reports shall be sent to the Coordinator for review. Upon request by the Coordinator, a commercial laboratory shall also submit for review all raw data including sample preparation and run logs, calibrations, chromatograms, calculations, etc. A commercial laboratory may use its standard data package to report PE sample results; however, the data package shall be sequentially numbered and contain, as a minimum, the following information:

- Table of contents.
- A case narrative including problems encountered with PE sample analysis.
- A chain-of-custody report.
- Sample preparation information.
- Analytical results for all target analytes plus method citations and quantitation limits.
- Summary of method-specific QC results for assessment of precision and accuracy.
- Phone conversation records on major issues related to PE sample analysis.

(c) Failure to submit the requested information within a required time frame will be considered as non-responsive and may result in termination of the validation procedure. It is the laboratory's responsibility to keep the Coordinator informed early of any problems with PE sample analyses that would affect the return of results within a required time frame.

(5) Evaluation of PE Sample Results.

(a) After receipt of PE sample data reports, the PE sample suppliers should immediately evaluate the analytical data quality based on statistically established confidence limits and generally accepted QC indicators for accuracy and precision. The PE sample results will be compared in the following manner:

- with the prepared concentrations of PE samples that are used as the absolute recovery comparators, and

- with the statistical mean and standard deviations reported by a group of referee and/or peer laboratories.

(b) The general acceptance limits for analyte quantitation will be established statistically at the 95 percent confidence based on referee laboratories and/or peer group results. The Committee shall review the evaluation reports and determine the pass/fail status for PE sample results. The general criteria for acceptance of PE sample results are as follows:

- All Chemical Analyses:

  All method-specific QC data are reported and within method-specified criteria.

- Multianalyte Organic Analyses:

  No more than one target compound outside three sigma confidence limits and no more than two target compounds between two and three sigma limits. False negatives and false positives are considered as outside three sigma.

- Metal Analysis:

  No metal elements outside three sigma confidence limits and no more than two metal elements between two and three sigma limits. False negatives and false positives are considered as outside three sigma.

- Classical Chemical Analyses:

  All data are within two sigma.

(c) Within ten working days after receipt of PE sample results, the PE sample suppliers shall send the Coordinator a written evaluation report. At a minimum, the report shall contain the: laboratory name; location (city and state); dates that PE samples were delivered; laboratory's PE sample results; dates results were received; true values and/or acceptable limits for each target analyte; narratives for special problems or issues; follow-ups on failed parameter; and recommendations for pass/fail. If requested by the Coordinator, the PE sample suppliers shall provide the Committee with verbal reports on PE sample results within five working days after receipt of PE sample results. In addition to a written evaluation report, the PE sample suppliers shall also send a cover memorandum in line-item summary format with the: names of PE samples within acceptable limits; names of target analytes correctly identified, but quantitated outside acceptable limits; and number of false

positives and/or negatives reported for each PE sample. The identities of false positives and/or negatives shall not be disclosed in the cover letter or memorandum.

(d) The majority of PE samples available from the USACE are in water and/or soil/sediment matrices. If only water PE samples are available for certain analytical parameters from the USACE, a commercial laboratory that passes the water PE samples will be considered for a multimedia validation of these parameters. However, if both water and soil/sediment PE samples are available for any parameters from USACE, a commercial laboratory must pass both matrices prior to consideration for a multimedia validation for these parameters. A commercial laboratory that passes water PE samples but fails the corresponding soil/sediment PE samples for any parameters will be considered for a validation of these parameters in water samples only. However, a laboratory that passes soil/sediment PE samples but fails the corresponding water PE samples will not be considered for validation of the failed parameters in any matrix type of samples, including soil/sediment samples.

(e) For volatile and semivolatile organic analyses, some compounds in the water or soil/sediment PE samples may not be the method-specific target compounds. A laboratory is required to use the NIST/EPA/MSDC or any other USEPA approved mass spectral library to tentatively identify and quantify up to ten non-target volatile organic compounds and twenty non-target semivolatile organic compounds that exhibit the strongest ion current signals. These compounds must not be system monitoring compounds. Identification of these compounds, based on spectral interpretation procedures, is evaluated and integrated into the evaluation process for volatile and semivolatile organic PE sample results. For metal analysis, the validation will be granted for one of the following four categories based on the number of metal elements in the PE samples passed:

- Category I: Eight RCRA metal elements (arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver.)

- Category II: Fourteen RCRA and Priority Pollutant (PP) metal elements (antimony, arsenic, barium, beryllium, cadmium, chromium, copper, lead, mercury, nickel, selenium, silver, thallium, and zinc.)

- Category III: Twenty-three USEPA CLP Target Analyte List (TAL) metal elements (aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury,

nickel, potassium, selenium, silver, sodium, thallium, vanadium, and zinc.)

- Category IV: Any other metal element(s) including the four metal elements (arsenic, cadmium, chromium, and lead) usually required for UST removal projects.

(f) Based on project requirements on metal analysis, one of the above four specific categories of metal PE samples will be selected for laboratory validation. A commercial laboratory may volunteer for any one of the four categories of metal PE samples as long as more metal elements than the project-required are analyzed. Normally, a commercial laboratory must satisfactorily pass all metal elements in a specific category prior to consideration for validation of the specific category of metal elements.

(9) If PE samples for a particular parameter such as dioxin, radioactivity, air toxics, etc. are not available from the USACE, the analysis of PE samples will be exempted until the appropriate PE samples for these particular parameters become available. The validation of a commercial laboratory for parameters without PE samples available will be based solely on the laboratory's qualification documents submitted to the Coordinator for review. The qualification documents shall include: copies of the laboratory’s LQMM; laboratory certificates or licenses; and the most recent two rounds of PE sample results from other government and/or private agencies. If the parameter is the only project-required chemical analysis, an on-site inspection may be waived.

(h) For the analysis of chemical warfare agents, their degradation products, and other scheduled compounds in the complex matrices, the primary contracts shall select chemical surety laboratories that have already been approved by the U.S. Army Edgewood Research, Development and Engineering Center (ERDEC) at Aberdeen Proving Ground, Maryland. The USACE will not send PE samples to or inspect the approved chemical surety laboratories. The USACE will contact the ERDEC for technical assistance and provide a list of approved chemical surety laboratories if requested.

(i) The acceptance of PE sample results also depends on whether the results are returned in a timely manner and no procedural problems are found during a follow-up laboratory inspection. The Coordinator will send a copy of the cover letter or memorandum from the PE sample suppliers evaluation reports to the laboratory for information and/or necessary action(s) by the laboratory. Due to confidentiality requirements, the true values

and/or two sigma confidence limits for any batch of volatile organic PE samples and soil/sediment PE samples shall not be released to commercial laboratories until the batch is discontinued. A commercial laboratory will be allowed to provide revised data for failed parameters if problems such as calculation or transcription errors can be identified. If a commercial laboratory is requested by the Coordinator to check its analytical data, the laboratory shall return revised data within five working days to the Coordinator.

(j) After data revisions, a commercial laboratory must pass, as a minimum, more than 50 percent of all PE samples, including project-specific and non-project-specific PE samples, within 40 working days from receipt of the first set of PE samples, or the validation process will be terminated. The Coordinator will notify all affected USACE TM/CORs immediately and suggest selection of another laboratory by the prime contractor for evaluation. After a commercial laboratory passes 50 percent of all PE samples within 40 working days, the Coordinator will contact the laboratory to schedule an on-site inspection within ten working days. Prior to an on-site inspection, the laboratory shall submit to the Coordinator a concise written statement describing the problems, solutions, and corrective actions taken or to be taken for the analytical parameters failed in its first attempt.

c. Step 3: On-Site Laboratory Inspection.

(1) Two Committee representatives will normally serve as the inspectors to inspect a commercial laboratory after Steps 1 and 2 have been satisfactorily completed. The inspectors shall contact and invite the USACE TM/COR(s) who initiated the evaluation request(s) and the USACE division laboratory(s) that serves as the QA laboratory(s) for the project(s) to send representatives to the inspection. The PE sample suppliers may also be requested to send technical experts if assistance is needed for the inspection. During an on-site laboratory inspection, the inspectors shall verify that:

- the organization and personnel are qualified to perform assigned tasks,

- adequate facilities and equipment are available,

- complete documentation, including chain-of–custody of samples, is being implemented,

- proper analytical methodology is being used without deviations,

- adequate analytical quality control (including reference samples, control charts, documented corrective actions, etc.) is being provided,

- acceptable data handling and documentation techniques are being used,

- adequate facilities and operations are installed to ensure laboratory health and safety, and

- proper waste disposal procedures are implemented.

(2) The on-site laboratory inspection helps to ensure that the laboratory is technically competent and that all the necessary quality control is being applied by the laboratory in order to deliver a quality product. The on-site inspection also serves as a mechanism for discussing weaknesses identified through PE sample analysis or other review of data deliverables. Lastly, the on-site inspection allows the inspector to monitor whether the laboratory has continuously and successfully implemented the recommended and/or required corrective actions that were made during previous on-site inspections by the USACE. Failure to have implemented past action items may be grounds for termination of the current validation process.

(3) Prior to the inspection, the inspectors shall review all appropriate project- and laboratory-specific documents including:

- scope of services, specifications, work plans, and/or chemical data acquisition plan, if available,

- LQMM and qualification documents,

- preliminary questionnaire,

- PE sample results and evaluation reports,

- previous inspection reports, if applicable, and

- previous performance on USACE HTRW projects based on the chemical quality assurance reports (CQARS) for projects that the laboratory has previously worked on.

(4) The on-site inspection generally takes eight hours and normally consists of three parts: entrance interview, laboratory tour, and exit interview. The entrance interview will be held with the upper laboratory management personnel (including laboratory director/managers, QA officer, and project personnel)

to discuss the upcoming USACE projects, the USACE QA program, the USACE review comments on the laboratory's LQMM, the PE sample results, and the laboratory's previous performance on USACE projects, if applicable. A copy of written comments on the LQMM shall be presented to the laboratory during the entrance interview. The inspectors will also present an overview of the laboratory's performance on PE sample analysis.

(5) A tour of the commercial laboratory will follow to examine the laboratory facilities, instrumentation, operation, maintenance, documentation, safety, waste compliance, etc. The audit tour is generally conducted in a manner that allows the following of a sample through the laboratory, and looking at all operations that a sample is exposed to during its transfer of custody, digestion/extraction, and analysis. This includes sample/digestate/extract storage, instrument calibration, SOPS, documentation, data review and reporting, etc. During the tour, the inspectors shall also examine the raw data of the PE samples and talk with the analysts who performed the analyses of any failed PE samples to determine the cause of failure and to decide if additional PE samples are needed for the failed parameters. The inspectors should adhere to the inspection guidelines and criteria in Appendix H and use the appropriate laboratory inspection checklists in Appendices I or J.

(6) At the conclusion of the laboratory tour, the inspectors shall request a 30-minute close door session to organize, review, and document the findings. After the close door session, an open exit interview will be held with laboratory personnel in which a summary of any deficiencies and recommendations is discussed. The format in Figure 2-2 can be used to document the meeting summary on deficiencies, recommendations, and/or any other findings, if applicable. The authorized representative of the laboratory shall be asked to sign the meeting summary to attest that the laboratory representative has reviewed the meeting summary with the inspectors. The laboratory has ten working days to submit written responses with supporting documentation to the deficiencies and/or recommendations to prevent possible validation termination. The responses shall address the corrective actions that have been taken or will be taken with proposed implementation and completion schedules. All deficiencies shall be corrected by the laboratory prior to performing USACE HTRW project work. Recommendations based on good laboratory practice for operations and management are for the laboratory's consideration.

2-4. Approval Procedures. Normally, within five working days after the inspection, the inspectors shall organize, document,

U.S. ARMY CORPS OF ENGINEERS
ON-SITE LABORATORY INSPECTION SUMMARY

LAB NAME/LOCATION: _____

DATE/TIME: _____

PURPOSE: This format documents any deficiencies and recommendations noted during the on-site laboratory inspection. The laboratory has ten working days to submit written responses with supporting documentation, including an implementation schedule for any corrective actions, to the deficiencies and recommendations to prevent possible validation termination.

MEETING ATTENDEES:

NAME	ORGANIZATION/TITLE	PHONE

Figure 2-2 On-Site Laboratory Inspection Summary

ON-SITE INSPECTION SUMMARY:

DEFICIENCIES :

Figure 2-2 On-Site Laboratory Inspection Summary (continued)

ON-SITE INSPECTION SUMMARY:

RECOMMENDATIONS:

OTHER FINDINGS:

ACKNOWLEDGMENT:

LABORATORY:_____

USACE INSPECTION TEAM:_____

Figure 2-2 On-Site Laboratory Inspection Summary (continued)

and verbally present the findings and the recommended validation status for the laboratory to the Committee for approval and/or concurrence. In the event that supportive documents from a laboratory are needed before a final decision by the Committee, the inspectors shall present a second presentation within five working days after receipt of the requested materials from the laboratory. A minimum of three members of the Committee must be present in the review meeting to determine the validation status of a commercial laboratory. The decisions of the Committee can be documented in the format shown in Figure 2-3. Normally, a parameter- and matrix-specific full validation for 18 months will be granted to a commercial laboratory after the laboratory has satisfactorily met all USACE HTRW laboratory validation criteria. The 18 months start from the date that the Committee first met after the inspection and agreed upon the laboratory's validation status. The guidelines for determination of validation status for a commercial laboratory are as follows:

a. For a commercial laboratory that passes all PE samples and has no deficiencies noted during the on-site inspection, a full validation status of 18 months will be granted for all analytical parameters that the laboratory has passed the associated PE samples.

b. For a commercial laboratory that passes all PE samples but has deficiencies noted during the on-site inspection, a full validation status of 18 months will be granted for all analytical parameters that the laboratory has passed the associated PE samples. However, validation will only be granted after the Committee reviews and accepts the written responses from the laboratory and the laboratory completes the implementation of corrective actions for the deficiencies.

c. For a commercial laboratory that does not pass all PE samples but has no other deficiencies noted during the on-site inspection, a full validation status of 18 months will be granted for all analytical parameters that the laboratory has passed the associated PE samples. Validation may also be granted for analytical parameters if it is determined during the on-site inspection that the failure was due to minor errors, such as errors in data calculation, transcription, etc. For any failed parameters caused by major errors (such as errors in analytical procedure, spectra interpretation, etc.) or unknown/unsure reasons, the laboratory must pass additional PE samples prior to consideration for validation of the additional parameters. In this case, one set of additional PE samples will be sent to the commercial laboratory that failed the first set of PE samples. Results of the additional set of PE sample analyses shall be returned to the PE sample suppliers and the Coordinator within

U.S. ARMY CORPS OF ENGINEERS
LABORATORY EVALUATION COMMITTEE
VALIDATION REVIEW MEETING SUMMARY

LAB NAME/LOCATION:_____

REVIEW MEETING DATE/TIME:_____

PURPOSE: This format documents the final committee decisions on the validation status of a contract laboratory inspected by the staffs of the Army Corps of Engineers.

MEETING ATTENDEES:

NAME	ORGANIZATION/TITLE
1.	
2.	
3.	
4.	
5.	
6.	
7.	
8.	
9.	
10.	
11.	
12.	

INSPECTOR'S RECOMMENDATIONS TO COMMITTEE:

Figure 2-3 Validation Review Meeting Summary

VALIDATION REVIEW MEETING SUMMARY:

MAJOR FACTORS SUPPORTING COMMITTEE DECISIONS:

FINAL COMMITTEE DECISIONS:

SIGNATURES OF COMMITTEE MEMBERS:

1.		2.	
3.		4.	
5.		6.	
7.		8.	
9.		10.	
11.		12.	

Figure 2-3 Validation Review Meeting Summary (continued)

five or ten working days, depending on the number of additional PE samples required. The Committee will make the final decision on the pass/fail status of PE sample analysis or any additional work needed to pass PE samples. If a commercial laboratory fails to pass the additional set of PE samples, no validation status will be granted for the additional parameters.

d. For a commercial laboratory that does not pass all PE samples and also has other deficiencies noted during the on-site inspection, similar procedures and criteria as described in paragraph 2-4.c. will be used to determine the laboratory's validation status. However, validation will only be granted after the Committee reviews and accepts the written responses from the laboratory and the laboratory completes the implementation of corrective actions for the deficiencies.

e. For a commercial laboratory that has deficiencies noted during the on-site inspection, but failed to submit acceptable responses or to satisfactorily complete corrective actions within the required time frame, no validation status will be granted. A commercial laboratory that is considered to have failed on attempted validation shall wait for six months prior to repeating the validation process, and then the process is only initiated by a written request from a USACE TM/COR. If a commercial laboratory fails the laboratory validation process during any of the three major steps mentioned previously, another commercial laboratory or a prevalidated commercial laboratory must be selected by the prime contractor for evaluation. If another non-validated laboratory is selected, the prime contractor will be responsible for the expense of this additional laboratory validation.

f. A commercial laboratory, that is exempted from PE sample analysis due to lack of suitable PE samples from USACE, will be granted a six month conditional validation. The performance of a commercial laboratory granted a conditional validation status will be closely monitored by the USACE TM/CORs, the USACE division laboratories that serve as the government QA laboratories, and the Committee during the conditional period. Prior to the end of the conditional validation, the Committee will review the case and determine the appropriate actions required for a full validation for an additional 12 months. Normally, if no performance problems are noted during the probation period, a full validation will be granted. The Coordinator shall keep all affected USACE TM/CORs informed of any changes of validation status of commercial laboratories.

g. For a commercial laboratory inspected by a "non-committee representative" inspector for UST removal

projects, the same guidelines addressed above apply. The inspector(s) shall send a written inspection report and all appropriate documents to the Committee for technical review and approval. The Committee will make the final decision on a laboratory validation status based on all information available including the inspectors' written inspection reports.

h. For a mobile laboratory, the above mentioned guidelines in paragraphs 2-4.a. through 2-4.f. apply. However, the validation status of a mobile laboratory will be terminated when the laboratory is demobilized.

2-5. Inspection/Evaluation Report.

a. If no deficiencies were noted, a laboratory inspection and evaluation report shall be prepared by the inspectors and submitted to the HTRW MCX management for approval within ten working days after the inspection date. If deficiencies were noted and the laboratory provided satisfactory responses, the report shall be submitted within five working days after receipt of the satisfactory responses.

b. The inspection and evaluation report shall contain, but not be limited to, the information listed in Table 2-1. Upon approval by the HTRW MCX management, a cover letter and the inspection report including review comments on LQMM, PE sample evaluation reports, and laboratory's written responses to deficiencies shall be immediately sent to the USACE TM/CORs and the commercial laboratory. The cover letter shall specify the methods, matrices, time period, and limitations for which the validation is granted, and the corrective actions that have to be taken by the laboratory if applicable. A commercial laboratory must rectify all deficiencies prior to the initiation of field studies and sample analyses. During the 18-month period, the Committee reserves the right to send additional PE samples or to conduct additional inspections as necessary. The laboratory validation does not guarantee the award of any contracts from a USACE TM/COR or a prime contractor. For UST removal projects, although the inspections may not be conducted by the Committee representatives, all reports generated by the inspectors shall follow the format given in this manual. All cover letters shall originate from the HTRW MCX.

Table 2-1. Sample Format for Inspection Report

1. General

   a. Date of Inspection.
   b. Name, office symbol, and phone number of inspector.
   c. Contract(s) for which the laboratory will be used.
   d. Description of contract.
   e. General information of the laboratory (Business name, street address, phone, how long in business, number employed, type of services offered, and other pertinent information.)

2. Summary of Inspection Results

   a. Overall comments on the laboratory's technical capabilities in meeting the project requirements.
   b. The validation status and expiration date of the laboratory.
   c. Major deficiencies or concerns to be corrected or be aware of for USACE HTRW projects.

3. Interviews

   a. Entrance

      - Introduction to the USACE QA program.
      - Overview of USACE HTRW laboratory validation procedures.
      - Discussion of the upcoming USACE project(s).
      - Presentation and discussion of the USACE comments on the laboratory's LQMM.
      - Overview and discussion of PE sample results.
      - Discussion of the laboratory's past performance on USACE HTRW projects, if applicable.

   b. Exit

      - Discussion of deficiencies to be corrected.
      - Recommendations based on good laboratory practice.
      - Action items for the laboratory's response.

Table 2-1. Sample Format for Inspection Report (continued)

4. General On-Site QA Evaluation

   a. Adequacy of organizational structure to maintain its stated capabilities in operation and management.
   b. Adequacy and maintenance of facilities and equipment.
   c. Quality, age, availability, scheduled maintenance, and performance of instrumentation.
   d. Staff qualifications, experience, and training programs.
   e. Availability, appropriateness, and utilization of SOPs .
   f. Reagents, standards, and sample storage facility.
   g. Bench sheets and analytical logbooks maintenance and review.
   h. Data package and data management.
   i. Availability and use of control charts.
   j. Waste disposal compliance.

5. Conclusions

   a. Deficiencies that must be corrected by the laboratory prior to approval for validation.
   b. Recommendations for laboratory's consideration.
   c. Other findings of interesting or important nature.
   d. Concerns from the laboratory on USACE HTRW projects.
   e. Laboratory's responses to deficiencies and recommendations, if available.
   f. Action items for the laboratory’s response.

Section II. Revalidation, Termination, and Appeal

2-6. Revalidation.

a. A commercial laboratory whose 18-month validation status has expired will be considered for revalidation upon receipt of a written request from a TM/COR based on the analytical requirements in the upcoming contract(s). The Committee will determine which of the three steps are required for the revalidation process based on the laboratory's previous performance. Normally, Steps 1 (review of qualifications) and 2 (analysis of PE samples) are always required. Step 3 (inspection of laboratory) could be waived if the following criteria are met.

(1) The laboratory's performance on PE samples has been satisfactory,

(2) the laboratory has had no performance problems with previous USACE HTRW projects,

(3) the laboratory has not moved to a new location or had major facility changes at the current location since the last USACE HTRW inspection, and

(4) the laboratory has been inspected by the Committee during a validation/revalidation process within the last three years.

b. During the 18-month validation period, a commercial laboratory shall inform the Coordinator immediately of any major changes in its personnel, equipment, or facilities that could impact the laboratory's performance on any USACE projects. Depending on the scope of changes, a revalidation may be required. The Committee will determine which of the three steps would be needed for the revalidation. The validation status of a commercial laboratory that fails to inform the Coordinator of any major changes may be suspended.

c. A revalidation may also be required when a fully validated laboratory obtains another contract(s) within its 18-month validation period. Based on the contract requirements, the laboratory's validation status, and its previous performance on USACE projects, the Committee will determine which of the three steps are required for a revalidation. Ordinarily, if different analytes or matrices are involved in another contract(s), analysis of additional PE samples is required. If its previous performance has been satisfactory and/or additional PE sample results are acceptable, no further actions are required.

d. A revalidation is also required for laboratories working on ongoing projects that will extend more than six months beyond the validation expiration date of the laboratory. The Coordinator will alert the affected USACE TM/CORs of the pending expiration three months prior to expiration date. A revalidation process should not interfere with the ongoing project unless performance problems are noted during the revalidation process.

2-7. Termination.

a. As a means of measuring a commercial laboratory's performance after validation, the Committee may send additional PE samples on a quarterly or as-needed basis. This depends on the laboratory's past performance on PE sample and/or field sample analyses and on whether the laboratory is currently working on an ongoing USACE HTRW project. The quarterly PE samples could be either single blind or double blind sent by the Committee directly or through a prime contractor. As a minimum, the results are evaluated for compound identification, quantitation, and sample contamination. Results from the analysis of the PE samples will be used by the Committee to verify the laboratory's continuing ability to produce acceptable analytical data. A commercial laboratory's results on these quarterly PE samples will determine its performance as follows:

(1) Acceptable, No Response Required:

Data meets most or all of evaluation criteria as previously described. No response is required.

(2) Acceptable, Response Explaining Deficiencies Required:

Deficiencies exist in the laboratory's performance. Within five working days of receipt of notification from the Coordinator, the laboratory shall send written response to describe the deficiencies and the action(s) taken to correct the deficiencies to the Coordinator.

(3) Unacceptable Performance, Response Explaining Deficiencies Required:

Deficiencies exist in the laboratory's performance to the extent that the Committee has determined that the commercial laboratory has lost its capability to meet the USACE project requirements. Within five days of receipt of notification from the Coordinator, the laboratory shall describe the deficiencies and the

action(s) taken to correct the deficiencies in a letter to the Coordinator.

b. Remedial PE samples may be sent for the failed parameters. It is the sole decision of the Committee to approve or disapprove the quarterly PE sample results and to send remedial PE samples. If a commercial laboratory fails to pass quarterly PE samples, the laboratory may expect, but the Committee is not limited to the following actions: suspension of the laboratory validation status, an additional on-site laboratory inspection, data package audit, a remedial PE sample, and/or contract sanctions.

c. During the 18-month validation period, the performance of the laboratory will be monitored by the USACE TM/CORs, the USACE division laboratories, and the Committee through review of appropriate CQARs prepared by the USACE division laboratories that serve as the project QA laboratories. If a commercial laboratory has performance problems with field sample analysis or data reporting, the USACE TM/CORs and the USACE Division Laboratories should contact the Coordinator immediately to work out necessary corrective and remedial actions. Figure 2-4 can be used to report performance problems with commercial laboratories. Depending on the scope of problems, a commercial laboratory's validation status may be suspended such that the laboratory will not be allowed to analyze any more project samples until the corrective actions are accepted by the Committee and the problems are corrected.

d. While a commercial laboratory is in the process of performing corrective actions, another validated laboratory shall be used until the problems are solved. Should a commercial laboratory fail to solve the problems satisfactorily in a timely manner, the validation status of the laboratory may be revoked for default. The validation status of any laboratory suspended or debarred by other government regulatory agencies may also be terminated by the Committee.

2-8. Appeal. The Coordinator shall advise a commercial laboratory of its right to appeal adverse validation decisions including suspension or termination of validation status. If a commercial laboratory decides to appeal, it should submit a written appeal to the Coordinator within 20 working days from receipt of the laboratory validation report. The Committee will review its decision and send a written response to the laboratory within 20 working days. All review decisions by the Committee are final.

TO: CEMRD-ED-EC FROM :_____ DATE:___/___/____

SUBJECT: PERFORMANCE PROBLEMS WITH COMMERCIAL LABORATORY

Project Name:_____
Location:_____ State:_____
Contract No:_____ TYPE: POL TANK REMOVAL:___HTRW:__

Program: SF:____FUDS:____IRP:____ AF(ACC):____ OTHER:_____
Phase: PA/SI:____ RI/FS:____RD:____ RA:____ RFA:____ RFI:____ CMS:____

USACE Technical Manager:_____
Phone:_____ Address:_____
Government QA Lab:_____ POC:_____
Phone:_____ Address:_____

A-E/Contractor:_____ State:_____
Lab Name:_____
Address:_____
Phone:_____
POC:_____

PROBLEMS ENCOUNTERED:

Figure 2-4 Laboratory Performance Problem Report

PERFORMANCE PROBLEMS WITH COMMERCIAL LABORATORY:

CORRECTIVE ACTIONS TAKEN:

TECHNICAL ASSISTANCE NEEDED FROM THE HTRW MCX:

Figure 2-4 Laboratory Performance Problem Report (continued)

Section III. Information Management

2-9. Record Files:

a. Centralized validation records for commercial laboratories are kept at the HTRW MCX. The record files include:

(1) Original laboratory evaluation requests (Figure 2-l),

(2) laboratory qualification documents including LQMM and preliminary questionnaire,

(3) PE sample evaluation reports,

(4) laboratory's responses to PE sample reports,

(5) inspection report and cover letters,

(6) laboratory inspection checklist,

(7) on-site laboratory inspection summary (Figure 2-2),

(8) laboratory's responses to inspection report,

(9) the Committee's validation review meeting summary (Figure 2-3),

(10) laboratory performance problem reports (Figure 2-4), and

(11) miscellaneous documents (e.g., raw data, chromatograms, correspondences, etc.) that the inspectors deems important for the current and/or future laboratory validation.

b. When there is a potential of litigation against the USACE on a particular laboratory validation; all documents pertaining to the particular laboratory validation shall be retained in the file until a final settlement or a revalidation request is received.

2-10. Database. A laboratory validation database is maintained at the MCX for program management. An example of a laboratory record in the database is shown in Appendix K. Updated lists of validated commercial laboratories in alphabetical order by laboratory name and state will be distributed to each Engineering, Construction, and Contracting office within the USACE on a monthly basis. Customized reports are also available

if requested by USACE TM/CORs. These reports are for government use only and will not be distributed to private sector.

CHAPTER 3

PROCEDURES FOR USACE DIVISION LABORATORIES

3-1. USACE Division Laboratories. Minor modifications to the validation procedures as described in Chapters 1 and 2 will be made for validation/revalidation of USACE division laboratories to ensure that all USACE division laboratories are able to provide analytical data of the highest accuracy and precision for validated parameters. The modifications include:

a. The Committee shall invite the HQUSACE to send a representative(s) to participate in the inspection of each USACE division laboratory and shall keep the HQUSACE informed of the current validation status of each USACE division laboratory on a regular basis. A copy of the inspection report for each USACE division laboratory shall be sent to the HQUSACE. The HTRW MCX will recommend the corrective actions for each USACE division laboratory. The HQUSACE will coordinate with the HTRW MCX to ensure that corrective actions for each USACE division laboratory are planned and implemented appropriately.

b. All USACE division laboratories will be revalidated every 18-months. The revalidation shall always include a two–day on-site inspection. During the 18-month period, at least one additional announced and/or unannounced site visit will be performed by the Committee whenever needed.

c. During the 18-month validation period, additional PE samples shall be provided to USACE division laboratories on a periodic basis to monitor the laboratory’s continuing ability to provide superior analytical performance. Results from these PE samples would be used primarily for the division laboratories to check and improve their method specific performance.

d. All division laboratories' technical SOPS that would affect data quality shall be received, reviewed, and approved by the Committee prior to implementation.

e. Appendix L, Supplemental Questionnaire for USACE Division Laboratories, shall be included as an appendix to the preliminary questionnaire and sent to USACE division laboratories for completion.

f. Upon request by USACE division laboratories, the Committee will provide technical support to assist USACE division laboratories to obtain a full validation on a continual basis.

g. All expenses for validation of USACE division laboratories will be covered by USACE HTRW program management funds.

3-2. Contract QA Laboratories: Designated commercial laboratories with contracts to provide technical support to the QA function of USACE division laboratories shall also be evaluated under higher standards and more stringent criteria. However, Appendix L is not needed for commercial laboratories.

APPENDIX A

REFERENCE

DEPARTMENT OF THE ARMY
U.S. Army Corps of Engineers
WASHINGTON. D.C. 20314-1000

REPLY TO
ATTENTION OF:

CEMP-RT (200-1a) 14 SEP 1993

MEMORANDUM FOR SEE DISTRIBUTION

Subject: Hazardous, Toxic & Radioactive Waste (HTRW) - Policy Guidance on Validation of Commercial Analytical Chemistry Laboratories

1. Reference ER 1110-1-263, Chemical Data Quality Management for Hazardous Waste Remedial Activities, 1 Oct 90.

2. The referenced ER requires that all commercial analytical chemistry laboratories which analyze samples in support of all HTRW projects be validated by CEMRD-ED-EC for project specific parameters prior to the analysis of those samples. This memo supplements the reference by providing policy guidance on:

a. The use of lists of validated laboratories (paragraph 3); and

b. Alternative validation procedures for laboratories supporting underground storage tank (UST) projects (paragraph 4).

3. A list of validated laboratories, which now includes about 160 laboratories, is distributed monthly by the HTRW MCX (CEMRD-ED-EC) to all Divisions and Districts. The following information, regarding laboratory validations and the use of these lists, must be provided to potential contractors in CBD announcements or subsequent solicitation packages:

Laboratories must be validated for project specific parameters prior to analyzing any samples under contract as part of USACE HTRW Program execution. Laboratories must be revalidated every eighteen months if they are actively supporting USACE projects.

b. Initial laboratory validations require eight to twelve weeks, depending on the responsiveness of the laboratory. Revalidation usually requires less time.

The list of currently validated laboratories and/or information (encl 2) on the validation process can be obtained from the contracting office of the procuring district.

d. Potential contractors may use laboratories on the list or propose to use laboratories not on the list. Proposed laboratories not on the list will be validated.

CEMP-RT (200-la)
SUBJECT: Hazardous, Toxic & Radioactive Waste (HTRW) - Policy Guidance on Validation of Commercial Analytical Chemistry Laboratories

A "Request for Evaluation of Commercial Laboratory" form (encl 1) must be submitted to the HTRW MCX (CEMRD-ED-EC) by a contracting Officer Representative for all projects/contracts, regardless of whether the laboratory is on the list. The form must be submitted as soon as a contract has been awarded to the prime contractor and may be faxed to Ms. Paulette Lewis at 402 221-7403.

4. For projects consisting of the removal of Underground storage tanks (UST) Which have contained only petroleum, oils, or lubricants, there are two alternatives to the validation process described in ER 1110-1-263. The following alternatives apply only to predesign sampling of UST organic phase contents and soil sampling during removal. They do not apply to investigations required by groundwater contamination or extensive soil contamination.

a. State certified laboratories may be used without USACE validation, if the state considers its certification to be applicable to UST removal. When this alternative is selected, a document in the project file must identify the individual responsible for coordination with the state.

b. The HTRW MCX will follow an abbreviated laboratory validation process if requested on the "Request for Evaluation of Commercial Laboratory" form (encl 1) and the analyses identified are applicable to UST removal. The laboratory's standard operating procedure for the required analyses must be submitted to the HTRW MCX for review. Based on the SOPs and other available information, the HTRW MCX will determine the remainder of the validation process. The HTRW MCX may delegate certain validation responsibilities to Division Chemistry Laboratories by mutual consent. Note that the SOPs are not copies of printed standard methods, but rather step-by-step procedures which are followed by the laboratories for sample analysis, quality control, and data reporting. Laboratories validated by this abbreviated procedure may be used only for the UST projects defined above.

5. The CEMP-RT POC is Dr. **Bruce Heitke, 202-272-8882.**

2 Encls

CARY JONES
Chief, Environmental Restoration
Division
Directorate of Military programs

CEMP-RT (200-la)
SUBJECT: Hazardous, Toxic & Radioactive Waste (HTRW) - Policy Guidance on Validation of Commercial Analytical Chemistry Laboratories

COMMANDER:
HUNTSVILLE DIVISION, ATTN: CEHND-CT
HUNTSVILLE DIVISION, ATTN: CEHND-ED-CS
HUNTSVILLE DIVISION, ATTN: CEHND-TD
HUNTSVILLE DIVISION, ATTN: CEHND-TD-TO
LOWER MISSISSIPPI VALLEY DIVISION, ATTN: CELMV-ED-W
LOWER MISSISSIPPI VALLEY DIVISION, ATTN: CELMV-CO-C
LOWER MISSISSIPPI VALLEY DIVISION, ATTN: CELMV-CT
MISSOURI RIVER DIVISION, ATTN: CEMRD-ED
MISSOURI RIVER DIVISION, ATTN: CEMRD-ED-L
MISSOURI RIVER DIVISION, ATTN: CEMRD-CT
NEW ENGLAND DIVISION, ATTN: CENED-CT
NEW ENGLAND DIVISION, ATTN: CENED-ED
NEW ENGLAND DIVISION, ATTN: CENED-ED-GL
NEW ENGLAND DIVISION, ATTN: CENED-PD-L
NORTH ATLANTIC DIVISION, ATTN: CENAD-CT
NORTH ATLANTIC DIVISION, ATTN: CENAD-EN-TS
NORTH ATLANTIC DIVISION, ATTN: CENAD-CO-CE
NORTH ATLANTIC DIVISION, ATTN: CENAD-PP-PM
NORTH CENTRAL DIVISION, ATTN: CENCD-CT
NORTH CENTRAL DIVISION, ATTN: CENCD-ED
NORTH CENTRAL DIVISION, ATTN: CENCD-CO-C
NORTH CENTRAL DIVISION, ATTL: CENCD-TE-ED-W
NORTH PACIFIC DIVISION, ATTN: CENPD-CT
NORTH PACIFIC DIVISION, ATTN: CENPD-PE-GT
NORTH PACIFIC DIVISION, ATTN: CENPD-PE-GT-L
NORTH PACIFIC DIVISION, ATTN: CENPD-PM-MP
OHIO RIVER DIVISION, ATTN: CEORD-CT
OHIO RIVER DIVISION, ATTN: CEORD-ED
OHIO RIVER DIVISION, ATTN: CEORD-PE-GL
OHIO RIVER DIVISION, ATTN: CEORD-DL-MS
PACIFIC OCEAN DIVISION, ATTN: CEPOD-CT
PACIFIC OCEAN DIVISION, ATTN: CEPOD-ED-ME
SOUTH ATLANTIC DIVISION, ATTN: CESAD-CT
SOUTH ATLANTIC DIVISION, ATTN: CESAD-EN
SOUTH ATLANTIC DIVISION, ATTN: CESAD-EN-FL
SOUTH ATLANTIC DIVISION, ATTN: CESAD-PM-H
SOUTH PACIFIC DIVISION, ATTN: CESPD-CT
SOUTH PACIFIC DIVISION, ATTN: CESPD-ED
SOUTH PACIFIC DIVISION, ATTN: CESPD-ED-GL
SOUTH PACIFIC DIVISION, ATTN: CESPD-CO-CM
SOUTHWESTERN DIVISION, ATTN: CESWD-CT
SOUTHWESTERN DIVISION, ATTN: CESWD-ED
SOUTHWESTERN DIVISION, ATTN: CESWD-ED-GL
SOUTHWESTERN DIVISION, ATTN: CESWD-PP-M
SOUTHWESTERN DIVISION, ATTN: CESWD-PP-MM

CEMP-RT (200-la)
SUBJECT: Hazardous, Toxic & Radioactive Waste (HTRW) - Policy Guidance on Validation of Commercial Analytical Chemistry Laboratories

CF:
CENPA-CT
CENPA-EN-G-M
CESWA-CT
CESWA-ED
CENAB-CT
CENAB-EN-HT
CENCB-CT
CENCB-PE-HQ
CESAC-CT
CESAC-EN-DF
CENCC-ED
CENCE-CT
CENCE-ED-D
CESWF-CT
CESWF-ED
CESWG-CT
CESWG-ED-DC
CEORH-CT
CEORH-ED-AE
CESAJ-CT
CESAJ-EN
CESAJ-CO-CQ
CEMRK-CT
CEMRK-ED
CESWL-CT
CESWL-ED-GH
CESPL-CT
CESPL-ED-GG
CEORL-CT
CEORL-ED-G
CEORL-CD-FB
CELMM-CT
CELMM-ED-HW
CENAN-CL-ME-E
CESAM-CT
CESAM-EN
CESAM-PD-ES
CEORN-CT
CEORN-ED
CELXN-CD
CELMN-CD-QM
CELMN-CT
CELMN-ED-EE
CENAN-CT

CEMP-RT (200-la)
SUBJECT: Hazardous, Toxic & Radioactive Waste (HTRW) - Policy Guidance on Validation of Commercial Analytical Chemistry Laboratories

CENAN-EN
CENAO-CT
CENAO-EN-MP
CEMRO-CT
CEMRO-ED-EG
CENAP-CT
CENAP-EN-C
CEORP-CT
CEORP-ED
CENPP-CT
CENPP-ED-DC
CENCR-CT
CENCR-ED-DG
CESPK-CT
CESPK-ED-M
CESPK-ED-G
CELMS-CT
CELMS-ED-HQ
CENCS-CT
CENCS-EN
CESPN-CT
CESPN-EN
CESAS-CT
CESAS-EN
CENPS-CT
CENPS-ED
CENPS-EN-GT-HW
CESWT-CT
CESWT-ED
CESWT-OW-AR
CELMK-CD
CELMK-CT
CELMX-ED-DR
CEIMK-CD-QM
CELMK-OD-M
CEWES-EE-A
CENPW-CT
CENPW-EN-EE
CESAW-CT
CESAW-EN

APPENDIX B

INTRODUCTION
TO
THE U.S. ARMY CORPS OF ENGINEERS
VALIDATION PROGRAM
FOR
COMMERCIAL ANALYTICAL CHEMISTRY LABORATORIES

(SAMPLE LETTER)

Dear Laboratory Director:

Thank you for your interest in the U.S. Army Corps of Engineers (USACE) Hazardous, Toxic, and Radioactive Waste (HTRW) laboratory validation program. I hope that the information enclosed will be helpful to you and answer any questions you may have.

If you have any further questions regarding this information or the USACE HTRW laboratory validation program in general, please contact the USACE Laboratory Validation Coordinator at (402) 221-7494.

Sincerely,

Chief, Environmental, HTRW Division
HTRW and Engineering Directorate

Enclosure

INTRODUCTION TO THE U.S. ARMY CORPS OF ENGINEERS VALIDATION PROGRAM FOR COMMERCIAL ANALYTICAL CHEMISTRY LABORATORIES

WHO NEEDS VALIDATION?

According to USACE Engineer Regulation 1110-1-263, CHEMICAL DATA QUALITY MANAGEMENT FOR HAZARDOUS WASTE REMEDIAL ACTIVITIES:

> Laboratory validation shall apply to all commercial laboratories directly or indirectly providing chemical analysis support to USACE HTRW investigative and remedial activities.

All commercial laboratories that support USACE HTRW response activities must obtain a USACE laboratory validation prior to field studies or sample analyses and must maintain the validated status throughout the contract/project/task order(s) (hereafter referred to as the contract or project) for the HTRW response activities.

HOW TO APPLY FOR A VALIDATION

After a prime architect-engineering firm or a construction contractor (hereafter referred to as the prime contractor) is awarded with a contract to support USACE HTRW remedial activities, the prime contractor will select a subcontract commercial laboratory for this contract and notify the USACE Technical Manager or Contracting Officer Representative (TM/COR) of its selection. The USACE TM/COR will then submit a written request for evaluation of the subcontract commercial laboratory to the USACE Laboratory Validation Committee (hereafter referred to as the Committee) to initiate the laboratory validation process. After receipt of the request, the Committee will contact the laboratory shortly. A commercial laboratory, itself, does not apply for a USACE HTRW laboratory validation. The Committee will only respond to a validation request from a USACE TM/COR.

WHAT IS THE VALIDATION PROCESS?

The laboratory validation process consists of three major sequential steps: (1) the Committee reviews the laboratory's qualification documents, (2) the laboratory analyzes a set of performance evaluation (PE) samples, and (3) the Committee conducts an on-site laboratory inspection. The Committee is

responsible for execution and management of the laboratory validation program.

WHAT KIND OF QUALIFICATION DOCUMENTS?

Typical qualification documents may be in the form of an off-the-shelf laboratory quality management manual (LQMM) or in some other format which includes a laboratory floor plan, organization chart, instrumentation list, staff resumes, certificates, in-house standard operating procedures, etc. The documents should provide proper information for the Committee to assess the laboratory's technical capabilities. Upon request, a laboratory should promptly submit its qualification statements to the Committee for review. If it appears that the laboratory has the adequate capabilities to meet project requirements, the Committee will initiate the next step, PE sample analysis.

WHAT KIND OF PE SAMPLES?

The Committee will provide the laboratory with project-specific PE samples for performance evaluation. The PE samples may be in water and/or soil/sediment matrices. Arrangements will be made with the laboratory for analysis and reporting of these samples. The results are considered passing if the results of a particular method are within statistically established acceptance limits as determined by the USACE and no procedural problems are found during a follow-up laboratory inspection. A laboratory may volunteer for additional non-project-specific PE samples at its own cost. A laboratory must pass more than 50 percent of all PE samples within 40 working days from receipt of the PE samples or the validation process will be terminated.

WHAT ARE INSPECTION PROCEDURES?

Two Committee representatives will inspect the laboratory only after Steps 1 and 2 have been satisfactorily completed. The on-site inspection which generally takes eight hours includes: (1) an entrance interview with the laboratory management to discuss USACE QA program, review comments on laboratory qualification submittals including LQMM, PE sample results, upcoming projects, etc., (2) a follow-up laboratory tour to examine laboratory facility, instrumentation, operation, maintenance, documentation, etc., and (3) an exit interview to summarize any deficiencies found and corrective actions required. A laboratory must rectify any deficiencies noted during the inspection prior to an approval for a full validation status.

After inspection, the Committee will meet to review and determine the validation status of a laboratory.

WHAT ARE VALIDATION CRITERIA?

The USACE basically follows Federal and/or State laws, regulations, and guidelines and good laboratory practices to evaluate laboratory performance. The validation status of a laboratory depends on whether the laboratory's PE sample results are within USACE established acceptance criteria and no procedural problems are found during a follow-up laboratory inspection. The laboratory's PE sample results will be compared in the following manner: (1) with the prepared concentrations of PE samples that are used as the absolute recovery comparators, and (2) with the statistical mean and standard deviations reported by a group of peer laboratories. The acceptable limits for analyte quantitation will be established statistically at 95 percent confidence based on referee laboratories' and/or peer group results.

HOW MUCH TIME DOES VALIDATION TAKE?

The entire process of laboratory validation generally takes up to 12 weeks depending on a laboratory's performance and responsiveness. The prime contractors should plan the project schedule to allow adequate time for laboratory validation process.

WILL A CERTIFICATE BE ISSUED?

USACE will not issue a certificate for validated laboratories. However, a letter and a copy of inspection report will be sent to each validated laboratory. The letter will specify the methods and matrices, the project(s), and the time period (usually 18 months) for which the validation is granted.

IS THE VALIDATION UNIVERSAL?

The validation is a parameter, method, and matrix-specific approval and only applies for USACE HTRW program. However, for each new contract awarded during the 18-month validation period, a project-specific evaluation is still required. The Committee will check the laboratory's validation status and previous performance to determine if any additional actions are needed. If different parameters, methods, and/or matrices are involved,

only those PE samples will be sent. If work done for the USACE by the laboratory has been satisfactory, no further actions will be necessary.

HOW ABOUT SUBCONTRACTING?

A validated laboratory may not subcontract any USACE samples to a second laboratory without the knowledge and approval of the USACE TM/COR and the concurrence of the Committee. The second laboratory must also be validated for methods, parameters, and matrices corresponding to the subcontract. Subcontract of PE sample analysis is totally prohibited.

WHAT ARE THE FEES REQUIRED FOR VALIDATION?

There are no direct fees for the laboratory besides the cost for additional PE samples required for failed parameters or non-project-specific parameters. The current cost for any additional or any non-project-specific PE samples ranges from $100 to $300 per method, per matrix, and per shipment. The cost shall be reviewed annually and adjusted as necessary without notice to reflect currency value fluctuations or changes in program administration costs. The USACE will not pay the costs for analysis of PE samples and preparation of any qualification documents.

WHERE TO GET MORE INFORMATION

The Laboratory Validation Committee at the USACE HTRW Mandatory Center of Expertise (MCX) of the USACE is responsible for all aspects of the USACE HTRW laboratory validation program. The Committee meets as needed to propose policy on USACE HTRW laboratory validation program and to make ultimate decisions on laboratory-specific validation status. Any questions concerning the validation program can be directed to the Laboratory Validation Coordinator.

U.S. Army Corps of Engineers
HTRW Mandatory Center of Expertise
ATTN: CEMRD-ED-EC (Laboratory Validation Coordinator)
12565 West Center Road
Omaha, NE 68144-3869

Voice: (402) 221-7494
FAX : (402) 221-7403

APPENDIX C

INFORMATION

FOR

COMMERCIAL ANALYTICAL CHEMISTRY LABORATORIES

UNDERGOING VALIDATION

BY

THE U.S. ARMY CORPS OF ENGINEERS

(SAMPLE LETTER)

Dear Laboratory Director:

Your laboratory has been submitted as a candidate for validation/revalidation in support of the U.S. Army Corps of Engineers (USACE) hazardous, toxic, and radioactive waste (HTRW) response activities. Prior to the field studies or sample analyses, your laboratory must be validated by the USACE. Enclosed for your information and action are:

(1) Information about the USACE Project(s) that leads to this validation/revalidation process,

(2) Information for Commercial Analytical Chemistry Laboratories Undergoing Validation by the USACE,

(3) Guidelines for Analyzing and Reporting Performance Evaluation Samples (Appendix D), and

(4) Preliminary Questionnaire (Appendix E).

If you decide to obtain a USACE HTRW laboratory validation, please be sure that:

(1) All instructions, including all time deadlines, are read and followed carefully.

(2) The preliminary questionnaire is completed and returned with original verification signature(s) within ten working days from receiving date.

I hope that the information provided in this packet will be helpful to you and answer any questions you may have. If you have any questions regarding this information or the USACE HTRW laboratory validation program in general, please contact the Laboratory Validation Coordinator at (402) 221-7494.

Sincerely,

_____
Chief, Environmental, HTRW Division
HTRW and Engineering Directorate

4 Enclosures

TO: Laboratory Director/Manager

FROM: USACE HTRW MCX

DATE: 01/21/92

SUBJECT: USACE HTRW Projects and Laboratory Validation

Listed below is some basic information about the USACE HTRW project(s) that your laboratory will provide analytical chemistry services. For the details, please contact the primary contractor and/or refer to the approved final Work Plan.

Laboratory Name: ABC Analytical Laboratory State: MD

1. Project Name: Elmwood County Landfill State: NJ
Contract No: DACWO1-91-C-2345
Sampling Date: 09/01/91 (approximate)

AE/Contractor: DEF, Inc. State: PA

USACE TM: John Dow
Phone No: (222) 333-4444

HTRW Analyses: VOA, BNA, PCB, PEST, TAL METALS, TRPH, CN.

2. Project Name: Any AFB; Fire Fighting Training 2A State: AZ
Contract No: DACAO1-91-B-1234
Sampling Date: 04/15/92 (approximate)

AE/Contractor: Any Environmental Services, Inc. State: CA

USACE TM: Paula Smith
Phone No: (333) 444-5555

HTRW Analyses: RCRA METALS, TRPH, AVO, TPH (Mod. 8015) .

Remarks: The HTRW analyses may involve samples of various matrices.

Figure C-1 Sample Laboratory Evaluation Request

INFORMATION FOR COMMERCIAL ANALYTICAL CHEMISTRY LABORATORIES UNDERGOING VALIDATION BY THE U.S. ARMY CORPS OF ENGINEERS

Please retain this information and a copy of your completed preliminary questionnaire in your file for future reference.

WHO NEEDS VALIDATION?

According to USACE Engineer Regulation 1110-1-263, CHEMICAL DATA QUALITY MANAGEMENT FOR HAZARDOUS WASTE REMEDIAL ACTIVITIES:

> Laboratory validation shall apply to all commercial laboratories directly or indirectly providing chemical analyses to support USACE HTRW investigative and remedial activities.

All commercial laboratories that support USACE HTRW response activities must obtain a USACE laboratory validation prior to field studies or sample analyses and must maintain the validated status throughout the contract/project/task order(s) (hereafter referred to as the contract or project) for the HTRW response activities.

WHAT IS THE VALIDATION PROCESS?

The laboratory validation process consists of three major sequential steps: (1) review of the laboratory's qualification documents, (2) analysis of performance evaluation (PE) samples, and (3) on-site inspection of laboratory's facility, instrumentation, operation, and management.

(1) Step 1. Upon request, a commercial laboratory should submit its qualification documents within five working days to the USACE Laboratory Validation Committee (hereafter referred to as the Committee) for review. This submittal may be in the form of an off-the-shelf quality assurance manual or in some other format that provides proper laboratory-specific information for the Committee to assess the laborator's technical capabilities. The information includes, but is not limited to, laboratory floor plan, organization chart, list of major instrumentation, copy of staff resumes, laboratory certificates, standard operating procedures for nonstandard/modified standard chemical testing, quality assurance/quality control (QA/QC) policy and practice, etc. If it appears that a laboratory has the adequate capabilities to meet project requirements, the Committee will initiate Step 2.

(2) Step 2. The Committee will provide a laboratory with project-specific PE samples for performance evaluation. A laboratory may volunteer for additional non-project-specific PE samples at its own cost. Arrangements will be made with the laboratory for the analysis and reporting of these samples. Enclosure 3 is a general guidance for PE sample analysis and reporting. Sample-specific instructions will be sent along with the PE samples and should be followed wherever applicable. Failure to analyze these samples correctly or within the required time frame may result in termination of the validation. The results are considered passing if the results of a particular method are within statistically established acceptance limits as determined by the USACE and no procedural problems are found during the Step 3 follow-up laboratory inspection. Normally, only one set of PE samples will be sent to each laboratory. A laboratory must pass more than 50 percent of all PE samples within 40 working days from receipt of the PE samples or the validation process will be terminated. Prior to an on-site inspection, a laboratory shall submit to the Committee a concise written statement describing the problems, solutions, and corrective actions taken or to be taken for the analytical parameters failed in the first attempt.

(3) Step 3. Two Committee representatives will inspect a laboratory only after Steps 1 and 2 have been successfully completed. The on-site inspection which generally takes eight hours involves:

(a) An entrance interview with the upper laboratory management staff (including laboratory director, managers, QA officer, and project personnel) to discuss upcoming USACE project(s), the USACE QA program, the USACE review comments on the laboratory's qualification documents, the PE sample results, and the laboratory's previous performance on USACE projects, if applicable.

(b) A laboratory tour to determine the adequacy of laboratory organization, personnel, facility, and equipment and the implementation of adequate analytical quality and document control, including use of proper analytical methodology, control charts, data and sample handling, documented corrective action measures, chain-of-custody, etc.

(c) An exit interview to discuss any deficiencies noted during the inspection and recommended corrective actions with the laboratory management staff. The corrective actions may include the analysis of a second set of PE samples for failed parameters.

During the exit interview, a laboratory will be requested to submit written responses with supporting documentation to the deficiencies within ten working days from the inspection date. The Committee will evaluate and determine the validation status. A laboratory must rectify any deficiencies noted during the inspection prior to approval for a full validation status.

WHAT ARE VALIDATION CRITERIA?

The USACE basically follows Federal and/or State laws, regulations, and guidelines and good laboratory practices to evaluate laboratory performance. The validation status of a laboratory depends on whether the laboratory's PE sample results are within USACE established acceptance criteria and no procedural problems are found during a follow-up laboratory inspection. The laboratory's PE sample results will be compared in the following manner: (1) with the prepared concentrations of PE samples that are used as the absolute recovery comparators, and (2) with the statistical mean and standard deviations reported by a group of referee and/or peer laboratories. The acceptance limits for analyte quantitation will be established statistically at 95 percent confidence based on peer group results.

WHAT DOES A LAB NEED TO PREPARE FOR THE INSPECTION?

Prior to the USACE on-site inspection, a laboratory should be familiar with all the materials that have been provided by the USACE. Laboratory key personnel including laboratory director/ manager, QA officer, group supervisors, etc., should be residing and available for answering questions during the inspection.

Results of any USACE PE samples should be reviewed prior to the inspection. Special attention should be placed on unacceptable results. Documented corrective actions for unacceptable results should be made available to the USACE inspector(s) during the inspection. Any data or information requested in advance by the USACE inspector(s) should be made readily available.

The preliminary questionnaire should have been filled out and returned within ten working days from receipt or at least one week before the inspection. A map and/or directions for getting to the laboratory should also be submitted along with the preliminary questionnaire.

HOW MUCH TIME DOES VALIDATION TAKE?

The entire process of laboratory validation generally takes up to 12 weeks depending on a laboratory's performance and responsiveness. A simplified flow diagram for the entire validation process is shown in Figure C-2 (Pages C-9 thru C-10).

WILL A CERTIFICATE BE ISSUED?

USACE will not issue a certificate for validated laboratories. However, a letter and a copy of inspection report will be sent to each validated laboratory. The letter will specify the methods and matrices, the project(s), and the time period (usually 18 months) for which the validation is granted.

IS THE VALIDATION UNIVERSAL?

The validation is a parameter, method, and matrix-specific approval and only applies for USACE HTRW program. However, for each new contract awarded during the 18-month validation period, a project-specific evaluation is still required. The Committee will check the laboratory’s validation status and previous performance to determine if any additional actions are needed. If different parameters, methods and/or matrices are involved, only those PE samples will be sent. If work done for the USACE by the laboratory has been satisfactory, no further actions will be necessary.

HOW ABOUT SUBCONTRACTING?

A validated laboratory may not subcontract any USACE samples to a second laboratory without the knowledge and approval of the USACE TM/COR and the concurrence of the Committee. The second laboratory must also be validated for methods, parameters, and matrices corresponding to the subcontract. Subcontract of PE sample analysis is totally prohibited.

WHAT ARE THE FEES REQUIRED FOR VALIDATION?

There are no direct fees for the laboratory besides the cost for additional PE samples required for failed parameters or non-project-specific parameters. The cost for any additional or non-project-specific PE samples range from $100 to $300 per analytical parameter, per matrix, and per shipment. The cost shall be reviewed annually and adjusted as necessary without

notice to reflect currency value fluctuations or changes in program administration costs. The USACE will not pay the cost for analysis of PE samples and preparation of any qualification documents.

HOW TO RENEW VALIDATION

On a monthly basis, the Committee will notify USACE TM/CORs of laboratories with expiring validation (i.e., within three months). If the USACE TM/CORs intend to use those laboratories beyond the expiration dates, the USACE TM/CORs will request revalidations. For a commercial laboratory with an expired validation status, its validation will be renewed when next contract is awarded. After considering use of the laboratory and its previous performance, the Committee will determine which of the three steps will apply to the revalidation process.

WHAT TO DO WITH THE PRELIMINARY QUESTIONNAIRE

The enclosed preliminary questionnaire shall be completed and returned to the Committee within ten working days from the date of receipt. Any supporting documents should be attached if available.

WHERE TO GET MORE INFORMATION

The Laboratory Validation Committee at the HTRW Mandatory Center of Expertise (MCX) of the USACE is responsible for all aspects of the USACE HTRW laboratory validation program. Any questions concerning the validation program can be directed to the Laboratory Validation Coordinator.

U.S. Army Corps of Engineers
HTRW Mandatory Center of Expertise
ATTN: CEMRD-ED-EC (Laboratory Validation Coordinator)
12565 West Center Road
Omaha, NE 68144-3869

Voice: (402) 221-7494
FAX: (402) 221-7403

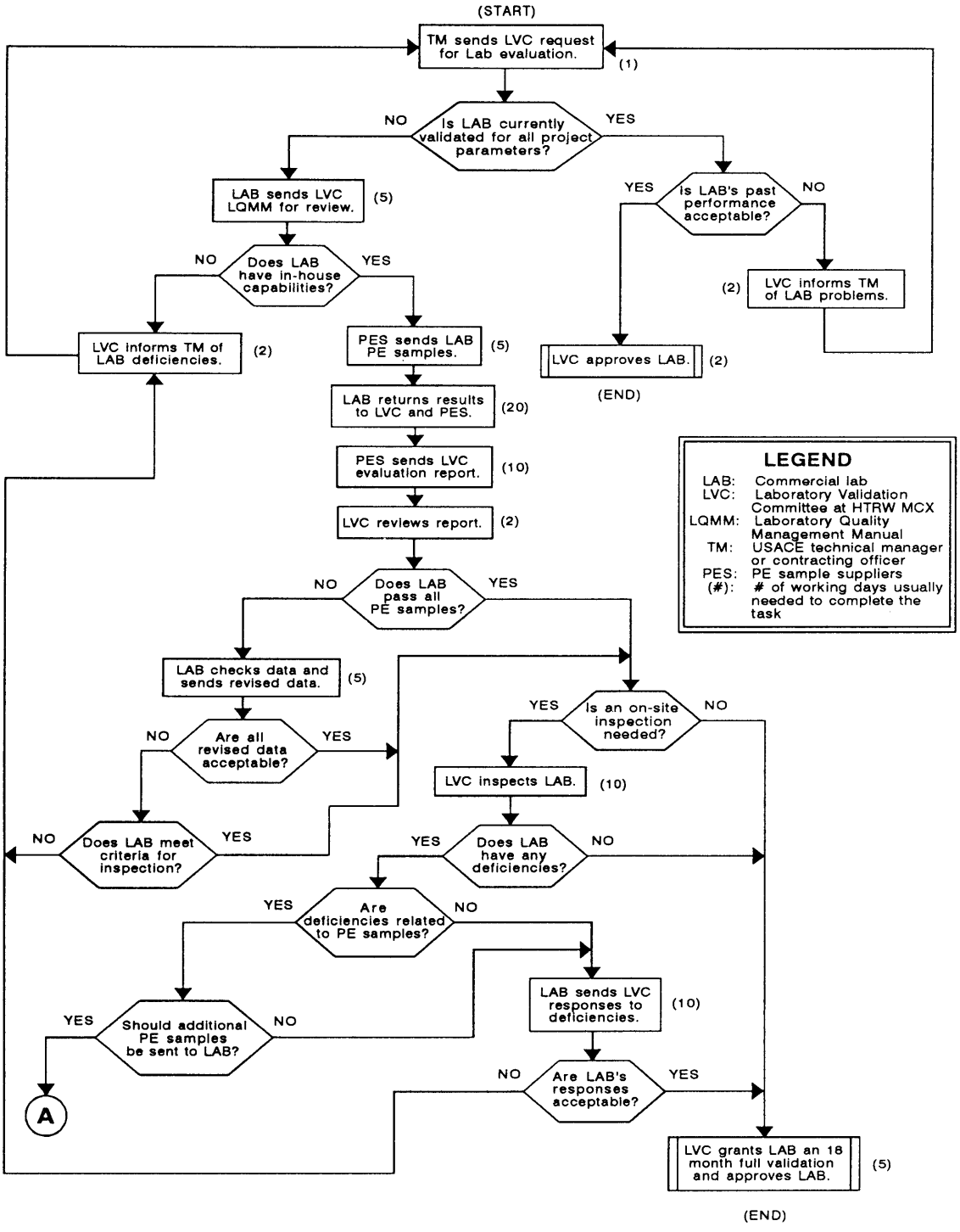


Figure C-2. Flow Diagram of Commercial Laboratory Validation Procedures

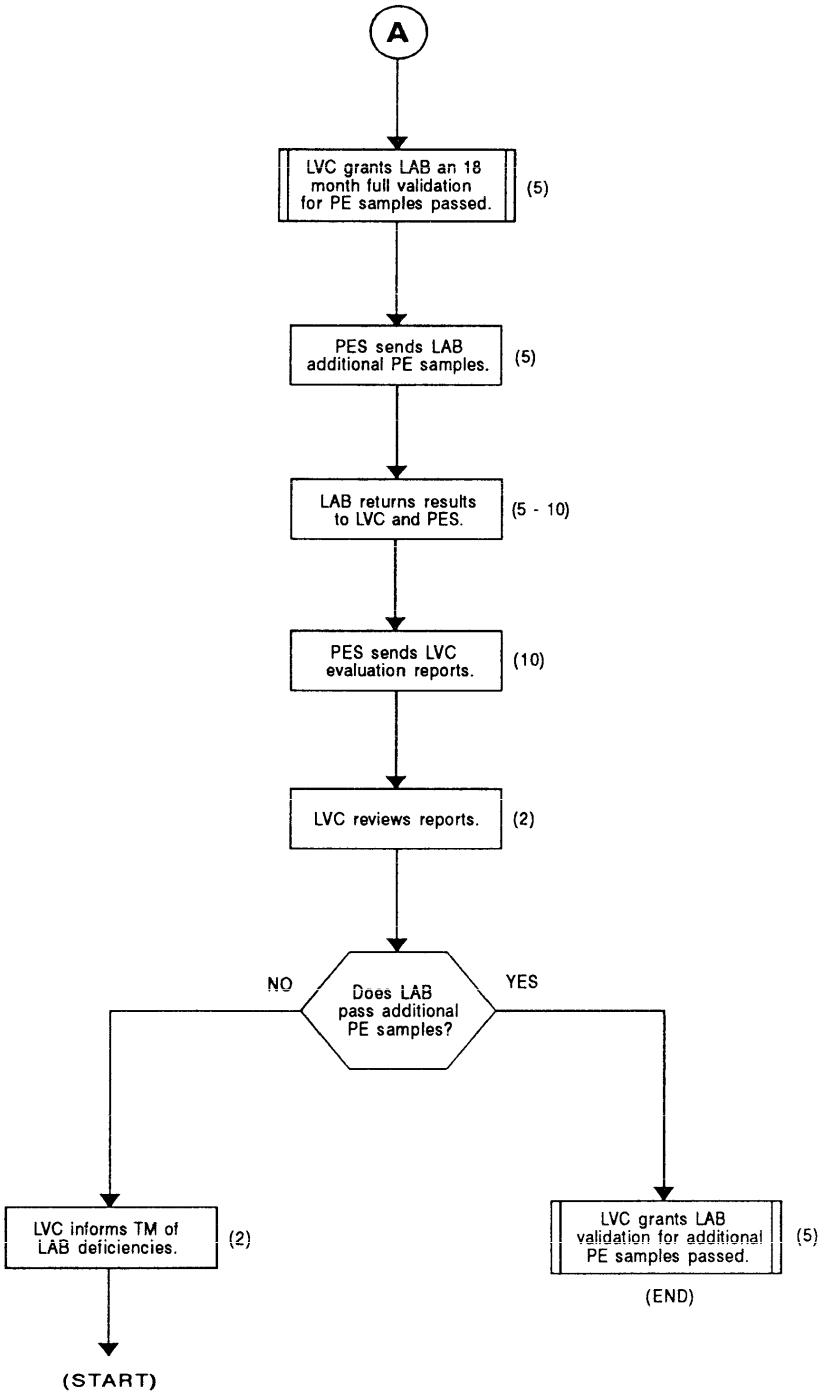


Figure C-2. Flow Diagram of Commercial Laboratory Validation Procedures (continued)

APPENDIX D

GUIDELINES
FOR
ANALYZING AND REPORTING
PERFORMANCE EVALUATION SAMPLES
FROM
THE U.S. ARMY CORPS OF ENGINEERS

GUIDELINES FOR ANALYZING AND REPORTING PERFORMANCE EVALUATION SAMPLES FROM THE U.S. CORPS OF ENGINEERS

Please read and follow these guidelines for analyzing and reporting performance evaluation samples and retain these guidelines in your file for future reference.

GENERAL INFORMATION

The guidelines addressed below are the general requirements for performance evaluation (PE) samples analysis and reporting. Please follow them explicitly. Sample-specific guidelines will be provided with each shipment of PE samples and shall be followed wherever applicable. The sample-specific guidelines supersede these general guidelines.

POINTS OF CONTACT

A Laboratory Validation Committee (hereafter referred to as the Committee) at the HTRW Mandatory Center of Expertise (MCX) of the U.S. Army Corps of Engineers (USACE) is responsible for all aspect of the USACE HTRW laboratory validation program. A Laboratory Validation Coordinator (hereafter referred to as the Coordinator) is the point of contact of the Committee. Any questions concerning the USACE HTRW laboratory validation program should be directed to the Coordinator at the following mailing address and phone number:

U.S. Army Corps of Engineers
HTRW Mandatory Center of Expertise
ATTN: CEMRD-ED-EC (Laboratory Validation Coordinator)
12565 West Center Road
Omaha, NE 68144-3869

Voice: (402) 221-7494
FAX: (402) 221-7403

PE SAMPLES

Most PE samples will be sent out from the analytical laboratory of USACE Waterways Experiment Station (WES) in Vicksburg, Mississippi, except for petroleum hydrocarbons, oil and grease, and explosives which will be sent out from the USACE Missouri River Division Laboratory (MRDL) in Omaha, Nebraska. PE samples are method- and matrix-specific. A laboratory has to

pass all PE samples of different matrices available from the USACE to be considered for multimedia approval.

ANALYTICAL METHODS

A commercial laboratory shall use contract-required analytical methods for all PE sample analyses unless otherwise instructed by the Coordinator. The contract-required analytical methods are usually specified in a project-specific Scope of Services or Chemical Data Acquisition Plan. The following analytical methods from SW-846 (1986 or the most recently promulgated version) and EPA-600/4-79-020 (revised 3/1983) are the most commonly specified methods for the respective analyses. Any changes in analytical methods from the contract-required analytical methods must be pre-approved by the Committee.

PARAMETERS	METHODS
Volatile Organic Compounds (VOA)	8240A
Halogenated Volatile Organic Compounds (HVO)	8010A
Aromatic Volatile Organic Compounds (AVO)	8020
Semivolatile Organic Compounds (BNA)	8250/8270A
Organochlorine Pesticides (PEST)	8080
Polychlorinated Biphenyls (PCB)	8080
Phenols (PHENO)	8040A
Chlorinated Herbicides (HERB)	8150A
Polynuclear Aromatic Hydrocarbons (PAH)	8100/8310
Nitroaromatics and Nitramines (EXPLO)	8330 (draft)
Total Recoverable Petroleum Hydrocarbons (TRPH)	418.1
Total Recoverable Oil and Grease (O&G)	413.1/413.2
Total Petroleum Hydrocarbons (TPH)	8015 (mod.)
Trace Metals (METAL)	6010A
Arsenic	7060/7061
Mercury	7470/7471
Selenium	7740/7741
Cyanide (CN)	9010A/9012
Total Organic Carbon (TOC)	9060
Common Anions (ANION)	300.0/300s
Phenolics (PHENL)	9065/9066/ 9067

PARAMETERS	METHODS
Total Hardness (HARD)	130s
Alkalinity (ALKAL)	310s
Chemical Oxygen Demand (COD)	410s
Total Dissolved Solids (TDS)	160.1
Total Suspended Solids (TSS)	160.2

ANALYSIS OF PE SAMPLES

A laboratory must use project-required analytical methods for analyses of all project-specific PE samples unless otherwise instructed by the Coordinator. A laboratory's practical quantitation limits for each analytical method must meet or be lower than those specified in the method. The soil/sediment PE samples could be real world environmental samples which contain certain analytes of high concentrations. Special attention is needed to reduce or correct the interference caused by the analytes of high concentrations. Subcontract of PE sample analysis is prohibited.

INTERNAL QC ANALYSES

A laboratory shall conduct and report all method-required internal QC analyses. The minimum internal QC analyses required for PE samples include:

- method blanks for all PE sample analyses,
- surrogate spikes for all organic PE sample analyses,
- laboratory control samples (LCSs), second column confirmation, etc., whenever applicable,
- replicates, matrix spikes, and matrix spike duplicates for all soil/sediment PE sample analyses, and
- replicates, matrix spikes, and matrix spike duplicates on spiked reagent water for all water PE samples.

DATA REPORTING PACKAGE

A laboratory may use its standard data package to report PE sample results, however, the data package should be sequentially numbered and contain as a minimum the following information:

a. Table of contents

b. A case narrative including a list of PE samples analyzed/reported and problems encountered with PE sample analysis.

c. A Chain-of-Custody report.

d. Sample preparation information including sample preparation date, method citations for sample digestion, extraction, solvent exchange, concentration, cleanup, etc.

e. Analytical results for all target analytes plus method citations and laboratory practical quantitation limits.

f. Summary of method-specific QC results and assessments of precision and accuracy.

g. Phone conversation records on major issues related to PE sample analysis.

The analysis results shall identify and quantify all target analytes listed in the required analytical method, including estimated values and the quantitation limits for target analytes not detected. Except for petroleum hydrocarbons PE samples, all soil/sediment PE sample analyses shall be reported on a dry-weight basis along with percent moisture. For petroleum hydrocarbons PE samples, the results shall be reported on an "as-received" basis, i.e., no correction should be made for moisture content. Neither should any data be corrected for spike recoveries nor for any contamination found in trip blank or laboratory's method blank. Raw data including sample preparation and run log, calibration, chromatograms, calculation, etc., are normally not required for PE sample data package unless requested by the Coordinator.

WHEN TO REPORT

Normally, written reports for all PE sample analyses are to be received by the sample originators at WES and/or MRDL within 20 working days after receipt of the samples. For fast turnaround projects or reanalysis of additional PE samples, a laboratory shall return the results within five or ten working days, depending on the number of PE samples to be analyzed. Failure to analyze these samples successfully or within the required time frame may result in termination of the validation process. It is a laboratory's responsibility to keep the

Committee informed of any problems with PE sample analyses that would affect return of the results in a required time frame.

WHERE TO REPORT

All PE sample results except for total recoverable petroleum hydrocarbons, total petroleum hydrocarbons, total recoverable oil and grease, and explosives shall be returned to WES:

U.S. Army Corps of Engineers
Waterways Experiment Station
ATTN: CEWES-EE-C (Ann B. Strong)
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

The PE sample results for total recoverable petroleum hydrocarbons, total petroleum hydrocarbons, total recoverable oil and grease, and explosives shall be returned to MRDL:

U.S. Army Corps of Engineers
Missouri River Division Laboratory
ATTN: CEMRD-ED-L (Doug Taggart)
420 S. 18th Street
Omaha, NE 68102-2586

A complete copy of all PE sample results shall be sent to the Committee:

U.S. Army Corps of Engineers
HTRW Mandatory Center of Expertise
ATTN: CEMRD-ED-EC (Laboratory Validation Coordinator)
12565 West Center Road
Omaha, NE 68144-3869

CRITERIA FOR EVALUATION OF PE SAMPLE RESULTS

The laboratory PE sample results will be compared in the following manner: (1) with the prepared concentrations of PE samples that are used as the absolute recovery comparators, and (2) with the statistical mean and standard deviations reported by a group of referee and/or peer laboratories. The acceptable limits for analyte quantitation will be established statistically at 95 percent confidence based on peer group results. If only minor errors which are attributable to data calculation, transcription, etc. appear in PE samples analysis, a laboratory will have an opportunity to provide revised data. If a

laboratory is asked to check its analytical data, the laboratory should return revised data within five working days.

WHAT IS NEXT AFTER PE SAMPLE ANALYSIS?

Possibly an on-site laboratory inspection. After data revisions, a commercial laboratory must pass, as a minimum, more than 50 percent of all PE samples, including project-specific and non-project-specific PE samples, within 40 working days from receipt of the first set of PE samples to trigger the on-site laboratory inspection process. Prior to an inspection, a laboratory shall promptly submit to the Coordinator a concise report about the problems, solutions, and corrective actions on the PE sample parameters failed on its first attempt. After receipt of this report, the Coordinator will contact the laboratory to schedule an on-site inspection within two weeks.

During an on-site laboratory inspection, the USACE inspectors will investigate the problems and solutions for the failed PE samples. Additional PE samples may be required, as recommended by the inspectors and concurred by the Committee, for a laboratory to demonstrate that all problems associated with the failed parameters have been satisfactorily corrected. If additional PE samples are analyzed, a laboratory shall return analytical results of the additional PE samples within five or ten working days after receipt of the PE samples depending on the number and type of the additional PE samples. The cost of additional PE samples will be borne by the laboratory (currently about $100 to $300 per method, per matrix, and per shipment.)

APPENDIX E

PRELIMINARY QUESTIONNAIRE

FOR

THE U.S. ARMY CORPS OF ENGINEERS

VALIDATION PROGRAM FOR

ANALYTICAL CHEMISTRY LABORATORIES

PRELIMINARY QUESTIONNAIRE

This questionnaire is designed to elicit all the information required prior to an on-site survey. Please make a concerted effort to furnish the information as accurately and concisely as possible. For convenience, the questionnaire has been divided into seven sections:

Section 1: General Laboratory Information

Section 2: Organization and Personnel

Section 3: Laboratory Facilities and Equipment

Section 4: Analytical Instrumentation

Section 5: Technical Services

Section 6: Chemical Analyses

Section 7: Federal RCRA Compliance

In each section, the questions are styled for the ease of the laboratory's response. In many cases only a check (✓) is required. Other questions call for a short answer; clarity and brevity should hallmark your response. If you need more space, please continue on blank sheets and attach them to the questionnaire.

Each section is independent, so that the different sections may be distributed to the most knowledgeable persons in the laboratory who can complete their parts independently. Finally, management should assemble and check all responses before returning the completed forms. The completed preliminary questionnaire shall be returned to the USACE within ten working days from the date of receipt or prior to the on-site laboratory inspection.

The completed questionnaire will be used by the USACE laboratory inspectors to prepare the upcoming on-site laboratory inspection. The time involved in the on-site inspection can be minimized by a thorough presentation of the information sought in the questionnaire. Therefore, it is advantageous to both your laboratory and the inspection team if these questions are answered precisely and completely.

Thank you for your cooperation.

SECTION 1. GENERAL LABORATORY INFORMATION

1. Laboratory Name: _____

2. Street Address: _____

_____

Mailing Address: _____

_____

3. Telephone No.: _____ FAX No.: _____

4. Name of Laboratory Director: _____

Name of Laboratory Manager: _____

Name of QA Officer: _____

5. Does your laboratory routinely participate in any of the following QA programs? If yes, please check the brackets, complete the attached CHART E-1 (Page E-6), and submit copies of the laboratory certificates, a list of approved analytical parameters and the two most recent results of any performance evaluation sample analyses. [ ] Check if attached.

a. Department of Defense QA Programs:

[ ] U.S. Army Corps of Engineers (USACE)
[ ] U.S. Army Environmental Center (USAEC)
[ ] U.S. Air Force Occupational and Environmental Health Laboratory QA/QC Audit (USAFOEHL)
[ ] U.S. Navy Energy and Environmental Support Activity (NEESA)
[ ] Naval Assessment and Control of Installation Pollutants (NACIP)

b. USEPA QA Programs:

[ ] EMSL/Cincinnati Water Supply QA Program
[ ] EMSL/Cincinnati Water Pollution QA Program
[ ] Office of Solid Waste Quarterly Audit Program
[ ] Remedial Engineering Management (REM) or Alternative Remedial Contracts Strategy (ARCS) Subcontract Laboratory
[ ] Radiochemistry Laboratory Intercomparison Study

[ ] Discharge Monitoring Program for NPDES Permitted Clients

c. Other Federal Agencies:

[ ] DOE Hazardous Waste Remedial Action Program (HAZWRAP)
[ ] USDA Plant Protection and Quarantine Program
[ ] NIST National Voluntary Laboratory Accreditation Program (NVLAP) for Asbestos
[ ] U.S. Geological Survey Performance Evaluation Program
[ ] NIOSH Proficiency Analytical Testing Program (PAT)
[ ] NIOSH Asbestos Analyst Registry
[ ] Nuclear Regulatory Commission (NRC) Broadscope Materials License

6. Does your laboratory currently participate in any state certification/accreditation programs? [ ] Check if yes and complete the attached CHART E-2 (Page E-7).

7. List major USEPA or USACE contracts held in the last two years that included soil/sediment/sludge analyses for hazardous, toxic, and radioactive wastes.

Agency	Project Name	Approx. No. of Samples	Analytes

8. Is your laboratory currently approved by the Small Business Administration (SBA) for Section 8(a) program? [ ] Check if yes and submit copies of the SBA approval letter/documents.

9. This questionnaire is completed/assembled by:

_____ Date___/___/____
NAME TITLE

and reviewed/approved by:

_____ Date ___/___/____
NAME TITLE

CHART E-1

Please check the brackets to indicate your laboratory's participation in the QA programs listed below. Indicate your laboratory's period of participation, identification number, and expiration date for each of these programs, in the space provided.

Lab Name: _____

	Period of Participation	Laboratory ID Number	Expiration Date
A. Department of Defense			
[ ] USACE			/ /
[ ] USAEC			/ /
[ ] USAFOEHL			/ /
[ ] NEESA			/ /
[ ] NACIP			/ /
B. USEPA			
[ ] USEPA WS			/ /
[ ] USEPA WP			/ /
[ ] USEPA OSW			/ /
[ ] REM/ARCS			/ /
[ ] RADCHEM			/ /
[ ] NPDES			/ /
C. Other Federal Agencies			
[ ] HAZWRAP			/ /
[ ] USDA			/ /
[ ] NVLAP			/ /
[ ] USGS			/ /
[ ] NIOSH PAT			/ /
[ ] NIOSH AAR			/ /
[ ] NRC			/ /

CHART E-2

Please indicate the State, type of certification, certifying organization, certification number, and the expiration date for each of certification programs in which your laboratory currently participates in.

Lab Name: _____

State	Type of Certification	Certifying Organization	Certification Number	Expiration Date
_____	1 2 3 4 5 6 7 8	_____	_____	_____
_____	1 2 3 4 5 6 7 8	_____	_____	_____
_____	1 2 3 4 5 6 7 8	_____	_____	_____
_____	1 2 3 4 5 6 7 8	_____	_____	_____
_____	1 2 3 4 5 6 7 8	_____	_____	_____
_____	1 2 3 4 5 6 7 8	_____	_____	_____
	1 2 3 4 5 6 7 8	_____	_____	_____
_____	1 2 3 4 5 6 7 8	_____	_____	_____
_____	1 2 3 4 5 6 7 8	_____	_____	_____
_____	1 2 3 4 5 6 7 8	_____	_____	_____
_____	1 2 3 4 5 6 7 8	_____	_____	_____
_____	1 2 3 4 5 6 7 8	_____	_____	_____

Type of Certifications:
(1) General/Environmental, (2) Drinking Water, (3) Waste Water, (4) Hazardous Waste, (5) State Contract Laboratory, (6) Air Analyses, (7) Asbestos Analysis, (8) Radiochemical Analysis.

SECTION 2. ORGANIZATION AND PERSONNEL

1. Provide an organization chart of the laboratory, including any field operations or other internal affiliations to show how the laboratory fits into the general organizational structure. If attached, please check. [ ]

2. How many years in operation?_____

3. What is the total number of laboratory employees?_____
   Has this number increased over the past five years?
   Check if yes [ ]

4. What portion of the laboratory employees are technical staff?
   Number_____ Percentage_____

5. What portion of your technical staff participated in a formal training program related to improving work performance during the past year? Number_____ Percentage_____

6. What was your turnover rate during the last 12 months?

   (A) Administrative Staff:

   Number_____ Percentage_____

   (B) Technical Staff:

   Number _____ Percentage_____

7. Complete CHART E-3 (Pages E-9 thru E-16) for all technical staff. Use a separate block for each employee and make additional copies if needed.

CHART E-3

1. QUALIFICATIONS OF MANAGEMENT STAFF:

Position Title	Name of Employee	Degree & Major	Years[1] of Exp	Analyses performed and Appropriate Training[2]
Lab Director (Exp: 10 yrs min.)[3]				
Lab Manager (Exp: 7 yrs min.)[3]				
Org. Lab Manager (Exp: 5 yrs min.)[3]				
Inorg. Lab Manager (Exp: 5 yrs min.)[3]				
System Manager (Exp: 3 yrs min.)[4]				
Project Manager (Exp: 1 yr min.)[4]				
Other (specify) _____				
_____				
_____				

1. Related to chemical analysis of hazardous, toxic, and radioactive wastes. Requirements for experience as listed are minimal.
2. Manufacturer sponsored class, ACS short course, or EPA symposiums, etc.
3. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.
4. Minimum of Bachelor's degree with four or more intermediate courses in programming, information, and system management.

CHART E-3

2. QUALIFICATIONS OF QA/QC AND TECHNICAL STAFF:

Position Title	Name of Employee	Degree & Major	Years[1] of Exp	Analyses performed and Appropriate Training[2]
Technical Director (Exp: 7 yrs min.)[3]				
QA Officer (Exp: 5 yrs min.)[3]				
QC Specialists (Exp: 3 yrs min.)[3]				
Sample Custodians[4] (Exp: 6 mos min.)				
Data Reporting and Delivery Officers[4] (Exp: 6 mos min.)				

1. Related to chemical analysis of hazardous, toxic, and radioactive wastes. Requirements for experience as listed are minimal.
2. Manufacturer sponsored class, ACS short course, EPA symposiums, etc.
3. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.
4. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline, or in lieu of the Bachelor's degree, three years of experience in sample receiving or data reporting, respectively.

CHART E-3

3. QUALIFICATIONS OF SAMPLE PREPARATION STAFF:

Position Title	Name of Employee	Degree & Major	Years[1] of Exp	Analyses performed and Appropriate Training[2]
Sample Prep Lab Supervisors (Exp: 3 yrs min.)[3]				
Org. Extraction and Concentration Experts (Exp: 1 yr min.)[4]				
Metal Digestion Experts[4] (Exp: 6 mos min.)				

1. Related to chemical analysis of hazardous, toxic, and radioactive wastes. Requirements for experience as listed are minimal.
2. Manufacturer sponsored class, ACS short course, EPA symposiums, etc.
3. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.
4. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline, or in lieu of the Bachelor's degree, three years of experience in organic or metal sample preparation, respectively.

CHART E-3

4. QUALIFICATIONS OF GC STAFF:

Position Title	Name of Employee	Degree & Major	Years[1] of Exp	Analyses performed and Appropriate Training[2]
GC Lab Supervisor[3] (Exp: 3 yrs min.)				
GC Operators (Exp: 1 yr min.)[4]				
Pesticide Residue Analysis Experts (Exp: 2 yrs min.)[3]				

1. Related to chemical analysis of hazardous, toxic, and radioactive wastes. Requirements for experience as listed are minimal.
2. Manufacturer sponsored class, ACS short course, EPA symposiums, etc.
3. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.
4. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline, or in lieu of the Bachelor's degree, three years of experience in operating and maintaining GC instruments.

CHART E-3

5. QUALIFICATIONS OF GC/MS STAFF:

Position Title	Name of Employee	Degree & Major	Years[1] of Exp	Analyses performed and Appropriate Training[2]
GC/MS Lab Supervisr (Exp: 3 yrs min.)[3]				
GC/MS Operators (Exp: 1 yr min.)[4]				
GC/MS Spectral Interpretation Experts (Exp: 2 yrs min.)[3]				

1. Related to chemical analysis of hazardous, toxic, and radioactive wastes. Requirements for experience as listed are minimal.
2. Manufacturer sponsored class, ACS short course, EPA symposiums, etc.
3. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.
4. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline, or in lieu of the Bachelor's degree, three years of experience in operating and maintaining GC/MS instruments.

CHART E-3

6. QUALIFICATIONS OF AA/ICP STAFF:

Position Title	Name of Employee	Degree & Major	Years[1] of Exp	Analyses performed and Appropriate Training[2]
Metal Lab Supervisr (Exp: 3 yrs min.)[3]				
AA Operators (Exp: 1 yr min.)[4]				
ICP Operators (Exp: 1 yr min.)[4]				
ICP Spectroscopists (Exp: 2 yrs min.)[3]				

1. Related to chemical analysis of hazardous, toxic, and radioactive wastes. Requirements for experience as listed are minimal.
2. Manufacturer sponsored class, ACS short course, EPA symposiums, etc.
3. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.
4. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline, or in lieu of the Bachelor's degree, three years of experience in operating and maintaining AA or ICP instruments, respectively.

CHART E-3

7. QUALIFICATIONS OF CLASSICAL AND OTHER ANALYSES:

Position Title	Name of Employee	Degree & Major	Years[1] of Exp	Analyses performed and Appropriate Training[2]
Wet Lab Supervisor (Exp: 3 yrs min.)[3]				
UV/VIS Specialists (Exp: 1 yr min.)[3]				
Cyanide Analyst[3]				
IR Specialists (Exp: 1 yr min.)[3]				
TRPH Analyst[3]				
HPLC Specialists (Exp: 1 yr min.)[3]				
Explosive Analyst[3]				

CHART E-3

7. QUALIFICATIONS OF CLASSICAL AND OTHER ANALYSES: (continued)

Position Title	Name of Employee	Degree & Major	Years[1] of Exp	Analyses performed and Appropriate Training[2]
Ion Chromatography Specialists (Exp: 1 yr min.)[3]				
Common Ion Analyst[3]				
Radiochemical Analysis Experts (Exp: 2 yrs min.)[3]				
Characteristics Testing Experts (Exp: 1 yr min.)[3]				

1. Related to chemical analysis of hazardous, toxic, and radioactive wastes. Requirements for experience as listed are minimal.
2. Manufacturer sponsored class, ACS short course, EPA symposiums, etc.
3. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

SECTION 3. LABORATORY FACILITIES AND EQUIPMENT

1. Please provide a laboratory floor plan and complete CHART E-4. (* Note: The adequacy of laboratory facilities will be checked by USACE inspectors.)

CHART E-4

Lab Name: _____ Page 1 of 2

Item	Description	Ade-* quate	Additional Information
Building in Use Total (Sq Ft)			
Office Space Total (Sq Ft)			
Lab Space Total (Sq Ft)			
Bench-top Space Total (Sq Ft)			
Bench Hoods No. _____ (Ft/min)			

Item	Avai - able	Ade-* quate	Additional Information
Storage Space - Chemicals			
Sample Storage - General			
Secured Space			
Refrigerated Space			
High Hazardous Samples			
Controlled Area - Temperature			
Humidity			
Shielded			
Clean Rooms			
Compressed Air			

CHART E-4

Item	Avai - able	Ade- quate*	Additional Information
Vacuum			
Water Supply - Distilled			
Deionized			
Ammonia - free			
$C0_2$ - free			
Bacteriologically Suitable			
Safety Equipment - Fire Alarm			
Fire Extinquishing Equipment			
Emergency Showers			
Eye Fountains			
Safety Glasses & Gloves			
Hazardous Area Escape			
Flammable Material Storage			
OSHA Signs			
Glassware Washing Equipment			
Disposal Equipment - Broken Glass, Contaminated Solvent, Material, etc.			
Laboratory Information Management System (LIMS)			
Building Security System			
Mobile Laboratories			
Facilities as a Whole			

2. FIELD SAMPLING/ANALYSIS. Please complete CHART E-5 if the laboratory conducts field sampling/analysis. (* Note: The adequacy of laboratory facilities and equipment shall be checked by USACE inspectors.)

CHART E-5

Lab Name:_____ Page 1 of 1

Item	Avai - able	Ade-* quate	Additional Information
Dedicated Lab Space & Hoods			
Bottle Preparation Area			
Sample Coolers			
Chain-of-Custody Record			
Sample Labels and Tags			
Sampling Tools - Soil			
Sediment			
Sludge			
Surface Water			
Ground Water			
Ambient Air			
Emission Source			
Other (specify) _____			
Field Testing/Monitoring Equipment - Sniffers			
Portable GCS			
Geiger Counters			
Other (specify) _____			
Mobile Laboratories			

SECTION 4. ANALYTICAL INSTRUMENTATION

CHART E-6

1. SUMMARY OF GC INSTRUMENTS:

No.	Manufacturer	Model Number	Age Yrs	Use for Method†	Ade-quate*	Comments‡
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						

† 8000 series GC methods in SW-846 (3rd Edition, 1986).
* The adequacy of analytical instrument will be checked by USACE inspectors.
‡ Detectors, condition, autosamplers, modifications, etc.

CHART E-6

2. SUMMARY OF GC/MS INSTRUMENTS: †

No.	Manufacturer	Model Number	Age Yrs	Use for Method ‡	Ade-* quate	Comments Φ
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						

† Iclude MS, LC/MS, GPC, etc., if available.
‡ 8000 series GC methods in SW-846 (3rd Edition, 1986).
* The adequacy of analytical instrument will be checked by USACE inspectors.
Φ Detectors, condition, autosamplers, modifications, etc.

CHART E-6

3. SUMMARY OF AA/ICP INSTRUMENTS: †

No.	Manufacturer	Model Number	Age Yrs	Use for Method ‡	Ade-* quate	Comments Φ
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						

† Include ICP/MS, microwave digester, etc., if available.
‡ 8000 series GC methods in SW-846 (3rd Edition, 1986).
* The adequacy of analytical instrument will be checked by USACE inspectors.
Φ Detectors, condition, autosamplers, modifications, etc.

CHART E-6

4. SUMMARY OF OTHER INSTRUMENTS:†

No.	Manufacturer	Model Number	Age Yrs	Use for Method‡	Ade-quate*	Comments Φ
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						

† Include autoanalyzer, UV/VIS, IR, HPLC, IC, SEM, X-ray instrument, radioactivity counter/system, analytical balance, etc.

‡ 8000 series GC methods in SW-846 (3rd Edition, 1986).

* The adequacy of analytical instrument will(be checked by USACE inspectors.

Φ Detectors, condition, autosamplers, modifications, etc.

SECTION 5. TECHNICAL SERVICES

1. Laboratory Name:_____

2. Please check the types of technical services routinely provided at this laboratory.

[ ] Environmental	[ ] Pharmaceutical	[ ] Metallurgical
[ ] Ecological	[ ] Clinical	[ ] R&D
[ ] Radiochemical	[ ] Agricultural	[ ] Other (specify)
[ ] Geotechnical	[ ] Food Quality	_____

3. Please check the types of samples routinely analyzed at this laboratory.

[ ] Drinking Water	[ ] Air	[ ] Hazardous Waste
[ ] Waste Water	[ ] Asbestos	[ ] Mixed Waste
[ ] Soil/Sludge	[ ] Fuel Oil	[ ] Other (specify)
[ ] Sediment	[ ] Wipe Sample	_____

4. Please check the types of analyses routinely conducted at this laboratory.

A. Organics:

[ ] Volatile Organic Compounds
[ ] Semivolatile Organic Compounds
[ ] Organic compounds using Isotope Dilution Techniques
[ ] Organochlorine Pesticides

[ ] Organophosphorus Pesticides
[ ] Polychlorinated Biphenyls
[ ] Congener Specific Polychlorinated Biphenyls
[ ] Polynuclear Aromatic Hydrocarbons

[ ] Chlorinated Herbicides
[ ] Dioxins and Furans
[ ] Nitroaromatics/Nitramines/Explosives
[ ] Other (specify)_____

[ ] Perform any of the above analyses on an oily matrix.
[ ] Perform any of the above analyses on a plant/animal tissue matrix.
[ ] Perform any of the above analyses on dioxin contaminated samples.
[ ] Perform any of the above analyses on mixed waste samples.

B. Metals:

[ ] General Metals Analysis
[ ] Microwave Digestion
[ ] Hexavalent Chromium
[ ] Organo-Lead, Tin and Mercury
[ ] Metals analyses using neutron activation
[ ] Other (specify)_____

[ ] Perform any of the above analyses on an oily matrix.
[ ] Perform any of the above analyses on a plant/animal tissue matrix.
[ ] Perform any of the above analyses on dioxin contaminated samples.
[ ] Perform any of the above analyses on mixed waste samples.

C. Wet Chemistry:

[ ] Anions ($C1^-$, F^-, NO_2^-, NO_3^-, SO_4^{2-}, PO_4^{3-}, etc.)
[ ] Physical (TDS, TSS, Conductivity, pH, etc.)
[ ] Oxygen Demands
[ ] Nutrients

[ ] Phenols
[ ] Oil and Grease
[ ] Petroleum Hydrocarbons
[ ] Total Organic Carbon

[ ] Total Organic Halides
[ ] Radioactivity
[ ] Other (specify)_____

D. RCRA Characteristics:

[ ] Ignitability
[ ] Reactivity
[ ] Corrosivity
[ ] Toxicity

E. Leaching Procedures:

[ ] Toxicity Characteristic Leaching Procedure
[ ] Extraction Procedure Toxicity
[ ] California Leach
[ ] ASTM Leach

F. Radiochemical:

[ ] Gross Alpha/Beta
[ ] Radium 226/228
[ ] Tritium
[ ] Gamma-Emitting Radionuclides

[ ] Total/Isotopic Uranium
[ ] Total/Isotopic Thorium
[ ] Transuranic Alpha-Emitters
[ ] Strontium 89/90

[ ] Isotopic Plutonium
[ ] Radon
[ ] Other (specify)_____

G. Physical:

[ ] Viscosity
[ ] Bulk Density
[ ] Proximate/Ultimate Analysis (percent moisture, percent ash, volatile matter, C, H, S, N, O)
[ ] Chlorine

[ ] Total Sulfur
[ ] Forms of Sulfur
[ ] Fuel Oil Fingerprinting
[ ] Specific Gravity

[ ] Percent Water (Karl Fisher Test)
[ ] Heat Contents
[ ] Other (specify)_____

[ ] Perform any of the above analyses on dioxin contaminated samples.
[ ] Perform any of the above analyses on mixed waste samples.

H. Air:

[ ] Summa Canisters
[ ] Tenax Tubes
[ ] Carbon Molecular Sieves/Charcoal Tubes
[ ] Tedlar Bags

[ ] Polyurethane Foam Filters
[ ] XAD Resins
[ ] other (specify)_____

[ ] Performs metals analyses on cellulose membrane filters.
[ ] Performs metals analyses on air samples using an annular denuder.

I. Asbestos:

[ ] Polarized Light or Phase Contrast Microscopy
[ ] Scanning Electron Microscopy
[ ] Transmission Electron Microscopy
[ ] X-Ray Diffraction

J. Biological:

[ ] AMES Mutagenicity Testing
[ ] Biological Oxygen Demand
[ ] Chlorophyll A
[ ] Bacteriological (fecal coliform/streptococcus, etc.)

[ ] Acute Toxicity Bioassay
[ ] Chronic Toxicity Bioassay
[ ] Other (specify)_____

K. Geotechnical:

[ ] Atterberg Limits
[ ] Permeability
[ ] Cation Exchange Capacity
[ ] Porosity

[ ] Shear Strength
[ ] Grain Size
[ ] Other (specify)_____

[ ] Perform any of the above analyses on dioxin contaminated samples.
[ ] Perform any of the above analyses on mixed waste samples.

5. Do you perform field sampling activities? Yes [ ] No [ ]

6. Do you perform field testing activities? Yes [ ] No [ ]

7. Do you perform field monitoring activities? Yes [ ] No [ ]
If yes, please check nature of field monitoring activity:

[ ] Water Quality [ ] Air-Ambient [ ] Radiation
[ ] Estuaries [ ] Air-Source [ ] Other (specify)
[ ] Oceans [ ] NPDES _____

8. Do you participate in enforcement actions, emergency episodes, or special studies? Please specify.

9. Are/were you an USEPA CLP RAS contract laboratory? If yes, please provide the following information.

[ ] Volatile Organics Expiration Date: ___/___/___

[ ] Organics Expiration Date: ___/___/___

[ ] Inorganic Expiration Date: ___/___/___

[ ] Dioxin Expiration Date: _____

10. Are you an USEPA SAS contract laboratory? If yes, please provide the following information.

Project Name	Approx. No. of samples	Analytes

11. Is your laboratory an AIHA accredited laboratory and/or have your laboratory successfully participated in NIOSH Proficiency Analytical Testing (PAT) Program for air sample analysis? Yes [ ] No [ ] If yes, please check the proficiency testing that your laboratory participated in and attach copies of the two most recent rounds of PAT results.

[ ] METALS [ ] SILICA [ ] ASBESTOS [ ] ORGANIC SOLVENTS

12. Does your laboratory conduct USEPA Compendium Air Sampling/ Analysis? If yes, please list below which methods (TO-1 thru TO-14).

Method	Project Name	Total No. of Samples

SECTION 6. CHEMICAL ANALYSES

All sample analyses of water, soil, sediment, sludge, or waste shall be performed with standard USEPA methods, if available and appropriate. All method specified procedures must be followed exactly with no deviations unless modifications are specifically authorized by the USACE TM/COR. When a standard USEPA method is not available, the USACE TM/COR may approve the use of other methods (USEPA CLP, ASTM, USGS, NIOSH, DOE, and APHA/AWWA/WPCF methods). The standard USEPA methods refer to methods in the following publications, including the latest approved or promulgated revisions from USEPA.

1. Test Methods for Evaluating Solid Wastes, SW-846, Third Edition, Revision 0, September 1986 and Revision 1, July 1992.

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

3. Guidelines Establishing Testing Procedures for the Analysis of Pollutants, 40 CFR Part 136, October 26, 1984.

4. Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, August 1980.

5. Radiochemical Analytical Procedures for Analysis of Environmental Samples, EMSL-LV-0539-17, March 1979.

6. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/600/4-89/017, June 1988.

The USEPA CLP methods refer to analytical parameters included in the appropriate USEPA Contract Laboratory Program Statement of Work and/or the most current revision:

1. Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, Document Number 0LM02.0 including Revision 0LM02.1.

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

3. Superfund Analytical Methods for Low Concentration Water for Organics Analysis, SOW Number Revision 10/92.

4. Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration, Document Number ILM03.0.

5. Statement of Work for Inorganics Analysis, Multi-Media, High-Concentration, Document Number IHCO1.3.

6. Superfund Analytical Methods for Low Concentration Water for Inorganics Analysis, SOW Number Revision 10/91.

7. Statement of Work for Analysis of Polychlorinated Dibenzo-P-Dioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF), Multi-Media, Multi-Concentration, Document Number DFLM01.0 including Revision DFLM01.1, September 1991.

The ASTM methods refer to the Annual Book of ASTM Standards, Section 11, Water and Environmental Technology, 1993 or the most current revision, published by the American Society for Testing and Materials.

The USGS methods refer to the Techniques of Water-Resources Investigations of the United States Geological Survey, Book 5, Third Edition, 1989 or the latest revised edition, published by the United States Geological Survey, U.S. Department of Interior.

The NIOSH methods refer to the Manual of Analytical Methods, Third Edition, 1984 and all supplements and revisions, published by the National Institute for Occupational Safety and Health, U.S. Department of Health and Human Services.

The DOE methods refer to the DOE Methods for Evaluating Environmental and Waste Management Samples, DOE/EM-0089T, Revision 1, March 1993 and the latest update or addendum, published by the U.S. Department of Energy.

The APHA/AWWA/WPCF methods refer to Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992 or the latest published edition, published jointly by the American Public Health Association, the American Water Works Association, and the Water Pollution Control Federation.

If your laboratory routinely uses an alternate method or a modification of a referenced method above, please provide the requested information for each such case in CHART E-7 (Page E-32), "ALTERNATE OR MODIFIED ANALYTICAL METHODS".

In CHART E-8 (Page E-33), "OTHER ANALYTICAL METHODS", please provide information on important tests performed by your laboratory that are not included in the reference methods above.

CHART E-7

ALTERNATE OR MODIFIED ANALYTICAL METHODS

Laboratory Name: _____

1. Test: _____

2. If this is a modification of a referenced method,
   A. Which reference method (give manual name and pages)?

   _____

   B. Purpose of modification:

   _____

   _____

   _____

   C. Brief description of modification:

   _____

   _____

   _____

3. If this an alternate method,
   A. Purpose of use of alternate method:

   _____

   _____

   _____

   B. Brief description of method:

   _____

   _____

   _____

4. Have you applied to USEPA for approval of this procedure? (c.f., FR, Vol. 38, No. 199, October 16, 1973, Page 28760)

   _____

5. If alternate or modified methods will be used for USACE projects, please attach all validation documentation to prove the method works.

CHART E-8

OTHER ANALYTICAL METHODS

Lab Name: _____ Page __ of __

Test and Unit		Method Reference	SOPs Avail-able?	No. of Sample per Mo

SECTION 7. FEDERAL RCRA COMPLIANCE

1. Lab Name: _____

   Hazardous Waste Coordinator: _____

2. Was a RCRA inspection ever done at the lab? ____ If yes, who performed the inspection? _____
   When was the inspection performed? _____
   (Attach a copy of the most recent inspection report.)

3. Generally, what were the results of the inspection? _____

4. Describe the way hazardous waste is stored at the lab: _____

5. How does the lab dispose of their waste? _____

6. Describe the way hazardous waste is managed at the lab: _____

Citation: 40 CFR 261 USEPA Regulations for Identifying Hazardous Waste

Subpart A - General

1. Does the lab generate any hazardous waste? _____

2. Does the lab generate any hazardous wastes that are excluded from regulations under 40 CFR 261.4? _____ Provide citation for exclusion:_____

3. Has the sample exclusion in 40 CFR 261.4(d) been invoked for the lab?_____ If yes, have all the requirements associated with this exemption been met? If not, explain:

_____

_____

_____

_____

4. Are treatability studies conducted by the lab? _____ Has the State adopted the Treatability Exclusion in 40 CFR 261.4(f)? _____ If yes, has the lab met the requirements of 40 CFR 261.4(e) and (f)? _____ If the State has not adopted the exclusion, does the lab have a RCRA Part B Permit for treatment? _____

5. Is the lab a conditionally exempted small quantity generator (SQG)?_____ Does the lab generate less than 100 kg/mo of hazardous waste and less than 1 kg/mo of acute hazardous waste?_____ How much waste does the lab produce each month? _____ Are there records available to substantiate the amount of waste generated each month?_____ Are there records available that substantiate how much waste is being stored on-site at any one time?_____ If the lab generates less than 100 kg/mo of hazardous waste and less than 1 kg/mo of acute hazardous waste, the lab is a conditionally exempted SQG. To qualify for this exemption, the lab must meet the following (40 CFR 261.5):

   a. Hazardous waste is characterized IAW 40 CFR 262.
   b. There is never more than 1,000 kg stored on site.
   c. Waste is sent to a TSDF or a facility that beneficially reuses the waste, or a state permitted facility.

   Are there records to substantiate the above claims? ____

6. Does the facility do any of the following (40 CFR 261.6):

   a. Recycle materials in a manner constituting disposal?_____
   b. Burn or send to be burned hazardous wastes in a boiler or industrial furnace for energy recovery?_____
   c. Recycle waste containing precious metals?_____
   d. Reclaim spent lead-acid batteries?_____
   e. Generate used oil?____

   If the lab does any of the above, they are regulated by the requirements of 40 CFR 266 (Standards for the Management of Specific Hazardous Wastes).

7. Containers previously holding a hazardous wastes may be reused for other purposes or discarded as a solid waste (40 CFR 261.7) if they are emptied by pouring, pumping, aspirating, etc. Containers that once contained an acute hazardous wastes must be tripled rinsed prior to reuse. What happens to empty hazardous waste containers? _____

   _____

   _____

   Are there empty hazardous waste containers on site?_____
   Have all residues been removed from the containers?_____
   Have all labels been removed from the containers?_____
   What happens to empty containers that once contained an acute hazardous waste? _____

   _____

   _____

Subpart B - Criteria for Identifying the Characteristic of Hazardous Waste and for Listing Hazardous Wastes

1. Does the lab have a Hazardous Waste Management Plan or an equivalent? _____

Subpart C - Characteristics of Hazardous Wastes

1. Does the facility generate any of the following types of characteristic wastes:

   a. Ignitable?____
   b. Corrosive?____
   c. Reactive? _____
   d. TCLP? _____

Subpart D - Lists of Hazardous Wastes

1. Does the facility lab generate any of the following listed hazardous wastes:

   a. F-listed? _____ c. P-listed? _____
   b. K-listed? _____ d. U-listed? _____

2. Does the facility understand how to characterize their waste? _____ Is there a plan that describes the procedure?_____ List examples of the types of waste generated by the lab:

_____

_____

_____

_____

_____

_____

Citation: 40 CFR 262 USEPA Regulations for Hazardous Waste Generators

Subpart A - General

1. Does the facility generate less than 100 kg/mo of hazardous waste and 1 kg/mo of acute hazardous waste?_____ If yes, the facility is a conditionally exempted SQG. Does the facility store more than 1,000 kg of waste or 1 kg of acute waste at any one time?_____If yes, the facility is NOT a conditionally exempted SQG.

2. Does the facility generates between 100 - 1,000 kg/mo of hazardous waste or store more than 1,000 kg of waste on site? _____ If yes, the facility is a SQG.

3. Does the facility generate more than 1,000 kg/mo?_____ If yes, the facility is a generator.

4. Does the generator or SQG have a USEPA identification number (40 CFR 262.12)? ____ What is that number? _____

   Has the facility filed USEPA Form 8700-12, “Notification of Hazardous Waste Activity”?_____ Does the USEPA number on this form match the USEPA number on the manifests?_____

Subpart B - Manifest

1. Does the SQG or generator use a manifest when shipping hazardous waste (40 CFR 262.20)?_____

2. Are efforts made to use the consignment states's manifest? _____If the consignment state does not have a state manifest, are efforts made to secure a manifest from the generator's state (40 CFR 262.21)?_____

3. Does the facility sign the manifests certifying that a waste minimization program is in place at the facility?_____ Is there a waste minimization program in place (40 CFR 262.20)?_____

4. Do the following land ban records accompany the manifests:

   a. USEPA Hazardous Waste Number?_____
   b. Corresponding Treatment Standard?_____
   c. Waste Analysis?_____
   d. Certification if waste meets land ban standards or if the lab is shipping lab packs for disposal (40 CFR 268.7)?_____

Subpart C - Pre-Transportation Requirements

1. Does the facility label, mark, and placard waste prior to transportation to disposal? _____ What training has been provided to those persons?_____

   _____

   _____

   _____

   _____

   Who is responsible for labelling, marking, and placarding the waste leaving your facility?_____

2. SQG Requirements (40 CFR 262.34(d)):

   A SQG may generate between 100 - 1,000 kg/mo of hazardous waste and store up to 6,000 kg of hazardous waste on site without a permit. If the quantity stored exceeds 6,000 kg or 180 days (270 days if waste must be transported over 200 miles to disposal), the SQG will need a TSDF permit for storage.

a. Does the facility dispose of its waste over 200 miles away from the facility?_____
b. Does the facility store hazardous waste more than 180 days?_____
c. Is more than 6,000 kg of waste stored on site at any one time?_____
d. Is the waste stored in containers (40 CFR 265.170)?_____
e. Are containers in good condition?_____
f. Is the waste compatible with the containers?_____
g. Is the container always kept closed except when adding or removing waste?_____
h. Are the containers inspected at least weekly?_____
i. Is the date of which accumulation began clearly marked on each container?_____
j. Is the hazardous waste stored in tanks (40 CFR 265.201)? _____
k. Are only compatible wastes stored in the tank?_____
1. Is there sufficient freeboard or containment around the tank?_____
m. If the tank is a continuous feed tank, is there a means to stop inflow?_____
n. Is the tank, discharge control equipment, and monitoring equipment inspected each operating day?_____

Preparedness and Prevention (40 CFR 265 Subpart C)

a. Does the facility have an internal communications or alarm system?_____
b. Does the facility have means to summons emergency assistance?_____
c. Does the facility have a portable fire extinguisher?_____
d. Is an adequate volume of water available to fire fighters?_____
e. Is adequate aisle space provided in container storage area?_____
f. Have arrangements been made with the local authorities to familiarize them with wastes stored at the site?_____
g. Has an emergency coordinator been designated? _____
Name:_____
h. Is the following information posted next to the phone?

- Name and telephone number of the emergency coordinator? _____
- Location of spill control equipment, fire alarm, fire extinguishers, etc.?_____
- Are all employees familiar with the proper waste handling and emergency procedures relevant to their responsibilities?_____

3. Generator Requirements (20 CFR 262.34):

A generator is a person who generates more than 1,000 kg/mo of waste. A generator may accumulate hazardous waste on site 90 days or less without a permit.

a. Does the facility store waste over 90 days?____
b. Is the waste stored in containers (40 CFR 265.170)? _____
c. Are the containers in good condition?_____
d. Is the waste compatible with the container?_____
e. Are the containers always kept closed except when adding or removing waste? _____
f. Are the containers inspected at least weekly? _____
g. Is accumulation start date marked on each container?_____
h. Is the container labeled “Hazardous Waste”?_____
i. Is the hazardous waste stored in tanks (40 CFR 265 Subpart J)?_____
j. Is the tank integrity good?_____
k. Has the tank been adequately designed to hold the waste both structurally and with respect to compatibility?_____
l. Has secondary containment been provided around the tank (40 CFR 265.192)?_____
m. Are only compatible wastes stored in the tank?____
n. Is there sufficient freeboard or containment around the tank?____
o. If the tank is a continuous feed tank, is there a means
p. to stop inflow?_____
Is tank, discharge control equipment and monitoring equipment inspected each operating day?_____
q. Is tank closure anticipated?_____
r. During tank closure how was the disposal of contaminated soil, structures, and debris handled (40 CFR 265.114)?

_____

_____

_____

_____

_____

Preparedness and Prevention (40 CFR 265 Subpart C)
a. Does the facility have an internal communications or alarm system?_____
b. Does the facility have means to summons emergency assistance?_____
Does the facility have a portable fire extinguisher?_____
c. Is an adequate volume of water available to fire fighters?_____

e. Is adequate aisle space provided in container storage area?
f. Have arrangements been made with the local authorities to familiarize them with wastes stored at the site? _____
g. Has an emergency coordinator been designated? _____
Name: _____

Contingency Plan and Emergency Procedures (40 CFR 265 Subpart D)
a. Does the facility have a Contingency Plan? _____
b. Does the plan include a list of emergency equipment? _____
c. Does the plan include a description of arrangements with local emergency authorities? _____
d. Does the plan include an evacuation plan? _____
e. Have copies of the plan been submitted to the local authorities? _____
f. Has an emergency coordinator been designated?_____
Name: _____

Training (40 CFR 265.16)
a. Has training been provided to each employee who handles hazardous waste?_____
b. Has an annual update been provided?_____
c. Are the following records maintained at the facility:

- Job title of each position involving hazardous waste?_____
- Name of person filling that job?_____
- Written job description describing hazardous waste related activities?_____
- Written description of the type of training that will be provided?_____
- Documentation that the employees had received training? _____

4. Satellite Accumulation (40 CFR 262.34(c))

a. Does the facility use satellite accumulations points?_____
b. Are the containers in good condition?_____
c. Are the containers compatible with the waste stored in them?_____
d. Are the containers kept closed except when adding of removing waste?_____
e. Are the containers marked "Hazardous Waste" or others words that identify the contents? _____

Subpart D - Recordkeeping and reporting requirements

1. Are manifests kept on file for at least three years?_____

2. Are Biennial (applicable to generators only) and Exception Reports kept on file for at least three years?_____

3. Are waste analysis, waste records, etc. kept on file for at least three years?_____

4. If the lab is a generator, has a Biennial Report been filed by 1 March of each even numbered year?_____ If yes, does the report include the following:

   - Name, address, USEPA ID number for the generator?_____
   - Calendar year covered by the report? _____
   - Name, address and USEPA ID number of each TSDF facility you shipped waste to?_____
   - Name, address and USEPA ID number of each transporter used?_____
   - Description of the waste?_____
   - Description of the effort for waste minimization?_____
   - Waste minimization comparison with previous years?_____
   - Generator's certification?_____

5. Has the facility filed any exception reports (40 CFR 262.42)? ____

6. Recordkeeping and reporting requirements for SQG (40 CFR 262.44):

   a. Are manifests kept on file for at least three years?_____
   b. Are waste analysis, waste records, etc. kept on file for at least three years?_____
   c. Has the lab filed any exception reports (40 CFR 262.42)? ____

Subpart E - Exports of Hazardous Waste

1. Does the lab export hazardous waste?_____ If yes, see 40 CFR 262 Subparts E for requirements.

Subpart F - Imports of Hazardous Waste

1. Does the lab import Hazardous waste?_____ If yes, see 40 CFR 262 Subpart F for requirements.

Citation: 40 CFR 266 USEPA Standards for Management of Specific Hazardous Wastes and Facilities

Subpart E - Used Oil Burned for Energy Recovery

1. Does the lab generate used or waste oil?_____

2. Is the used oil sent to disposal?_____ If yes, the used oil must be sampled and if characteristic or mixed with a listed hazardous waste the used oil must be managed and disposed of as a hazardous waste.

3. If the used oil exceeds the parameters listed in the table below, the used oil is considered to be off-specification:

Arsenic	5 ppm maximum
Cadmium	2 ppm maximum
Chromium	10 ppm maximum
Lead	100 ppm maximum
Total Halogens	4,000 ppm maximum
Flash Point	100 °F minimum

Does analysis of the used oil indicate all levels less than those presented in the table? _____ If yes, the used oil is specification used oil and hence is not regulated under RCRA. If the used oil exceeds the levels presented in the table, it is considered to be off-specification used oil and the oil must be burned in an industrial furnace (40 CFR 260.10) or a boiler (40 CFR 260.10).

4. Does the used oil typically contain over 1,000 ppm total halogens?_____ If yes, the used oil is presumed to be a hazardous waste unless the generator can prove otherwise. Thus, this used oil becomes a hazardous waste fuel and must be burned in boilers and furnaces that are permitted under 40 CFR 264. If the hazardous waste fuel oil is stored on site, all generator regulations apply to this waste.

5. Is the oil properly disposed of? _____

   a. Specification used oil is not regulated under RCRA, however, the state may have special handling and disposal requirements.
   b. Off-specification used oil must be burned in an industrial furnace or boiler.
   c. Hazardous waste fuel must be burned in permitted furnaces and boilers.

Is the disposal method documented?_____ Are there records to substantiate the characterization of the used oil?_____ Are there records identifying the energy recovery facility used or the disposal facility used?_____

6. Does the lab sell/distribute their used oil directly to a burner? _____ Does the lab sell/distribute their used oil to another marketer?_____ If yes to either question, the lab is considered a marketer of used oil.

7. If the lab is a marketer of off-specification used oil, are the following requirements fulfilled:

   a. Analysis of used oil kept on file for both on-spec and off-spec used oil?_____
   b. Notification to USEPA of off-spec used oil management activities? _____
   c. Invoice system used?_____ The following items must be included in the invoice system:

      - An invoice number.
      - The lab's USEPA ID number and the receiving facility's number.
      - The names and address of the generator's facility and the receiving facility.
      - The quantity of off-spec used oil to be delivered.
      - The dates of shipment or delivery.
      - The following statement: "This used oil is subject to USEPA regulation under 40 CFR Part 266."

   d. Has the lab secured a signed notice from the burner or marketer certifying: that the facility has notified USEPA of their location and management activities; and that the burner will only burn the off-spec oil in an industrial furnace or boiler?_____

8. Recordkeeping requirements for generators of used oil that meets specifications:

   a. Are copies of the analyses kept for three years?_____
   b. Does the record include the name and address of the facilities receiving the used oil?_____
   c. Does the record include the dates of shipment or delivery? _____

9. Recordkeeping requirements for generators of off-specification used oil:

a. Are copies of the invoices kept for three years?_____
b. Are copies of the required notices kept on file for three years? _____

10. Does the lab burn used oil for energy recovery? _____ If yes, the requirements of 40 CFR 266.44 must also be met. Check the regulations to ensure compliance.

Subpart F - Recyclable Materials Utilized for Precious Metal Recovery

1. Does the lab accumulate precious metals for reclamation?_____ If yes, has the lab notified USEPA of the reclamation activities?_____ Does the lab use a manifest when transporting precious metals for reclamation?_____

2. Does the lab store recyclable materials?_____

a. Does the lab maintain records showing the volume of materials stored at the beginning of the calendar year? _____
b. Does the lab maintain records showing the volume of materials generated during the calendar year?_____
c. Does the lab maintain records showing the volume of materials remaining at the end of the calendar year? _____

3. Are these materials being speculatively accumulated?_____ If yes, all generator standards apply to these materials. Basically the material is being speculatively accumulated if the material is being stored and there is no real plans or market for reclamation. See 40 CFR 261.1 (c) for exact definition.

Subpart G - Spent Lead-Acid Batteries Being Reclaimed

1. Does the lab store spent batteries? _____ If yes, are these batteries destined for disposal? _____ If yes, these batteries are to be managed and disposed of as hazardous waste. If no, are these batteries destined for reclamation? _____ If yes, has the lab notified USEPA of this activity? _____ If the lab is storing spent batteries for reclamation, 40 CFR Part 264 applies.

SECTION 7. FEDERAL RCRA COMPLIANCE (continued)

Review the State regulations and list below any differences between the Federal RCRA requirements and the State's requirements:

SECTION 7. FEDARAL RCRA COMPLIANCE (continued)

The lab shall prepare to have the following documents, if applicable, ready for review during the inspection.

1. USEPA Notification Form 8700-12
2. USEPA Identification Number
3. SQG Permit
4. RCRA Part A Permit
5. RCRA Part B Permit
6. NPDES Permit
7. Manifests
8. Waste Analysis Records
9. Land Ban Records
10. Exception Reports
11. Biennial Reports
12. Annual Reports
13. Training and Personnel Files
14. Contingency Plan/Spill Prevention Control and Countermeasure (SPCC) Plan
15. Agreements with Local Emergency Authorities
16. Used Oil Records
17. Hazardous Waste Management Plan

APPENDIX F

ANALYTICAL PARAMETERS, METHODS, AND FEE SCHEDULE FOR THE PERFORMANCE EVALUATION SAMPLES FROM THE U.S. ARMY CORPS OF ENGINEERS

ANALYTICAL PARAMETERS, METHODS, AND FEE SCHEDULE FOR THE PERFORMANCE EVALUATION SAMPLES FROM THE U.S. ARMY CORPS OF ENGINEERS

The USACE PE samples are parameter, matrix, and method specific. Listed below are: the common analytical parameters and matrices for PE samples that are currently available from the USACE, the analytical methods that are normally required for the PE sample analyses, and the fee schedule that are currently charged for additional or non-project-required PE samples. The fee schedule of the PE samples is on a per method, per matrix, and per shipment basis.

ANALYTICAL PARAMETERS	MATRIX	METHODS	COST
Volatile Organics	water	8240A	$100
Halogenated Volatile Organics	water	8010A	$100
Aromatic Volatile Organics	water	8020	$100
Semivolatile Organics	water	8250/8270A	$150
Semivolatile Organics	soil	8250/8270A	$150
Organochlorine Pesticides	water	8080	$100
Polychlorinated Biphenyls	water	8080	$100
Polychlorinated Biphenyls	soil	8080	$100
Chlorinated Herbicides	water	8150A	$100
Phenols	water	8040A	$100
Polynuclear Aromatic Hydrocarbons	water	8100/8310	$100
Nitroaromatics and Nitramines	water	8330 (draft)	$150
Nitroaromatics and Nitramines	soil	8330 (draft)	$150
Petroleum Hydrocarbons	water	418.1	$100
Petroleum Hydrocarbons	soil	9071/418.1	$100
Petroleum Hydrocarbons	water	8015 (mod.)	$150
Petroleum Hydrocarbons	soil	8015 (mod.)	$150
Oil and Grease	water	413.1/413.2	$100
Trace Metals	water	6010A/7000s	$100
Trace Metals	soil	6010A/7000s	$100
Cyanide	water	9010A/9012	$100
Total Organic Carbon	water	9060	$100
Phenolics	water	9065/9066/9067	$100
Common Anions	water	300.0/300s	$100
Total Hardness	water	130s	$100
Alkalinity	water	310s	$100
Chemical Oxygen Demand	water	410s	$100

APPENDIX G

GUIDANCE
FOR
PREPARATION, HANDLING, AND VALIDATION
OF
PERFORMANCE EVALUATION SAMPLES

GUIDANCE FOR PREPARATION, HANDLING, AND VALIDATION OF PERFORMANCE EVALUATION SAMPLES

G-1. Introduction. PE samples are an integral part of a comprehensive laboratory validation program and are used to evaluate the performance of the entire laboratory system for a specific parameter and matrix. This includes sample tracking, preparation, analysis, method selection (i.e., selection of particular options within specified standard operating procedures), record keeping, and data reduction and reporting. The USACE's HTRW Quality Assurance (QA) Program routinely employs PE samples to validate the performance of a contract laboratory and to evaluate the quality of data produced by a validated laboratory. The USACE has developed a number of PE samples in water, soil, and sediment matrices for various environmental analyses. Other new PE samples in the above matrices and air matrix are under development to fulfill the USACE’s environmental mission needs.

a. PE samples used for performance evaluation could be either single blind or double blind. A single blind PE sample is known to be an audit sample, but its composition is not known to the analyst. A double blind PE sample is intended to be indistinguishable from a routine field sample such that a laboratory will not devote more attention to produce non-routine analytical performance. Use of double blind PE samples is perhaps the most ideal approach to the assessment of laboratory performance. However, stability considerations for aqueous samples and homogeneity concerns for soil samples present substantive obstacles to the effective use of double blind PE samples. Given these concerns, use of single blind PE samples is currently the most effective and economical mechanism for monitoring laboratory performance.

b. The preparation process for PE samples should be carefully planned to ensure the precision, accuracy, and reproducibility of each batch of PE samples. Detailed preparation procedures should be documented and maintained in-house per proper USEPA and USACE guidance for legal defensibility. All chemicals, reagents, and solvents used should be pre-analyzed to ensure that they meet high purity requirements. Each gravimetric and volumetric measurement devices such as titrant, balance, and calibrant should be certified against the National Institute of Standards and Technology (NIST) standards whenever available. Only ASTM class A volumetric glassware should be used for PE sample preparation. Each batch of resulting PE samples should be checked to confirm concentrations. All PE samples should be refrigerated and stored in the dark to ensure maximum storage life.

c. Ideally, all PE samples should have the following characteristics:

- Physical similarity to field samples.

- Analyte and interference content similar to field samples.

- Analyte concentrations near the levels expected in field samples, or, in absence of this information, concentrations that span the range of the analytical method.

- Behavior similar to actual field samples throughout laboratory handling and method manipulations.

- Ability to provide useful information on laboratory performance as well as documentation of associated data quality.

d. During the design and development of PE samples, the USACE must ensure that the following goals are considered and met.

- Suitability of the materials to mimic real world environmental samples for performance evaluation of sample processing and analysis.

- Homogeneity of the materials in terms of the target analyte profile.

- Stability of the materials in terms of the target analyte profile over an extended time no less than specified holding time.

- Long-term availability of a sufficient and reliable supply of PE samples.

- Legal defensibility of the data associated with PE samples.

- Minimization of the cost and time required to produce these materials.

G-2. Determinig PE Sample Requirements and Specifications. As aforementioned, ideal PE samples should be site-specific. The constituents (analytes and matrices), concentrations, and associated acceptance limits for PE samples should be selected based on certain key aspects of the specific project: project goals and objectives, data quality objectives (DQOs), and analytical methods to be employed. However, due to the great number and broad variety types of environmental projects and

programs that the USACE is involved in, site-specific PE samples are not cost effective and may not be available in a timely manner. Furthermore, the USACE PE samples are mainly used for validation of contract laboratories prior to field sample analysis. The continuous monitoring of contract laboratory's performance during the time period of active field sample analysis is mainly achieved through analysis of split field QA samples by government QA laboratories, with supplemental PE samples if needed. Therefore, the USACE PE samples are basically designed and prepared on a non-site-specific basis.

a. Matrix. Ideally, the matrix for the PE samples should be relevant to the problem at hand and must be accurately characterized. The matrix can generally be categorized into water, soil, sediment, sludge, ash, oil, waste, etc. However, significant matrix differences can be found, for example, between two soil or even two water samples. The design of PE samples should include consideration of the origin, mineralogy, and pretreatment of the field samples. Because total site-specific PE samples are not cost effective and/or available in a timely manner, the USACE normally uses reagent water and real world soil and sediment as matrix materials for preparation of PE samples. By special requests, the USACE can also prepare PE samples with site-specific sample matrices, such as spiked field samples or spiked well-defined field matrices.

b. Methods. The analytical method or instrumentation to be used for analysis must be considered when selecting or preparing PE samples. PE samples prepared for a highly sensitive instrument, such as graphite furnace atomic absorption (GFAA) spectrophotometer, may not be appropriate for a less sensitive instrument, such as flame atomic absorption (FLAA) spectrophotometer or inductively coupled plasma (ICP) atomic emission spectrometer. Because most USACE environmental projects request USEPA SW-846 methods for sample analysis, the majority of USACE PE samples are designed and prepared for evaluation of a laboratory's capability in SW-846 methods. PE samples for the USEPA CLP or drinking water methods are also available.

c. Quality Assurance/Quality Control. The laboratory validation process is usually focused on certain specific problems with a laboratory’s quality assurance/quality control (QA/QC). With proper use of different types of PE samples, specific QA/QC problems can be detected and corrected. For example, analytical precision could be verified by duplicate PE samples that are extremely homogeneous (such as water) and contains many analytes at midrange concentrations. Matrix spike recovery problems can be verified by sending a spiked field sample and a spiked extract or digest of the same field sample.

Differences in recoveries between pre- and post-extraction/digestion spikes will demonstrate whether the laboratory's extraction/digestion process is at fault. Precision data based on PE samples of clean matrices and on PE samples of real world matrices provide information about the true laboratory precision against the precision difficulty associated with the method on complex matrices.

d. Analytes. A PE sample must contain target analytes, but it also should contain components that cause known interferences when the target analytes are measured. This approach will uncover whether or not the laboratory is performing interference correction and the extent to which the correction is effective. Sometimes the difficulties encountered by a laboratory in the analysis of PE samples may be due to limitations of a method or an instrument. When considering candidate PE samples, one should obtain as much information as possible about the analytes of interest, all possible interfering species, and the limitations of the method or instrument.

(1) It is common to include problematic and non-problematic analytes and to evaluate a laboratory's performance proficiency on an analyte-by-analayte basis. PE samples that contain problematic analytes that are unstable, reactive, or interfering under optional preparation/analysis conditions can be used to check whether a laboratory takes proper precautions and corrective actions. Examples of these include: breakdown of DDT and endrin in a dirty gas chromatography (GC) injection port; loss of dichlorobenzene (the most volatile of the semivolatile compounds) by a poor nitrogen blow-down technique; loss of phenols caused by incomplete acidification of the sample, a less than required extraction time, excess drying out of the extract, etc.

(2) False-positive problems can be identified by looking for detection of analytes that are purposely left absent. False-negative problems can be identified by adding low level analytes and watching for non-detects. Or, the PE samples may contain isomers of analytes that elute close together and share a common GC/MS ion (for example, 2,4,5 & 2,4,6-trichlorophenol, 4-nitrophenol & dibenzofuran, benzo(a)anthracene & chrysene, benzo(b) & benzo(k) fluoranthene, anthracene & phenanthrene), high level of transition metals (especially iron) that exhibit potentially interfering spectral lines, or excess phthalate esters or elemental sulfur that interferes with pesticide or PCB analysis. These conditions are designed to mimic problems that would occur in analyzing routine field samples.

(3) PE samples should be designed to evaluate the entire analytical process. Specific modifications of the composition of PE samples provide additional checks of specific procedures. For example, semivolatile PE samples should contain acid, base, and neutral extractable over the full retention time range. However, the addition of isomeric pairs to organic PE samples will check GC resolution; the addition of phthalates to pesticide PE samples will test extract cleanup methods; the addition of oil to soil PE samples will verify whether gel permeation chromatographic cleanup was performed as contract required; and the use of potassium ferricyanide, instead of potassium cyanide, to prepare aqueous cyanide PE samples will check whether distillation was conducted. Various other analytes may be added to gauge instrument performance, such as addition of chloromethane to volatile PE samples to check for correct purge flow, addition of di-n-octyl phthalate to semivolatile PE samples to determine if the GC/MS transfer line temperature was set too low, use of specific xylene isomers to indicate if proper standards and response factors were used to set up instrument criteria, etc.

(4) Certain groups of compounds should not be combined since they will react together. For example, semivolatile acids (phenols) should not be combined with bases (anilines), because these compounds will react with each other causing subsequent loss of analytes. Silver and low to medium levels of chloride are incompatible and should not be mixed. Certain compounds may not even be compatible with some instruments and should not be used. For example, it is difficult to use GFAA to analyze a PE sample with a high concentration of chloride because of analyte signal suppression.

G-3. Preparation of PE Samples. PE samples can be prepared either by spiking known amounts of analytes into a well defined homogeneous matrix or by defining well homogenized real world samples. The USACE PE samples can generally be categorized into two groups based on preparation methods: fortified PE samples and "real world" PE samples. The fortified PE samples are prepared by spiking high purity reagent water or control solid materials with solvated target analytes of high purity. Fortified PE samples cost less to prepare and allow qualitative and quantitative variations in the compositions of final PE samples. Real world PE samples are usually soil or sediment collected from contaminated sites, which are dried, ground, mixed, and analyzed prior to use. Real world PE samples are used for validation of laboratory performance in soil analysis for two reasons: (a) it is very difficult to prepare an absolutely homogeneous sample that can then be subsampled for PE samples and (b) a spiked sample can never truly represent the weathering and complexities

of a naturally contaminated matrix. Because the constituents are integrated into the matrices as naturally as possible, the real world PE samples present special analytical challenges of matrix interferences. The USACE is continually seeking suitable real world samples that represent typical environmental samples and contain a broad spectrum of target analytes at adequate concentrations.

a. General Preparation Procedure. Regardless of the type of PE samples, the general USACE procedure for preparing PE samples is outlined below.

(1) Determine matrix type, analytical method, and instrumentation.

(2) Calculate the amount of PE samples needed by volume or weight.

(3) Select analytes, interferences, solvents, and preservatives.

(4) Decide on the concentration of each component.

(5) Select stock materials and calculate appropriate amounts to add.

(6) Write step-by-step instructions (i.e., standard operating procedures).

(7) Perform an error analysis and determine performance requirements.

(8) Obtain materials.

(9) Prepare the PE sample.

(l0) Verify the concentration of each component in the PE samples.

(11) Verify PE samples by multi-laboratory referee analyses.

(12) Establish the performance acceptance limits of PE samples.

When real world materials are used for preparation of fortified or non-fortified PE samples, additional intra- and interlaboratory analyses are needed to verify the compositions of the real world materials. Any indigenous levels of analytes and interferents that are present in the real world materials must be

accurately determined. Depending on the levels and types of analytes and interferents, the real world materials may be used for preparation of fortified or non-fortified PE samples.

b. Starting Materials and Stock Solutions. Starting materials and stock solutions must fulfill several criteria in order to be suitable for preparation of PE samples. All critical information about starting materials and stock solutions should be recorded in logbooks such that the PE samples are traceable to NIST or other reliable reference materials.

(1) Purity is the first requirement, especially if the final sample's true values are going to be based on the material added to the sample. Only chemical sources of known high quality will be used for PE sample preparation. The purity of all reagents, acids, and solvents should be checked prior to use. Purity is not as much of a factor if the PE sample is going to be characterized with consensus values from reputable laboratories.

(a) For inorganic PE samples, contaminant levels should be in the low ppm range if the PE sample is to contain only one analyte. Higher purity (low ppb range) starting materials should be used if multianalyte PE samples are prepared (to avoid contamination) or if sensitive instrumentation is used. The amount of target analytes in the starting material should be certified to within ±0.5 percent for most cases. Materials that are sold without certified purity information should not be used. Individual metal solutions are either purchased from NIST or from vendors whose materials are traceable to NIST.

(b) For organic PE samples, only the highest purity solvent should be used. Purge-and-trap grade methanol is necessary for preparation of volatile spiking solution because lower grades frequently contain toluene and xylene as impurities. Standards for use in preparing organic PE samples may be purchased neat, as single component solutions, or as multiple standard mixes from reliable vendors.

(2) Stability and chemical compatibility are other important criteria for starting materials and stock solutions. Specific reagents for each analyte are selected on the basis of availability and chemical characteristics, such as stability and reactivity. An expiration date must be specified for all prepared materials.

(3) For solid PE samples, unless the entire sample is to be analyzed, homogeneity is one of the most important factors to be considered. Natural solid matrices, such as soil or sediment, should always be dried, ground, sieved, and mixed thoroughly

prior to spiking. For solid PE samples, the smallest sample aliquot that will provide reproducible analysis results could be estimated with Pierre Gy's sampling theory and confirmed by replicate analyses. (See F. F. Pitard, Pierre Gy's Sampling Theory and Sampling Practice, 2 volumes, 1989, CRC press, Inc., Boca Raton, Florida.) For liquid PE samples, homogeneity is inherent unless adhesion of analytes to the container wall or multiple phases are present. Normally, multiple phase PE samples should be avoided because sampling errors may overwhelm all other errors, thus limiting a study's usefulness.

(4) Starting materials and stock solutions should also be obtained at appropriate concentration levels to minimize the amounts required and remain in the realm of accurate laboratory ware measurements. For example, weights of solid materials should be between 0.1 and 500g and volumes of liquids should be between 50 µL and 500 mL. Avoid dilutions that would require odd sizes of volumetric ware. If several levels for a given analyte are available, the more concentrated solution should be chosen to minimize potential contamination from stock materials.

(5) When commercially available reference materials are utilized, only certified reference materials (CRMS) should be used for PE sample preparation. The term "certified" means that documentation supports the reference material. Using a CRM may assure the capability of the measurement system to determine the analyte in the sample. NIST is the most widely used supplier of CRMs. However, using NIST values for solid materials can lead to comparison errors on data obtained using USEPA inorganic and organic extraction methods. NIST expresses CRM values as "total" concentrations but many USEPA methods use values based upon "extractable" concentrations. Because of this, certified NIST values for solid CRMs usually cannot be used. Using NIST values do not pose a problem in performance evaluation of laboratories for water analysis, where "total" extractables approximate true values. The acceptance criteria of the USACE PE samples should be established based on extractable concentrations.

(6) Each lot of standards used for preparation or verification of PE samples should be analyzed by the PE sample suppliers to verify its concentration prior to use. Reanalysis of the standards is required periodically to verify stability, according to a schedule optimized for each standard. The probable error of each step in the preparation of PE samples should be evaluated and used to assess the overall probable error and implications on confidence levels. Details analysis and validation procedures should be documented and maintained in-house per appropriate USEPA and USACE guidance for legal defensibility.

(7) All PE sample suppliers must actively participate in State and/or Federal proficiency testing programs and provide the USACE Laboratory Validation Committee with their most recent results for review on a quarterly basis.

(8) Safety must also be considered. Material Safety Data Sheets (MSDS) should be obtained with each material and should be read and followed carefully. As good laboratory practice, handle and weigh out all toxic materials in a well ventilated fume hood.

c. Calculations. The calculations involved in preparing fortified PE samples are relatively simple. It is best to start with the final volume or weight of PE samples to be prepared and work backward to determine the amounts of individual analyte stocks needed. Care should be taken in calculations with reagent purity values, gravimetric factors, dilution and concentration factors, significant figures, and unit manipulations. The most common types of concentration units are weight/weight for solid PE samples and weight/volume for liquid PE samples. Reagent bottles should be labeled with specific units, such as µg/mL or µg/kg. Ambiguous units such as ppb or ppm should not be used because these units do not differentiate between weight/weight or weight/volume. Depending on when they are noticed, calculation errors can have serious ramifications when PE samples are involved. Therefore, it is best to double check all calculations leading up to the final concentrations of PE samples before the samples are prepared. Good laboratory practice should include a second person's review of the calculation.

d. Standard Operating Procedures. In order to prepare reliable PE samples, adherence to prescribed preparation procedures is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of standard operating procedures (SOPs). This is especially true for preparing PE samples that will be used to determine laboratory performance. An SOP is defined as a written, narrative, and stepwise description of laboratory operating procedures including examples of laboratory documentation. An SOP should accurately describe the actual procedures used in the laboratory to ensure that reproducible results can be achieved by following the SOP. The SOP for PE sample preparation should be prepared as part of the planning process and should be at or near completion before PE sample preparation work begins. The SOP should be reviewed before preparing actual PE samples. Ambiguous statements or terminology like "air dried at ambient temperature" or "1:10 dilution" should not be used when "18 to 22°C" or "ten-fold dilution" is meant. The "l:l0" may be confused with one part concentrate diluted with ten parts of diluent, which is really an 11-fold dilution. As a

PE sample is prepared, changes and observations are documented so that significant information will be available if needed later.

e. Fortified PE samples. Fortified PE samples are usually prepared with spiking techniques. Either large volumes or small units of fortified PE samples of any matrix can be prepared by spiking analytes of choice at selected concentrations. Normally, it is preferable to spike a large volume and create individual units from it, unless there is a major concern of analyte loss to container wall. PE samples should be prepared by designated, experienced senior chemists to improve batch-to-batch reproducibility and reliability.

(1) Fortified aqueous PE samples. Aqueous PE samples should be prepared on the day of shipment, usually early in the week to allow adequate preparation time for the contract laboratory to perform digestions, extractions, cleanups, etc. before the weekend.

(a) Reagent water which is free of contaminants at the method detection limits is normally used for PE samples preparation. Reagent water can be prepared by passing tap water through a reverse osmosis water system and then through an ultraviolet and activated carbon cartridge or equivalent system to produce analyte-free reagent water. The quality of reagent water should be monitored and documented on a routine basis.

(b) ASTM class A pipets and calibrated microsyringes should be used for delivering and spiking during PE sample preparation. Variable pipetters can be used if they are verified to be in calibration; however, glass pipets are preferred. Sample containers (high density polyethylene for inorganic and amber glass for organics) are purchased as “certified pre–cleaned” according to USEPA standards.

(c) Gravimetric measurements can be used on less volatile liquids, such as water. If weights are used for calculations, density of the liquid also must be determined so that weight-to-volume units can be calculated. Volatile liquids have to be prepared by volume, using minimal headspace and minimal exposure to the atmosphere. Diluents should already contain any required preservatives so that final volumes are not altered by preservation.

(d) Full-volume PE samples of one liter are normally used for aqueous organic PE samples except volatiles which are 40 mL. A trip blank should always accompany volatile samples for each different analytical method. Volumes for the inorganic analyses

vary from 200 to 1,000 mL, depending on the target analytes and analytical methods.

(e) Multiple sets of spiking solutions are maintained with varying constituents and concentrations to avoid sending the same PE samples to the same laboratory twice or to affiliated laboratories of the same parent organization.

(f) Organic spiking solutions (except volatiles) are prepared by dilution of reference stocks. Records and certificates of all stock solutions and dilutions are maintained in standard logbooks. Aqueous PE samples for organics (except volatiles) are spiked individually into the sample bottles since the entire sample is used for analysis.

(g) Volatile spikes are purchased as mixed solutions designed for laboratory evaluations and certificates are maintained in laboratory files. Aqueous volatile PE samples are prepared in a volumetric flask with sufficient volume to prepare the day's shipment and then transferred to 40-mL VOA vials for submission to contract laboratories.

(h) Aqueous PE samples for inorganic are prepared in volumetric flasks and aliquots are then transferred to individual sample bottles for shipment.

(i) All PE samples should be properly preserved per method requirements. PE samples with critical holding times should be shipped immediately after preparation to allow adequate time for the contract laboratory to prepare and analyze the PE samples.

(j) Only one aliquot of each aqueous PE sample will be sent to each contract laboratory. Because the aqueous PE sample is prepared with reagent water, the laboratory will be instructed to perform method-specific QC analyses with its own reagent water.

(2) Fortified solid PE samples. Various types of soil samples are collected and prepared to serve as a solid matrix. The soil could be clayey, silty, or sandy with different alkalinity, organic, and metal contents. However, care must be taken to avoid using soils that are very reactive to acids or other reagents used for sample preparation. Except for volatile organics, solid PE samples can be prepared by solid or liquid addition. Due to the high volatility of volatile organics, soil PE samples for volatile organics can be prepared by a vapor fortification technique. (See A. D. Hewitt, P. H. Miyares, D. C. Leggett, and T. F. Jenkins, *Comparison of Analytical Methods for Determination of Volatile Organic Compounds in Soils,* Environ.

Sci. Technol., **1992,** 26, 1932.) The USACE is looking into this technique.

(a) After removal of extraneous materials such as rocks, sticks, etc., the soil will be air dried, ground, and mixed with mills or grinders. Mixing mills or grinders capable of grinding and mixing large volumes of soil (up to 1 gallon) per batch are preferred. Separate batches can be combined, sieved to pass 150 mesh (<100 µm), and blended in a larger container. A 1 g sample aliquot should have a relative sampling error of about two percent at this particle size if the total batch is 100 g. The grinding and mixing times are established by short interval runs and examining the particle size and physical consistency of the soil. The homogenized soils should be stored in a cool, dark, and dry place. If needed, the potential influence of laboratory relative humidity can be removed by conditioning an air dried, sieved, and thoroughly mixed soil with $CaSO_4$ desiccation.

(b) The concentrations of any target analytes and interferents in the homogenized soil should be thoroughly and accurately determined. It is preferred that the concentrations of natural contaminants in the soil are below method detection limits or relatively low compared with the concentrations of spiked analytes.

(c) The spiking can be done by solid addition. The two solids that are to be mixed should be reduced to approximately the same small particle size (at least <150 mesh) before mixing. This reduction leads to easier blending and components will be less prone to segregate during storage and transit. Relative amounts of each component should not be extreme because it is very difficult to evenly distribute small amounts of one material within large amounts of another. If extremes in relative amounts cannot be avoided, the blending can be done in stages. That is a small quantity of the main component can be spiked and blended, then mixed and blended with the rest of the main component.

(d) The spiking can also be done by liquid addition. Analyte solutions (except volatiles) are sprayed over the homogenized soil in small increments. After vaporization of the solvent, mix the soil thoroughly and spray again. The above process is repeated until all analyte solutions are used up. Rinse the spray bottles with more solvent and spray over the soil again to ensure all target analytes are quantitatively transferred to the homogenized soil. When liquid spikes are used to modify solid matrix, the solvent must be removed by drying. Since local deposits of analyte can be left after drying, thorough mixing after drying is crucial. Mixing can be improved by using enough solvent to form a runny paste or mud. The paste

is occasionally stirred while drying and, when completely dry, must be re-ground and blended.

(e) Prior to packing the homogenized bulk PE samples into small units for use, the homogeneity of the PE samples should be reassessed to determine the minimum subsample size for each target analyte. A general approach is first selecting aliquots from the homogenized bulk PE samples and measuring the concentrations of target analytes. A two-way analysis of variance is then carried out by comparing results from aliquots within subsamples with those between subsamples. If the means do not differ significantly at 95 percent confident level, the bulk PE samples is considered homogeneous. Homogeneity could further be assessed by analyzing aliquots from certain percentage of the individual subsamples at a variability of, say, five percent relative standard deviation. Not all target analytes need to be tested, and a single measurement technique may be used, However, the selected analytes and technique should include be representative and conclusive.

f. Real world PE samples. Real world soil and sediment PE samples are collected from locations that have significant levels of numerous contaminants of concern. Numerous low levels of analytes that may cause problems in assessing laboratory performance should be avoided. Large volumes of materials are collected and shipped to USACE PE sample suppliers for processing. The materials are mixed thoroughly and extraneous materials are removed. Approximately five to ten gallons of materials are air dried to three to four percent moisture. The materials are then ground in a large volume grinder to pass through a 0.5-mm sieve. Materials are mixed and passed through the grinder a second time to desired particle size (i.e., 45-75 µm) and stored at 4°C in the dark.

(1) Extraneous materials such as rocks, sticks, etc. should first be removed from the solid materials. The materials are then air dried, ground, and mixed with mills or grinders. Mixing mills or grinders capable of grinding and mixing large volumes of soil (up to one gallon) per batch are preferred. Separate batches can be combined, sieved to pass 150 mesh (<100 µm), and blended in a larger container. A l-g sample aliquot should have a relative sampling error of about two percent at this particle size if the total batch is 100 g. The grinding and mixing times are established by short interval runs and examining the particle size and physical consistency of the soil. The homogenized soils should be stored in a cool, dark, and dry place.

(2) The content of natural PE samples can be altered by spiking to fulfill special needs. The same spiking technique as

previously described can be used. After spiking and drying, an additional blending step is necessary.

(3) Most real world PE samples used by the USACE are very stable. The stability of PE samples should be studied and monitored by analyzing random PE samples of each production batch according to a proper kinetics-based schedule. Some real world solid PE samples have been used as long as five years with no significant changes in concentrations in metals and semivolatile organics.

(4) Multiple sets of real world PE samples with different constituents and/or concentrations should be available and ready for use to avoid sending the same PE samples to the same laboratory twice or to affiliated laboratories belonging to the same parent organization.

G-4 . Handling. All PE samples should be handled and stored with extreme care to ensure the sample stability, integrity, purity, and authenticity.

a. Generally, containers are selected for their inertness to their contents and their ability to prevent sample loss. Samples for organic analyses are stored in amber glass to avoid the plasticizers and organics found in plastic containers. Amber glass is recommended since some analytes are ultraviolet (UV) light sensitive. Plastic bottles are suggested for metals to avoid leaching of trace impurities from glass containers. Bottle caps should be tightly closed to avoid leakage during shipment.

b. A PE sample must maintain its stability. If values change significantly before the sample can be analyzed, the PE sample is worthless. Short holding times are common practice for unstable species such as mercury, cyanide, and volatile organics. In addition to observance of holding times, preservatives and refrigeration are used to retard sample degradation. In addition, PE samples for cyanides and organic analysis should be kept in the dark to avoid degradation by UV light. PE samples must be preserved according to the required analysis. For example, aqueous PE samples for volatile organics should only be acid preserved depending on the analytical method to be used. Normally, all PE samples should be preserved and stored at 4°C in the dark to retard degradation processes. Analytes requiring different preservatives cannot be grouped together in the same sample container. Bottles for volatile organic samples should be completely filled to retard loss of volatiles.

c. All PE samples should be appropriately preserved, packed, and shipped by overnight express delivery service to commercial

laboratories according to USEPA, USACE, and DOT regulations and guidelines. Chain-of-custody form should be used for all PE samples.

d. PE samples are usually provided as single blind, although double blind are occasionally provided. When double blind PE samples are shipped, special precautions on labeling and packing should be taken to make the PE samples indistinguishable from regular field samples. The packaging and container must be identical with that used by field personnel sending the same sample type to the contract laboratory. Special arrangements, such as arranging for a "consulting firm" to contract with the laboratory to be evaluated or using the same bottles, labels, chain-of-custody forms, sample coolers, shipping location, etc. as used in the field, will be made to simulate actual environmental samples.

G-5 . Validation. Because PE samples may be used to disqualify a laboratory's performance or to challenge a laboratory's results, the analyte concentrations in PE samples must be validated with legal defensibility prior to use. All PE samples should be meticulously tested internally and externally to determine the true values and statistically establish the acceptance limits prior to use.

a. Two approaches, the consensus interlaboratory approach and the multiple techniques/definitive techniques approach, are usually used for validation of PE samples. In the multiple techniques/definitive techniques approach, the PE samples are tested by independent techniques with different measurement principles and by definitive techniques whose measurement principles are based on or are directly traceable to physical measurements such as weight and radioactive decay to reduce random or systematic variabilities of chemical measurement techniques. Nearly all of NIST's environmental standard reference materials are certified by this approach. However, there are few definitive techniques and none for organics. The majority of USACE PE samples are validated by interlaboratory consensus in performing a single methodology where the mean value approximates the true value. When there is no definitive technique available to check, the mean value obtained by interlaboratory consensus could be nothing more than a statistical average. Therefore, only reliable laboratories of high performance should be used for validation of PE samples.

b. Fortified PE samples. Depending on the type of fortified PE samples, the true concentrations and acceptance limits of each target analyte can be determined by three different methods:

referee laboratory analysis, error propagation analysis, or performance data estimation.

(1) Fortified aqueous PE samples. The true values and acceptance limits of fortified aqueous samples can be determined by all three methods. Normally, consensus values by referee laboratory analysis should be used. If the other two methods are used, a triplicate for each batch of PE samples should be analyzed by the USACE PE sample supplier to check the accuracy and precision.

(a) Referee laboratory analysis. For analytes with critical holding times, PE samples should be sent to the contract laboratory being tested at the same time they are sent to a minimum of four referee laboratories. The uncertainty of the mean value based on referee laboratory's results decreases with increasing number of laboratories. Therefore, it is preferred to have more laboratories (e.g., 12 referee laboratories) to improve the confidence level of the mean value. The determined concentration from each independent referee laboratory should be within ten percent of prepared concentrations or the causes of excess high/low recovery should be investigated. Consensus values within 95 percent confidence level from the referee laboratories can then be used for evaluation of the contract laboratories. Stable analytes can be characterized before shipment to contract laboratory.

(b) Error propagation analysis. If a material is not characterized (i.e., round-robin data not available), acceptance limits can be calculated. Sometimes calculation is the only way to determine the true values and acceptance limits. The calculation for the expected or true concentration for each analyte in fortified aqueous PE samples is very accurate and straightforward. The acceptance limits of fortified aqueous PE samples can be determined through an error analysis of the steps caused by analytical sample preparation and by sample analysis. Error propagation rules are used as guidelines to estimate determinate and indeterminate errors that should be experienced by the laboratory being evaluated. The indeterminate errors are always judgement calls and should be based on experience. The Factor-2 criterion (i.e., indeterminate errors = 2 x determinate errors) can be used as a good approximation for inclusion of indeterminate errors. The result is a relative error that can be multiplied by the expected target values for each analyte to get acceptance limits. If biases are known to exist but cannot be reliably accounted for, the PE sample may have to be characterized by several reputable laboratories and consensus values used for acceptance windows.

(c) Performance data estimation. The performance data for a number of USEPA methods, based on multiple laboratories testing results, are published in the methods. The acceptance limits for each analyte can therefore be estimated by the calculated target values and the precision formula. The estimated acceptance limits usually are very reliable.

(2) Fortified solid PE samples. A difficulty with fortified solid PE samples is matrix interaction with the analytes. Analyte accuracy of the spiking solution may be very well known, but that accuracy is lost after spiking, when the analytes react with the solid matrix. For example, adsorption of metal ions in solution by the clay matrix of a soil is a well known phenomenon. Since most USEPA extractions are designed to remove leachable rather than true totals, all the analyte that was introduced by spiking may or may not be removable by the sample preparation method. The result is a reduced recovery for affected analytes. Analytes like antimony, silver, and selenium are especially susceptible. To complicate matters further, if indigenous levels of analytes are present in the solid matrix, their leachable levels must be known before total levels or percent recovery can be calculated accurately. Given these difficulties, a fortified solid PE sample is best characterized by consensus rather than by calculation or estimation of analyte levels from individual components.

c. Real world PE samples. For real world PE samples, the true values of target analytes are usually unknown. The mean of reported values from a round-robin testing is usually considered a "consensus" value and would be used as the true analytical value. Confidence intervals for the consensus values of target analytes are based on reported values using standard population statistics. The initial acceptance limits for PE samples are statistically determined by consensus values of the participating laboratories which include reputable government and contract laboratories. The acceptance limits for each target analyte will be established statistically at 95 percent confidence level. The acceptance limits for each target analyte are matrix- and method-specific.

(1) Any method of evaluating real world PE samples may present problems of accuracy that depend upon the amount of data used to set acceptance limits. Thus, it would be best to send split PE samples to a minimum of four round-robin testing laboratories. Although a consensus value resulting from a small number of determinations may have significant uncertainties, the consensus value from the round-robin testing laboratories should be a better estimate of true value than any single measurement.

(2) A round-robin analysis is used to certify analytes of interest. In order to ensure the integrity of PE samples, one or two PE samples should periodically be resubmitted to the referee laboratories to evaluate any possible degradation or trends in the analyte concentrations. This information is also used to evaluate possible extension of the useful life of real world PE samples.

d. The pool of PE sample results produced by all contract laboratories should be carefully analyzed on a regular basis. The mean values and the associated uncertainties of target analytes should always be documented. The program-wide statistical results for PE sample analyses by contract laboratories should also be used to adjust the acceptance limits in order to observe the relative performance of each laboratory using a given protocol against its peers. The USACE may adjust the acceptance limits on any given PE sample to compensate for unanticipated difficulties with a particular sample or analysis.

e. All PE samples must be analyzed with the same methodology (i.e., USEPA SW-846 of the most recently promulgated revisions) by both the contract laboratories and the referee laboratories. Deviations from the standard methods will make the data noncomparable. The results of all PE sample analyses should be used to develop control charts displaying the true concentration and ranges of recovery and bias for each target analyte.

APPENDIX H

GUIDELINES AND CRITERIA
FOR
ON-SITE INSPECTION
OF
COMMERCIAL ANALYTICAL CHEMISTRY LABORATORIES

GUIDELINES AND CRITERIA FOR ON-SITE INSPECTION OF COMMERCIAL ANALYTICAL CHEMISTRY LABORATORIES

H-1. On-Site Laboratory Inspection Procedures. This document outlines the procedures to be used by the USACE inspectors to conduct an on-site inspection and evaluation of a commercial laboratory. On-site laboratory inspections are carried out to monitor a commercial laboratory's ability to meet selected terms and conditions specified in a USACE HTRW contract and to identify laboratory problems that adversely impact performance. The frequency of on-site inspection is dictated by a commercial laboratory's performance. An on-site inspection generally takes eight hours and normally consists of three parts: entrance interview, laboratory tour, and exit interview. Prior to the inspection, the inspectors shall thoroughly review all project- and laboratory-specific documents. The Pre-Inspection Checklist shown in Figure H-1 can be used as a guidance for preparation of on-site inspection.

a. Entrance Interview. The entrance interview will be held with the laboratory management personnel, including laboratory director/managers, QA officer, and project personnel, to discuss the upcoming USACE projects, USACE Chemical Data Quality Management (CDQM) requirements, PE sample results, USACE review comments on laboratory quality management manual (LQMM), and laboratory's previous performance on USACE projects, if applicable. A copy of written comments on the LQMM should be presented to the laboratory during the entrance interview. The Entrance Interview Checklist shown in Figure H-2 can be used a guidance.

b. Laboratory Tour. A tour of the commercial laboratory will follow to examine the laboratory facilities, instrumentation, operation, maintenance, documentation, safety, waste compliance, etc. The laboratory tour will emphasize on two separate aspects: Quality Assurance Evaluation and Evidentiary Audit. The questionnaire and checklist presented in Appendices E and I should be used during the laboratory tour.

(1) Quality Assurance Evaluation: The inspectors shall inspect a commercial laboratory's facilities to verify the adequacy and maintenance of instrumentation, the continuity of personnel meeting experience or education requirements, and the acceptable performance of analytical and QC procedures. The items to be monitored will include, but not be limited to, the following items:

PRE-INSPECTION CHECKLIST

1. Gather all appropriate laboratory information from files.

   a. Preliminary questionnaire.

   b. Laboratory's LQMM.

   c. PE sample results and evaluation reports.

   d. Chemical quality assurance reports (CQARs) on past projects.

2. Gather and review project information.

   a. Project summary based on specifications, scope of work, work plans, chemical data acquisition plan, etc.

   b. Analytical parameters and number of samples.

   c. Project data quality objectives (DQOs).

3. Contact laboratory to set up audit date.

   a. Get directions to laboratory by FAX.

   b. Suggest tentative on-site inspection date.

   c. Briefly review inspection procedures.

4. Contact USACE TM/CORs and district chemists.

   a. Get most current project information.

   b. Extend an invitation for them to attend the inspection.

5. Contact laboratory to confirm inspection date and make travel arrangements.

6. Review laboratory’s LQMM, questionnaire, and any other qualification documents. Generate written comments.

7. Review laboratory's PE sample results.

   a. Review and confirm all PE sample results.

Figure H-1 Pre-Inspection Checklist

b. Gather any missing information.

c. Update database with current information.

d. Prepare a summary of PE sample status for review at the laboratory.

8. Review the CQARs on past USACE projects that laboratory previously worked on.

a. Check with QA laboratory(s) for any detail or missing information.

b. Extend an invitation for QA laboratory(s) to attend the inspection.

9. Prepare a list of problem areas in laboratory.

a. Based on PE sample results.

b. Based on past performance on USACE projects.

10. Gather general information and forms for distribution at the laboratory.

a. ER 1110-1-263.

b. Copy of laboratory evaluation request(s).

c. "On-site Inspection Summary" format.

d. Entrance interview checklist.

e. Laboratory inspection checklist.

f. Exit interview checklist.

g. Cooler receipt checklist.

h. USACE minimum data reporting requirements.

i. Sample CQAR and data comparison table.

j. Your business card.

Figure H-1 Pre-Inspection Checklist (continued)

ENTRANCE INTERVIEW CHECKLIST

1. Personnel introductions.

   a. Give background of inspectors and USACE HTRW MCX.

   b. Pass out/gather business cards.

   c. Pass out and have everyone sign "On-site Inspection Summary" format.

2. Validation process.

   a. Describe USACE laboratory validation process.

      - Step 1: Preliminary review and screening based on qualification submittals.

      - Step 2: Performance evaluation based on PE sample analysis.

      - Step 3: On-site inspection.

   b. Explain the approval process after on-site inspection.

   c. Emphasize that laboratory validation is a parameter, matrix, and method-specific approval.

   d. A project-specific evaluation is needed for each new project.

3. Project information.

   a. Is laboratory aware of the project?

      - Is CDAP available? Laboratory should get copies of CDAPs for upcoming projects.

      - Are DQOs available?

      - Make available to laboratory a copy of evaluation request(s).

   b. Describe projects.

Figure H-2 Entrance Interview Checklist

4. USACE QA Program.

   a. Pass out a copy of ER 1110-1-263.

      - Describe USACE QA program and special features.

      - It is consistent and complies with Federal and State regulations.

   b. Describe field split QA sample program

   c. Describe government QA Laboratory and its role:

      - Examines incoming field samples against CDAP. Pass out copy of "Cooler Receipt Checklist". Notify TM/CORs immediately if errors noted.

      - Analyzes QA samples. QA Laboratory can be used as a resource to answer questions.

      - Reviews project laboratory's data. Pass out a copy of "USACE Minimum Data Reporting Requirements."

      - Generates CQAR. Describe this report. Pass out a copy of sample "Data Comparison Table" and describe the key elements that are focused on.)

5. Review comments on laboratory's LQMM.

   a. Pass out and review comments.

   b. Discuss corrective actions, if needed.

6. Status of PE sample results.

   a. Summarize current status of all PE samples.

   b. Discuss deficiencies and corrective actions, if needed.

7. Laboratory's performance on past USACE projects.

   a. Discuss data quality based on precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS).

   b. Discuss corrective actions, if needed.

Figure H-2 Entrance Interview Checklist (continued)

(a) Size and appearance of the facility.

(b) Quantity, age, availability, scheduled maintenance, and performance of instrumentation.

(c) Availability, appropriateness, and utilization of SOPs.

(d) Staff qualifications, experience, and personnel training programs.

(e) Reagents, standards, and sample storage facilities.

(f) Standard preparation logbooks and traceability.

(g) Sample analysis, raw data, bench sheets, and analytical logbooks maintenance and review.

(h) Data package review and data management procedures.

(2) Evidentiary Audit: The inspectors conducts an evidentiary audit to determine if the laboratory's QA/QC policies and SOPS are implemented to warrant required data quality and legal defensibility. The evidentiary audit is comprised of the following three activities:

(a) Procedural Audit: The procedural audit consists of review and examination of actual operating procedures and accompanying documentation for the following laboratory operations: sample receiving, storage, identification, security, tracking (from receipt to completion of analysis), and analytical project file organization and assembly.

(b) Written SOPs Audit: The written SOPs audit consists of review and examination of the written SOPs to determine if they are accurate and complete for the following laboratory operations: sample receiving, storage, identification, security, tracking (from receipt to completion of analysis), and analytical project file organization and assembly.

(c) Analytical Project File Audit: The analytical project file audit consists of review and examination of the analytical project file documentation. The inspectors shall review the files to determine:

- the accuracy of the document inventory,

- the completeness of the file,

the traceability of sample activity,

the identification of activity recorded on the documents, and

the error correction methods.

c. Exit Interview. At the conclusion of the laboratory tour, the inspectors discuss their findings and recommendations for any corrective actions with the laboratory management staff during an exit interview. A commercial laboratory shall prepare a written report regarding the corrective actions implemented or to be implemented with schedule for completion to the Committee for review and approval. The written report must provide detail on corrective actions for all deficiencies discussed during the exit interview and must be sent within ten working days from the on-site inspection. The Exit Interview Checklist shown in Figure H-3 can be used as guidance.

H-2 . Guidance and Criteria for Sample Management, Data Management, Document Control, and Standard Operating Procedure

a. Sample Management. Sample management procedures are defined as procedures specifying the sample receiving, log-in, storage, and disposal. A sample is a physical evidence collected from a facility or from the environment. Controlling evidence is an essential part of the hazardous waste investigation effort. A commercial laboratory shall establish SOPs to maintain the integrity, authenticity, and legal defensibility of samples from initial receiving to proper disposal. To accomplish this, laboratories are required to develop and implement the following sample identification, chain-of-custody, sample receiving, and sample tracking procedures:

(1) Sample Identification: To assure traceability of the samples while in possession of a laboratory, the laboratory shall have a specified method for maintaining identification of samples throughout the laboratory. Each sample and sample preparation container shall be labeled with a USACE field sample ID number or a unique laboratory identifier. If a unique laboratory identifier is used, it shall be cross-referenced to the USACE field sample ID number.

(2) Chain-of-Custody Procedures: The custody of USACE samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. A commercial laboratory shall have procedures ensuring that USACE sample custody is maintained and documented. A sample is under custody if:

EXIT INTERVIEW CHECKLIST

1. Express gratitude for laboratory's efforts, cooperation and time

2. Present inspection findings

   a. Strong and weak areas.

   b. Deficiencies and corrective actions.

   c. Recommendations for improvements.

3. Complete the "Inspection Summary" sheets

   a. Ask laboratory director/manager to review and sign summary sheet.

   b. Pass out a copy of signed summary sheet.

   b. Request written responses within ten working days.

4. Invite questions and comments

5. Meeting adjourned

Figure H-3 Exit Interview Checklist

- it is in your possession,

- it is in your view after being in your possession,

- it was in your possession and you locked it up, or

- it is in a designated secure area that is accessible only to authorized personnel.

(3) Sample Receiving Procedures:

(a) A commercial laboratory must designate a sample custodian responsible for receiving all samples. A representative should also be designated to receive samples in the event that the sample custodian is not available. The sample custodian must inspect the condition of the shipping containers, sample bottles, and the custody seals (intact/not intact) upon receipt. The sample custodian shall also check for the presence or absence of the following documents accompanying each sample shipment:

- Airbills or airbill stickers

- Chain-of-Custody forms

- Sample labels

- Sample tags (if required for a project)

(b) The sample custodian must sign and date all forms (e.g., custody records, packing lists, and airbills) accompanying the samples at the time of sample receipt. A commercial laboratory must immediately contact the prime contractor and/or USACE TM/COR to resolve any discrepancies and problems such as absent documents, conflicting information, broken custody seals, and unsatisfactory sample condition (e.g., leaking sample bottle, improper preservation, etc.) A commercial laboratory shall record the resolution of discrepancies and problems on a phone conversation log. All records and logs shall become part of the project file records.

(c) The following information shall be recorded in sample logbook by the sample custodian or his/her representative as samples are received and inspected:

- Condition of the shipping container

- Presence or absence and condition of custody seals on shipping and/or sample containers

- Custody seal numbers, when present
- Condition of the sample bottles
- Presence or absence of airbills or airbill stickers
- Airbill or airbill sticker numbers, when present
- Presence or absence of packing lists
- Presence or absence of sample tags
- Sample tag identification numbers, when present
- Verification of agreement or non-agreement of information recorded on shipping documents and sample containers
- Problems or discrepancies
- Resolutions for problems or discrepancies

(d) The Cooler Receipt Checklist as shown in Figure H-4 or a similar one is strongly recommended.

(4) Sample Tracking Procedures: A commercial laboratory shall maintain records documenting all phases of sample handling from receipt, analysis, and final sample disposal.

(5) Sample Disposal Procedures: A commercial laboratory shall treat all USACE samples, including residual samples, digested or extracted samples, samples with analyses cancelled, sample containers, waste generated during sample preparation or analysis, etc., as potential hazardous and toxic material or substance until proven otherwise. SOPs for disposal USACE samples shall comply with all Federal and State regulations such that the USACE will not be legally liable for improper sample or waste disposal by the laboratory.

b. Data Management. Data management procedures are defined as procedures specifying the acquisition or entry, update, correction, deletion, storage, and security of computer readable data and files. These procedures should be in written form and contain a clear definition for all databases and files used to generate or submit deliverables. Key areas of concern include: system organization (including personnel and security), documentation operations, and traceability. The system should prevent entry of incorrect or out-of-range data and alert data entry personnel of errors through a multilevel review process.

LIMS #:_____ Chain-of-Custody No: _____ Date received: _____

Project: _____

USE OTHER SIDE OF THIS FORMAT TO NOTE DETAILS CONCERNING CHECK-IN PROBLEMS.

A. PRELIMINARY EXAMINATION PHASE: Date cooler was opened: _____

by (print): _____ (sign): _____

1. Did cooler come with a shipping slip (airbill, etc.)? ..... YES NO

If YES, enter carrier name & airbill nunber here: _____

2. Here custody seals on outside of cooler? ..... YES NO

How many & where: _____, seal date: _____, seal name: _____

3. Were custody seals unbroken and intact at the date and time of arrival? ..... YES NO

4. Did you screen samples for radioactivity using a Geiger Counter ..... YES NO

5. Were custody papers sealed in a plastic bag & taped inside to the lid? ..... YES NO

6. Were custody papers filled out properly (ink, signed, etc.)? ..... YES NO

7. Did you sign custody papers in the appropriate place? ..... YES NO

8. Was project identifiable from custody papers? ..... YES NO
If YES, enter project name at the top of this form.

9. If required, was enough ice used? ..... Type of ice: _____ ..... YES NO

10. Have designated person initial here to acknowledge receipt of cooler: _____ (date): _____

B. LOG-IN PHASE: Date samples were logged-in: _____

by (print): _____ (sign): _____

11. Describe type of packing in cooler: _____

12. Were all bottles sealed in separate plastic bags? ..... YES NO

13. Did all bottles arrive unbroken and were labels in good condition? ..... YES NO

14. Were all bottle labels complete (ID, date, time, signature, preservative, etc.)? ..... YES NO

15. Did all bottle labels agree with custody papers? ..... YES NO

16. Were correct containers used for the tests indicated? ..... YES NO

17. Were correct preservatives added to samples? ..... YES NO

18. Was a sufficient amount of sample sent for tests indicated? ..... YES NO

19. Were bubbles absent in VOA samples? If No, list by sample #:..... YES NO

20. Was the USACE Technical Manager called and status discussed? ..... YES NO
If YES, give details on the back of this form.

21. Who was called? _____ By whom? _____ date: _____

Figure H-4 Cooler Receipt Checklist

The record of changes in the form of corrections and updates to data originally generated, submitted, and/or resubmitted must be documented to allow traceability of updates. Documentation must include the following for each change:

- Justification or rationale for the change.

- Initials of the person making the change or changes. Data changes must be implemented and reviewed by a person or group independent of the source generating the deliverable.

- The laboratory manager must approve changes to originally submitted deliverables.

c. Document Control. The goal of a laboratory document control program is to assure that all documents for a specified project will be accounted for when the project is completed. Accountable documents used by commercial laboratories shall include, but not be limited to, logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses. The following document control procedures should be established to assure that all laboratory records are assembled, stored, and ready for delivery to USACE when requested by USACE:

(1) Preprinted Laboratory Forms and Logbooks:

(a) All observations and results recorded by a commercial laboratory but not on preprinted laboratory forms shall be entered into permanent laboratory logbooks. The laboratory shall identify the activity recorded on all laboratory documents that are directly related to the preparation and analysis of USACE samples.

(b) Preprinted laboratory forms shall contain the name of the commercial laboratory and be dated (month/day/year) and signed by the person responsible for performing the activity at the time an activity is performed. Logbook entries shall also be dated, signed, and entered in chronological order. Pages in both bound and unbound logbooks shall be sequentially numbered. Instrument run logs shall be maintained so as to enable a reconstruction of the run sequence of individual instruments.

(c) Corrections to raw data and supporting documents shall be made by drawing a single line through the error and entering the correct information. Corrections and additions to raw data and supporting documents shall be dated and initialed. No information shall be obliterated or rendered unreadable. All

notations shall be recorded in ink. Unused portions of documents shall be crossed out.

(2) Consistency of Documentation: A commercial laboratory should assign a document control officer responsible for the organization and assembly of all project related files. All copies of laboratory documents shall be complete and legible. Before releasing analytical results, the document control officer shall assemble and cross-check the information on sample tags, custody records, laboratory bench sheets, personal and instrument logs, and other relevant deliverables to ensure that data pertaining to each particular sample is consistent throughout the specific USACE project.

(3) Storage of USACE Files: A commercial laboratory shall maintain USACE laboratory documents in a secure location that has a limited access. All documents that are directly related to the preparation and analysis of USACE samples shall be stored and made available to USACE upon request within a contract specified time limit.

d. Specifications for SOPs. In order to obtain reliable results, adherence to prescribed analytical methodology is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of SOPs . An SOP shall be functional: i.e., clear, comprehensive, up-to-date , and sufficiently detailed to permit duplication of results by qualified analysts. All SOPs must accurately reflect actual procedures used in the laboratory, and copies of the written SOPs shall be available to the appropriate laboratory personnel. In addition, all SOPs must be consistent with appropriate, current Federal and/or State regulations and guidelines and with manufacturer's specific instruction manuals.

(1) SOP Format: An SOP is defined as a written document that provides step-by-step description of a laboratory operation, analysis, or actions. The format of an SOP may vary depending upon the kind of activity for which they are prepared; however, at a minimum, the following sections must be included:

(a) Title page

(b) Scope and Application

(c) Definitions

(d) Procedures

(e) QC Criteria

(f) Corrective Action Procedures, including secondary review of information being generated

(g) Documentation description and example forms

(h) Miscellaneous notes and precautions

(i) References

(2) SOPs Required: The followings are the minimum number of SOPs required:

(a) Sample receipt and logging

(b) Chain-of-custody procedures

(c) Sample storage

(d) Prevention of sample contamination

(e) Security for laboratory and samples

(f) Standard purity and preparation

(g) Instrument maintenance records and logbooks

(h) Sample analysis and data control system

(i) Glassware cleaning

(j) Internal review of QA/QC data for each data package

(k) Data reduction and reporting

(l) Laboratory data validation

- Data flow and chain-of-command for data review.
- Procedures for measuring precision and accuracy.
- Control chart generation and utilization.
- Evaluation parameters for identifying systematic errors.
- Internal QA inspection procedures.
- Documentation of problem identification, corrective actions, and resumption of analytical processing.

(m) Data management and handling

e. Handling of Confidential Information. A commercial laboratory conducting work under the USACE HTRW contract may receive USACE-designated confidential information. Confidential information must be handled separately from other documentation. To accomplish this, a commercial laboratory should establish the following procedures for the handling of confidential information:

(1) All confidential documents shall be under the supervision of a designated document control officer. In order to provide document accountability of the confidential documents, each item in a specific USACE project file should be inventoried and assigned a serialized number. All documents relevant to each sample delivery group should be inventoried. This includes: logbook pages, bench sheets, mass spectra, chromatograms, screening records, re-preparation records, re-analysis records, records of failed or attempted analysis, custody records, library research results, etc. The designated document control officer shall be responsible for ensuring that all documents generated are placed in the specified project file for inventory.

(2) Any samples or information received with a request of confidentiality shall be handled as "confidential." A separate locked file shall be maintained to store this information and shall be segregated from other nonconfidential information. Data generated from confidential samples shall be treated as confidential. Upon receipt of confidential information, the document control officer will log these documents into a Confidential Inventory Log. The information will then be available to authorized personnel but only after it has been signed out to that person by the document control officer. The documents shall be returned to the locked file at the end of each working day.

(3) Confidential information may not be reproduced except upon approval by the USACE TM/COR. The document control officer shall enter all copies into the document control system described above. In addition, this information may not be disposed of except upon approval by the USACE TM/COR. The document control officer shall remove and retain the cover page of any confidential information disposed of for one year and shall keep a record on the disposition in a Confidential Inventory Log.

APPENDIX I

CHECKLISTS

FOR

ON-SITE LABORATORY INSPECTIONS

CHECKLISTS FOR ON-SITE LABORATORY INSPECTION

CHARTS I-1 through **I-36** contain checklists on which the adequacy of laboratory organization, facility, equipment, operation, and QA/QC policy and practice shall be checked by the inspector(s) during an on-site inspection. The titles of these checklists are:

CHART I-1	Organization and Personnel
CHART I-2	Facilities
CHART I-3	Equipment
CHART I-4	General QA/QC
CHART I-5	Report Generation
CHART I-6	Field Sampling
CHART I-7	Sample Receipt and Storage
CHART I-8	Sample Preparation for Organic Analysis
CHART I-9	General QA/QC for Organic Analysis by GC
CHART 1-10	Organic Analysis by GC: HVO (8010A)
CHART I-11	Organic Analysis by GC: TPH (Modified 8015)
CHART I-12	Organic Analysis by GC: AVO (8020)
CHART I-13	Organic Analysis by GC: PHENOLS (8040A)
CHART I-14	Organic Analysis by GC: PEST/PCB (8080)
CHART I-15	Organic Analysis by GC: PAH (8100)
CHART I-16	Organic Analysis by GC: HERB (8150A)
CHART I-17	General QA/QC for Organic Analysis by GC/MS
CHART I-18	Organic Analysis by GC/MS: VOA (8240A)
CHART I-19	Organic Analysis by GC/MS: BNA (8270A)
CHART I-20	Organic Analysis by GC/MS: DIOXINS (8280)
CHART I-21	Organic Analysis by HPLC: PAH (8310)
CHART I-22	Organic Analysis by HPLC: EXPLOSIVES (8330)
CHART I-23	Sample Preparation for Metal Analysis
CHART I-24	General QA/QC for Metal Analysis
CHART I-25	Metal Analysis by ICP: METALS (6010A)
CHART I-26	Metal Analysis by AA: METALS (7000s)
CHART I-27	General QA/QC for Classical Analysis
CHART I-28	Classical Analysis: COMMON ANIONS (300s)
CHART I-29	Classical Analysis: OIL AND GREASE (413.1)
CHART I-30	Classical Analysis: TRPH (418.1)
CHART I-31	Classical Analysis: CYANIDE (9010A)
CHART I-32	Classical Analysis: TOC (9060)
CHART I-33	Waste Characteristics: Ignitability (1010/1020)
CHART I-34	Waste Characteristics: Corrosivity (1110)
CHART I-35	Waste Characteristics: Reactivity (Section 7.3)
CHART I-36	Waste Characteristics: Toxicity (1311)

An inspector(s) should use those checklists that are applicable to the laboratory to be inspected. Depending on the projects and/or the laboratory, an inspector(s) shall determine which sections of each checklist should be used. The inspector(s) should check those items, under the “YES” column, which he/she believes to be adequately practiced and documented in the laboratory. Additional information should be entered in the “COMMENT” columns with an “N/A” for items not applicable to the work for the USACE. The detail of any observations, comments, or problems should be recorded in the blank space provided at the end of each checklist. Any deficiences noted on the checklist shall be discussed with and acknowledged by the laboratory management staff during an Exit Interview.

The checklists will be revised or augmented when revised or new analytical methods are officially approved by the USEPA or other regulatory agencies. All revisions of the checklists shall be approved by the HQUSACE.

CHART I-1

ORGANIZATION AND PERSONNEL:

ITEM	YES	COMMENT
Is the lab legally identifiable?		
Has the lab provided supervision by persons familiar with the test methods, the objective of the test, and the assessment of the results?		
Has the lab specified and documented the responsibility, authority, and relationship of all personnel who manage, perform, or verify work affecting the quality of tests?		
Does the lab have a QA Officer who has responsibility for the quality system and its implementation?		
Is the QA Officer familiar with all test procedures and QC requirements?		
Does the QA Officer have direct access to the highest level of management at which decisions are taken on lab policy or resources?		
Does the lab nominate deputies in case of absence of the QA Officer?		
lees the lab have documented protocol for training in QC methods?		
Do personnel assigned to this project have the appropriate background to successfully accomplish the objectives of tests?		
Is each analyst accountable for performing tasks in any of the following areas meet the specified minimum experience: a. Inorganic sample preparation – 6 months? b. Organic sample preparation – 1 year?		

CHART I-1

ORGANIZATION AND PERSONNEL: Page 2 of 3

ITEM	YES	COMMENT
c. Classical analysis – 1 year? d. Trace metal analysis – 1 year? e. Gas chromatography – 1 year? f. Pesticide residue analysis – 2 years? g. Mass spectrometry – 1 year? h. Spectrum interpretation – 2 years? i. Radiochemical analysis – 2 years?		
Is each analyst's performance audited and approved prior to work without close supervision by a senior chemist?		
Is there documented evidence of analyst proficiency for each test method performed?		
Does the lab have an in-house training program or send staff to training schools?		
Are staff's qualification, training, and experience recorded?		
Is backup provided for technical staff?		
Does the lab have documented policy and procedures to ensure the protection of clients' confidential information and proprietary right?		

CHART I-1

ORGANIZATION AND PERSONNEL:

ITEM
Additional observations, comments, or problems:

CHART I-2

FACILITIES:

ITEM	YES	COMMENT
Does the lab building have a security system?		
Is access to the test and sample storage area controlled?		
Is a guest logbook available and used?		
Is equipment protected and environment monitored as needed?		
Does the lab have adequate work space, ventilation, light, and access to stable power sources at workstations?		
Is the lab clean and organized?		
Is the lab free of dust, drifts, and temperature extremes?		
Is reagent water free of contamination used for preparation of standards and blanks?		
Is the conductivity of water routinely checked and recorded on a daily basis?		
Is a separate conductivity meter (capable of being calibrated) used to measure the conductivity of the reagent water? (Meters built into the water purification system are not acceptable.)		
Is a corrective action taken when the conductivity of the reagent water is two micromho or greater at 25°C?		
Are exhaust hoods provided to allow contamination-free work with volatile and hazardous materials?		

CHART I-2

FACILITIES:

ITEM	YES	COMMENT
Is the air flow of the hoods periodically checked and recorded?		
Are adequate facilities, including cold storage, provided for separate storage of samples, extracts, reagents, solvents, reference materials and standards to preserve their identity, concentration, purity, and stability?		
Is adequate chemical storage space available and are chemicals properly segregated according to class?		
Are solvent storage cabinets properly vented as appropriate for the prevention of possible lab contamination?		
Does the lab have adequate safety devices such as eye wash stations, spill control stations, showers, first-aid stations, etc.?		
Are these safety devices checked routinely to ensure they are still working properly?		
Are special facilities (e.g., glove box, controlled air) provided for handling extremely toxic materials such as dioxin?		
Are adequate filing space available for storage of manuals, SOPs, raw data, and reports?		
Are chemical waste disposal policies and procedures well-defined and followed by the lab?		

CHART I-2

FACILITIES:

ITEM
Additional observations, comments, or problems:

CHART I-3

EQUIPMENT:

ITEM	YES	COMMENT
Is appropriate equipment available for use in accordance with required methodology?		
Is equipment adequately maintained with sufficient spare parts and are maintenance instructions available?		
Is out-of-service equipment clearly labelled?		
Are equipment maintenance logs maintained?		
Are standard curves prepared to cover the expected concentration ranges of samples?		
Are calibration logs maintained?		
Is a new curve prepared annually (or more frequently if specified by the method) or whenever new reagents are prepared, whichever is more frequent?		
Are calibration labels used as applicable?		
Is proper backup equipment available?		
Balances: a. Analytical Balances: (1) Are analytical balances capable of weighing 0.1 mg in use? (2) Is there a record of balance calibration in two ranges with Class S weights? (Please specifiy the ranges.) (3) Do records show daily functional and calibration checks (<±0.1%) for analytical balances?		

CHART I-3

EQUIPMENT:

ITEM	YES	COMMENT
(4) Have the balances been calibrated at least annually? b. Top Loading and/or Pan Balances: (1) Is a top loading and/or pan balances capable of accurately detecting a 100 mg weight at a load of 150 g available? (2) Is there a record of the balance having been serviced within the previous 12 months? (3) Is there a record of balance calibration in two ranges with Class S weights? (Please specify the ranges.) (4) Do records show weekly functional and calibration checks (<±0.1%) for pan balances? (5) Have the balances been calibrated at least annually?		
Thermometers: a. Certified Thermometer: (1) Does the lab have, or have access to, an NIST-traceable factory certified thermometer? (2) Is a copy of factory certificate for the thermometer available for inspection? (3) Is there a record of the annual check of the certified thermometer at the ice point?		

CHART I-3

EQUIPMENT:

ITEM	YES	COMMENT
b. Working Thermometers: (1) Are sufficient working thermometers available so that each has a dedicated use? (2) Does each working thermometer have a unique identifying number? (3) Is the calibration of each working mercury thermometer checked annually against an NIST-traceable thermometer? (4) Is the calibration of each dial type thermometer checked at least quarterly against an NIST-traceable thermometer? (5) Are digital thermometers calibrated quarterly at their temperature of use against an NIST-traceable thermometer? (6) Is a record of thermometer calibration maintained?		
pH Meters: a. Is a clean pH meter with appropriate electrode with scale graduations at least 0.1 pH units in use? b. Is a thermometer or temperature sensor for automatic compensation in use? c. Do records show daily, or before each use, calibration, whichever is less frequent? d. Are three standard buffers used for the calibration?		

CHART I-3

EQUIPMENT:

ITEM	YES	COMMENT
e. Are aliquots of standards of pH 4, pH 7, and pH 10 used only once? f. Are acceptance limits in place? g. Is the meter recalibrated if not within the limits of 0.05 for two point calibration and within 0.2 for one point calibration? h. If the limits cannot be achieved, is the problem determined and resolved?		
Conductivity Meters: a. Are a conductivity meter and probe of sufficient sensitivity in use? b. Do records show a daily, or before each use, calibration check, whichever is less frequent? c. Do records show cell constant is determined annually? d. If the cell constant has a large deviation from the expected value, is the cause determined and corrected?		
Refrigerators/Walk-in Coolers: a. Is a thermometer in each refrigerator with bulb immersed in liquid? b. Are thermometers graduated in increments no larger than 1°C? c. Are temperatures for each refrigerator recorded daily?		

CHART I-3

EQUIPMENT:

ITEM	YES	COMMENT
d. Do records show that refrigerator temperatures are maintained in the range of 2 to 6°C?		
Ovens: a. Are thermometers graduated in increments no larger than 1°C? b. If the oven temperature cannot be read without opening the door, is the bulb of the thermometer in a sand bath? c. Is oven temperature adequately monitored (e.g., beginning and end of each use cycle)? d. Is a record documenting date, time of use, nature of use, and temperature maintained? e. Do the records indicate the oven holds temperature at the appropriate drying temperature?		
Glassware: a. Is the lab stocked with sufficient volumetric glassware for the analyses performed? b. Is Class A volumetric glassware available for standard preparation? c. Is glassware cleaned in a manner appropriate for the analytical procedures for which it is to be used? d. Is glassware cleaning procedure posted next to the cleaning station?		

CHART I-3

EQUIPMENT:

ITEM	YES	COMMENT
e. For organics, are the following basic cleaning steps used?		
(1) Removal of surface residuals immediately after use?		
(2) Flush with methanol before it is placed in hot detergent soak?		
(3) Hot soak (>50°C) in a synthetic detergent bath to loosen and float most particulate material?		
(3) Hot-water rinse to flush away floated particulates?		
(4) Soak with oxidizing agent such as chromic acid solution made up of sulfuric acid and potassium or sodium bichromate at 40-50°C to destroy traces of organic compounds?		
(5) Hot-water rinse to flush away materials loosened by the deep penetrant soak?		
(6) Distilled-water rinse to remove metallic deposits from the tap water?		
(7) Methanol or isopropanol rinse to flush off any final traces of organic materials and remove the water?		
(8) Flushing the item immediately before use with some of the same solvent that will be used in the analysis?		

CHART I-3

EQUIPMENT:

ITEM	YES	COMMENT
f. Is glassware for organics dried at 100°C?		
g. As an alternative to solvent rinsing, is glassware for organics heated to a minimum of 300°C to vaporize any organics?		
h. Is volumetric glassware, glassware with ground glass joints, or sintered glassware not heated to high temperature to avoid deformation?		
i. For trace metals, is the plastic or glassware cleaned with detergent, tap water, 1:1 nitric acid, tap water, 1:1 hydrochloric acid, tap water, and reagent water?		
j. Is chromic acid not used to clean glassware and plastic bottles for trace metal analysis?		
k. Is clean glassware properly covered and stored to prevent recontamination by dust?		

CHART I-3

EQUIPMENT: Page 8 of 8

ITEM
Additional observations, comments, or problems:

CHART I-4

GENERAL QA/QC:

ITEM	YES	COMMENT
Does the lab maintain a QA Manual?		
Does the manual address the important elements of a QA/QC program, including the following: a. QA Policy and Objectives? b. organization? c. Personnel? d. Facilities and Equipment? e. Document Control? f. Sample Receiving and Storage? g. Analytical Methodology? h. Instrument Operation? i. Instrument Calibration? j. Preventive Maintenance? k. Certification of Regents/Standards? l. Data Generation/Reduction/Validation? m. Data Reliability? n. Feedback and Corrective Action? o. Recordkeeping and Archives? p. Internal QC Audits? q. Performance and External Audits? r. Training/Certification of Personnel?		

CHART I-4

GENERAL QA/QC: Page 2 of 10

ITEM	YES	COMMENT
Is the manual available to all laboratory personnel?		
Is the manual updated regularly?		
Is line authority for all referenced organizations explained by including an organization chart?		
Is the organizational structure appropriate to accomplish the project QA objectives?		
Are QA/QC responsibilities and reporting relationships clearly defined?		
Are all staff aware of QA/QC and its application?		
Is the QA Officer a full-time employee?		
Does the QA Officer operate independently of the analyses?		
Does the QA Officer report directly to a senior officer?		
Are internal QA reviews conducted at least annually and recorded including any corrective action taken?		
QA Objectives and Criteria: a. Are the terms and definitions for precision, accuracy, comparability, representativeness, and completeness properly used? b. Have the following been defined for each parameter and matrix: (1) Level of QA effort (frequency and type of QC, etc.)?		

CHART I-4

GENERAL QA/QC:

ITEM	YES	COMMENT
(2) Accuracy (matrix spikes, surrogate spikes, reference samples, etc.)? (3) Precision (replicate samples)? (4) Sensitivity or MDL? (5) Statistical reporting units? c. Are quantitative limits established for each parameter and matrix? d. Are field and lab both covered? e. If appropriate, are completeness objectives quantitatively stated? f. Are representativeness and comparability appropriately addressed?		
Is a sample batch clearly defined and determined as a group of samples of $\leq$ 20, with similar matrix, prepared and analyzed with same technique and reagents at same time or within same time sequence?		
Is at least the following minimum QC practiced in the lab? a. For Inorganic/Classical Analysis: (1) Minimum three concentrations of standards plus blank, and one check standard in ten; the lab shall repeat all samples if check standard is outside ±10%. (2) One method blank per batch. (3) One matrix spike per batch. (4) One lab duplicate per batch.		

CHART I-4

GENERAL QA/QC:

ITEM	YES	COMMENT
(5) One control (consists of a control matrix spiked with analytes representative of the target analytes) per batch. b. For Organic Analysis: (1) Minimum five concentrations of standards plus blank and one check standard in ten; if any are outside control limits repeat all samples. (2) One method blank per batch. (3) One matrix spike per batch. (4) One lab duplicate/matrix spike duplicate per batch. (5) One control (consists of a control matrix spiked with analytes representative of the target analytes) per batch. (6) Surrogates for all samples.		
Are there any exceptions to the above minimum QC practice in the lab?		
Has the lab established control limits for all the above types of QC samples? (Control limits should be at least as tight as those stated in the methods.)		
Are quality control data (e.g., standard curve, results of duplicates and spikes) accessible for all analytical results?		
Are method detection limits empirically determined and documented?		

CHART I-4

GENERAL QA/QC:

ITEM	YES	COMMENT
Are control charts maintained for each routine analysis?		
Do lab records indicate what corrective action has been taken when results fail to meet QC criteria?		
Are documented methods/procedures available for assurance of field and lab equipment functioning optionally?		
Is a program of initial and periodic calibration established for each method?		
Does the QA Manual include calibration documentation requirements: a. Date of calibration? b. Identification of standards used? c. Personnel performing calibration? d. Results of calibration (raw data and summary statistics)? e. Corrective actions taken?		
Are primary reference standards used for calibration only?		
Are all working standards versus primary standards verified and documented?		
Are reference materials traceable to NIST standards?		
Are reagent grade or higher purity chemicals used to prepare standards?		

CHART I-4

GENERAL QA/QC:

ITEM	YES	COMMENT
Are fresh analytical standards prepared at a frequency consistent with good QC?		
Are reference materials/reagents properly labeled with concentrations, dates of preparation and expiration, and identity of the person preparing the reagent?		
Are updated equipment operating instructions available?		
Are analytical procedures written as SOPs available for review?		
Are all procedural steps and options described?		
Are the criteria of method selection included (e.g., in order to obtain a specific data quality objective?)		
If method choice is governed by regulatory requirement (e.g., NPDES, SDWA, RCRA), have the appropriate methods been chosen?		
Are approved methods being used as specified?		
Are procedures documented for data handling, reporting, and recordkeeping?		
Are documented validation procedures applied at appropriate levels for all measurement procedures?		
Are documented procedures available for checking the validity of reported analysis values?		

CHART I-4

GENERAL QA/QC:

ITEM	YES	COMMENT
Are predetermined limits available for data acceptability beyond which corrective action is required?		
Are documented procedures available for correcting erroneously reported results?		
Are the following SOPs available for review? a. Sample collection, preservation, storage, and handling. b. Sample preparation and analysis. c. Purity and preparation of standards. d. Instrument operation and calibration. e. Preventive maintenance and corrective actions. f. Quality control for each type of test. g. Quality control chart. h. Data reduction and reporting. i. Recordkeeping and archives. j. Personnel training/certification. k. Procurement and inventory procedures. l. Glassware cleaning. m. Waste disposal. n. Technical and managerial review of lab operation.		

CHART I-4

GENERAL QA/QC: Page 8 of 10

ITEM	YES	COMMENT
Are procedures in place for making and controlling revisions to in-house SOPs?		
Are there internal audits for both field and lab activities?		
Are there designated persons who will conduct the audits? Auditor's Name: _____ _____ _____		
Is there a documented protocol which will be used for audit?		
Are acceptance criteria defined?		
Are audit reports prepared and distributed? To whom? _____ _____ _____		
Are the type and frequency of audit reports specified? Type/frequency: _____ _____ _____		
Do the reports address: a. status of project (time table)? b. results of performance and system audits? c. data quality assessment? d. significant QA problems and proposed corrective action?		

CHART 1-4

GENERAL QA/QC:

ITEM	YES	COMMENT
Does the lab participate in any external proficiency testing programs such as EPA performance evaluation studies for water supply and water pollution?		
Are there corrective actions taken and documented?		
Does the lab have a laboratory information management system (LIMS) currently in use? If yes, manufacturer: _____ Model No.: _____ Brief description of hardware & software:		
Does the LIMS have an audit trail feature?		

CHART I-4

GENERAL QA/QC: Page 10 of 10

ITEM
Additional observations, comments, or problems:

CHART I-5

REPORT GENERATION: Page 1 of 4

ITEM	YES	COMMENT
Are there documented procedures for internal field and lab checks of: a. precision and accuracy? b. routine duplicates, spikes, and standard samples? c. statistical methods, including control chart and computer methods?		
Is there a written description of the lab record system including data management, review, validation, and audit?		
Is there written description of the lab reporting system?		
Is there a system in place that provides for retrievability and traceability of the sample source, analytical methods, results, person performing analysis, and date?		
Are records and reports adequately secured for the required amount of time to ensure the integrity per regulatory requirements?		
Are permanently bound notebooks with consecutively numbered pages being used?		
Is a unique serial number clearly displayed on each notebook cover or spine?		
Are logbook entries made in permanent fashion with indelible ink?		
Are logbook entries legible?		
Are all raw data signed and dated by the chemist who performed the analysis?		

CHART I-5

REPORT GENERATION: Page 2 of 4

ITEM	YES	COMMENT
Are there evidence of entries being tampered with?		
Has data been altered?		
If yes, was a single line drawn through the entry and corrections made without obliterating original entries?		
Was the new entry initialed and dated?		
Were technical reviews conducted on all logbook entries and deliverables?		
Was a minimum of three-levels of technical reviews conducted by chemist, supervisor, QA Officer?		
Have QC measures been utilized to ensure the quality of the work performed?		
Can all signatures be clearly identified?		
Is a central file being maintained for all project documents?		
Is a system of document control numbers in place?		
Are all completed lab notebooks, raw data, analytical reports, electronic tapes and disks, and other pertinent documentation filed in a secure, controlled archives area?		
Has the supervisor personally examined and reviewed each notebook periodically and signed and dated the review?		
Do the lab's reports accurately, clearly, and unambiguously present results and all other relevant information?		

CHART I-5

REPORT GENERATION:

ITEM	YES	COMMENT
Does each test report include the following information: a. Names and addresses of laboratory and client? b. Unique identification and page number? c. Case narrative? d. Sample identification and description? e. Dates of sample receipt and test performed, as appropriate? f. Identification of sample preparation and analysis methods used? g. Description of any deviations from test method? h. Disclosure of any subcontractor used? i. Results including all method required QC data? j. Description of any problems or failures identified? k. Measurement uncertainty, if relevant?		
Is the lab's report format acceptable?		
Does the length of storage time for all sample related information comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)		

CHART I-5

REPORT GENERATION:

ITEM
Additional observations, comments, or problems:

CHART I-6

FIELD SAMPLING: (Complete if this lab conducts field sampling for USACE projects.)

ITEM	YES	COMMENT
Is a site specific Chemical Data Acquisition Plan (CDAP) available to lab personnel?		
Are lab personnel familiar with the QC requirements of the CDAP?		
Do sampling procedures follow contract specifications?		
Do field documentation procedures: a. document the sources and lot numbers of reagents and supplies? b. include procedures/forms for recording the exact location and specific considerations associated with sample acquisition? c. document specific preservative methods? d. include labels containing all necessary information? e. include forms for tracking custody?		
Is there a unique identification on each sample?		
Is sampling information properly recorded such as sample ID numbers, type (grab versus composite), preservatives, analytes, location, date and time of collection, and name of sample collector?		
Are written chain-of-custody procedures available for review? Are they in accordance with USACE/EPA guidelines?		

CHART I-6

FIELD SAMPLING:

ITEM	YES	COMMENT
Are there written sampling SOPs covering sampling plan, sampling equipment, sample collection, preservation, identification, storage, and lab handling?		
Are there written descriptions of chain-of-custody of samples? (Attach a copy of chain-of-custody form.)		
Are there written procedures for field measurement of flow, dissolved oxygen, residual chlorine, etc.?		
Are there written procedures for monitoring water supply, effluent, ambient air, stacks, radiation, etc.?		
Are proper preservation techniques being used for the analytical methods and sample types concerned?		
Are provisions made for the collection of QA/QC split samples?		
Are provisions made for field blanks and duplicate samples at an appropriate rate (normally 10% or minimum of one per matrix type, whichever is greater, or as specified in contract?)		
Are adequate facilities available to do compatibility testing?		

CHART I-6

FIELD SAMPLING:

ITEM
Additional observations, comments, or problems:

CHART I-7

SAMPLE RECEIPT AND STORAGE: Page 1 of 3

ITEM	YES	COMMENT
Are there adequate written procedures for receipt and storage of samples to ensure sample integrity?		
Do the written procedures address sample handling, storage, and dispersement for analysis and disposal?		
Do the written procedures accurately reflect procedures in use?		
Is separate area and facility including hoods available for sample receipt?		
Is a dedicated sample custodian available? Custodian's name: _____		
Are appropriate chain-of-custody procedures documented and followed in the lab?		
Does the lab maintain internal custody procedures?		
Does a permanent record exist for sample log-in?		
Are samples assigned unambiguous sample ID numbers when logged in?		
Is a checklist used to document problems or deficiencies noted during sample log-in?		
Is sample temperature properly measured and recorded during log-in?		
Are pH values of aqueous samples for the following analyses checked and adjusted in a hood during log-in? (Metals, phenols, oil and grease, TRPH, TOC, TOX, COD, hardness, ammonia, nitrate-nitrite, total phosphorus, Kjeldahl and organic nitrogen, radiological testing, cyanide, and sulfide.)		

CHART I-7

SAMPLE RECEIPT AND STORAGE:

ITEM	YES	COMMENT
Is the pH value properly measured to avoid sample contamination and to minimize waste generation?		
Are corrective actions properly documented?		
Are clients notified if problems are noted?		
Are there adequate facilities for sample storage?		
Are samples stored in such a way as to maintain their identity, integrity, stability, and concentration?		
Are volatile organic samples stored in separate refrigerators from other samples?		
Are temperature logs of storage coolers and refrigerators properly maintained?		
Are acceptable temperature ranges used and posted? (4 ± 2°C)		
Are coolers and refrigerators locked when unattended?		
Is final disposition of samples documented?		

CHART I-7

SAMPLE RECEIPT AND STORAGE:

ITEM
Additional observations, comments, or problems:

CHART I-8

SAMPLE PREPARATION FOR ORGANIC ANALYSIS:

ITEM	YES	COMMENT
General:		
a. Are written SOPs available and adequate for sample preparation?		
b. Do these SOPs accurately reflect procedures in use?		
c. Are all sample preparations conducted in a hood?		
d. Are a group of samples (up to a maximum of 20) which behave similarly with respect to the procedures being employed and which are processed as a unit with the same method sequence and the same lots of reagents and with the reagents and with the manipulations manipulations common to each samples within the same time period or in continuous sequential time periods considered as a batch?		
e. Are the following lab internal QC samples prepared for each batch of samples?		
(1) Method blanks?		
(2) Matrix spikes?		
(3) Matrix spike duplicates?		
(4) Matrix duplicates?		
(5) Laboratory control samples?		
f. If the quantity of field samples is not sufficient for internal QC analyses, are blank spike/blank spike duplicate or duplicate laboratory control samples analyzed?		

CHART I-8

SAMPLE PREPARATION FOR ORGANIC ANALYSIS:

ITEM	YES	COMMENT
g. Is a purified solid matrix used for preparation of method blanks for soil and sediment volatile organics?		
h. Is a purified sodium sulfate used for preparation of method blanks for soil and sediment semivolatile organics including pesticides, herbicides, and PCBs?		
i. If sample extracts are cleaned up with Methods 3600s, are the associated QC samples also processed through the corresponding cleanup methods?		
j. Is the water meniscus of aqueous samples marked on the side of sample container for later determination of sample volume?		
k. Are the rates of internal QC samples consistent with method requirements or, at a minimum, 5% per batch of no more than 20 samples with similar matrix, whichever is greater?		
l. Is the appropriateness of a particular preparation for a specific sample type determined by the completeness of extraction and by spike recoveries?		
m. Are logbooks for sample preparation used and well maintained?		
n. Are permanently bound notebooks with consecutively numbered pages used?		
o. Is a unique serial number clearly displayed on each notebook?		
p. Are critical times entered in logbooks?		

CHART I-8

SAMPLE PREPARATION FOR ORGANIC ANALYSIS:

ITEM	YES	COMMENT
q. Are spiking solutions traceable to NIST or other reliable standards?		
r. Are spiking solutions labeled properly with date of preparation, composition, concentration and identity of preparer?		
s. Have entries been made in permanent fashion and corrections made without obliterating original entries?		
t. Are corrections reviewed and initialed by a supervisor?		
u. Does the logbook of sample preparation contain the following information?		
(1) Date/time?		
(2) Sample ID number?		
(3) Sample preparer?		
(4) Matrix noted?		
(5) Spiking standards?		
(6) Pretreatment?		
(7) Volume/weight of sample?		
(8) Final volume?		
(9) Preparation methods?		
Separatory Funnel Liquid-Liquid Extraction Method 3510A):		
a. Is the following equipment available?		

CHART I-8

SAMPLE PREPARATION FOR ORGANIC ANALYSIS:

ITEM	YES	COMMENT
(1) Separator funnel (2-L with Teflon stopcock)?		
(2) Drying tube with Pyrex glass wool at bottom and a Teflon stopcock?		
(3) Sets of Kuderna-Danish glassware (including concentration tubes, evaporation flasks, and macro and micro Snyder columns)?		
(4) Water bath capable of temperature control within 5°C?		
b. Are enough sets of separator funnels (2,000 mL with Teflon stopcock) and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples?		
c. Are the following reagents available?		
(1) Sodium hydroxide solution (10 N)?		
(2) Sulfuric acid solution (1:1)?		
(3) Anhydrous sodium sulfate?		
(4) Methylene chloride?		
(5) Hexane?		
(6) 2-Propanol?		
(7) Cyclohexane?		
(8) Acetonitrile?		
d. Are surrogate standards and spiking solutions added to the samples in the separator funnel prior to the addition of methylene chloride?		

CHART I-8

SAMPLE PREPARATION FOR ORGANIC ANALYSIS: Page 5 of 12

ITEM	YES	COMMENT
e. Is the Kuderna-Danish concentration process conducted with a hot water bath at 80-90°C? f. If concentrated extracts are to be stored more than two days are they transferred to Teflon-1ined screw-cap or crimp-top vials, labeled appropriately, and refrigerated?		
Continuous Liquid-Liquid Extraction (Method 3520A): a. Is the following equipment available? (1) Continuous liquid-liquid extractor equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication? (2) Drying column with Pyrex glass wool at bottom and a Teflon stopcock? (3) Sets of Kuderna-Danish glassware (including concentration tubes, evaporation flasks, and macro and micro Snyder columns)? (4) Water bath capable of temperature control within 5°C? (5) Heating mantle (Rheostat controlled)? b. Are enough sets of continuous liquid-liquid extractors and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples? c. Are the following reagents available? (1) Sodium hydroxide solution (10 N)?		

CHART I-8

SAMPLE PREPARATION FOR ORGANIC ANALYSIS: Page 6 of 12

ITEM	YES	COMMENT
(2) Sulfuric acid solution (1:1)? (3) Anhydrous sodium sulfate? (4) Methylene chloride? (5) Hexane? (6) 2-Propanol? (7) Cyclohexane? (8) Acetonitrile? d. Are surrogate standards and spiking solutions added to the samples prior to extraction? e. Is twice the volume of spiking solution added when GPC cleanup will be used? f. Are samples extracted for 18-24 hours at a specific pH value? g. Is the Kuderna-Danish concentration process conducted with a hot water bath at 80-90°C? h. If concentrated extracts are to be stored more than two days are they transferred to Teflon-lined screw-cap or crimp-top vials, labeled appropriately and refrigerated?		
Soxhlet Extraction (Method 3540A): a. Is the following equipment available? (1) Soxhlet extractor with 500-mL round bottom flask?		

CHART I-8

SAMPLE PREPARATION FOR ORGANIC ANALYSIS: Page 7 of 12

ITEM	YES	COMMENT
(2) Drying column with Pyrex glass wool at bottom and a Teflon stopcock?		
(3) Sets of Kuderna-Danish glassware (including concentration tubes, evaporation flasks, and macro and micro Snyder columns)?		
(4) Heating mantle (Rheostat controlled)?		
(5) Grinding apparatus?		
b. Are enough sets of Soxhlet extractors and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples?		
c. Are the following reagents available?		
(1) Anhydrous sodium sulfate?		
(2) Toluene/Methanol (10:1) solvent?		
(3) Acetone/Hexane (1:1) solvent?		
(4) Methylene chloride?		
(5) Hexane?		
(6) 2-Propanol?		
(7) Cyclohexane?		
(8) Acetonitrile?		
d. If a dry waste sample will not pass through a l-mm standard sieve or cannot be extruded through a l-mm opening, is it processed into a homogeneous sample that meet these requirements?		

CHART I-8

SAMPLE PREPARATION FOR ORGANIC ANALYSIS: Page 8 of 12

ITEM	YES	COMMENT
e. Are surrogate standards and spiking solutions added to the samples prior to extraction? f. Is twice the volume of spiking solution added when GPC cleanup will be used? g. Are samples extracted for 16-24 hours? h. Is the Kuderna-Danish concentration process conducted with a hot water bath at 80-90°C? i. If concentrated extracts are to be stored more than two days are they transferred to Teflon-lined screw-cap or crimp-top vials, labeled appropriately and refrigerated?		
Sonication Extraction (Method 3550): a. Is the following equipment available? (1) Grinding apparatus? (2) Horn-type sonicator equipped with a titanium tip (475 W)? (3) Sets of Kuderna-Danish glassware (including concentration tubes, evaporation flasks, and macro and micro Snyder columns)? (4) Drying column with Pyrex glass wool at bottom and a Teflon stopcock? (5) Water bath capable of temperature control within 5°C? b. Are the following reagents available? (1) Anhydrous sodium sulfate?		

CHART I-8

SAMPLE PREPARATION FOR ORGANIC ANALYSIS:

ITEM	YES	COMMENT
(2) Methylene chloride/Acetone (1:1)?		
(3) Methylene chloride?		
(4) Hexane?		
(5) 2-Propanol?		
(6) Cyclohexane?		
(7) Acetonitrile?		
c. If the sample will not pass through a l-mm standard sieve or cannot be extruded through a l-mm opening, is it processed into a homogeneous sample that meet these requirements?		
d. Are samples mixed with anhydrous sodium sulfate to form a free flowing powder?		
e. Are surrogate standards and spiking solutions added to the samples prior to the addition of the extraction solvent?		
f. Is twice the volume of spiking solution added when GPC cleanup will be used?		
g. Are samples that are expected to contain low concentrations of organics sonicated three times for three minutes with fresh solvent each time?		
h. Are samples that are expected to contain high concentrations of organics sonicated once for two minutes?		
i. Is the Kuderna-Danish concentration process conducted with a hot water bath at 80-90°C?		

CHART I-8

SAMPLE PREPARATION FOR ORGANIC ANALYSIS: Page 10 of 12

ITEM	YES	COMMENT
j. If concentrated extracts are to be stored more than two days are they transferred to Teflon-lined screw-cap or crimp-top vials, labeled appropriately and refrigerated?		
Purge-and-Trap (Method 5030A): a. Is a purge-and-trap device available? b. Are purge-and-trap systems subjected to a periodic bake-out and cleaning process and are these actions documented? c. Is a purge chamber designed to accept 5-mL samples available? d. Is a 25-mL all glass purge chamber available for GC/MS Methods 524.1, 524.2, and 8260 (optional)? e. Is the gaseous headspace less than 15 mL? f. Is the trap a minimum of 25-cm long? g. For Method 8010A, is the trap packed with 1.0-cm 3% OV-1 on Chromosorb-W 60/80 mesh, 7.7-cm Tenax GC, 7.7-cm silica gel, and 7.7-cm charcoal or equivalent? h. For Method 8015A, is the trap packed with l.0-cm 3% OV-1, 15-cm Tenax GC, and 7.7-cm silica gel or equivalent? i. For Methods 8020 and 8030A, is the trap packed with l.0-cm 3% OV-1 on Chromosorb-W and 23-cm Tenax GC or equivalent?		

CHART I-8

SAMPLE PREPARATION FOR ORGANIC ANALYSIS: Page 11 of 12

ITEM	YES	COMMENT
j. Is methanolic extraction of purge-and-trap only used for medium-concentration soils or sediments? k. Is the methanol purge-and-trap quality or equivalent? l. Are surrogate standards and spiking solutions added to the purging chamber along with the sample? m. Is a method blank carried through all of sample preparation and measurement before any sample are processed?		
Headspace (Method 3810): a. Is this method only used as a screening procedure? b. Is a hot bath capable of maintaining a 90°C temperature available? c. Are 125-mL hype-vials with seals and septa used for the equilibration? d. Are both a 1-ppm spike and a 1-ppm standard analyzed along with samples? e. Are the vials with the samples (the 1-ppm spike and the 1-ppm standard) equilibrated in a 90°C water bath for one hour? f. Are the vials maintained at 90°C while 2-mL of headspace gas is withdrawn for direct injection into a GC? g. Is the GC operated using the same GC conditions listed in the method being screened (8010A, 8015A, 8020, 8030A, or 8040A)?		

CHART I-8

SAMPLE PREPARATION FOR ORGANIC ANALYSIS: Page 12 of 12

ITEM
Additional observations, comments, or problems:

CHART I-9

GENERAL QA/QC FOR ORGANIC ANALYSIS BY GC:

ITEM	YES	COMMENT
Are the following detectors available and are they used appropriately? a. Flame Ionization (603, 604, 609, 610, 8015A, 8030A, 8040A, 8060, 8090, 8100)? b. Photoionization (602, 8020)? c. Electron Capture (604, 606, 608, 609, 612, 8040A, 8060, 8080, 8090, 8120, 8150A)? d. Electrolytic Conductivity (601, 611, 801OA, 8080, 8140)? e. Microcoulometric (601, 611, 8140)? f. Thermal Energy Analyzer (607)? g. Nitrogen/Phosphorus (607, 8140)? h. Flame Photometric (8140)?		
Are the column ovens, at a minimum, capable of temperature control within ±0.2°C at 220°C?		
Are the injection ports glass lined?		
Are manufacturer's operating manuals readily available to bench chemists?		
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		
Have any instruments been modified in any way?		
Are the instruments properly vented?		

CHART I-9

GENERAL QA/QC FOR ORGANIC ANALYSIS BY GC: Page 2 of 4

ITEM	YES	COMMENT
Is glassware for organics solvent rinsed or heated to a minimum of 300°C to vaporize any organics in a muffle furnace after careful cleaning?		
Is this high temperature treatment avoided for volumetric glassware, glassware with ground joints, or sintered glassware?		
Is there a calibration protocol available to bench chemists?		
Is there a calibration protocol available to bench chemists?		
Is a 5-point calibration used?		
Is the calibration curve or calibration factor verified each working day?		
Are calibration results kept in a permanent logbook?		
Is the MDL for each analyte and matrix type determined every six months or whenever there is a significant change in background or instrument response?		
Is the linear calibration range determined for each analyte when there is significant change in instrument response and every six months for those analytes that periodically approach their linear limits?		
Is a method blank included with each sample batch and carried through the entire preparation and analysis?		
Is a matrix spike and a matrix spike duplicate run with each batch at a rate of 5% or one per batch, whichever is greater?		

CHART I-9

GENERAL QA/QC FOR ORGANIC ANALYSIS BY GC: Page 3 of 4

ITEM	YES	COMMENT
Is corrective action taken if matrix spike recoveries exceed QC limits?		
Is a matrix duplicate run with each batch at a rate of 5% or one per batch, whichever is greater?		
Is corrective action taken if percent differences based on duplicated analyses exceed QC limits?		
Are surrogate recoveries run on each sample?		
Is corrective action taken if surrogate recoveries exceed QC limits?		
Is an LCS prepared with standards independent of calibration standards analyzed for each batch of samples?		
Is corrective action taken if the LCS recovery exceeds QC limits?		
Are QC data statistically analyzed and charted for quality control?		
Are control charts maintained and readily available to bench chemists?		

CHART I-9

GENERAL QA/QC FOR ORGANIC ANALYSIS BY GC: Page 4 of 4

ITEM
Additional observations, comments, or problems:

CHART I-10

ORGANIC ANALYSIS BY GC: HVO (8010A)

ITEM	YES	COMMENT
General: a. Are written SOPs available and adequate for HVO sample preparation/analysis? b. Do these SOPs accurately reflect procedures in use? c. Are all target analytes, at a minimum, that have retention times published in Table 1 of Method 8010A, routinely analyzed at the lab? d. Are manufacturer's operating manuals readily available to bench chemists? e. Are prenumbered, bound notebooks used for data entry? f. Are all records written in indelible ink? g. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible? h. Are corrections initialed and dated by the responsible individual? i. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff: a. Do bench chemists appear knowledgeable and experienced in operation of a purge-and-trap and GC system and in interpretation of chromatograms? b. Are backup bench chemists available?		

CHART I-10

ORGANIC ANALYSIS BY GC: HVO (8010A) Page 2 of 13

ITEM	YES	COMMENT
c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		
Apparatus and Facilities: a. Is working space adequate and clean? b. Does the lab have adequate air handling system to avoid cross contamination of samples? c. Is a temperature-programmable gas chromatography equipped with an purge-and-trap device and electrolytic conductivity detector available? d. Is oven temperature stable to ±0.5°C or better at desired setting? e. Is one of the following GC column available? (1) 8-ft x 0.1-in ID SS or glass column packed with 1% SP-1OOO on Carbopack-B 60/80 mesh or equivalent? (2) 6-ft x 0.1-in ID SS or glass column packed with chemically bonded n-octane on Porasil-C 100/200 mesh or equivalent? f. If an "equivalent" column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated? g. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		

CHART I-10

ORGANIC ANALYSIS BY GC: HVO (8010A)

ITEM	YES	COMMENT
h. Is helium used as carrier gas? i. Is a hood available for sample preparation? j. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available? k. Are backup instruments available?		
Reagents: a. Is reagent water used free from interferents at the MDL of target analytes? b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available? c. For standard preparation, is a waiting period of ten minutes allowed for drying the alcohol-wetted surface before measuring the weight of methanol? d. Are stock standards stored in bottles with minimal headspace and Teflon-lined screw-cap at -10 to -20°C and protected from light? e. Are stock standards replaced after six months, or sooner if comparison with check standards indicates a problem? f. Are stock standards for target analytes of low boiling points (<30°C) and high reactivity prepared fresh every two months or sooner?		

CHART I-10

ORGANIC ANALYSIS BY GC: HVO (8010A)

ITEM	YES	COMMENT
g. Are secondary standards stored with minimal headspace and check frequently for degradation or evaporation? h. For the initial calibration, are aqueous calibration standards, at a minimum of five concentrations, prepared fresh and discarded after one hour, unless properly sealed in a vial and stored at 4°C with no headspace (up to 24 hours)? i. Is a 25-µL Hamilton 702N microsyringe or equivalent used for standard preparation? (Pipets should never be used to dilute or transfer volatile samples or aqueous standards.) j. Are volatile organic standards stored in a separated freezer/refrigerator from samples or other standards? k. Is “purge-and trap”, “pesticide quality” or equivalent methanol stored away from other solvents? l. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage: a. Are volatile organic samples stored at 4°C in separate refrigerators from other samples? b. Are low concentration volatile organic samples stored separately from high concentration volatile organic samples?		

CHART I-10

ORGANIC ANALYSIS BY GC: HVO (8010A)

ITEM	YES	COMMENT
Instrument Calibration and Maintenance:		
a. Is there a calibration protocol readily available to bench chemists?		
b. Are calibration results kept in permanent logbooks?		
c. Is an initial calibration performed with a minimum of five concentration levels for each target analyte?		
d. Is one of the calibration standards at a concentration near, but above, the MDL?		
e. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?		
f. Is a linear calibration curve with a correlation coefficient $\geq$ 0.995 prepared for each analyte?		
g. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working ranges?		
h. Is the calibration curve or factor verified at the beginning and end of each analysis sequence with a mid-concentration standard?		
i. Is a new calibration curve prepared for any target analyte when the response for the target analyte varies from the predicted response by more than ±15% or exceeds the acceptance criteria listed in the Table 3 of Method 8010A?		

CHART I-10

ORGANIC ANALYSIS BY GC: HVO (8010A)

ITEM	YES	COMMENT
j. Is the retention time window established with three injections of all target analytes throughout the course of a 72-hour period? k. Is the retention time window checked on a quarterly basis or whenever a new GC column is installed?		
Sample Preparation: a. Is a combination of bromochloromethane, 2-bromo-l-chloropropane, and 1,4-dichlorobutane used as surrogate standards? b. Are samples routinely introduced into the GC using purge-and-trap (Method 5030)? c. Is methanolic extraction of purge-and-trap only used for medium-concentration soils or sediments? d. Is direct injection used only for water soluble compounds that do not purge or when concentrations are expected to exceed 10,000 µg/L? e. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?		
Sample Analysis: a. Is daily calibration checked with a mid-concentration standard at the beginning and the end of an analysis sequence?		

CHART I-10

ORGANIC ANALYSIS BY GC: HVO (8010A)

ITEM	YES	COMMENT
b. If the calibration factor calculated from daily calibration check at the end of an analysis sequence exceeds ±15% when compared with the initial standard of the analysis sequence, is the GC system recalibrated and reanalysis performed for all samples, in the sequence, that contain target analytes that exceed the criteria?		
c. Are daily retention time windows established for each analyte prior to sample analysis?		
d. Is the retention time for each analyte in the daily mid-concentration standard used as the midpoint of the window for that day?		
e. Is the same sample introduction method used for calibration standards and samples? (i.e., either purge-and-trap or direct injection, but not mixed methods.)		
f. If a peak response exceeds the linear range of the system, is a dilution performed on a second aliquot of the sample that has been properly sealed and stored prior to use?		
g. Are peak height measurements used for quantitation when overlapping peaks caused errors in area integration?		
h. Is a second GC column used to resolve the analytes from co-eluting non-target compounds?		
i. Are positive hits routinely confirmed by a second GC column?		

CHART I-10

ORGANIC ANALYSIS BY GC: HVO (8010A)

ITEM	YES	COMMENT
Quality Control:		
a. Are all QC data maintained and available for easy reference and inspection?		
b. Is a three-level data review carried out within the lab prior to data release?		
c. Is a lab specific MDL empirically established and updated on a semiannually basis?		
d. Is the lab specific MDL equal to or lower than the method specified MDL?		
e. Is a mid-concentration standard analyzed for each group of ten samples in the analysis sequence?		
f. Is a method blank run at a minimum rate of 5% or one per batch, whichever is greater?		
g. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations?		
(1) Is an LCS, prepared with standards independent of calibration standards, analyzed for each batch?		
(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?		

CHART I-10

ORGANIC ANALYSIS BY GC: HVO (8010A)

ITEM	YES	COMMENT
(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?		
h. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?		
(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever is higher?		
(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever is higher?		
(3) If it is not possible to determine the background concentration, is the spike concentration - the regulatory limit, if any; or - the larger of either five times the expected background or LCS concentrations?		

CHART I-10

ORGANIC ANALYSIS BY GC: HVO (8010A)

ITEM	YES	COMMENT
(4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit?		
(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?		
(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?		
i. Is the performance of purge-and-trap, analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with surrogates that encompass the method specified temperature range?		
j. Are the average percent recovery and standard deviation of the percent recovery for each surrogate calculated, when surrogate data from 25 to 30 samples for each matrix is available?		
k. Are control limits for each surrogate in a given matrix calculated based on the above data?		
l. Do the control limits fall within the control limits of Method 8240 if applicable?		
m. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?		

CHART I-10

ORGANIC ANALYSIS BY GC: HVO (8010A)

ITEM	YES	COMMENT
n. Are corrective actions of reanalysis or reextraction/reanalysis taken if any surrogates for a sample are out of control limits? o. Are control charts for internal QC data plotted and available to bench chemists? p. Are control limits for internal quality control empirically established and updated on a regular basis?		
Data Package: a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.) b. Does the data package contain all method required QC data and meet the USACE contract requirements? c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		
Waste Disposal: a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?		

CHART I-10

ORGANIC ANALYSIS BY GC: HVO (8010A)

ITEM	YES	COMMENT
b. Are lab wastes disposed of properly such that no secondary pollution is generated from sample analysis and the USACE will not be liable for any pollution problems in the future?		
Overall Evaluation: a. Does the lab have sound technical capability for HVO analysis? b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____ c. Could the lab handle quick turnaround samples? d. Overall, is the lab acceptable for HVO analysis?		

CHART I-10

ORGANIC ANALYSIS BY GC: HVO (8010A)

ITEM
Additional observations, comments, or problems:

CHART I-11

ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

ITEM	YES	COMMENT
General: a. Are written SOPs available and adequate for TPH sample preparation and analysis as gasoline range organics (GRO) and diesel range organics (DRO)? b. Do these SOPs accurately reflect procedures in use? c. Are manufacturer's operating manuals readily available to bench chemists? d. Are prenumbered, bound notebooks used for data entry? e. Are all records written in indelible ink? f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual? g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff: a. Do bench chemists appear knowledgeable and experienced in operation of a purge-and-trap and GC system and in interpretation of chromatograms? b. Are backup bench chemists available? c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		

CHART I-11

ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

ITEM	YES	COMMENT
Apparatus and Facilities: a. Is working space adequate and clean? b. Does the lab have adequate air handling system to avoid cross contamination of samples? c. Is a temperature-programmable gas chromatography equipped with a purge-and-trap device and flame ionization detector available for GRO? d. Is oven temperature stable to ±0.5°C or better at desired setting? e. Is a data system available for determination of peak area sums using forced baseline and baseline projection? f. Are the following GC columns available? GRO Analysis: (1) 105-m x 0.53-mm ID Restek RTX 502.2 0.3-micron film thickness? (2) Other capillary columns which can resolve 2-methylpentane from the methanol solvent front in a 25 µg/L LCS and to resolve ethylbenzene from m/p-xylene (<25% valley)? DRO Analysis: (1) 25-m x 0.25-mm Quadrex 007 5% methyl phenyl 0.5-micron film thickness? (2) 30-m x 0.53-mm ID Quadrex RTX-5, 1.5-micron film thickness?		

CHART I-11

ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

ITEM	YES	COMMENT
(3) Other capillary columns which can resolve C_{17}/pristane and C_{18}/phytane (>50% resolution)?		
g. If an "equivalent" column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?		
h. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		
i. Has any instrument been modified in any way?		
j. Is a hood available for sample preparation?		
k. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available?		
l. Is a horn-type sonicator equipped with a titanium tip and 475 Watt available in the lab?		
m. Are backup apparatus available?		
Reagents:		
a. Is reagent water used free from interferents at the MDL of target analytes?		
b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?		
c. Are "pesticide quality" or equivalent solvents used for TPH analysis?		

CHART I-11

ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

ITEM	YES	COMMENT
d. Is granular, anhydrous sodium sulfate purified by heating at 400°C for four hours prior to use?		
e. Does the lab use a calibration standard composed of a blend of the following typical ten gasoline compounds for GRO?		
2-methylpentane 15% wt. 2,2,4-trimethylpentane 15% heptane 5% benzene 5% toluene 15% ethylbenzene 5% m-xylene 10% p-xylene 10% o-xylene 10% l,2,4-trimethylbenzene 10%		
f. Does the lab use a calibration standard composed of a blend of the following typical 14 C_{10}-C_{28} even normal alkane standards, plus n-C_{17}, pristane, and phytane for DRO?		
decane ≈7% wt. dodecane ≈7% tetradecane ≈7% hexadecane ≈7% heptadecane ≈7% pristane ≈7% octadecane ≈7% phytane ≈7% eicosane ≈7% decosane ≈7% tetracosane ≈7% hexacosane ≈7% octacosane ≈7% 5 –α–androstane (I.S.) ≈7%		

CHART I-11

ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

ITEM	YES	COMMENT
g. Are the materials of interest, if available, or the same type of petroleum fraction, if it is known and original sample is unavailable, used for preparation of calibration standards?		
h. Is an internal standard used for DRO samples to correct for injection variances and matrix interferences?		
i. Does the lab have, at a minimum, the following Pattern Recognition Standards for identification of petroleum hydrocarbons? Gasoline, aviation fuel, (JP-4), kerosene, and diesel fuel (#2).		
j. Does the lab use a well characterized gasoline (e.g., API PS-6 or equivalent) and a commercial diesel #2 as LCSs for GRO and DRO, respectively?		
k. Are stock standards for GRO prepared in methanol and replaced after six months, or sooner, if comparison with check standards indicates a problem?		
l. Are stock standards for DRO prepared in acetone and replaced after six months, or sooner, if comparison with check standards indicates a problem?		
m. Are secondary dilution standards in methanol stored with minimum headspace for volatiles and frequently checked for signs of degradation/evaporation?		
n. Are working standards at a minimum of five concentration levels prepared in reagent water?		

CHART I-11

ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

ITEM	YES	COMMENT
o. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage: a. Are triplicate water samples in 40 mL VOA vials received for GRO analysis? b. Are water GRO samples inverted and checked for existence of air bubbles? c. Are soil GRO samples in wide mouth glass jars with Teflon-lined septa checked (without opening the container) for existence of excess headspace? d. Are duplicate samples collected for the alternate methanol extraction method? e. Are low level GRO samples stored at 4°C in a separate refrigerators from high level GRO samples? f. Are GRO samples analyzed within 14 days from collection? g. Are water DRO samples stored at 4°C, and extracted within seven days from collection and analyzed within 40 days from extraction? h. Are soil DRO samples stored at 4°C, and extracted within 14 days from collection and analyzed within 40 days from extraction?		

CHART I-11

ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

ITEM	YES	COMMENT
Instrument Calibration and Maintenance:		
a. Is there a calibration protocol readily available to bench chemists?		
b. Are calibration results kept in permanent logbooks?		
c. Is the GC system calibrated with a minimum of five concentration levels of calibration standard blenders?		
d. Is one of the calibration standards at a concentration near, but above, the MDL?		
e. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?		
f. Are the calibration standards for GRO injected using a purge-and-trap?		
g. For DRO, is a methylene chloride blank run in every batch to determine the area generated on normal baseline bleed between C_{10} and C_{28} and subtracted from the total areas of DR0 standards and samples?		
h. Is a linear calibration curve with a correlation coefficient $\geq$ 0.995 prepared for each analyte?		
i. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working ranges?		

CHART I-11

ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

ITEM	YES	COMMENT
j. Is the calibration curve or factor verified with, at a minimum, a midpoint calibration standard at the beginning and end of each analysis sequence? k. Is a new calibration curve prepared for any target analyte when the response factors for the daily calibrations vary from the initial response factors by more than 15%? l. Is the retention time window established with three injections of each calibration standard over the course of a 72-hour period? m. Are the retention time windows, specially for surrogates, internal standards, and the first and the last components in calibration standards, checked on a quarterly basis or whenever a new GC column is installed? n. Are the retention times for surrogates, internal standards, and the first and the last components in the daily mid-concentration standard used as the midpoints of the windows for that day?		
Sample Preparation: a. Is a purge-and-trap device used to inject water GRO samples to a GC? b. Are soil and solid GRO samples extracted by methanol extraction, diluted in water and injected with purge-and-trap to a GC? c. Are all supernatant liquids retained in methanol extraction process for GRO samples?		

CHART I-11

ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

ITEM	YES	COMMENT
d. Are water DRO samples extracted by EPA Methods 3510/3520 and soil DRO samples by EPA Methods 3540/3550? e. Is the water meniscus on the side of bottle marked for later determination of the sample volume? f. Is the pH of water DRO samples checked and adjusted with 10 N NaOH or 1:1 H_2SO_4 to 5-9? g. Is the water DRO sample containers rinsed with methylene chloride? (Do not cap and shake the bottle.) h. Is continuous extraction method used if emulsion forms and cannot be broken during separatory funnel method such that the recovery of methylene chloride is less than 80% after correction for water volubility of methylene chloride? i. For soil DRO samples, are large rocks or foreign materials removed and any vegetation chopped into small pieces? j. Are soil DRO samples sonicated for 1.5 minutes at 475 watts, one second pulse mode with a 50% duty cycle? k. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?		
Sample Analysis: a. Are the concentrations of all analytes within the initial calibration ranges?		

CHART I-11

ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015) Page 10 of 15

ITEM	YES	COMMENT
b. Is a method/instrument blank analyzed after a sample that produces a saturated response from a compound?		
c. If the method/instrument blank is not free from interferences, is the system decontaminated before sample analysis?		
d. Is the quantitation of GRO based on the area summation of all peaks that are above instrument blank baseline and elute between 2-methylpentane and l,2,4-trimethylbenzene?		
e. Are non-petroleum hydrocarbons, such as chlorinated solvents, ketones, and esters, excluded from the GRO quantitation?		
f. Is the total peak area for C_{10}-C_{28} from baseline-to-baseline used for DRO quantitation?		
g. Are non-petroleum hydrocarbons, such as chlorinated solvents, phenols, and phthalates, excluded from the DRO quantitation?		
h. Are retention times and patterns of the peaks used in identification of the type of petroleum hydrocarbons?		
i. Are comments provided for contaminants that appear in the GRO and DRO windows but do not match the reference fuels?		
j. Is internal standard, 5 –α–androstane, used as a retention time marker for DRO samples?		

CHART I-11

ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015) Page 11 of 15

ITEM	YES	COMMENT
Quality Control: a. Are all QC data maintained and available for easy reference and inspection? b. Is a three-level data review carried out within the lab before data release? c. Are lab specific MDL or PQL empirically established and updated on a semiannually basis? d. Is the lab specific PQL equal to or lower than the method specified PQL? **GRO:** water 100 µg/L soil 5 mg/kg **DRO:** water 100 µg/L (diesel #2) soil 4 mg/kg (diesel #2) e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent? f. For GRO/GRO samples, are duplicate LCSs analyzed at a minimum rate of 5% or one per batch, whichever is more frequent? g. Is the percent recovery of LCS larger than 50% and the percent difference less than 20%? h. Is a column bleed profile run for each batch of DRO samples to determine the area from normal baseline bleeding and subtracted from the area of DRO samples?		

CHART I-11

ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

ITEM	YES	COMMENT
i. Is a methylene chloride blank run after samples of highly concentrated to prevent carryover?		
j. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?		
(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever concentration would be higher?		
(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?		
(3) If it is not possible to determine the background concentration, is the spike concentration - the regulatory limit, if any; or - the larger of either five times the expected background or LCS concentrations?		
(4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit?		

CHART I-11

ORGANIC ANALYSIS BY GC TPH (MODIFIED 8015)

ITEM	YES	COMMENT
(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?		
(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?		
k. Does the lab use one or two surrogate compounds, p-chlorofluorobenzene, bromofluorobenzene, or trifluoro-toluene, to monitor the system performance and effectiveness of the of the GRO method in dealing with each matrix?		
l. Does the lab use one or two surrogate compounds, n-pentacosane (n-C_{25}) or ortho-terphenyl, to monitor the system performance and effectiveness of the of the DRO method in dealing with each matrix?		
m. Has the lab established control limits for surrogate recoveries?		
n. Are corrective actions of reanalysis or reextraction/reanalysis taken if surrogate(s) for a sample are out of control limits?		
o. Are control charts for internal QC data plotted and available to bench chemists?		
p. Are control limits for internal quality control empirically established and updated on a regular basis?		

CHART I-11

ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

ITEM	YES	COMMENT
Data Package: a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.) b. Does the data package contain all method required QC data and meet the USACE contract requirements? c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		
Waste Disposal: a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled? b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		
Overall Evaluation: a. Does the lab have sound technical capability for TPH analysis?		

CHART I-11

ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____		
c. Could the lab handle quick turnaround samples?		
d. Overall, is the lab acceptable for TPH analysis?		
Additional observations, comments, or problems:		

CHART I-12

ORGANIC ANALYSIS BY GC: AVO (8020)

ITEM	YES	COMMENT
General: a. Are written SOPs available and adequate for AVO sample preparation/analysis? b. Do these SOPs accurately reflect procedures in use? c. Are manufacturer's operating manuals readily available to bench chemists? d. Are prenumbered, bound notebooks used for data entry? e. Are all records written in indelible ink? f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible? g. Are corrections initialed and dated by the responsible individual? h. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff: a. Do bench chemists appear knowledgeable and experienced in operation of a purge-and-trap and GC system and in interpretation of chromatograms? b. Are backup bench chemists available? c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		

CHART I-12

ORGANIC ANALYSIS BY GC: AVO (8020)

ITEM	YES	COMMENT
Apparatus and Facilities:		
a. Is working space adequate and clean?		
b. Does the lab have adequate air handling system to avoid cross contamination of samples?		
c. Is a temperature-programmable gas chromatography equipped with an purge-and-trap device and photo-ionization detector available?		
d. Is oven temperature stable to ±0.5°C or better at desired setting?		
e. Is one of the following GC column available?		
(1) 6-ft x 0.082-in ID SS or glass column packed with 5% SP-1OOO and 1.75% Bentone-34 on 100/120 mesh Supelcoport or equivalent?		
(2) 8-ft x 0.1-in ID SS or glass column packed with 5% l,2,3-Tris(2-cyano-ethoxy)propane on 60/80 mesh Chromosorb W-AW or equivalent?		
(3) IS column one used as the primary analytical column and column two as a confirmation column?		
f. If an “equivalent” column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?		
g. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		

CHART I-12

ORGANIC ANALYSIS BY GC: AVO (8020)

ITEM	YES	COMMENT
h. Has any instrument been modified in any way? i. Is a hood available for sample preparation? j. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available? k. Are backup instruments available?		
Reagents: a. Is reagent water used free from interferents at the MDL of target analytes? b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available? c. For standard preparation, is a waiting period of ten minutes allowed for drying the alcohol-wetted surface before measuring the weight of methanol? d. Are stock standards stored in bottles with minimal headspace and Teflon-lined screw-cap at -4°C and protected from light? e. Are stock standards replaced after six months, or sooner if comparison with check standards indicates a problem? f. Are secondary standards stored with minimal headspace and check frequently for degradation or evaporation?		

CHART I-12

ORGANIC ANALYSIS BY GC: AVO (8020)

ITEM	YES	COMMENT
g. For the initial calibration, are aqueous calibration standards, at a minimum of five concentrations, prepared fresh and discarded after one hour, unless properly sealed in a vial and stored at 4°C with no headspace (up to 24 hours)? h. Is a 25 µL Hamilton 702N microsyringe or equivalent used for standard preparation? (Pipets should never be used to dilute or transfer volatile samples or aqueous standards.) i. Are volatile organic standards stored in a separated freezer/refrigerator from samples or other standards? j. Is “purge-and-trap”, “pesticide quality”, or equivalent methanol stored away from other solvents? k. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage: a. Are volatile organic samples stored at 4°C in separate refrigerators from other samples? b. Are low concentration volatile organic samples stored separately from high concentration volatile organic samples?		
Instrument Calibration and Maintenance: a. Is there a calibration protocol readily available to bench chemists?		

CHART I-12

ORGANIC ANALYSIS BY GC: AVO (8020) Page 5 of 13

ITEM	YES	COMMENT
b. Are calibration results kept in permanent logbooks?		
c. Is an initial calibration performed with a minimum of five concentration levels for each target analyte?		
d. Is one of the calibration standards at a concentration near, but above, the MDL?		
e. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?		
f. Is a linear calibration curve with a correlation coefficient $\geq$ 0.995 prepared for each analyte?		
g. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working range?		
h. Is the calibration curve or factor verified at the beginning and end of each analysis sequence with a mid-concentration standard?		
i. Is a new calibration curve prepared for any target analyte when the response for the target analyte varies from the predicted response by more than ±15% or exceeds the acceptance criteria listed in the Table 3 of Method 8020?		
j. Is the retention time window established with three injections of all target analytes throughout the course of a 72-hour period?		

CHART I-12

ORGANIC ANALYSIS BY GC: AVO (8020)

ITEM	YES	COMMENT
k. Is the retention time window checked on a quarterly basis or whenever a new GC column is installed?		
Sample Preparation: a. Are surrogate compounds, bromochlorobenzene, bromofluorobenzene, 1,1,1-trifluorotoluene, fluorobenzene, and difluorobenzene, which encompass the temperature range of this method used for all samples? b. Are samples routinely introduced into the GC using purge-and-trap (Method 5030)? c. Is methanolic extraction of purge-and-trap only used for medium-concentration soils or sediments? d. Is direct injection used only for water soluble compounds that do not purge or when concentrations are expected to exceed 10,000 µg/L? e. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?		
Sample Analysis: a. Is daily calibration performed with a mid-concentration standard at the beginning and the end of an analysis sequence?		

CHART I-12

ORGANIC ANALYSIS BY GC: AVO (8020)

ITEM	YES	COMMENT
b. If the calibration factor calculated from daily calibration check at the end of an analysis sequence exceeds ±15% when compared with the initial standard of the analysis sequence, is the GC system recalibrated and reanalysis performed for all samples, in the sequence, which contain target analytes that exceed the criteria?		
c. Are daily retention time windows established for each analyte prior to sample analysis?		
d. Is the retention time for each analyte in the daily mid-concentration standard used as the midpoint of the window for that day?		
e. Is the same sample introduction method used for calibration standards and samples? (i.e., either purge-and-trap or direct injection, but not mixed methods.)		
f. If a peak response exceeds the linear range of the system, is a dilution performed on a second aliquot of the sample that has been properly sealed and stored prior to use?		
g. Are peak height measurements used for quantitation when overlapping peaks caused errors in area integration?		
h. Is a second GC column used to resolve the analytes from co-eluting non-target compounds?		
i. Are positive hits routinely confirmed by a second GC column?		

CHART I-12

ORGANIC ANALYSIS BY GC: AVO (8020)

ITEM	YES	COMMENT
Quality Control:		
a. Are all QC data maintained and available for easy reference and inspection?		
b. Is a three-level data review carried out within the lab prior to data release?		
c. Is a lab specific MDL empirically established and updated on a semiannually basis?		
d. Is the lab specific MDL equal to or lower than the method specified MDL?		
e. Is a mid-concentration standard analyzed for each group of 10 samples in the analysis sequence?		
f. Is a method blank run at a minimum rate of 5% or one per batch, whichever is greater?		
g. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations?		
(1) Is an LCS prepared with standards independent of calibration standards analyzed for each batch?		
(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?		

CHART I-12

ORGANIC ANALYSIS BY GC: AVO (8020)

ITEM	YES	COMMENT
(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?		
h. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?		
(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever concentration would be higher?		
(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?		
(3) If it is not possible to determine the background concentration, is the spike concentration - the regulatory limit, if any; or		

CHART I-12

ORGANIC ANALYSIS BY GC: AVO (8020)

ITEM	YES	COMMENT
- the larger of either five times the expected background or LCS concentrations?		
(4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit?		
(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?		
(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?		
i. Is the performance of purge-and-trap, analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with surrogates which encompass the method specified temperature range?		
j. Are the average percent recovery and standard deviation of the percent recovery for each surrogate calculated, once a minimum of 30 samples of the same matrix have been analyzed?		
k. Are control limits for each surrogate in a given matrix calculated based on the above data?		
l. Do the control limits fall within the control limits of Method 8240 if applicable?		

CHART I-12

ORGANIC ANALYSIS BY GC: AVO (8020)

ITEM	YES	COMMENT
m. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis? n. Are corrective actions of reanalysis or reextraction/reanalysis taken if any surrogates for a sample are out of control limits? o. Are control charts for internal QC data plotted and available to bench chemists? p. Are control limits for internal quality control empirically established and updated on a regular basis?		
Data Package: a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.) b. Does the data package contain all method required QC data and meet the USACE contract requirements? c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		

CHART I-12

ORGANIC ANALYSIS BY GC: AVO (8020)

ITEM	YES	COMMENT
Waste Disposal: a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled? b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		
Overall Evaluation: a. Does the lab have sound technical capability for AVO analysis? b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____ c. Could the lab handle quick turnaround samples? d. Overall, is the lab acceptable for AVO analysis?		

CHART I-12

ORGANIC ANALYSIS BY GC: AVO (8020)

ITEM
Additional observations, comments, or problems:

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A) Page 1 of 13

ITEM	YES	COMMENT
General: a. Are written SOPs available and adequate for phenols sample preparation and analysis? b. Do these SOPs accurately reflect procedures in use? c. Are manufacturers operating manuals readily available to bench chemists? d. Are prenumbered, bound notebooks used for data entry? e. Are all records written in indelible ink? f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual? g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff: a. Do bench chemists appear knowledgeable and experienced in operation of a GC system and interpretation of chromatograms? b. Are backup bench chemists available? c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

ITEM	YES	COMMENT
Apparatus and Facilities:		
a. Is working space adequate and clean?		
b. Are enough sets of separator funnels, continuous liquid-liquid extractors, Soxhlet extractors, and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples?		
c. Is oven temperature stable to ±0.5°C or better at desired setting?		
d. Is a 1.8-m x 2-mm ID glass column packed with 1% SP-1240 DA on Supercoport (80/100 mesh) or an equivalent column in use for the determination of underivatized phenols?		
e. Is a flame ionization detector available for the determination of underivatized phenols?		
f. Is nitrogen carrier gas available for use with the FID?		
g. Is a 1.8-m x 2-mm ID glass column packed with 5% OV-17 on Chromosorb W-AW-DMCS (80/100 mesh) or an equivalent column in use for the determination of derivatized phenols?		
h. Is an electron capture detector (ECD) available for the determination of derivatized phenols?		
i. Is 5% methane/95% argon carrier gas available for use with the ECD?		

CHART 1-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

ITEM	YES	COMMENT
j. If an "equivalent" column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated? k. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records? l. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records? m. Has any instrument been modified in any way? n. Is a hood available for sample preparation? o. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available? p. Are backup apparatus available?		
Reagents: a. Is reagent water used free from interferents at the MDL of target analytes? c. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available? c. Are the following reagents available for use in derivatization: (1) Pentafluorobenzene bromide (α –Bromopentafluorotoluene)?		

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

ITEM	YES	COMMENT
(2) 18-crown-6-ether (1,4,7,10,13,16-Hexaoxacyclooctadecane)? d. Are derivatization reagents prepared fresh weekly and stored at 4°C from light? e. Are "pesticide quality" or equivalent solvents used for sample analysis? f. Does the lab have calibration standards for all method specified target analytes? g. Are calibration standards prepared with 2-propanol as a solvent? h. Are stock standard solutions stored at 4°C and protected from light? i. Are stock standard solutions replaced after one year, or sooner if comparison with check standards indicates a problems? j. Are working standards replaced after six months, or sooner if comparison with calibration standards indicates a problems? k. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage: a. Are aqueous samples stored at 4°C, and extracted within seven days from collection and analyzed within 40 days from extraction?		

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

ITEM	YES	COMMENT
b. Are soil samples stored at 4°C, and extracted within 14 days from collection and analyzed within 40 days from extraction?		
Instrument Calibration and Maintenance: a. Is there a calibration protocol readily available to bench chemists? b. Are calibration results kept in permanent logbooks? c. Is an initial calibration performed with a minimum of five concentration levels for each target analyte? d. Is one of the calibration standards at a concentration near, but above, the MDL? e. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector? f. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working range? g. Is the calibration curve or factor verified at the beginning and end of each analysis sequence with a mid-concentration standard? h. Is a new calibration curve prepared for any target analyte when the response for the target analyte varies from the predicted response by more than ±15%?		

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

ITEM	YES	COMMENT
i. Is the retention time window established with three injections of all target analytes throughout the course of a 72-hour period? j. Is the retention time window checked on a quarterly basis or whenever a new GC column is installed?		
Sample Preparation: a. Are aqueous samples extracted at a pH ≤2 with methylene chloride, using Method 351OA or 3520A? b. Are solid samples extracted using either Method 3540A or 3550? c. Are extracts from either Method 3520A or 3550 undergone acid-base partition cleanup, using Method 3650A? d. Is the extraction solvent exchanged to 2-propanol prior to GC analysis? e. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?		
Sample Analysis: a. Is daily calibration performed with a mid-concentration standard prior to sample analysis? b. Is daily calibration checked at the end of an analysis sequence?		

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

ITEM	YES	COMMENT
c. If the calibration factor calculated from daily calibration check at the end of an analysis sequence exceeds ±15% when compared with the initial standard of the analysis sequence, is the GC system recalibrated and reanalysis performed for all samples, in the sequence, which contain target analytes that exceed the criteria?		
d. Are daily retention time windows established for each analyte prior to sample analysis?		
e. Is the retention time for each analyte in the daily mid-concentration standard used as the midpoint of the window for that day?		
f. Is solvent flush technique used to inject samples to GC?		
g. If interferences prevent measurement of peak area during analysis by an FID, is the phenol extract derivatized by pentafluorobenzylbromide (PFB) and the derivatized extract cleaned up using Method 3630A (silica gel cleanup) and analyzed by an ECD?		
h. If the peak areas exceed the linear range of the system, is the extract diluted and reanalyzed?		
i. Are peak height measurements used for quantitation when overlapping peaks caused errors in area integration?		
j. Are any positive hits confirmed by a second GC column (or by GC/MS if the concentration of each positive hit exceeds 10 ng/µL in the final extract)?		

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

ITEM	YES	COMMENT
k. If sample extracts are cleaned up with Methods 3630A/3650A, are the associated QC samples processed through the same methods?		
Quality Control: a. Are all QC data maintained and available for easy reference and inspection? b. Is a three-level data review carried out within the lab prior to data release? c. Is a lab specific MDL empirically established and updated on a semiannually basis? d. Is the lab specific MDL equal to or lower than the method specified MDL? e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent? f. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations? (1) Is an LCS prepared with standards independent of calibration standards analyzed for each batch? (2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?		

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

ITEM	YES	COMMENT
(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the sample analysis halted until the system performance is back in control?		
g. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?		
(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever concentration would be higher?		
(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?		
(3) If it is not possible to determine the background concentration, is the spike concentration – the regulatory limit, if any; or - the larger of either five times the expected background or LCS concentrations?		

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

ITEM	YES	COMMENT
(4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit?		
(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?		
(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?		
h. Is the performance of extraction, cleanup (when used), analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with phenolic surrogates using 2-fluorophenol and 2,4, 6-tribromophenol to encompass the range of temperature used in this method?		
i. Are the average percent recovery and standard deviation of the percent recovery for each surrogate calculated, when surrogate data from 25 to 30 samples for each matrix is available?		
j. Are control limits for each surrogate in a given matrix calculated based on the above data?		
k. Do the control limits fall within the control limits of Method 8270 if applicable?		
l. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?		

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A) Page 11 of 13

ITEM	YES	COMMENT
m. Are corrective actions of reanalysis or reextraction/reanalysis taken if surrogates for a sample are out of control limits? n. Are control charts for internal QC data plotted and available to bench chemists? o. Are control limits for internal quality control empirically established and updated on a regular basis?		
Data Package: a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.) b. Does the data package contain all method required QC data and meet the USACE contract requirements? c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		
Waste Disposal: a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?		

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

ITEM	YES	COMMENT
b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		
Overall Evaluation: a. Does the lab have sound technical capability for phenols analysis? b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____ c. Could the lab handle quick turnaround samples? d. Overall, is the lab acceptable for phenols analysis?		
Additional observations, comments, or problems:		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

ITEM	YES	COMMENT
General: a. Are written SOPS available and adequate for PEST/PCB sample preparation and analysis? b. Do these SOPS accurately reflect procedures in use? c. Are manufacturer's operating manuals readily available to bench chemists? d. Are prenumbered, bound notebooks used for data entry? e. Are all records written in indelible ink? f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual? g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff: a. Do bench chemists appear knowledgeable and experienced in operation of a GC system and interpretation of chromatograms? b. Are backup bench chemists available? c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

ITEM	YES	COMMENT
d. Does the lab have experienced residue analysis experts on staff?		
Apparatus and Facilities: a. Is working space adequate and clean? b. Are enough sets of separatory funnels (2,000 mL with Teflon stopcock), Soxhlet extractors, and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples? c. Is gas chromatography equipped with an glass-lined injection port, and an electron capture or electrolytic conductivity detector available? d. Is oven temperature stable to ±0.5°C or better at desired setting? e. Is carrier-gas line equipped with a molecular sieve drying cartridge and a trap for removal of oxygen from the carrier gas? f. Is one of the following glass GC column available? (1) 1.8-m x 4-mm ID glass, packed with 1.5% SP-2250/l.95% SP-2401 on Supelcoport (100/120 mesh) or equivalent? (2) 1.8-m x 4-mm ID glass, packed with 3% OV-1 on Supelcoport (100/120 mesh) or equivalent? g. If an “equivalent” column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

ITEM	YES	COMMENT
h. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records? i. Has any instrument been modified in any way? j. Is a hood available for sample preparation? k. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available? l. Are backup apparatus available? m. Is glassware properly cleaned and finally rinsed with pesiticide-quality hexane? n. Is volumetric glassware cleaned with “No Chromix” or equivalent? o. Is heavily contaminated glassware heated in a muffle furnace at 400°C for 15 to 30 minutes? p. Is glassware contaminated with high-boiling-point materials, such as PCBS, heated at 500°C overnight? (Borosilicate glassware shall not be heated above this temperature.) q. Is high temperature treatment on volumetric glassware, glassware with ground joints, or sintered glassware avoid?		
Reagents: a. Is reagent water used free from interferents at the MDL of target analytes?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

ITEM	YES	COMMENT
b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available? c. Are all chemical reagents for pesticide and PCB analyses stored in glass containers? d. Are “pesticide quality” or equivalent solvents used for pesticide analysis? e. Are all solvents stored in glass containers and transferred with all glass system? f. Is 5% methane/95% argon carrier gas available? g. Are solvent extracted silicon carbide or equivalent used as boiling chips? h. Does the lab have calibration standards for all method specified target PCBs? i. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage: a. Are aqueous samples stored at 4°C, and extracted within seven days from collection and analyzed within 40 days from extraction? b. Are soil samples stored at 4°C, and extracted within 14 days from collection and analyzed within 40 days from extraction?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

ITEM	YES	COMMENT
Instrument Calibration and Maintenance:		
a. Is there a calibration protocol readily available to bench chemists?		
b. Are calibration results kept in permanent logbooks?		
c. If the GC is not used for a day or more, is the GC column primed or deactivated by injecting a PCB or pesticide standard mixture about 20 times more concentrated than the mid-level standard, prior to instrument calibration?		
d. Is a calibration blank run following the system prime to ensure no carryover contamination?		
e. Is a mid-level standard contain only 4,4'-DDT and endrin injected to check the degradation problem at injection port or front of the column prior to calibration?		
f. If the degradation of either DDT or endrin exceeds 20% (or 15% for capillary column) based on peak areas, is corrective action taken before proceeding with calibration?		
g. Is an initial calibration performed with a minimum of five concentration levels for each target analyte?		
h. Is one of the external standards at a concentration near, but above, the MDL?		

CHART I-14

ORGANIC AMALYSIS BY GC: PEST/PCB (8080)

ITEM	YES	COMMENT
i. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?		
j. Is a linear calibration curve with a correlation coefficient ≥ 0.995 prepared for each analyte?		
k. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working range?		
l. Is the total area of all peaks measured from the common baseline under all peaks used for quantitation of multiresponse analytes?		
m. Is the calibration curve or factor verified at the beginning and end of each analysis sequence with a mid-concentration standard?		
n. Is a new calibration curve prepared for any target analyte when the response or the target analyte varies from the predicted response by more than ±5%?		
o. Is the retention time window established with three injections of all single component standard mixtures and multiple response products throughout the course of a 72-hour period?		
p. Is the retention time window checked on a quarterly basis or whenever a new GC column is installed?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

ITEM	YES	COMMENT
Sample Preparation: a. Are aqueous samples extracted at a neutral, or as is, pH with methylene chloride, using Method 3510 or 3520? b. Are solid samples extracted using either Method 3540 or 3550? c. Is entire aqueous sample consumed for analysis and no analysis performed on aliquots of samples? d. Is sample bottle rinsed with extraction solvent and the rinsate combined with extract? e. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?		
Sample Analysis: a. Is daily calibration performed with a mid-concentration standard prior to sample analysis? b. Is daily calibration checked at the end of an analysis sequence? c. If the calibration factor based on daily calibration check at the end of an analysis sequence exceeds ±15% when compared with the initial standard of the analysis sequence, is the GC system recalibrated and reanalysis performed for all samples which contain target analytes that exceed the criteria? d. Are daily retention time windows established for each analyte prior to sample analysis?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

ITEM	YES	COMMENT
e. Is the retention time for each analyte in the daily mid-concentration standard used as the midpoint of the window for that day?		
f. Is the volume of sample injected recorded to the nearest 0.05 µL?		
g. If the peak areas exceed the linear range of the system, is the extract diluted and reanalyzed?		
h. Are peak height measurements used for quantitation when overlapping peaks caused errors in area integration?		
i. If peak detection and identification are prevented due to interference, does the extract routinely undergo a Florisil column cleanup (Method 3620A) and/or sulfur cleanup (Method 3660A) to eliminate interferences?		
j. Is mercury, activated copper powder, or tetrabutylammonium (TBA)-sulfite reagent used for sulfur cleanup?		
k. Is microcoulometric or halogen specific (i.e., electrolytic conductivity) detector used to eliminate interference caused by phthalate esters?		
l. Are any positive hits confirmed by a second GC column (or by GC/MS if the concentration of each positive hit exceeds 10 ng/µL in the final extract)?		
m. If the early portion of toxaphene chromatogram is interfered with by other substances, is area of the last four peaks in both sample and standard used for quantitation?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

ITEM	YES	COMMENT
n. If chlordane residue does not resemble technical chlordane, but instead consists primarily of individual, identifiable peaks, is each peak quantitated separately against appropriate reference materials and reported as individual residues? o. Is the total area of all peaks measured from the common baseline under all peaks used for PCB quantitation? p. Are only those peaks that can be attributed to chlorobiphenyls used for PCB quantitation? q. If there are interference peaks within the Aroclor pattern, is the PCB quantitation determined with three to five major peaks that are ≥25% of the height of the largest Aroclor peak in the Aroclor standards? r. Is the amount of Aroclor calculated with the individual response factor for each of the major peaks and are the results of the three to five determinations averaged?		
Quality Control: a. Are all QC data maintained and available for easy reference and inspection? b. Is a three-level data review carried out within the lab prior to data release? c. Is a lab specific MDL empirically established and updated on a semiannually basis?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

ITEM	YES	COMMENT
d. Is the lab specific MDL equal to or lower than the method specified MDL?		
e. Is a separate set of internal QC samples including method blanks, LCS, matrix spikes, matrix spike duplicates and matrix duplicates run for each analytical batch of pesticides or PCB?		
f. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?		
g. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations?		
(1) Is an LCS prepared with standards independent of calibration standards analyzed for each batch?		
(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?		
(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

ITEM	YES	COMMENT
h. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples? (1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever concentration would be higher? (2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher? (3) If it is not possible to determine the background concentration, is the spike concentration - the regulatory limit, if any; or - the larger of either five times the expected background or LCS concentrations? (4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit? (5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

ITEM	YES	COMMENT
(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?		
i. Is the performance of extraction, cleanup (when used), analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with pesticide surrogates using 2,4,5,6-tetrachloro-meta-xylene (TCMX) and decachlorobiphenyl (DCBP) as specified by the method?		
j. Are the average percent recovery and standard deviation of the percent recovery for each surrogate calculated, when surrogate data from 25 to 30 samples for each matrix is available?		
k. Are control limits for each surrogate in a given matrix calculated based on the above data?		
l. Do the control limits fall within the control limits of Method 8270 if applicable?		
m. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?		
n. Are corrective actions of reanalysis or reextraction/reanalysis taken if both surrogates for a sample are out of control limits?		
o. Are control charts for internal QC data plotted and available to bench chemists?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080) Page 13 of 15

ITEM	YES	COMMENT
p. Are control limits for internal quality control empirically established and updated on a regular basis?		
Data Package: a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.) b. Does the data package contain all method required QC data and meet the USACE contract requirements? c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		
Waste Disposal: a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled? b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080) Page 14 of 15

ITEM	YES	COMMENT
Overall Evaluation: a. Does the lab have sound technical capability for PEST/PCB analyses? b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____ c. Could the lab handle quick turnaround samples? d. Overall, is the lab acceptable for PEST/PCB analyses?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

ITEM
Additional observations, comments, or problems:

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

ITEM	YES	COMMENT
General: a. Are written SOPS available and adequate for PAH sample preparation/analysis? b. Do these SOPS accurately reflect procedures in use? c. Are manufacturer's operating manuals readily available to bench chemists? d. Are prenumbered, bound notebooks used for data entry? e. Are all records written in indelible ink? f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual? g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff: a. Do bench chemists appear knowledgeable and experienced in operation of a GC system and interpretation of chromatograms? b. Are backup bench chemists available? c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

ITEM	YES	COMMENT
Apparatus and Facilities:		
a. Is working space adequate and clean?		
b. Are enough sets of separatory funnels, continuous liquid-liquid extractors, Soxhlet extractors, and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples?		
c. Is oven temperature stable to ±0.5°C or better at desired setting?		
d. Is one of the following glass GC column available?		
(1) 1.8-m x 2-mm ID glass column packed with 3% OV-17 on Chromosorb W-AW-DCMS (100/120 mesh) or equivalent?		
(2) 30-m x 0.25-mm ID SE-54 fused silica capillary column?		
(3) 30-m x 0.32-mm ID SE-54 fused silica capillary column?		
e. If capillary column is in use, is helium used as the carrier gas?		
f. If packed column is in use, is nitrogen used as the carrier gas?		
g. Is a flame ionization detector available?		
h. If an “equivalent” column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

ITEM	YES	COMMENT
i. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records? j. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records? k. Has any instrument been modified in any way? l. Is a hood available for sample preparation? m. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available? n. Are backup apparatus available?		
Reagents: a. Is reagent water used free from interferents at the MDL of target analytes? b. Are reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available? c. Are "pesticide quality" or equivalent solvents used for sample analysis? d. Does the lab have calibration standards for all method specified target analytes? e. Are calibration standards prepared with isooctane as a solvent?		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

ITEM	YES	COMMENT
f. Are stock standard solutions stored at 4°C and protected from light? g. Are stock standard solutions replaced after one year, or sooner if comparison with check standards indicates a problem? h. Are working standards replaced after six months, or sooner if comparison with check standards indicates a problem? i. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage: a. Are aqueous samples stored at 4°C, and extracted within seven days from collection and analyzed within 40 days from extraction? b. Are soil samples stored at 4°C, and extracted within 14 days from collection and analyzed within 40 days from extraction?		
Instrument Calibration and Maintenance: a. Is there a calibration protocol readily available to bench chemists? b. Are calibration results kept in permanent logbooks? c. Is an initial calibration performed with a minimum of five concentration levels for each target analyte?		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

ITEM	YES	COMMENT
d. Is one of the external standards at a concentration near, but above, the MDL? e. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector? f. Is a linear calibration curve with a correlation coefficient $\geq$0.995 prepared for each analyte? g. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working range? h. Is the calibration curve or factor verified at the beginning and end of each analysis sequence with a mid-concentration standard? i. Is a new calibration curve prepared for any target analyte when the response for the target analyte varies from the predicted response by more than ±15%? j. Is retention time window established with three injections of all single component standard mixtures and multiple response products throughout the course of a 72-hour period? k. Is retention time window checked on a quarterly basis or whenever a new GC column is installed?		
Sample Preparation: a. Are aqueous samples extracted at a neutral pH with methylene chloride, using Method 3510 or 3520?		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

ITEM	YES	COMMENT
b. Are solid samples extracted using either Method 3540 or 3550? c. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?		
Sample Analysis: a. Is daily calibration performed with a mid-concentration standard prior to sample analysis? b. Is daily calibration checked at the end of an analysis sequence? c. If the calibration factor calculated from daily calibration check at the end of an analysis sequence exceeds ±15% when compared with the initial standard of the analysis sequence, is the GC system recalibrated and reanalysis performed for all samples, in the sequence, which contain target analytes that exceed the criteria? d. Are daily retention time windows established for each analyte prior to sample analysis? e. Is the retention time for each analyte in the daily mid-concentration standard used as the midpoint of the window for that day? f. If peak detection and identification are prevented due to interferences, is the extract undergone Method 3630 (Silica Gel Cleanup)?		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

ITEM	YES	COMMENT
g. If the peak areas exceed the linear range of the system, is the extract diluted and reanalyzed? h. Is peak height measurement used for quantitation when overlapping peaks caused errors in area integration? i. Are any positive hits confirmed by a second GC column (or by GC/MS if the concentration of each positive hit exceeds 10 ng/µL in the final extract)?		
Quality Control: a. Are all QC data maintained and available for easy reference and inspection? b. Is a three-level data review carried out within the lab prior to data release? c. Is a lab specific MDL empirically established and updated on a semiannually basis? d. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent? e. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations? (1) Is an LCS prepared with standards independent of calibration standards analyzed for each batch?		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

ITEM	YES	COMMENT
(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?		
(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?		
f. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?		
(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever concentration would be higher?		
(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever would be higher?		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

ITEM	YES	COMMENT
(3) If it is not possible to determine the background concentration, is the spike concentration - the regulatory limit, if any; or - the larger of either five times the expected background or LCS concentrations? (4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit? (5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria? (6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration? g. Is the performance of extraction, cleanup (when used), analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with one or two surrogates, e.g., 2-fluorobiphenyl & l-fluoronaphthalene, to encompass the range of temperature used in this method? h. Are the average percent recovery and standard deviation of the percent recovery for each surrogate calculated to establish control limits, when surrogate data from 25 to 30 samples for each matrix is available?		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

ITEM	YES	COMMENT
i. Do the control limits fall within those of Method 8270 if applicable?		
j. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?		
k. Are corrective actions of reanalysis or reextraction/reanalysis taken if surrogates for a sample are out of control limits?		
l. Are control charts for internal QC data plotted and available to bench chemists?		
m. Are control limits for internal quality control empirically established and updated on a regular basis?		
n. Because of coelution problems, is the use of this method avoided and the sample analyzed by either HPLC or GC/MS when the four pairs of compounds listed below are encountered?		
(1) Anthracene and phenanthrene		
(2) Chrysene and benzo(a)anthracene		
(3) Benzo(b) fluoroanthene and benzo(k) fluoranthene		
(4) Dibenzo(a,h) anthracene and indeno(l,2,3-cd)pyrene		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8200)

ITEM	YES	COMMENT
Data Package: a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.) b. Does the data package contain all method required QC data and meet the USACE contract requirements? c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		
Waste Disposal: a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled? b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		
Overall Evaluation: a. Does the lab have sound technical capability for PAH analysis?		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____ c. Could the lab handle quick turnaround samples? d. Overall, is the lab acceptable for PAH analysis?		
Additional observations, comments, or problems:		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

ITEM	YES	COMMENT
General: a. Are written SOPS available and adequate for HERB sample preparation/analysis? b. Do these SOPS accurately reflect procedures in use? c. Are manufacturer's operating manuals readily available to bench chemists? d. Are prenumbered, bound notebooks used for data entry? e. Are all records written in indelible ink? f. Are all errors corrected by drawing a single line through the error with correction written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual? g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff: a. Do bench chemists appear knowledgeable and experienced in operation of a GC system and interpretation of chromatograms? b. Are backup bench chemists available? c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

ITEM	YES	COMMENT
d. Do bench chemists have proper experience in working with diazomethane which is explosive and carcinogenic?		
Apparatus and Facilities: a. Is working space adequate and clean? b. Is a temperature-programmable gas chromatography equipped with an electron capture detector, microcoulometric detector, or electrolytic conductivity detectors? c. Is oven temperature stable to ±0.5°C or better at desired setting? d. Is one of the following glass GC column available? (1) 1.8-m x 4-mm ID glass, packed with 1.5% SP-2250/l.95% SP-2401 on Supelcoport (100/120 mesh) or equivalent? (2) 1.8-m x 4-mm ID glass, packed with 5% OV-210 on Gas Chrom Q (100/120 mesh) or equivalent? (3) 1.98-m x 2-mm ID glass, packed with 0.1% SP-1000 on Carbopack C (80/100 mesh) or equivalent? (4) Is column one used as the primary analytical column and columns two or three as a confirmation column? e. If an “equivalent” column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

ITEM	YES	COMMENT
f. Is a diazomethane generator available at the lab? g. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records? h. Has any instrument been modified in any way? i. Are glassware and glass wool acid rinsed prior to use? j. Are boiling chips solvent extracted? k. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available? l. Are backup instruments available?		
Reagents: a. Is reagent water used free from interferents at the MDL of target analytes? b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available? c. Are pesticide-quality or equivalent solvents (i.e., acetone, methanol, and hexane) used? d. Is diethyl ether of pesticide quality or equivalent and free of peroxides used?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

ITEM	YES	COMMENT
e. Is 20 mL of ethyl alcohol preservative added to each liter of cleaned diethyl ether? f. Is sodium sulfate purified by heating at 400°C for four hours or by precleaning with methylene chloride? g. Is sodium sulfate acidified with sulfuric acid prior to use to avoid reaction with herbicides? h. Are stock standards stored in bottles with Teflon-lined screw caps or crimp tops at 4°C and protected from light? i. Are stock standards replaced after one year, or sooner if comparison with check standards indicates a problem? j. Are working standards replaced after six months or sooner, if comparison with check standards indicates a problem? k. Does the lab use one or two herbicides, that are not expected to be presented in the sample and that elute over the temperature range of this method, as surrogate(s)? l. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage: a. Are herbicide samples stored at 4°C and extracted within seven days (water) or 14 days (soil) from collection?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

ITEM	YES	COMMENT
b. Are extracts stored under refrigeration and analyzed within 40 days from extraction?		
Instrument Calibration and Maintenance: a. Is there a calibration protocol readily available to bench chemists? b. Are calibration results kept in permanent logbooks? c. Is an initial calibration performed with a minimum of five concentration levels for each target analyte? d. Is one of the calibration standards at a concentration near, but above, the MDL? e. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector? f. Is a linear calibration curve with a correlation coefficient ≥ 0.995 prepared for each analyte? g. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working range? h. Is the calibration curve or factor verified at the beginning and end of each analysis sequence with a mid-concentration standard?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

ITEM	YES	COMMENT
i. Is a new calibration curve prepared for any target analyte when the response for the target analyte varies from the predicted response by more than ±15%? j. Is the retention time window established with three injections of all target analytes throughout the course of a 72-hour period? k. Is the retention time window checked on a quarterly basis or whenever a new GC column is installed?		
Sample Preparation: a. Is the pH of aqueous samples adjusted to <2 with sulfuric acid prior to extraction? b. Is diethyl ether of pesticide-quality or equivalent and free of peroxides used for extraction of aqueous samples? c. For soil/sediment samples, is the pH of sample adjusted to two with HC1 and monitored and adjusted, if needed, for 15 minutes prior to extraction? d. Are multiple extractions with acetone and diethyl ether used for soil and sediment samples? e. Is cold (4°C) sulfuric acid used to adjust the pH to two prior to solvent cleanup? f. Is acidified sodium sulfate used to dry the diethyl ether for a minimum of two hours prior to esterification?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

ITEM	YES	COMMENT
g. Is a bubble method or a Diazald kit method used at the lab to generate diazomethane? h. Are the following precautions taken during esterification with diazomethane? (1) Use a safety screen? (2) Use mechanical pipetting aides? (3) Do not heat above 90°C? (4) Avoid grinding surfaces, ground glass joint, sleeve bearing, glass stirrers? (5) Store away from alkali metals? (6) Avoid contact with copper powder, calcium chloride, and boiling chips? i. Is methylated extracts analyzed immediately to minimize trans-esterification and other potential reactions?		
Sample Analysis: a. Is GC column 1 selected for majority of herbicide analysis, except for Dalapon which is analyzed with GC column 3? b. Is daily calibration performed with a mid-concentration standard at the beginning and the end of an analysis sequence?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A) Page 8 of 14

ITEM	YES	COMMENT
c. If the calibration factor calculated from injection of a mid-concentration standard at the end of an analysis sequence exceeds ±15% when compared with the initial standard of the analysis sequence, is the GC system recalibrated and reanalysis performed for all samples, in the sequence, which contain target analytes exceed the criteria?		
d. Are daily retention time windows established for each analyte prior to sample analysis?		
e. Is the retention time for each analyte in the daily mid-concentration standard used as the midpoint of the window for that day?		
f. Have calibration standards undergone the same hydrolysis and esterification processes as the samples?		
g. If calibration is done with standards made from methyl ester compounds, is the final concentration corrected for molecular weight of methyl ester versus the acid herbicides?		
h. If a peak response exceeds the linear range of the system, is a dilution performed on a second aliquot of the sample which has been properly sealed and stored prior to use?		
i. Is peak height measurement used for quantitation when overlapping peaks caused errors in area integration?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

ITEM	YES	COMMENT
j. Is further extract cleanup routinely conducted if interferences prevent peak detection and identification?		
Quality Control: a. Are all QC data maintained and available for easy reference and inspection? b. Is a three-level data review carried out within the lab prior to data release? c. Is a lab specific MDL empirically established and updated on a semiannually basis? d. Is the lab specific MDL equal to or lower than the method specified MDL? e. Are GC/MS techniques routinely used to confirm positive hits? f. If GC/MS fails, are additional steps including alternative packed or capillary GC columns or additional cleanup routinely taken for qualitative confirmation? g. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent? h. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations? (1) Is an LCS prepared with standards independent of calibration standards analyzed for each batch?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A) Page 10 of 14

ITEM	YES	COMMENT
(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?		
(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?		
i. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?		
(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is checked against a regulatory limit, is the spike at that regulatory limit or one to five times higher than the background concentration, whichever concentration would be higher?		
(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

ITEM	YES	COMMENT
(3) If it is not possible to determine the background concentration, is the spike concentration - the regulatory limit, if any; or - the larger of either five times the expected background or LCS concentrations? (4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit? (5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria? (6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?		
j. Is the performance of extraction, cleanup, analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with surrogates which encompass the method specified temperature range?		
k. Are the average percent recovery and standard deviation of the percent recovery for each surrogate calculated, when surrogate data from 25 to 30 samples for each matrix is available?		
l. Are control limits for each surrogate in a given matrix calculated based on the above data?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A) Page 12 of 14

ITEM	YES	COMMENT
m. Do the control limits fall within the control limits of Method 8270 if applicable? n. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis? o. Are corrective actions of reanalysis or reextraction/reanalysis taken if any surrogates for a sample are out of control limits? p. Are control charts for internal QC data plotted and available to bench chemists? q. Are control limits for internal quality control empirically established and updated on a regular basis?		
Data Package: a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational poilicy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.) b. Does the data package contain all method required QC data and meet the USACE contract requirements? c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

ITEM	YES	COMMENT
Waste Disposal: a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled? b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		
Overall Evaluation: a. Does the lab have sound technical capability for HERB analysis? b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____ c. Could the lab handle quick turnaround samples? d. Overall, is the lab acceptable for HERB analysis?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

ITEM
Additional observations, comments, or problems:

CHART I-17

GENERAL QA/QC FOR ORGANIC ANALYSIS BY GC/MS: Page 1 of 3

ITEM	YES	COMMENT
Is the MS capable of scanning from 35 to 450 amu every seven seconds or less?		
Is tuning compound, FC-43, used to verify mass calibration?		
Do the mass spectra of BFP and DFTPP meet all criteria before each batch of volatile and semivolatile samples is run?		
Are standards containing all of the analytes of interest analyzed to verify response factors and update retention time?		
Is glassware for organics solvent rinsed or heated to a minimum of 300°C to vaporize any organics in a muffle furnace after careful cleaning?		
Is this high temperature treatment avoided for volumetric glassware, glassware with ground joints, or sintered glassware?		
Is glassware sealed and stored in a clean environment?		
Are magnetic tapes stored in a secure area?		
Are extensive in-house replacement parts available?		
Are manufacturer’s operating manuals readily available to bench chemists?		
Is there a calibration protocol available to the bench chemists?		
Are calibration results kept in a permanent logbook?		

CHART I-17

GENERAL QA/QC FOR ORGANIC ANALYSIS BY GC/MS: Page 2 of 3

ITEM	YES	COMMENT
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		
Has the instrument been modified in any way?		
Are the instruments properly vented?		
Is a 5-point calibration used?		
Are continuing calibration checks done on a 12-hour basis?		
Are system performance response factors checked on a 12-hour basis?		
Are BFB and DFTPP tuning checks done on a 12-hour basis?		
Is low-level method routinely used for environmental soil/sediment samples?		
For tentatively identified compounds, are library searches done for the ten volatile organics and the 20 semivolatile organics of highest concentration?		
Are surrogate recoveries run on each sample?		
Is a corrective action taken if surrogate recoveries exceed QC limits?		
Is a method blank included with each batch of samples and carried through the entire preparation and analysis?		
Is a lab duplicate run at a rate of 5% or one per batch, whichever is greater?		

CHART I-17

GENERAL QA/QC FOR ORGANIC ANALYSIS BY GC/MS:

ITEM	YES	COMMENT
Is a corrective action taken if matrix spike recoveries exceed QC limits?		
Is a spiked sample run at a rate of 5% or one per batch, whichever is greater?		
Is an LCS analyzed with every tenth sample or each batch?		
Additional observations, comments, or problems:		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
General: a. Are written SOPS available and adequate for VOA sample preparation/analysis? b. Do these SOPS accurately reflect procedures in use? c. Are all target analytes, at a minimum, listed in Table 2 of Method 8240A routinely analyzed at the lab? d. Are manufacturer's operating manuals readily available to bench chemists? e. Are prenumbered, bound notebooks used for data entry? f. Are all records written in indelible ink? g. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual? h. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff: a. Do bench chemists appear knowledgeable and experienced in operation of a purge-and-trap and GC/MS system and in interpretation of chromatograms and mass spectra? b. Are backup bench chemists available?		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		
Apparatus and Facilities: a. Is working space adequate and clean? b. Does the lab have adequate air handling system to avoid cross contamination of samples? c. Is a temperature-programmable gas chromatography equipped with a purge-and-trap device available? d. Is oven temperature stable to ±0.5°C or better at desired setting? e. Is a GC column of 6-ft x O.1-in ID glass, packed with 1% SP-1OOO on Carbopack-B (60/80 mesh) or equivalent, available? f. If an "equivalent" column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated? g. Are enough sets of purge-and-trap devices available for all samples in an analytical batch? h. Is the mass spectrometer capable of scanning from 35 - 260 amu every three seconds or less, using 70-volt electron energy in the electron impact mode? i. Is a computer data system that allows continuous acquisition and storage on machine-readable media of all mass spectra available?		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
j. Is the most recent version of the EPA/ NIST Mass Spectral Library available? k. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records? l. Is sample preparation conducted in a hood? m. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available? n. Are backup instruments available?		
Reagents: a. Is reagent water used free from interferents at the MDL of target analytes? b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available? c. For standard preparation, is a waiting period of ten minutes allowed for drying the alcohol-wetted surface before measuring the weight of methanol to the nearest 0.1 mg? d. Are stock standards stored in bottles with minimal headspace and Teflon-lined screw cap at -10 to -20°C and protected from light? e. Are stock standards replaced after six months, or sooner if comparison with check standards indicates a program?		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
f. Are stock standards for target analytes of low boiling points (<30°C) and high reactivity prepared fresh every two months or sooner?		
g. Are secondary standards stored with minimal headspace and check frequently for degradation or evaporation?		
h. Is 2 mL of GC/MS system tuning standard, containing 25 ng/µL of 4-bromofluorobenzene (BFB) in methanol injected or purged for hardware tuning?		
i. Are method recommended surrogates, toluene-d_8, 4-bromofluorobenzene, and 1,2-dichlgroethane-d_4 spiked into each sample undergoing GC/MS analysis?		
j. Are method recommended internal standards, bromochloromethane, 1,4-dichlorobenzene, and chlorobenzene-d_5 or other compounds with retention times similar to the compounds being detected by GC/MS?		
k. For the initial calibration, are aqueous calibration standards, at a minimum of five concentrations, prepared fresh and discarded after one hour, unless properly sealed in a vial and stored at 4°C with no headspace (up to one week)?		
l. Are method recommended matrix spike standards (1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene in methanol at 25 µg/mL) available?		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
m. Are all non-aqueous standard solutions stored at -10 to -20°C in screw-cap amber bottles with Teflon liners?		
n. Are volatile organic standards stored in a separated freezer/refrigerator from samples or other standards?		
o. Is "purge-and-trap", "pesticide quality", or equivalent methanol stored away from other solvents?		
p. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage:		
a. Are volatile organic samples stored at 4°C in separate refrigerators from other samples?		
b. Are low concentration volatile organic samples stored separately from high concentration volatile organic samples?		
Instrument Calibration and Maintenance:		
a. Is there a calibration protocol readily available to bench chemists?		
b. Are calibration results kept in permanent logbooks?		
c. Is the trap of a purge-and-trap device conditioned overnight at 180°C in the purge mode with an inert gas flow of at least 20 mL/min?		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
d. Prior to use, is the trap conditioned daily for 10 minutes while backflushing at 180°C with the column at 220°C?		
e. Is manufacturer's recommendations used for conditioning of the purge-and trap device?		
f. Initial Calibration:		
(1) Is each GC/MS system hardware-tuned to meet the criteria for 50-ng injection or purging of BFB prior to sample analysis?		
Mass — Ion Abundance Criteria 50 — 15% to 45% of mass 95 75 — 30% to 60% of mass 95 95 — base peak, 100% relative abundance 96 — 5% to 9% of mass 95 173 — 0% to <2% of mass 174 174 — >50% of mass 95 175 — 5% to 9% of mass 174 176 — >95% but <101% of mass 174 177 — 5% to 9% of mass 176		
(2) Is the initial calibration performed with a minimum of five concentration levels for each target analyte?		
(3) Is one of the calibration standards at a concentration near, but above, the MDL?		
(4) Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
(5) Is a system performance check made with five System Performance Check Compounds (SPCCs) for a minimal average response factor (RF) of 0.300 for each SPPC (0.250 for bromoform)? The SPCCs are: Chloromethane, 1,1-Dichloroethane, Bromoform, 1,1,2, 2-Tetrachloroethane, and Chlorobenzene. (a) Chloromethane will be lost if the purge flow is too fast. (b) Bromoform will be purged very poorly if purge flow is too slow. Cold spots and/or active sites may adversely affect response. (c) Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines and/or active sites.		
(6) Is percent relative standard deviation for each Calibration Check Compound (CCC), less than 30%, based on the RFs from the initial calibration? The CCCs are: 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene, and Vinyl chloride.		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
g. Daily Calibration:		
(1) Is each GC/MS system hardware-tuned to meet BFB tuning criteria for each 12-hour shift prior to sample analysis?		
(2) Is the initial calibration curve for each target analyte checked and verified by checking SPCC and CCC of a midpoint calibration standard every 12-hour shift?		
(3) Do the RFs of SPCCs meet the initial SPCC criteria for each 12-hour shift?		
(4) Is the percent difference on RFs less than 25% for any one CCC?		
(5) If the criteria in (3) and (4) are not met, is corrective action taken to solve possible problems such as standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or GC system?		
(6) If no source of problem can be determined after corrective action has been taken, is a new 5-point calibration generated?		
(7) Are the retention times of the internal standards in the check calibration standard within 30 seconds from the last daily calibration check (12 hour)?		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
(8) Is the response of the internal standards in the check calibration standard within a factor of two (-50% to +100%) from the last daily calibration standard check (12 hour)? (9) If the criteria in (7) and (8) are not met, is the mass spectrometer inspected and corrected? (10) If corrections are made, is reanalysis conducted for samples analyzed while the system was malfunctioning?		
Sample Preparation: a. Are purge-and-trap (Method 5030) used for the extraction and injection of standards and samples? b. Before initial use, is the trap conditioned overnight at 180°C by back flushing with an inert gas flow of at least 20 mL per minute? c. Prior to daily use, is the trap conditioned for 10 minutes at 180°C with back flushing?		
Sample Analysis: a. Are all samples and standard solutions allowed to warm to ambient temperature before analysis? b. Is the flow rate of helium purge for best response for chloromethane and bromoform? (≈30-40 mL per minute)		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A) Page 10 of 18

ITEM	YES	COMMENT
c. If a second analysis is needed from sample stored in a syringe, is the analysis completed within 24 hours?		
d. Is the purging chamber washed with two 5-mL flushes of reagent water or methanol followed by reagent water to avoid carryover?		
e. If the concentration of analytes in a sample exceeds the calibration ranges, is the sample diluted and reanalyzed? (Diluted to upper half of curve.)		
f. If sample dilution is needed, is an aliquot of sample which is not less than 1 mL used for dilution and the mixture only inverted and shake three times to minimize loss?		
g. Is proper dilution conducted to keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of calibration curve?		
h. Is secondary ion quantitation used only when there are sample interferences with primary ion quantitation?		
i. Is there a method blank analyzed after a sample that has saturated ions from a compound?		
j. If the blank is not free of interferences, is the system cleaned prior to resuming sample analysis?		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
k. Are sediment/soil and waste samples screened by headspace (Method 3810) or hexadecane extraction (Method 3820) to determine whether the high-level method should be used? l. Is the low-level method used for samples containing individual compounds of <1 mg/kg and the high-level method used only for samples with an expected concentration of >1 mg/kg? m. Is a 5-g sample used if the expected concentration is <0.1 mg/kg or a l-g sample for expected concentration between 0.1 and 1 mg/kg? n. Is a heated purge calibration curve (40°C) prepared and used for the quantitation of all low-level sediment/ soil samples? o. Do the standards and method blank for high-level method contain 100 µL of methanol to simulate the sample conditions?		
Data Interpretations: a. Is the relative retention window (RRT) for each compound set at ±0.06 RRT units of the RRT of the standard compound analyzed within the same 12 hours as the sample? b. Are major ions in the standard mass spectra at a relative intensity >10% present in the sample spectra?		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
c. Do the relative intensities of the major ions agree within 20% between the standard and sample spectra? d. Are molecular ions present in the reference spectrum also present in the sample spectrum? e. Is the lab capable to conduct a computer library search to identify and quantify tentatively identified compounds (TICs)? f. Is the identification of TICS determined only after visual comparison of a sample with the closest library search? g. Is the internal standard of nearest retention time of that of a given compound used for quantification?		
Quality Control: a. Are all QC data maintained and available for easy reference and inspection? b. Is a three-level data review carried out within the lab prior to data release? c. Are lab specific MDL and PQL empirically established and updated on a semiannually basis? d. Is the lab specific PQL equal to or lower than the method specified PQL?		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?		
f. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations?		
(1) Is an LCS prepared with standards independent from calibration standards analyzed for each batch?		
(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?		
(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?		
g. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples? (If a lab analyzes one to ten samples per month, at least one spiked sample per month is required.)		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limit or one to five times higher than the background concentration, whichever concentration would be higher?		
(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?		
(3) If it is not possible to determine the background concentration, is the spike concentration - the regulatory limit, if any; or - the larger of either five times the expected background or LCS concentrations?		
(4) For other matrices, is the spike concentration at ten times the estimated quantitation limit?		
(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?		
h. Is the performance of purge-and-trap, analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with surrogates which encompass the method specified temperature range?		
i. Are control limits for internal quality control empirically established and updated on a regular basis?		
j. Are lab's control limits for surrogates within the method specified limits?		
k. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?		
l. Are the average percent recovery and standard deviation of percent recovery for each surrogate standard calculated once a minimum of 30 samples of same matrix have been analyzed?		
m. Is the method accuracy for each matrix studied assessed and recorded after the analysis of five spiked samples?		
n. Is the accuracy assessment for each analyte updated after each five to ten new accuracy measurements?		
o. Are control charts for internal QC date plotted and available to bench chemists?		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
p. Are corrective actions of reanalysis or reextraction/reanalysis taken if any surrogates for a sample are out of control limits?		
Data Package: a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.) b. Does the data package contain all method required QC data and meet the USACE contract requirements? c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		
Waste Disposal: a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled? b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM	YES	COMMENT
Overall Evaluation: a. Does the lab have sound technical capability for VOA analysis? b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____ c. Could the lab handle quick turnaround samples? d. Overall, is the lab acceptable for VOA analysis?		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

ITEM
Additional observation, comments, or problems:

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

ITEM	YES	COMMENT
General: a. Are written SOPs available and adequate for BNA sample preparation/analysis? b. Do these SOPs accurately reflect procedures in use? c. Are all target analytes, at a minimum, listed in Table 2 of Method 8270A routinely analyzed at the lab? d. Are manufacturer's operating manuals readily available to bench chemists? e. Are prenumbered, bound notebooks used for data entry? f. Are all records written in indelible ink? g. Are all errors corrected by drawing a single line through the error with correction written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual? h. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff: a. Do bench chemists appear knowledgeable and experienced in operation of a GC/MS system and in interpretation of chromatograms and mass spectra? b. Are backup bench chemists available?		

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

ITEM	YES	COMMENT
c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		
Apparatus and Facilities: a. Is working space adequate and clean? b. Are enough sets of separator funnels, continuous liquid-liquid extractors, Soxhlet extractors, and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples? c. Is a temperature-programmable gas chromatography equipped available? d. Is oven temperature stable to ±0.5°C or better at desired setting? e. Is the following GC column available? 30-m x 0.25-mm ID (or 0.32-mm ID) l-µm film thickness silicone-coated fused silica capillary column or equivalent. f. If an “equivalent” column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated? g. Is the mass spectrometer capable of scanning from 35 - 500 amu every one second or less, using 70-volt electron energy in the electron impact mode? h. Is a computer data system that allows continuous acquisition and storage on machine-readable media of all mass spectra available?		

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

ITEM	YES	COMMENT
i. Is the most recent version of the EPA/ NIST Mass Spectral Library available? j. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records? k. Has any instrument been modified in any way? l. Is sample preparation conducted in a hood? m. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available? n. Are backup instruments available?		
Reagents: a. Is reagent water used free from interferents at the MDL of target analytes? b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available? c. Are stock standards stored in bottles with minimal headspace and Teflon line screw-cap at 4°C and protected from light? d. Are stock standards replaced after one year, or sooner if comparison with check standards indicates a program?		

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

ITEM	YES	COMMENT
e. Is a GC/MS system tuning standard, containing 50 ng/µL of decafluorotriphenylphhosphine (DFTPP) in methylene chloride, prepared?		
f. Are method recommended surrogates, phenol-d_5, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d_5, 2-fluorobiphenyl, and d-terphenyl$_{14}$-d into each sample undergoing GC/MS analysis?		
g. Are method recommended internal standards, 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10} phenanthrene-d_{10} chrysene-d_{12}, and perylene-d_{12} or other compounds with retention times similar to the compounds (within ±20% of internal standards') being detected by GC/MS?		
h. Are daily calibration standards, at a minimum of five concentrations, stored at 4°C and freshly prepared weekly or sooner if comparison with check standards indicates a problem?		
i. Are method recommended matrix spike standards (pentachlorophenol, phenol, 2-chlorophenol, 4-nitrophenol, 4-chloro-3-methylphenol, l,2,4-trichlorobenzene, acenaphthene, pyrene, 2,4-dinitrotoluene, N-nitroso-di-n-propylamine, and 1,4-dichlorobenzene) in methanol available?		
j. Are all non-aqueous standard solutions stored at -lO°C to -20°C in screw-cap amber bottles with Teflon liners?		

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

ITEM	YES	COMMENT
k. Are "pesticide quality" or equivalent methanol stored away from other solvents? l. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage: a. Are aqueous samples stored at 4°C, and extracted within seven days from collection and analyzed within 40 days from extraction? b. Are soil samples stored at 4°C, and extracted within 14 days from collection and analyzed within 40 days from extraction? c. Are all samples and sample extracts stored in the dark at 4°C?		
Instrument Calibration and Maintenance: a. Is there a calibration protocol readily available to bench chemists? b. Are calibration results kept in permanent logbooks? c. Initial Calibration: (1) Is each GC/MS system hardware-tuned to meet the criteria for 50-ng injection or purging of DFTPP prior to sample analysis?		

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

ITEM	YES	COMMENT
Mass — Ion Abundance Criteria 51 — 30% to 60% of mass 198 68 — <2% of mass 69 70 — <2% of mass 69 127 — 40% to 60% of mass 198 197 — <1% of mass 198 198 — Base peak, 100% relative abundance 195 — 5% to 9% of mass 198 275 — 10% to 30% of mass 198 365 — >1% of mass 198 441 — Present but less than mass 443 442 — >40% of mass 198 443 — 17% to 23% of mass 442 (2) Does the DFTPP tuning standard also contain 50 ng/µL each of 4,4'-DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance? (<20% of DDT degradation and no visible peak tailing for benzidine and pentachlorophenol.) (3) Is the initial calibration performed with a minimum of five concentration levels for each target analyte? (4) Is one of the calibration standards at a concentration near, but above, the MDL? (5) Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?		

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A) Page 7 of 16

ITEM	YES	COMMENT
(6) Is a system performance check made with four System Performance Check Compounds (SPPCs) for a minimal average response factor (RF) of 0.050 for each compound? The SPCCS are: N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. (a) Degradation of DDT to DDE and DDD should not exceed 20%. (b) Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible. (7) Is percent relative standard deviation for each Calibration Check Compound (CCC), less than 30%, based on the RFs from the initial calibration? The CCCs are: 4-chloro–3-methylphenol, 2,4-dichlorophenol, 2-nitrophenol, phenol, pentachlorophenol, 2,4, 6-trichlorophenol, acenaphthene, 1,4-dichlorobenzene, hexachlorobutadiene, N-nitroso-di-n-phenylamine, di-n-octylphthalate, fluoranthene, benzo(a)pyrene.		

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

ITEM	YES	COMMENT
d. Daily Calibration:		
(1) Is each GC/MS system hardware-tuned to meet DFTPP tuning criteria for each 12-hour shift prior to sample analysis?		
(2) Is the initial calibration curve for each target analyte checked and verified by checking SPCC and CCC of a midpoint calibration standard every 12-hour shift?		
(3) Do the RFs of SPCCs meet the initial SPCC criteria for each 12-hour shift?		
(4) Is the percent difference on RFs less than 30% for any one CCC?		
(5) If the criteria in (3) and (4) are not met, is corrective action taken to solve possible problems such as standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or GC system?		
(6) If no source of problem can be determined after corrective action has been taken, is a new five-point calibration generated?		
(7) Are the retention times of the internal standards in the check calibration standard within 30 seconds from the last daily calibration check (12 hours)?		

CHART I-91

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

ITEM	YES	COMMENT
(8) Is the response of the internal standards in the check calibration standard within a factor of two (-50% to +100%) from the last daily calibration standard check (12 hours)? (9) If the criteria in (7) and (8) are not met, is the mass spectrometer inspected and corrected? (10) If corrections are made, is reanalysis conducted for samples analyzed while the system was malfunctioning? e. Is the retention time window established with three injections of all target analytes throughout the course of a 72-hour period? f. Is the retention time window checked on a quarterly basis or whenever a new GC column is installed?		
Sample Preparation: a. Are samples extracted by Methods 3510, 3520, 3540, 3550, or 3580 prior to analysis? b. Are proper extract cleanup methods routinely used prior to analysis? c. Is direct injection used only for samples with concentrations in excess of 10,000 µg/L?		

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

ITEM	YES	COMMENT
Sample Analysis: a. Is the extract screened on a GC/FID or GC/PID using the same type of capillary column to minimize contamination of GC/MS system from unexpected high concentrations of organic compounds? b. If the concentration of analytes in a sample exceeds the calibration ranges, is the sample diluted and reanalyzed? c. Is additional internal standard added to the diluted extract to maintain the required 40 ng/µL of each internal standard in the extract volume? d. Is secondary ion quantitation used only when there are sample interferences with primary ion quantitation? e. Is there a method blank analyzed after a sample that has saturated ions from a compound? f. If the blank is not free of interferences, is the system cleaned prior to resuming sample analysis?		
Data Interpretations: a. Is the relative retention window (RRT) for each compound set at ±0.06 RRT units of the RRT of the standard compound analyzed within the same 12 hours as the sample? b. Are major ions in the standard mass spectra at a relative intensity >10% present in the sample spectra?		

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A) Page 11 of 16

ITEM	YES	COMMENT
c. Do the relative intensities of the major ions agree within 20% between the standard and sample spectra? d. Are molecular ions present in the reference spectrum also present in the sample spectrum? e. Is the lab capable to conduct a computer library search to identify and quantify tentatively identified compounds (TICs)? f. Is the identification of TICs determined only after visual comparison of a sample with the closest library search? g. Is the internal standard of nearest retention time of that of a given compound used for quantification?		
Quality Control: a. Are all QC data maintained and available for easy reference and inspection? b. Is a three-level data review carried out within the lab prior to data release? c. Are lab specific MDL and PQL empirically established and updated on a semiannually basis? d. Is the lab specific PQL equal to or lower than the method specified PQL? e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?		

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

ITEM	YES	COMMENT
f. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations? (1) Is an LCS prepared with standards independent from calibration standards analyzed for each batch? (2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance? (3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the sample analysis halted until the system performance is back in control?		
g. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples? (If a lab analyzes one to ten samples per month, at least one spiked sample per month is required.) (1) If the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limit or one to five times higher than the background concentration, whichever concentration would be higher?		

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

ITEM	YES	COMMENT
(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?		
(3) If it is not possible to determine the background concentration, is the spike concentration - the regulatory limit, if any; or - the larger of either five times the expected background or LCS concentrations?		
(4) For other matrices, is the spike concentration at 10 times the estimated quantitation limit?		
(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?		
(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?		
h. Is the performance of sample extraction, analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with surrogates which encompass the method specified temperature range?		

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

ITEM	YES	COMMENT
i. Are control limits for internal quality control empirically established and updated on a regular basis?		
j. Are lab's control limits for surrogates within the method specified limits?		
k. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?		
l. Are the average percent recovery and standard deviation of percent recovery for each surrogate standard calculated once a minimum of 30 samples of same matrix have been analyzed?		
m. Is the method accuracy for each matrix studied assessed and recorded after the analysis of five spiked samples?		
n. Is the accuracy assessment for each analyte updated after each five to ten new accuracy measurements?		
o. Are control charts for internal QC data plotted and available to bench chemists?		
p. Are corrective actions of reanalysis or reextraction/reanalysis taken if any surrogates for a sample are out of control limits?		

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

ITEM	YES	COMMENT
Data Package: a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.) b. Does the data package contain all method required QC data and meet the USACE contract requirements? c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		
Waste Disposal: a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled? b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		
Overall Evaluation: a. Does the lab have sound technical capability for BNA analysis?		

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month _____ c. Could the lab handle quick turnaround samples? d. Overall, is the lab acceptable for BNA analysis?		
Additional observations, comments, or problems:		

CHART I-20

ORGANIC ANALYSIS BY GC/MS: DIOXINS (8280)

ITEM	YES	COMMENT
Is a HRGC/LRMS system available for Method 8280?		
Does the lab have a HRGC/HRMS system?		
Is the column oven temperature programmable?		
Is the GC column 60-m long x 0.025-cm ID glass or fused silica, coated with a 0.2 micron film of SP-2330?		
Is the MS low or high resolution with an ion source of 70 volts (norminal)?		
Is a data system interfaced with the mass spectrometer?		
Is the mass spectrometer capable of selected ion monitoring (SIM)?		
If operating conditions such as GC column have changed, has the acceptance criteria for the start up QC been met?		
Are all samples preserved by cooling at 4°C?		
Are all samples extracted within seven days of collection and analyzed within 40 days?		
Is the standard 2,3,7,8-TCDD available?		
Is labeled 2,3,7,8-TCDD available (either $^{37}Cl_4$ or $^{13}C_{12}$)?		
Is a record of standard preparation available?		
Are stock standard solutions stored in Teflon sealed screw cap bottles, at 4°C, protected from light?		

CHART I-20

ORGANIC ANALYSIS BY GC/MS: DIOXINS (8280)

ITEM	YES	COMMENT
Are stock standard solutions prepared fresh every six months?		
Is a standard curve available?		
Is a method blank included with each sample batch and carried through the entire preparation and analysis?		
Is a lab duplicate run at a rate of 5% or one per batch, whichever is greater?		
Is a spiked sample run at a rate of 5% or one per batch, whichever is greater?		
Is an LCS analyzed with every tenth sample?		
Are results for LCSs charted?		
Are control limits for LCSs established?		
Are charts for LCSs current?		
Are results for spiked sample charted?		
Are control limits established for spiked samples?		
Are charts for spiked samples current?		
Is a temperature controlled (±2°C) hot water bath available?		

CHART I-20

ORGANIC ANALYSIS BY GC/MS: DIOXINS (8280)

ITEM
Additional observations, comments, or problems:

CHART I-21

ORGANIC ANALYSIS BY HPLC: PAH (8310)

ITEM	YES	COMMENT
General: a. Are written SOPs available and adequate for PAH sample preparation/analysis? b. Do these SOPs accurately reflect procedures in use? c. Are manufacturer's operating manuals readily available to bench chemists? d. Are prenumbered, bound notebooks used for data entry? e. Are all records written in indelible ink? f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual? g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff: a. Do bench chemists appear knowledgeable and experienced in operation of an HPLC and interpretation of chromatograms? b. Are backup bench chemists available? c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		

CHART I-21

ORGANIC ANALYSIS BY HPLC: PAH (8310)

ITEM	YES	COMMENT
Apparatus and Facilities:		
a. Is working space adequate and clean?		
b. Are enough sets of separator funnels, continuous liquid-liquid extractors, Soxhlet extractors, and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples?		
c. Is an HPLC equipped with a pump capable of achieving 4,000 psi available?		
d. Can the pump produce a gradient?		
e. Is a fluorescence detector for excitation at 280 nm and emission greater than 389 nm cutoff available?		
f. Is a UV detector at 254 nm coupled to the fluorescence detector available?		
g. Is a reverse phase column, HC-ODS Si-X, 5-micron particle size diameter, in a 250-mm x 2.6-mm ID SS column or equivalent available?		
h. If an “equivalent” column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?		
i. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		
j. Has any instrument been modified in any way?		

CHART I-21

ORGANIC ANALYSIS BY HPLC: PAH (8310)

ITEM	YES	COMMENT
k. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available? l. Are backup apparatus available?		
Reagents: a. Is reagent water used free from interferents at the MDL of target analytes? b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available? c. Is “HPLC grade” or equivalent solvent, acetonitrile, used for PAH analysis? d. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage: a. Are aqueous samples stored at 4°C, and extracted within seven days from collection and analyzed within 40 days from extraction? b. Are soil samples stored at 4°C, and extracted within 14 days from collection and analyzed within 40 days from extraction? c. Are all samples and sample extracts stored in the dark at 4°C?		

CHART I-21

ORGANIC ANALYSIS BY HPLC: PAH (8310)

ITEM	YES	COMMENT
Instrument Calibration and Maintenance:		
a. Is there a calibration protocol readily available to bench chemists?		
b. Are calibration results kept in permanent logbooks?		
c. Are stock standards stored in bottles with Teflon-lined screw caps or crimp tops at 4°C and protected from light?		
d. Are stock solutions replaced after one year, or sooner if comparison with check standards indicates a problem?		
e. Are working standards replaced after six months or sooner, if comparison with check standards indicates a problem?		
f. Is an initial calibration performed with a minimum of five concentration levels for each target analyte?		
g. Is one of the calibration standards at a concentration near, but above, the MDL?		
h. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?		
i. Is a linear calibration curve with a correlation coefficient $\geq$ 0.995 prepared for each analyte?		

CHART I-21

ORGANIC ANALYSIS BY HPLC: PAH (8310)

ITEM	YES	COMMENT
j. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working range? k. Is the calibration curve or factor verified at the beginning and end of each analysis sequence with a mid-concentration standard? l. Is a new calibration curve prepared for any target analyte when the response for the target analyte varies from the predicted response by more than 15%? m. Is the retention time window established with three injections of all target analytes throughout the course of a 72-hour period? n. Is the retention time window checked on a quarterly basis or whenever a new GC column is installed?		
Sample Preparation: a. Are aqueous samples extracted at a neutral, or as is, pH with methylene chloride, using Method 3510 or 3520? b. Are solid samples extracted using either Method 3540 or 3550? c. Is the entire aqueous sample consumed for analysis and no analysis performed on aliquots of samples? d. Is the sample bottle rinsed with extraction solvent and the rinsate combined with extract?		

CHART I-21

ORGANIC ANALYSIS BY HPLC: PAH (8310)

ITEM	YES	COMMENT
e. Is the extraction solvent exchanged to acetonitrile and concentrated to 1 mL with Kuderna-Danish apparatuses and micro-Snyder column prior to HPLC analysis? f. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?		
Sample Analysis: a. Is the HPLC elution isocratic with acetonitrile/water (4:6 by volume) for five minutes, then linear gradient to 100% acetonitrile for 25 minutes? b. Is a daily calibration performed with a mid-concentration standard prior to analysis? c. Are daily retention windows established for each analyte prior to sample analysis? d. If the peak areas/heights exceed the linear range of the system, is the extract diluted and reanalyzed? e. Is peak height measurement used for quantitation when overlapping peaks caused errors in area integration?		
Quality Control: a. Are all QC data maintained and available for easy reference and inspection? b. Is a three-level data review carried out within the lab prior to data release?		

CHART I-21

ORGANIC ANALYSIS BY HPLC: PAH (8310)

ITEM	YES	COMMENT
c. Is a lab specific MDL empirically established and updated on a semiannually basis?		
d. Is the lab specific MDL equal to or lower than the method specified MDL?		
e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?		
f. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations?		
(1) Is an LCS prepared with standards independent from calibration standards analyzed for each batch?		
(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?		
(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?		
g. Does the matrix spike solution contain all target analytes?		

CHART I-21

ORGANIC ANALYSIS BY HPLC: PAH (8310)

ITEM	YES	COMMENT
h. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples? (1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever concentration would be higher? (2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher? (3) If it is not possible to determine the background concentration, is the spike concentration - the regulatory limit, if any; or - the larger of either five times the expected background or LCS concentrations? (4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit? (5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?		

CHART I-21

ORGANIC ANALYSIS BY HPLC: PAH (8310)

ITEM	YES	COMMENT
(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?		
i. Does the lab use one or two analytes which are not expected to be presented in the sample as surrogates? (e.g., decafluorobiphenyl or other PAHs which encompass the retention time ranges.)		
j. Are the average percent recovery and standard deviation of percent recovery for each surrogate standard calculated when surrogate data from 25 to 30 samples for each matrix is available?		
k. Are control limits for each surrogate in a given matrix calculated based on the above data?		
l. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?		
m. Are corrective actions of reanalysis or reextraction/reanalysis taken if surrogate(s) for a sample are out of control limits?		
n. Are control charts for internal QC data plotted and available to operators?		
o. Are control limits for internal quality control empirically established and updated on a regular basis?		

CHART I-21

ORGANIC ANALYSIS BY HPLC: PAH (8310)

ITEM	YES	COMMENT
Data Package: a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.) b. Does the data package contain all method required QC data and meet the USACE contract requirements? c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		
Waste Disposal: a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled? b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		
Overall Evaluation: a. Does the lab have sound technical capability for PAH analysis?		

CHART I-21

ORGANIC ANALYSIS BY HPLC: PAH (8310)

ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____ c. Could the lab handle quick turnaround samples? d. Overall, is the lab acceptable for PAH analysis?		
Additional observations, comments, or problems:		

CHART I-22

ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

ITEM	YES	COMMENT
General: a. Are written SOPs available and adequate for explosives sample preparation and analysis? b. Are the SOPs consistent with the EPA's draft SW-846 Method 8330, Revision 0, November 1992? c. Do these SOPs accurately reflect procedures in use? d. Are manufacturer's operating manuals readily available to bench chemists? e. Are prenumbered, bound notebooks used for data entry? f. Are all records written in indelible ink? g. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error so that it remains legible, and initialed and dated by the responsible individual? h. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff: a. Do bench chemists appear knowledgeable and experienced in operation of an HPLC and interpretation of chromatograms? b. Are backup bench chemists available?		

CHART I-22

ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

ITEM	YES	COMMENT
c. Are bench chemists? performance audited and approved prior to work without close supervision by a senior chemist?		
Apparatus and Facilities: a. Is working space adequate and clean? b. Is an HPLC equipped with a pump capable of achieving 4,000 psi, a 100 µL loop injector, and 254-nm UV detector available? c. Is the detector capable to achieve a stable baseline at 0.001 absorbance units full scale? d. Are the following HPLC columns available? (1) C-18 reverse phase HPLC column, 25-cm x 4.6-mm (5-µm), Supelco LC-18 or equivalent? (2) CN reverse phase HPLC column, 25-cm x 4.6-cm (5-µm), Supelco LC-CN or equivalent? e. If an “equivalent” column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated? f. Is the HPLC column temperature controlled? If not, is special care taken to ensure that temperature shifts do not cause peak misidentification? g. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		

CHART I-22

ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

ITEM	YES	COMMENT
h. Has any instrument been modified in any way? i. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available? j. Is a temperature controlled ultrasonic bath available? k. Are backup apparatus available?		
Reagents: a. Is reagent water used free from interferents at the MDL of target analytes? b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available? c. Are “HPLC grade” or equivalent solvents used for explosives analysis? d. Is sodium chloride stored in glass container? e. Are all solvents stored in glass containers and transferred with all glass system? f. Does the lab have calibration standards for all method specified target analytes? g. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		

CHART I-22

ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

ITEM	YES	COMMENT
Sample Handling and Storage: a. Are aqueous samples stored at 4°C, and extracted within seven days from collection and analyzed within 40 days from extraction? b. Are soil samples stored at 4°C, and extracted within 14 days from collection and analyzed within 40 days from extraction? c. Are all samples and sample extracts stored in the dark at 4°C?		
Instrument Calibration and Maintenance: a. Is there a calibration protocol readily available to bench chemists? b. Are calibration results kept in permanent logbooks? c. Are solid analyte standards dried to constant weight in a vacuum desiccator in the dark prior to use? d. Are stock standard solutions stored in refrigerator at 4°C in the dark and replaced after one year or sooner, if comparison with check standards indicates a problem? e. Are intermediate standard solutions prepared in acetonitrile for both water and soil samples? f. Are intermediate standard solutions stored in refrigerator at 4°C in the dark and replaced after six months or sooner, if comparison with check standards indicates a problem?		

CHART I-22

ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

ITEM	YES	COMMENT
g. Are standards for low level methods and working standards prepared fresh on the day of calibration and stored in the dark?		
h. Is a 5 g/L calcium chloride solution added to each working standard?		
i. Is one of the calibration standards at a concentration near, but above, the MDL?		
j. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?		
k. Is an initial calibration performed with a minimum of five concentration levels for each target analyte?		
l. Does the initial calibration contain triplicate injections of each calibration standard?		
m. Is the response factor for each analyte taken as the slope of the best-fit linear regression line with correlation coefficient $\geq$ 0.995?		
n. Is the calibration curve or factor verified with, at a minimum, a midpoint calibration standard in triplicate at the beginning of the day, singly at the midpoint of the run and after the last sample of the day, assuming a sample group of ten or less?		
o. Is an additional mid-level standard checked after each ten samples in the analytical batch?		

CHART I-22

ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

ITEM	YES	COMMENT
p. Is a new calibration curve prepared for any target analyte when the response factors for the daily calibrations vary from the initial response factors by more than 15%? q. Is the retention time window established with three injections of two standard mixtures, (1) HMX, RDX, 135-TNB, 13-DNB, NB, 246-TNT, and 24-DNT, and (2) Tetryl, 26-DNT, 2-NT, 3-NT, and 4-NT, through the course of a 72-hour period? r. Is the retention time window checked on a quarterly basis or whenever a new HPLC column is installed? s. Is the retention time for each analyte in the daily mid-concentration standard used as the midpoint of the window for that day?		
Sample Preparation: a. Are process waste samples screened with the high-level method to determine if the low-level method (1-50 µg/L) is required? b. Is low-level method routinely used for most groundwater samples? c. Are soil samples dried in air at room temperature or colder to a constant weight without exposure to direct sunlight? d. Are dried soil samples ground and homogenized to pass a 30 mesh sieve?		

CHART I-22

ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

ITEM	YES	COMMENT
e. Are soil samples extracted in a cooled ultrasonic bath (<30°C) for 18 hours? f. Is a salting-out procedure used for extraction and concentration of water samples? g. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?		
Sample Analysis: a. Does the mobile phase consist of 50/50 (v/v) methanol/organic-free reagent water? b. Are peak heights used for quantitation of target analytes? (Peak height is recommended to improve the reproducibility of low level samples.) c. Are all positive measurements observed on the C-18 column confirmed with the CN column?		
Quality Control: a. Are all QC data maintained and available for easy reference and inspection? b. Is a three-level data review carried out within the lab prior to data release? c. Is a lab specific MDL empirically established and updated on a semiannually basis? d. Is the lab specific MDL equal to or lower than the method specified MDL?		

CHART I-22

ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

ITEM	YES	COMMENT
e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?		
f. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations?		
(1) Is an LCS prepared with standards independent from calibration standards analyzed for each batch?		
(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?		
(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?		
g. Does the matrix spike solution contain at least one isomer of all target analytes?		
h. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?		

CHART I-22

ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

ITEM	YES	COMMENT
(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever concentration would be higher?		
(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?		
(3) If it is not possible to determine the background concentration, is the spike concentration - the regulatory limit, if any; or - the larger of either five times the expected background or LCS concentrations?		
(4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit?		
(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?		

CHART I-22

ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

ITEM	YES	COMMENT
(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?		
i. Does the lab use one or two analytes which are not expected to be presented in the sample as surrogates?		
j. Are the average percent recovery and standard deviation of percent recovery for each surrogate standard calculated when surrogate data from 25 to 30 samples for each matrix is available?		
k. Are control limits for each surrogate in a given matrix calculated based on the above data?		
l. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?		
m. Are corrective actions of reanalysis or reextraction/reanalysis taken if surrogate(s) for a sample are out of control limits?		
o. Are control charts for internal QC data plotted and available to operators?		
p. Are control limits for internal quality control empirically established and updated on a regular basis?		

CHART I-22

ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330) Page 11 of 12

ITEM	YES	COMMENT
Data Package: a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.) b. Does the data package contain all method required QC data and meet the USACE contract requirements? c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		
Waste Disposal: a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled? b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		
Overall Evaluation: a. Does the lab have sound technical capability for explosives analysis?		

CHART I-22

ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____ c. Could the lab handle quick turnaround samples? d. Overall, is the lab acceptable for explosives analysis?		
Additional observations, comments, or problems:		

CHART I-23

SAMPLE PREPARATION FOR METAL ANALYSIS:

ITEM	YES	COMMENT
General:		
a. Are written SOPs available and adequate for sample preparation?		
b. Do these SOPs accurately reflect procedures in use?		
c. Are all sample preparations conducted in a hood?		
d. Are a group of samples (up to a maximum of 20) which behave similarly with respect to the procedures being employed and which are processed as a unit with the same method sequence and the same lots of reagents and with the reagents and with the manipulations manipulations common to each samples within the same time period or in continuous sequential time periods considered as a batch?		
e. Are the following lab internal QC samples prepared for each batch of samples?		
(1) Method blanks?		
(2) Matrix spikes?		
(3) Matrix spike duplicates?		
(4) Matrix duplicates?		
(5) Laboratory control samples?		
f. If the quantity of field samples is not sufficient for internal QC analyses, are blank spike/blank spike duplicate or duplicate laboratory control sample: analyzed?		

CHART I-23

SAMPLE PREPARATION FOR METAL ANALYSIS:

ITEM	YES	COMMENT
g. Are the rates of internal QC samples consistent with method requirements or, at a minimum, 5% per batch of no more than 20 samples with similar matrix, whichever is greater?		
h. Is the appropriateness of a particular preparation for a specific sample type determined by the completeness of extraction and by spike recoveries?		
i. Are logbooks for sample preparation used and well maintained?		
j. Are permanently bound notebooks with consecutively numbered pages used?		
k. Is a unique serial number clearly displayed on each notebook?		
l. Are critical times entered in logbooks?		
m. Are spiking solutions traceable to NIST or other reliable standards?		
n. Are spiking solutions labeled properly with date of preparation, composition, concentration, and identity of preparer?		
o. Have entries been made in permanent fashion and corrections made without obliterating original entries?		
p. Are corrections reviewed and initialed by a supervisor?		
q. Does the logbook of sample preparation contain the following information?		
(1) Date/time?		

CHART I-23

SAMPLE PREPARATION FOR METAL ANALYSIS: Page 3 of 8

ITEM	YES	COMMENT
(2) Sample ID number? (3) Sample preparer? (4) Matrix noted? (5) Spiking standards? (6) Pretreatment? (7) Volume/weight of sample? (8) Final volume? (9) Preparation methods?		
Acid Digestion of Mercury Samples for CVAA: a. Are mercury in liquid samples prepared according to Method 7470? b. Are mercury in solid or semisolid samples prepared according to Method 7471? c. Are all blanks, spiked samples, and laboratory control samples carried through the same digestion process?		
Acid Digestion of Aqueous Samples for FLAA and ICP (Method 3005A): a. Is this digestion used to prepare surface and ground water samples for analysis of total recoverable metals and dissolved metals by FLAA and ICP? b. For dissolved metals, is the samples filtered through a 0.5-µm filter at the time of collection, prior to acidification with nitric acid?		

CHART I-23

SAMPLE PREPARATION FOR METAL ANALYSIS:

ITEM	YES	COMMENT
c. Are samples digested with a mixture of concentrated nitric acid and hydrochloric acid? d. Is the sample heated at 90 to 95°C to avoid boiling and loss of antimony? e. Is filtration of digestate done only there is concern of insoluble materials may clog the nebulizer? f. Are the reagent water, nitric acid, and hydrochloric acid monitored to determine levels of impurities? g. Are all method blanks, spiked samples, and laboratory control samples carried through the same digestion process?		
Acid Digestion of Aqueous and Extract Samples for FLAA and ICP (Method 3010A): a. Is this digestion used to prepare aqueous samples, TCLP extracts, and wastes that contain suspended solid for analysis of total metals by FLAA and ICP? b. Are samples digested with concentrated nitric acid? c. After the digestion is complete, is the sample warmed with 1:1 hydrochloric acid to dissolve any precipitate or residue? d. Is filtration of digestate done only there is concern of insoluble materials may clog the nebulizer?		

CHART I-23

SAMPLE PREPARATION FOR METAL ANALYSIS:

ITEM	YES	COMMENT
e. Are the reagent water, nitric acid, and hydrochloric acid monitored to determine levels of impurities? f. Are all method blanks, spiked samples, and laboratory control samples carried through the same digestion process? g. Is the use of this digestion method avoided when samples are to be analyzed by the GFAA technique?		
Acid Digestion of Aqueous and Extract Samples by GFAA (Method 3020A): a. Is this digestion used to prepare aqueous samples, TCLP extracts, and wastes that contain suspended solid for analysis of total metals by GFAA? b. Is the digestion based on the use of nitric acid alone? c. Are the reagent water and nitric acid monitored to determine levels of impurities? d. Are all method blanks, spiked samples and laboratory control samples carried through the same digestion process? e. Are aqueous samples of arsenic and selenium prepared according to Methods 7060 and 7740, respectively?		
Acid Digestion of Oils, Greases, or Waxes ICP (Method 3040): a. Is the use of this preparation method limited to samples being analyzed only for Sb, Be, Cd, Cr, Cu, Fe, Mn, Ni, and V?		

CHART I-23

SAMPLE PREPARATION FOR METAL ANALYSIS:

ITEM	YES	COMMENT
b. Is xylene or methyl isobutyl ketone used as the solvent in this method? c. Are organic metallic standards used? d. Are method blanks (e.g., Conostan base oil or mineral oil plus reagents) spike samples, and laboratory control samples carried through the same preparation and analytical processes? e. Are samples and standards diluted as closely as possible to the time of analysis? f. Is the method of standard additions employed for all samples? g. Is background correction employed to account for additive interferences?		
Acid Digestion of Sediments, Sludges, and Soils (Method 3050A): a. Are nonaqueous samples refrigerated upon receipt and analyzed as soon as possible? b. Are the samples mixed thoroughly to achieve homogeneity prior to digestion? c. Is the initial phase of the digestion accomplished with nitric acid and hydrogen peroxide? d. Is hydrochloric acid used as the final reflux acid for (1) the ICP analysis of As and Se, and (2) the FLAA and ICP analyses of Al, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Mo, Pb, Ni, K, Na, Tl, V, and Zn?		

CHART I-23

SAMPLE PREPARATION FOR METAL ANALYSIS:

ITEM	YES	COMMENT
e. Is the use of hydrochloric acid avoided and nitric acid employed as the final dilution acid for GFAA analysis of As, Be, Cd, Cr, Co, Pb, Mo, Se, Tl, and V? f. Are the reagent water, nitric acid, hydrochloric acid, and hydrogen peroxide monitored to determine levels of impurities? g. Are all method blanks, spiked samples, and laboratory control samples carried through the same digestion process? h. Is the method of standard additions employed whenever a new sample matrix is analyzed?		
Alkaline Digestion for Hexavalent Chromium (Method 3060): a. Are samples digested with 3% sodium carbonate and 2% sodium hydroxide solution? b. Is the digestion solution stored in a tightly capped polyethylene bottle and prepared fresh monthly? c. Are the sample and digestate stored at 4°C until analyzed? d. Are all positive samples spiked with Cr (VI) to double the concentration found in the original aliquot, but with the increase no less than 0.10 mg/g? e. If spike recovery is not within 85% and 115%, is an interference regarded to be presented and the results invalid?		

CHART I-23

SAMPLE PREPARATION FOR METAL ANALYSIS: Page 8 of 8

ITEM
Additional observations, comments, or problems:

CHART I-24

GENERAL QA/QC FOR METAL ANALYSIS:

ITEM	YES	COMMENT
Atomic Absorption Spectrophotometer: a. Are fuels and oxidants commercial grade? b. Is there a filter moisture trap between the air source and the spectrometer? c. Is nitrous oxide reagent grade? d. Are flash-back arrestors and heaters in use where needed? e. Are all lamps dated when first put into use? f. Are lamps available for all elements analyzed? g. Does the lab have a Zeeman background correction system? h. Does the lab have a deuterium background correction system? i. Does the lab have a Smith-Hieftje background correction system?		
ICP-Atomic Emission Spectrometer: a. Is a background correction technique in use and documented according to sample matrix at least quarterly? b. Has the instrument detection limit and method detection limit for each element been established and documented at least semiannually? c. Where required, has the effect of high dissolved solids and/or acid concentration been controlled?		

CHART I-24

GENERAL QA/QC FOR METAL ANALYSIS:

ITEM	YES	COMMENT
d. Has salt buildup on the nebulizer been controlled? f. When a new matrix is encountered, is a serial dilution, spike addition, or an alternate method technique in use to eliminate potential interference? g. Is the spectrometer equipped with an argon gas supply? h. Are ultra high purity grade nitric acid, hydrochloric acid, and deionized or distilled water used for sample processing and preparation?		
Are manufacturer's operating manuals readily available to bench chemists?		
Is there a calibration protocol available to bench chemists?		
Are calibration results kept in permanent logbooks?		
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		
Is ICP calibration checked using a blank and the highest mixed calibration standard prior to sample analysis?		
Is ICP calibration verified every ten samples and at the end of the analytical run, using a calibration blank and a check standard?		
Does the result of check standard agree within ±10% of expected value?		

CHART I-24

GENERAL QA/QC FOR METAL ANALYSIS:

ITEM	YES	COMMENT
Are interelement and background correction factors at the beginning and end of an analytical run or twice during every 8-hour work shift, whichever is more frequent?		
Does the result of interelement check sample agree within ±20% of expected value?		
Has the instrument been modified in any way?		
Are the instruments properly vented?		
Is an initial 5-point calibration run to check instrument linearity?		
Is the MDL for each element and matrix type determined every six months or whenever there is a significant change in background or instrument response?		
Is the linear calibration range determined for each element when there is significant change in instrument response and every six months for those elements that periodically approach their linear limits?		
Is a matrix spike run at a rate of 5% with each batch of samples?		
Is a corrective action taken if matrix spike recoveries exceed QC limits?		
Is an internal QC duplicate run at a rate of 5% with each batch of samples?		
Is a corrective action taken if the internal QC duplicate exceed QC limits?		
Is the method of standard addition in use where needed?		

CHART I-24

GENERAL QA/QC FOR METAL ANALYSIS:

ITEM
Additional observations, comments, or problems:

CHART I-25

METAL ANALYSIS BY ICP: METALS (6010A)

ITEM	YES	COMMENT
General: a. Are written SOPs available and adequate for ICP sample preparation/analysis? b. Do these SOPs accurately reflect procedures in use? c. Are manufacturer's operating manuals readily available to bench chemists? d. Are prenumbered, bound notebooks used for data entry? e. Are all records written in indelible ink? f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual? g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff: a. Do bench chemists appear experienced with operation of an ICP system and knowledgeable in the correction of spectral, chemical, and physical interferences? b. Are backup bench chemists available? c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		

CHART I-25

METAL ANALYSIS BY ICP: METALS (6010A)

ITEM	YES	COMMENT
Apparatus and Facilities: a. Is working space adequate and clean? b. Does the lab have a simultaneous multielement ICP? c. Does the lab have a sequential multi-element ICP? d. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records? e. Has any instrument been modified in any way? f. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available? g. Are backup instruments available? h. Are hoods used in sample preparation areas free of rust?		
Reagents: a. Is reagent water used free from interferents at the MDLs of target analytes? b. Is reagent grade water of at least 16 mega-ohm quality used for metal analysis? c. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?		

CHART I-25

METAL ANALYSIS BY ICP: METALS (6010A)

ITEM	YES	COMMENT
d. Are ultra-high purity chemicals or metals (99.99 to 99.999% pure) used for in-house preparation of standard stock solutions? e. Are all salts used for preparation of standard stock solutions dried for one hour at 105°C, unless otherwise specified? f. If standard stock solutions are purchased, are the concentrations of the analytes verified in-house? g. Are stock standards replaced after one year, or sooner if comparison with check standards indicates a problem? h. Are calibration standards initially verified using check standards and monitored weekly for stability? i. Are silver standards limited to 2 mg/L and prevented from exposure to light? j. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage: a. Are aqueous samples preserved at pH $\leq$ 2 with nitric acid? b. Are solid samples stored at 4°C?		
Instrument Calibration and Maintenance: a. Is there a calibration protocol readily available to bench chemists?		

CHART I-25

METAL ANALYSIS BY ICP: METALS (601OA)

ITEM	YES	COMMENT
b. Are calibration results kept in permanent logbooks? c. Is the linearity of ICP calibration range established with a minimum of five levels of calibration standards? d. Is an initial calibration performed with a minimum of three concentration levels for each target analyte? e. Does the lab empirically establish the detection limits, sensitivity, and optimum ranges of the metals for each model of spectrometer and type of matrices? f. Are multiple exposures conducted to secure a reliable average reading for each solution? g. Is one of the calibration standards at a concentration near, but above, the MDL? h. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector? i. Are all mixed calibration standard solutions scanned with a sequential spectrometer to verify the absence of interelement spectral interference?		
Sample Preparation: a. Is an appropriate sample preparation method, Methods 3005A, 3010A, 3020A, 3040, or 3050A, used for sample digestion?		

CHART I-25

METAL ANALYSIS BY ICP: METALS (6010A)

ITEM	YES	COMMENT
b. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?		
Sample Analysis: a. Is an ICP allowed to become thermally stable before beginning calibration or analysis (usually requiring at least 30 minutes)? b. Are the average intensity of multiple exposures for both standardization and sample analysis used to reduce random error? c. Before beginning the sample run, is the highest mixed calibration standard reanalyzed to check if the deviation is within 5% from actual value? d. Is daily calibration checked with a mid-concentration standard at the beginning and the end of an analysis sequence? e. Is sufficient quantity of calibration blank solution used to flush the system for at least one minute before the analysis of each standard or sample? f. If a peak response exceeds the linear range of the system, is a dilution performed with calibration blank solution on a second aliquot of the sample that has been properly sealed and stored prior to use? g. Is an alternate less sensitive spectral line used only when all QC data are already established?		

CHART I-25

METAL ANALYSIS BY ICP: METALS (6010A)

ITEM	YES	COMMENT
Quality Control: a. Are all QC data maintained and available for easy reference and inspection? b. Is a three-level data review carried out within the lab prior to data release? c. Are lab specific IDL and MDL empirically established and updated on a semiannually basis? d. Is the lab specific IDL or MDL equal to or lower than the method specified IDL or MDL, respectively? e. Is a calibration blank used in establishing the calibration curve? (A calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples.) f. Is a minimum of one method blank per sample batch used to determine any memory effects or possible contaminations resulting from varying amounts of the acids used in the sample processing? (A method blank must contain all reagents in the same volumes as used in the processing of the samples and must be carried through the complete procedure and contain the same acid in the final solution as the sample solution used for analysis.)		

CHART I-25

METAL ANALYSIS BY ICP: METALS (6010A) Page 7 of 11

ITEM	YES	COMMENT
g. When a new or unusual sample matrix is encountered, are the following series of tests conducted to check interferences? (1) Serial Dilution: If the analyte concentration is minimally 50 times higher than the IDL, is a fivefold dilution analyzed and compared with the original determinations within l0%? (2) Post Digestion Spike Addition: Is an analyte spike added to a prepared sample, or its dilution to produce a minimum level of ten times and a maximum of 100 times of the IDL recovered to within 25% of the known value? h. If the above tests fail and interferences are suspected, are corrective actions such as use of a standard-addition analysis, computerized compensation, an alternate wavelength, or comparison with an alternative method used? i. Is the ICP calibration checked using a calibration blank and two appropriate standards? j. Is ICP calibration verified every ten samples and at the end of the analytical run, using a calibration blank and a check standard? k. Is the check standard prepared with reference materials independent of calibration standards analyzed for each batch?		

CHART 1-25

METAL ANALYSIS BY ICP: METALS (6010A)

ITEM	YES	COMMENT
l. Does the result of the calibration blank agree within 30 of mean blank value? If not, are the blank analysis repeated twice and the results averaged and checked against the 30 of the background mean?		
m. If the check standard is not within 10% of the expected value or the average background is not within 30, is the analysis terminated, the problem corrected, the instrument recalibrated, and the analysis of previous ten samples repeated?		
n. Are the interelement and background correction factors verified at the beginning and end of an analytical run or twice during every 8-hour work shift, whichever is more frequent? (The results should be within 20% of true values.)		
o. To demonstrate that a lab can generate data of acceptable accuracy and precision, does the lab routinely perform matrix spike, matrix spike duplicate, and matrix duplicate per batch of no more than 20 samples?		
p. Is a control limit of ±20% RPD used for sample values greater than ten times the IDL?		
q. Is the control limit for spike duplicate sample within 20% of the actual value?		
r. Are control charts for internal QC data plotted and available to bench chemists?		

CHART I-25

METAL ANALYSIS BY ICP: METALS (6010A)

ITEM	YES	COMMENT
s. Are control limits for internal quality control empirically established and updated on a regular basis? t. Are all results reported with up to three significant figures?		
Data Package: a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.) b. Does the data package contain all method required QC data and meet the USACE contract requirements? c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		
Waste Disposal: a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled? b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		

CHART I-25

METAL ANALYSIS BY ICP: METALS (6010A)

ITEM	YES	COMMENT
Overall Evaluation: a. Does the lab have sound technical capability for ICP analysis? b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month:_____ c. Could the lab handle quick turnaround samples? d. Overall, is the lab acceptable for ICP analysis?		

CHART I-25

METAL ANALYSIS BY ICP: METALS (6010A)

ITEM
Additional observations, comments, or problems:

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s) Page 1 of 12

ITEM	YES	COMMENT
General: a. Are written SOPS available and adequate for AA sample preparation and analysis? b. Do these SOPS accurately reflect procedures in use? c. Are manufacturer's operating manuals readily available to bench chemists? d. Are prenumbered, bound notebooks used for data entry? e. Are all records written in indelible ink? f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual? g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff: a. Do bench chemists appear experienced with operation of an AA system and knowledgeable in the correction of spectral, chemical, and physical interferences? b. Are backup bench chemists available? c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s)

ITEM	YES	COMMENT
Apparatus and Facilities:		
a. Is working space adequate and clean?		
b. Does the lab have in-house capability for metal analysis by FLAA, GFAA, CVAA, and HGAA?		
c. Does the lab have a Zeeman background correction system for GFAA?		
d. Does the lab have other background correction systems for GFAA (e.g., deuterium and/or Smith-Hieftje)?		
e. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		
f. Has any instrument been modified in any way?		
g. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available?		
h. Are backup instruments available?		
i. Are all glassware, polypropylene/ or Teflon containers, including sample bottles and flasks, washed in the following sequence: detergent, tap water, 1:1 nitric acid, tap water, 1:1 hydrochloric acid, tap water, and reagent water?		
j. Are pipet tips acid soaked with 1:5 HNO_3 and rinsed thoroughly with tap and deionized water (Type II ASTM D1193)?		

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s)

ITEM	YES	COMMENT
Reagents:		
a. Is reagent water used free from interferents at the MDLs of target analytes?		
b. Is reagent grade water of at least 16 mega-ohm quality used for metal analysis?		
c. Do reagent grade chemicals used conforn to the-specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?		
d. Are all reagents analyzed to prove that all constituents are below the MDLs?		
e. Are spectrograde hydrochloric and nitric acids certified for AA analysis used for metal analysis?		
f. Are redistilled nitric or hydrochloric acids used for preparation of stock standard metal solutions?		
g. Are sulfuric or phosphoric acids avoided for standard preparation?		
h. If standard stock solutions are prepared in-house, are all salts dried for one hour at 105°C, unless otherwise specified?		
i. If standard stock solutions are purchased, are the concentrations of the analytes verified in-house?		

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s) Page 4 of 12

ITEM	YES	COMMENT
j. Are stock standards replaced after one year, or sooner if comparison with check standards indicates a problem? k. Are calibration standards initially verified using check standards and monitored weekly for stability? l. Is the check standard prepared with reference materials independent of calibration standards analyzed for each batch? m. Are silver standards limited to 2 mg/L and prevented from exposure to light? n. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified? o. Is the acetylene tank grounded, safely strapped, and >100 psi?		
Sample Handling and Storage: a. Are aqueous samples preserved at pH $\leq$ 2 with nitric acid? b. Are solid samples stored at 4°C?		
Instrument Calibration and Maintenance: a. Is there a calibration protocol readily available to bench chemists? b. Are calibration results kept in permanent logbooks?		

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s)

ITEM	YES	COMMENT
c. Is a calibration curve prepared each day with a minimum of three (except five for mercury) concentration levels for each analyte? d. Are equal amounts of permanganate reagents added to mercury calibration standards and blanks? e. Is a calibration curve made for every hour of continuous sample analysis of mercury, arsenic, or selenium by CVAA or GFAA, respectively? f. Are freshly prepared calibration standards used each time a batch of samples is analyzed? g. Are the absorbance readings of calibration standards within 0.0 and 0.7? h. Are multiple exposures conducted to secure a reliable average reading for each solution? i. Is one of the calibration standards at a concentration near, but above, the MDL? j. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?		
Sample Preparation: a. Is an appropriate sample preparation method, Methods 3005A, 3010A, 3020A, 3040, or 3050A, used for sample digestion?		

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s)

ITEM	YES	COMMENT
b. Are the digestion procedures in Section 7.1 of Methods 7060 and 7740 used for preparation of aqueous arsenic and selenium samples, respectively? c. Are the digestion procedures in Section 7.0 of Methods 7470 and 7471 used for preparation of aqueous and solid mercury samples, respectively? d. For seawater, brines, and industrial effluents high in chlorides, are additional hydroxylamine sulfate and permanganate reagents (25 mL) used to prevent chlorine interference? e. Are soil samples dried at ambient temperature, ground, and sieved, prior to subsampling? f. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?		
Sample Analysis: a. Are the instructions provided by the manufacturer followed for each AA? b. After choosing the proper lamp for analysis, is the lamp allowed to warm up for a minimum of 15 minutes, unless operated in a double-beam mode? c. Is an instrument blank run and the instrument zeroed? d. Is a lanthanum solution added to samples that are to be analyze for calcium and magnesium?		

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s)

ITEM	YES	COMMENT
e. Is a calcium solution added to samples that are to be analyzed for iron and magnesium?		
f. Is a potassium chloride solution added to samples before atomization in the determination of aluminum, barium, and titanium?		
g. Is an aluminum nitrate solution added to samples before atomization in the determination of molybdenum and vanadium?		
h. Is a cyanogen iodide solution added to samples that are to be analyzed for silver?		
i. Is an unused cyanogen iodide solution discarded after two weeks and fresh solution prepared?		
j. Is a cyanogen iodide solution kept away from any acid solution?		
k. If a nitrous oxide/acetylene flame is used, is the nitrous oxide cylinder fitted with a non-freezable regulator or is a heating coil wrapped around an ordinary regulator?		
l. After a nitrous oxide/acetylene flame has been ignited, is the burner allowed to come to thermal equilibrium before the analysis is begun?		
m. Are the average intensity of multiple exposures for both standardization and sample analysis used to reduce random error?		

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s) Page 8 of 12

ITEM	YES	COMMENT
n. If the concentration found is greater than the highest standard, is the sample diluted in the same acid matrix and reanalyzed? o. Is same injection volumes used for samples and standards? p. Is a magnesium perchlorate drying tube or a small 60-W light bulb used to prevent condensation of moisture inside a mercury absorption cell?		
Quality Control: a. Are all QC data maintained and available for easy reference and inspection? b. Is a three-level data review carried out within the lab prior to data release? c. Are lab specific IDL and MDL empirically established and updated on a semiannually basis? d. Is the lab specific IDL or MDL equal to or lower than the method specified IDL or MDL, respectively? e. Is a calibration curve prepared each day with a minimum of a calibration blank and three standards? f. Is the calibration curve verified with at least a calibration blank and a mid-range check standard made from reference material or other independent standard material? (The check standard must be within 10% of its value for the curve to be considered valid.)		

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s)

ITEM	YES	COMMENT
g. If more than ten samples per day are analyzed, is the calibration curve verified with a mid-range calibration standard or check standard after every ten samples? (This sample value must be within 20% of the true value, or the previous ten samples need to be reanalyzed.)		
h. For mercury, arsenic, or selenium analysis by CVAA or GFAA, is the calibration curve verified with a mid-range, independently prepared check check standard every 15 samples?		
i. For mercury, arsenic, or selenium analysis by CVAA or GFAA, are the samples diluted if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve?		
j. Are the following interference tests conducted for each analytical batch?		
(1) Dilution Test: Select one typical sample with concentration of analytes at $\geq$ 25 times of the MDL. Dilute the sample by a minimum of fivefold and analyze. If the concentrations between the diluted and the undiluted are within 10%, the absence of interferences can be assumed and samples may be analyzed without using method of standard additions.		

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s)

ITEM	YES	COMMENT
(2) Recovery Test: If all samples in the batch are below ten times the MDL or the Dilution Test fails, a spiked sample should be analyzed. Add a known amount of analyte to bring the concentration to two to five times the original concentration or to 20 times of the MDL if all analytes in the batch are below MDL. The spike recovery should be within 15%, otherwise the method of standard additions shall be used for all samples in the batch.		
k. To demonstrate that a lab can generate data of acceptable accuracy and precision, does the lab routinely perform matrix spike, matrix spike duplicate, and matrix duplicate at a minimum rate of 5% or one per batch, whichever is greater?		
l. Is a control limit of ±20% RPD used for sample values greater than ten times the IDL?		
m. Is the control limit for spike duplicate sample within 20% of the actual value?		
n. Are control charts for internal QC data plotted and available to bench chemists?		
o. Are control limits for internal quality control empirically established and updated on a regular basis?		
p. Are all results reported with up to three significant figures?		

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s)

ITEM	YES	COMMENT
Data Package: a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.) b. Does the data package contain all method required QC data and meet the USACE contract requirements? c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		
Waste Disposal: a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled? b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		
Overall Evaluation: a. Does the lab have sound technical capability for AA analysis?		

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s)

ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____ c. Could the lab handle quick turnaround samples? d. Overall, is the lab acceptable for AA analysis?		
Additional observations, comments, or problems:		

CHART I-27

GENERAL QA/QC FOR CLASSICAL ANALYSIS:

ITEM	YES	COMMENT
Is the wavelength accuracy and repeatability of all spectrophotometers checked at several wavelengths for each batch of samples?		
Is photometric accuracy and repeatability checked and documented with NIST-traceable standards?		
Is acid washed glassware retained for phosphorus analyses only?		
Is ammonia free water used in preparation of standards and samples for nitrogen analyses?		
Are manufacturer's operating manuals available to bench chemists?		
Is there a calibration protocol available to bench chemists?		
Are calibration results kept in permanent logbooks?		
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		
Has any instrument been modified in any way?		
Is a minimum of 4-point calibration used?		
Are continuing calibration checks done on a regular basis?		
Is the MDL for each analyte and matrix type determined every six months or whenever there is a significant change in background or instrument response?		

CHART I-27

GENERAL QA/QC FOR CLASSICAL ANALYSIS:

ITEM	YES	COMMENT
Is the linear calibration range determined for each analyte when there is significant change in instrument response and every six months for those analytes that periodically approach their linear limits?		
Are internal QC samples run per method requirements?		
Is a method blank run for each batch?		
Additional observations, comments, or problems:		

CHART I-28

CLASSICAL ANALYSIS: COMMON ANIONS (300s)

ITEM	YES	COMMENT
Does the lab use an ion chromatography (IC) to analyze chloride, fluoride, nitrate, nitrite, ortho-phosphate, and sulfate?		
Does the IC system have appropriate anion guard column, separator column, suppressor column, and conductivity detector?		
Is the maximum loading to a separator column kept below 400 µg/L to avoid column overloading and nonlinear response?		
Are nitrate, nitrite, ortho-phosphate, and sulfate samples stored at 4°C?		
Are the lab's holding times for nitrate, nitrite, and ortho-phosphate by IC method 48 hours from sampling to analysis? (28 days for chloride, fluoride, and sulfate.)		
Is the eluent solution made of sodium bicarbonate (0.003 M) and sodium carbonate (0.0024 M)?		
Is the regeneration solution made of sulfuric acid (0.025 N)?		
Is a filtration conducted on samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns?		
Is a reagent water analyzed before processing any standards or samples to demonstrate that all glassware and reagent interferences are under control?		
Is a reagent blank processed each time there is a change in reagents?		

CHART I-28

CLASSICAL ANALYSIS: COMMON ANIONS (300s)

ITEM	YES	COMMENT
Are calibration standards prepared from sodium chloride, sodium fluoride, sodium nitrate, sodium nitrite, potassium sulfate, and potassium dihydrogen phosphate dried at 105°C for 30 minutes?		
Are stock standards stored at 4°C?		
Are working standards prepared at a minimum on a weekly basis, except those for nitrite and phosphate which should be prepared fresh daily?		
Is a minimum of three concentration levels and a blank used for calibration of each analyte of interest?		
Is one of the calibration standards near, but above, the MDL?		
Is the injection loop flush thoroughly using each new standard or sample?		
Is the same size of injection loop used for standards and samples?		
Unless the attenuator range settings are proven to be linear, is each setting calibrated individually?		
If the working range exceeds the linear range of the system, is a sufficient number of standards analyzed to allow an accurate calibration curve to be established?		
Is the water dip or negative peak that elutes near and interferes with fluoride peak eliminated by the addition of concentrated eluent to each standard and sample?		

CHART I-28

CLASSICAL ANALYSIS: COMMON ANIONS (300s)

ITEM	YES	COMMENT
Are the retention times of each analyte documented during the calibration? (Retention time is inversely proportional concentration.)		
Is the working calibration curve verified on each working day, or when the anion eluent is changed, and after every 20 samples?		
If the response or retention time for any analyte varies from the expected values by more than ±l0%, is the test repeated with fresh calibration standards?		
If the results are still more than ±l0%, is an entirely new calibration curve prepared for that analyte?		
Is the width of retention time window determined based upon three times of standard deviation of measurements of actual retention time variations over the course of a day?		
If the response of a peak exceeds the working range of the system, is the sample diluted with reagent water and reanalyzed?		
Is an initial demonstration of laboratory capability conducted with a minimum of four LCS?		
Is a continuing check on laboratory performance conducted with spiked samples at a minimum rate of 10% of all samples?		
Are LCS, lab duplicates, and other QC check samples routinely analyzed for each sample batch?		

CHART I-28

CLASSICAL ANALYSIS: COMMON ANIONS (300s) Page 4 of 7

ITEM	YES	COMMENT
Are method performance criteria empirically determined for each spike concentration of analyte being measured?		
Does the lab develop and maintain separate accuracy statements, $\%R \pm \sigma$, for water and wastewater samples? (The average percent recovery, $\%R$, and the standard deviation of of the percent recovery, σ, are developed by analyses of four aliquots of water and wastewater.)		
Is a confirmatory technique such as sample dilution and spiking used to confirm anion identification?		
Fluoride: a. Distillation: (1) Before a sample is run, is the distillation apparatus flushed by distilling the sulfuric acid-distilled water mixture until the temperature reaches 180°C? (2) Is the sample and acid-water mixture distilled until the flask temperature reaches 180°C? (3) Is the heating the contents of the distilling flask above 180°C avoided? (4) Are all water and wastewater sample distilled? b. Calorimetric-SPADNS: (1) If residual chlorine is present, is it removed with sodium arsenite solution?		

CHART I-28

CLASSICAL ANALYSIS: COMMON ANIONS (300s) Page 5 of 7

ITEM	YES	COMMENT
(2) Are all samples (including potable water) subjected to preliminary distillation?		
(3) Are standards prepared in the range of 0 to 1.40 mg/L?		
(4) Are samples and standards at the same temperature for color development?		
(5) Is color development carried out with SPADNS solution and zirconyl-acid reagent (or, alternatively acid-zirconyl-SPADNS reagent)?		
(6) Is the absorbance of samples and standards read at 570 nm?		
(7) Is a standard curve drawn based on the absorbance of the standards?		
(8) Are the fluoride concentrations of the samples read directly from the curve without extrapolation?		
(9) Are standard curves retained as part of the record?		
c. Potentiometric Ion Selective Electrode:		
(1) Is a series of fluoride standards covering the range of 0 to 2.0 mg/L fluoride prepared?		
(2) Is an equal volume of total ionic strength adjustment buffer mixed with the sample or standard to be measured?		
(3) Are samples and standards measured at room temperature?		

CHART I-28

CLASSICAL ANALYSIS: COMMON ANIONS (300s)

ITEM	YES	COMMENT
(4) When a measurement is made, are the electrodes allowed to remain in the solution for three minutes (or longer if necessary) before a reading is made?		
(5) When an electrometer is used, is a standard curve prepared on semi-logarithmic graph paper with the fluoride concentration in mg/L on the log axis and the electrode potential developed in the standard on the linear axis?		
(6) Are the samples diluted and remeasured if they fall outside the working range of the standard curve?		
(7) Is a 1.00 mg/L fluoride standard read after each known sample and each standard?		
(8) If a selective-ion meter is used, is it calibrated in accordance with the manufacturer’s instruction?		
(9) Are all standard curves and calibration data retained as part of the record?		

CHART I-28

CLASSICAL ANALYSIS: COMMON ANIONS (300s)

ITEM
Additional observations, comments, or problems:

CHART I-29

CLASSICAL ANALYSIS: OIL AND GREASE (413.1) Page 1 of 2

ITEM	YES	COMMENT
Are samples collected in glass containers?		
Are samples preserved at the time of collection by adjusting the pH to two or less with hydrochloric acid or sulfuric acid and cooling to 4°C?		
Are samples analyzed within 28 days of collection?		
Are the samples at a pH of two or less when the analysis is begun?		
Is the sample level marked on the sample container for later determination of sample volume?		
Is the entire sample transferred to a separator funnel?		
Is the sample bottle carefully rinsed with fluorocarbon 113 for two minutes and the layers allow to separate?		
Is the solvent layer drained through a funnel containing solvent moistened filter paper and (if necessary) anhydrous sodium sulfate into clean tared distilling flask?		
Is the extraction repeated twice more and the extracts combined in the distilling flask?		
Is the solvent distilled from the distilling flask using a 70°C water bath as a source of heat?		
After the distillation is completed, is the distilling flask swept out with air by inserting a glass tube connected to a vacuum source?		

CHART I-29

CLASSICAL ANALYSIS: OIL AND GREASE (413.1)

ITEM	YES	COMMENT
Is the flask wiped clean and dry on the outside, cooled in a desiccator for 30 minutes, and then weighted?		
Is a solvent blank run with each set of samples?		
Quality Control Requirements: a. Is a laboratory blank analyzed daily or with each batch of sample run? b. Is a reference standard analyzed with every tenth sample? c. Is a spiked sample analyzed with every 20th sample? d. Are duplicate analyses performed on a minimum of 10% of all positive samples?		
Additional observations, comments, or problems:		

CHART I-30

CLASSICAL ANALYSIS: TRPH (418.1)

ITEM	YES	COMMENT
General: a. Are written SOPs available and adequate for TRPH sample preparation/analysis? b. Do these SOPs accurately reflect procedures in use? c. Are manufacturer's operating manuals readily available to bench chemists? d. Are prenumbered, bound notebooks used for data entry? e. Are notebooks reviewed and initialed by supervisors on a regular basis? d. Is an error crossed out with a line and correction entered, dated, and initialed?		
Technical Staff: a. Do bench chemists appear knowledgeable and experienced in TRPH analysis? b. Are backup bench chemists available? c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		
Apparatus and Facilities: a. Is working space adequate and clean? b. Are enough sets of separator funnels (2,000 mL with Teflon stopcock) and Soxhlet extractors available for simultaneous extraction of all batch samples?		

CHART I-30

CLASSICAL ANALYSIS: TRPH (418.1)

ITEM	YES	COMMENT
c. Is a hood available for sample preparation? d. Are IR spectrophotometers suitable for measurements around 2930 cm^{-1}? e. Does lab have sodium chloride or IR grade optical cells of 1-cm, 5-cm, and 10-cm pathlength? f. Are backup apparatuses available?		
Reagents: a. Is reagent water used free from interferents at the method detection limits of target analytes? b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available? c. Is magnesium sulfate monohydrate prepared by drying the heptahydrate salt in an oven at 150°C overnight? d. Is granular, anhydrous sodium sulfate purified by heating at 400°C for four hours, or by precleaning with Freon-113 (1,1,2-trichloro-1,2,2-trifluoro-ethane)? e. Is silica gel, 60-200 mesh, containing 1-2% water as defined by residue test at 130°C available? (Dried at 110°C for 24 hours and stored in a tightly sealed container.)		

CHART I-30

CLASSICAL ANALYSIS: TRPH (418.1)

ITEM	YES	COMMENT
f. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage: a. Is the pH of aqueous and sludge samples checked and adjusted to <2 during sample log-in? b. Are aqueous and sludge samples stored at 4°C and analyzed within 28 days? c. Are soil samples stored at 4°C and analyzed with minimum delay upon receipt in the lab?		
Instrument Calibration and Maintenance: a. Is there a calibration protocol available to the bench chemists? b. Are calibration results kept in permanent logbooks? c. Are IR spectrophotometric accuracy and repeatability checked and documented with NIST-traceable standards? d. Are the materials of interest, if available, or the same type of petroleum fraction, if it is known and original sample is unavailable, used for preparation of calibration standards?		

CHART I-30

CLASSICAL ANALYSIS: TRPH (418.1)

ITEM	YES	COMMENT
e. Does the lab normally attempt to determine the petroleum fraction type for unknowns prior to instrument calibration? (Reference oil is to be used as a last resort for unknowns, as it generates low values for diesel, kerosene, and other known petroleum hydrocarbon types.)		
f. Does reference oil contain a mixture of n-hexadecane, isooctane, and chloro-benzene in the appropriate proportions? (i.e., 15.0 mL + 15.0 mL + 10.0 mL)		
g. Is Freon-113, b.p. 48°C, used for standard and sample preparation?		
h. Is a minimum of a four-point calibration curve (a blank plus three standards) prepared for calibration?		
i. Do working ranges and cell pathlengths comply with method requirements?		
j. Is a calibration plot prepared for absorbance versus mg petroleum hydrocarbons in 100 mL solution?		
k. Are standards scanned from 3200 cm^{-1} to 2700 cm^{-1} with solvent in the reference and results recorded on absorbance paper?		
l. Are absorbance of standards measured by constructing a base line over the scan range and measuring absorbance of the peak maximum at 2930 cm^{-1} and subtracting absorbance at that point?		
m. Are continuing calibration checks done on a regular basis for each batch of samples?		

CHART I-30

CLASSICAL ANALYSIS: TRPH (418.1)

ITEM	YES	COMMENT
n. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		
Sample Preparation: a. For aqueous samples, are the sample bottles marked at the water meniscus for later determination of sample volume? b. Is the entire aqueous sample consumed for analysis and no analysis performed on aliquots of samples? c. Is the pH value of aqueous samples checked and adjusted to $\leq$ 2 prior to extraction? d. Are sample bottle, tip of separator funnel, filter paper, and funnel rinsed with solvent and the rinsate combined with extract? e. Is the aqueous sample sequentially extracted with three 30 mL portion of fresh Freon-113? f. Is sodium sulfate, anhydrous crystal, used when emulsion occurs? g. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven? h. Are sludge samples acidified to a pH of two and dried with magnesium sulfate monohydrate? i. Are sediment/soil samples decanted and dried with anhydrous sodium sulfate?		

CHART I-30

CLASSICAL ANALYSIS: TRPH (418.1)

ITEM	YES	COMMENT
j. For solid samples, is Soxhlet method (Method 9071, steps 7.1 thru 7.11), instead of sonication method, used for sample extraction? k. Is Soxhlet extraction conducted at a rate of 20 cycles per hour for four hours? l. Is the water bath kept at 70°C? m. Is extract filtered with grease-free cotton or glass wool that is cleaned with solvent? n. Is 3-g silica gel used to remove polar fatty matter by stirring the solution with a Teflon coated magnetic stirrer for a minimum of five minutes? o. Is the absorptive capacity of silica gel checked by repeating the silica gel treatment procedure?		
Sample Analysis: a. Are samples scanned from 3200 cm^{-1} to 2700 cm^{-1} with solvent in the reference beam and results recorded on absorbance paper? b. Is a straight baseline constructed over the scan range and subtracted from the peak maximum at 2930 cm^{-1}? c. If the absorbance exceeds 0.8, is a shorter pathlength cell or a diluted extract used? d. Does the lab strictly adhere to the method without any deviations?		

CHART I-30

CLASSICAL ANALYSIS: TRPH (418.1)

ITEM	YES	COMMENT
Quality Control:		
a. Are all QC data maintained and available for easy reference and inspection?		
b. Is a three-level data review conducted within the lab prior to data release?		
c. Is a lab specific MDL empirically established and updated on a semiannually basis?		
d. Does the lab specific MDL meet or exceed the method specified MDL?		
e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?		
f. Is calibration curve verified within ±10% of an independent, mid-range check standard for each batch?		
g. Are duplicate analyses performed at a minimum rate of 5% or one per batch, whichever is more frequent?		
h. Is one pair of matrix spike and matrix spike duplicate samples run at a minimum rate of 5% or one per batch, whichever is more frequent?		
i. Are control charts for internal QC data plotted and available to bench chemists?		
j. Are control limits for internal quality control empirically established and updated on a regular basis?		

CHART I-30

CLASSICAL ANALYSIS: TRPH (418.1)

ITEM	YES	COMMENT
Data Package: a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.) b. Does the data package contain all method required QC data and meet the USACE contract requirements? c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		
Waste Disposal: a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled? b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future? c. Does the lab recycle Freon-113?		
Overall Evaluation: a. Does the lab have sound technical capability for TRPH analysis?		

CHART I-30

CLASSICAL ANALYSIS: TRPH (418.1)

ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____		
c. Could the lab handle quick turnaround samples?		
d. Overall, is the lab acceptable for TRPH analysis?		
Additional observations, comments, or problems:		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A) Page 1 of 10

ITEM	YES	COMMENT
General: a. Are written SOPs available and adequate for cyanide sample preparation and analysis? b. Do these SOPs accurately reflect procedures in use? c. Are manufacturer's operating manuals readily available to bench chemists? d. Are prenumbered, bound notebooks used for data entry? e. Are all records written in indelible ink? f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual? g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff: a. Do bench chemists appear knowledgeable and experienced in cyanide analysis? b. Are backup bench chemists available? c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A)

ITEM	YES	COMMENT
Apparatus and Facilities: a. Is working space adequate and clean? b. Are enough sets of reflux distillation apparatuses available for simultaneous distillation for all batch samples? c. Is a hood available for sample preparation? d. Are spectrophotometers suitable for measurements at 578 nm with a 1.0-cm cell or larger? e. Are backup apparatus available?		
Reagents: a. Is ASTM Type II water (ASTM D1193) monitored and used for analysis? b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available? c. Is KCN used for standard preparation in good physical condition? d. Is chloramine-T solution prepared fresh daily and refrigerated until ready to use? e. Is pyridine-barbituric acid reagent stored in a cool, dark place and discarded after six months (one month if stored at room temperature in the light) or upon formation of a precipitate?		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A)

ITEM	YES	COMMENT
f. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage: a. Are the pH values of aqueous samples checked and adjusted to $\geq$ 12 in a hood during log-in? b. If aqueous samples are not run immediately, are oxidizing agents, such as chlorine, in the samples checked with acidified KI-starch paper and preserved with ascorbic acid during sample log-in? c. Are samples stored at 4°C and prepared within 14 days?		
Instrument Calibration and Maintenance: a. Is there a calibration protocol available to the bench chemists? b. Are calibration results kept in permanent logbooks? c. Are photometric accuracy and repeatability checked and documented with NIST-traceable standards? d. Are calibration standards traceable to NIST or other reliable standards? e. Is a minimum of one 7-point calibration curve (a blank plus six standards) prepared for calibration?		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A)

ITEM	YES	COMMENT
f. Is method blank, consisting of sodium hydroxide dilution solution (1.25 N) and all reagents, used to adjust the photometer zero?		
g. Is a calibration curve ranging from 20 to 400 µg/L prepared?		
h. Are the cyanide standards prepared fresh daily and kept in glass-stoppered bottles?		
i. Are all calibration standards prepared with sodium hydroxide dilution solution for all dilution?		
j. Is a calibration curve prepared covering the range of the method by plotting absorbance of standards against cyanide concentrations (0-1.0 mg/L?)		
k. For samples without sulfide, is a minimum of two standards (a high and low) distilled and compared with similar values on the curve to test the distillation technique?		
l. Do the distilled standards agree within ±10% of the undistilled standards?		
m. For samples with sulfide, are all standards distilled in the same manner as the samples?		
n. Are continuing calibration checks done on a regular basis?		
o. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A)

ITEM	YES	COMMENT
p. Has any instrument been modified in any way?		
Sample Preparation: a. Is pretreatment for cyanides amenable to chlorination performed in a hood to avoid the very toxic gas cyanogen chloride and under amber light to avoid false positive from $K_3[Fe(CN)_6]$ decomposed by UV light? b. During the chlorination procedure, is the pH maintained between 11 and 12, and residual chlorine checked and maintained for one hour while the samples are agitated by magnetic stirring bars? c. After chlorination, is excess reducing agent, ascorbic acid or sodium arsenite, added to remove chlorine? d. Are all samples distilled before cyanide determination? e. Is a 500 mL aliquot (or small aliquot diluted to 500 mL if necessary) containing not more than 100 mg/L of cyanide taken for distillation? f. Is sodium hydroxide used as the absorbing solution? g. Is a fritted glass disc used to disperse HCN in absorbing solution? h. Is the vacuum adjusted so that about two air bubbles per second enter the boiling flask?		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A) Page 6 of 10

ITEM	YES	COMMENT
i. Is lead acetate paper used to check the sample for the presence of sulfide?		
j. If the test is positive, is bismuth nitrate solution used to remove sulfide?		
k. If samples are known or suspected to contain nitrate/nitrite, is adequate amount of sulfamic acid solution added, after the air rate is set, to remove nitrate/nitrite?		
l. Are sulfuric acid and magnesium chloride added, with washing, through the air inlet tube?		
m. Is the sample heated to boiling and then refluxed for one hour?		
n. After the reflux period is completed, is heat turned off and the airflow continued for at least 15 minutes?		
o. Are the contents of the gas absorber drained into a 250 mL volumetric flask?		
p. Are the gas absorber and the tube connecting the reflux condenser with the gas absorber rinsed with distilled water and combined with the drained liquid in the volumetric flask and the contents diluted to 250 mL?		
q. If incomplete recovery is suspected, is a fresh charge of sodium hydroxide placed in the gas washer and the sample refluxed for one more hour?		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A)

ITEM	YES	COMMENT
r. If samples contain appreciable amount of solid, oil, or grease to interfere with homogenization and agitation of the sample mixture in the distillation flask, is Method 9013 used to extract cyanide? s. Is Method 9013 used for the extraction of soluble cyanides from oil, solid, and multiphase samples?		
Sample Analysis: a. Is the amount of sodium hydroxide in the standards and the samples analyzed the same? b. Is the chlorine demand of any compounds in the distillate tested with KI-starch paper? c. Do standards bracket the concentration of the samples? d. If dilution is required, is distillate diluted with method blank solution? e. If pyridine-bartituric acid is used, are the reagents mixed and the color allowed to develop for 8 minutes before reading being taken within 15 minutes?		
Quality Control: a. Are all QC data maintained and available for easy reference and inspection? b. Is a three-level data review carried out within the lab prior to data release?		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A) Page 8 of 10

ITEM	YES	COMMENT
c. Is a lab specific MDL empirically established and updated on a semiannually basis?		
d. Does the lab specific MDL meet or exceed the method specified MDL?		
e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?		
f. Is calibration curve verified within ±15% of an independent, mid-range check standard for each batch?		
g. Is a matrix spike sample at a level of 40 µg/L analyzed per batch to check the efficiency of distillation?		
h. Are duplicate analyses performed at a minimum rate of 5% or one per batch, whichever is more frequent?		
i. Is one pair of matrix spike and matrix spike duplicate samples run at a minimum rate of 5% or one per batch, whichever is more frequent?		
j. Is method of standard additions used for the analysis of all samples that suffer from matrix interferences?		
k. Are control charts for internal QC data plotted and available to bench chemists?		
l. Are control limits for internal quality control empirically established and updated on a regular basis?		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A)

ITEM	YES	COMMENT
Data Package: a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.) b. Does the data package contain all method required QC data and meet the USACE contract requirements? c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		
Waste Disposal: a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled? b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		
Overall Evaluation: a. Does the lab have sound technical capability for cyanide analysis?		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A)

ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____ c. Could the lab handle quick turnaround samples? d. Overall, is the lab acceptable for cyanide analysis?		
Additional observations, comments, or problems:		

CHART I-32

CLASSICAL ANALYSIS: TOC (9060)

ITEM	YES	COMMENT
Are samples preserved on collection by adjusting the pH to two or less with sulfuric acid or hydrochloric acid and cooling to 4°C?		
Are samples analyzed within 28 days of collection?		
Based on the preliminary treatment of the samples prior to analysis, is a notation made defining the type of carbon to analysis?		
Is carbon dioxide-free double distilled water used on the preparation of standards and dilution of samples?		
Is the use of ion exchanged water avoided?		
Is the potassium hydrogen phthalate stock solution prepared using primary standard grade reagent?		
Is the hypodermic needle size selected so as to obtain the most reproducible results?		
Are injections repeated until three consecutive peaks are obtained that are reproducible to within ±3%?		
Does the series of standards run encompass the expected concentration range of the samples to be run?		
Is a dilution water blank run?		
Quality Control Requirements: a. Is a laboratory blank analyzed daily or with each sample run?		

CHART I-32

CLASSICAL ANALYSIS: TOC (9060)

ITEM	YES	COMMENT
b. Is a reference standard analyzed with every tenth sample?		
c. Is a spiked sample analyzed with every 20th sample?		
d. Are duplicate analyses performed on a minimum of 10% of all positive samples?		

Additional observations, comments, or problems:

CHART I-33

WASTE CHARACTERISTICS: IGNITABILITY (1010/1020) Page 1 of 3

ITEM	YES	COMMENT
Pensky-Martins Closed Method (1010): a. Is the Pensky-Martins closed-cup method used to determine the flash point of liquids that tend to form surface films under test conditions or contain non-filterable suspended solids? b. Are two standard thermometers available? c. Is a barometer capable of measuring ambient pressure available (barometers precorrected to give sea-level reading are not acceptable)? d. Are results documented with the following information? (1) Observed flash point? (2) Ambient barometric pressure? (3) Corrected flash point? e. Is a duplicate sample included with every tenth sample? f. Is a p-xylene reference standard determined in duplicate with every sample batch? g. Is the average of the duplicate p-xylene reference standard flash point determination 27 ± 0.8°C (81 ± 1.5°F)?		

CHART I-33

WASTE CHARACTERISTICS: IGNITABILITY (1010/1020) Page 2 of 3

ITEM	YES	COMMENT
Setaflash Closed-Cup Method (1020):		
a. Is the Setaflash closed-cup method used to determine the flash point of liquids that have flash points between 0° and llO°C (32° and 230°F) and viscosities lower than 150 stokes at 25°C (77°F)?		
b. Are ASTM grade thermometers available?		
c. Is heat transfer paste available?		
d. Is a barometer capable of measuring ambient pressure available (barometers precorrected to give sea-level reading are not acceptable)?		
e. Are results documented with the following information?		
(1) Observed flash point?		
(2) Ambient barometric pressure?		
(3) Corrected flash point?		
f. Is a duplicate sample included with every tenth sample?		
g. Is a p-xylene reference standard determined in duplicate with every sample batch?		
h. Is the average of the duplicate p-xylene reference standard flash point determination 27 ± 0.8°C (81 ± 1.5°F)?		

CHART I-33

WASTE CHARACTERISTICS: IGNITABILITY (1010/1020) Page 3 of 3

ITEM
Additional observations, comments, or problems:

CHART I-34

WASTE CHARACTERISTICS: CORROSIVITY (1110) Page 1 of 1

ITEM	YES	COMMENT
Are the steel coupons of SAE Type 1020 steel?		
Are the steel coupons suspended and supported with a non-conducting material such as glass, fluorocarbon, or coated metal?		
Are the areas of coupons known to ±l%?		
Is a blank run with each test sample?		
Are duplicates run with every tenth sample?		
Is cleaning done by either mechanical, chemical, or electrolytic means?		
Additional observations, comments, or problems:		

CHART I-35

WASTE CHARACTERISTICS : Toxicity (1311)

ITEM	YES	COMMENT
Is reactivity determined by the written criteria and an impact apparatus based on an 8-lb weight?		
Total Reactive Cyanide: a. Is the approved test method (Method 9010A) in place? b. Are the samples collected with minimum aeration and headspace, kept in a cool dark place, and analyzed as soon as possible? c. Equipment: Are the following pieces of equipment available? (1) Three neck round bottom flask of 500-mL capacity? (2) Separator funnel with pressure equalizing tube and 24/80 ground glass joint and Teflon sleeve? (3) Water pumped or oil pumped nitrogen gas? (4) Rotometer? d. Method Verification: (1) Has the system been checked with a reference solution yielding a recovery greater than 50%? (2) Has this been documented?		
Total Reactive Sulfides: a. Is the approved test method (Method 9030) in place?		

CHART I-35

WASTE CHARACTERISTICS: REACTIVITY (SECTION 7.3)

ITEM	YES	COMMENT
b. Are the samples collected with minimum aeration and headspace, kept in a cool dark place,and analyzed as soon as possible?		
c. Equipment:		
(1) Is the apparatus required for Method 9030 available?		
(2) Has the absorber been replaced with an “Industrial Hygiene” type detection tube for sulfide (100 to 2,000 ppm)?		
d. Method Verification:		
(1) Has the system been checked with a reference solution yielding a recovery greater than 50%?		
(2) Has this been documented?		
Additional observations, comments, or problems:		

CHART I-36

WASTE CHARACTERISTICS: TOXICITY (1311)

ITEM	YES	COMMENT
Are approved methods in place for analysis of toxicity characteristic leaching procedure (TCLP) extracts for volatiles, BNAs , RCRA metals, pesticides, and herbicides?		
Are zero-headspace extraction (ZHE) vessels available?		
Is the piston within the ZHE able to move with approximately 15 Psi or less?		
Are enough sets of ZHE and bottle extractors available for simultaneous extraction of all samples in one batch?		
Are borosilicate glass bottles used for TCLP extraction bottles?		
Is borosilicate glass filter containing no binder materials used for TCLP extraction?		
Are enough aliquots of samples collected for preliminary evaluation of which extraction fluid to be used, actual extraction of nonvolatiles, ZHE of volatiles, and QC measures?		
Is a separation procedure used for the solid and liquid phase?		
For liquid wastes containing less than 0.5% dry solid materials, are the liquid wastes, after filtration through a 0.6 to 0.8-µm glass fiber filter defined as the TCLP extract?		
Is an agitation apparatus capable of rotating the extraction vessels in an end-over-end fashion at 30 ± 2 rpm available?		

CHART I-36

WASTE CHARACTERISTICS: TOXICITY (1311)

ITEM	YES	COMMENT
Is vacuum filtration used for wastes with low solid content (<10%) and for highly granular, liquid-containing wastes? (Positive pressure filtration should be used for all other types of wastes.)		
Are filters for determination of mobility of metals acid washed prior to use with 1 N nitric acid followed by consecutive rinses with deionized distilled water? (A minimum of 1 L per rinse is recommended.)		
Are preservatives not added to samples before TCLP extraction?		
Are TCLP extracts analyzed as soon as possible following extraction?		
Are TCLP extracts for metal analysis acidified with nitric acid to pH<2, unless precipitation occurs?		
Are TCLP extracts for organic analyses preserved at 4°C without headspace to prevent loss?		
Are solid samples passed through a 9.5 mm standard sieve?		
If not, are solid samples crushed, cut, or ground to meet the size criteria?		
Is the method specified procedure followed to determine the appropriate extraction fluid for nonvolatile TCLP target analytes?		
Is extraction fluid Number 1 always used for TCLP volatiles?		
Is a minimum of 100 g samples extracted for nonvolatile?		

CHART I-36

WASTE CHARACTERISTICS: TOXICITY (1311)

ITEM	YES	COMMENT
Is a maximum of 25 g samples used for a 500 mL ZHE vessel?		
Is the pH meter used accurate t, ±O.05 units at 25°C?		
Is the balance used accurate to within ±O.01 g? (All weight measurements are to be within ±O.1 g.)		
Is TCLP extraction procedure carried out for 18 ± 2 hours at 22 ± 3°C?		
Are TCLP extractor bottles for nonvolatiles periodically opened to relieve excess pressure?		
Is TCLP extract filtered and combined with any liquid from the original separation, if compatible (i.e., no multiple phases form)?		
If the initial liquid phase is not or may not be compatible with the filtered liquid, are these liquids analyzed separately and results combined mathematically?		
Is TCLP toxicity determined by comparison with the levels identified in the appropriate regulations?		
Is a method blank run with each batch of extractions?		
Is a matrix spike performed for each waste type?		
Are matrix spikes added after filtration of the TCLP extract and before preservation? (Matrix pikes should not be added prior to TCLP extraction of samples.)		

CHART I-36

WASTE CHARACTERISTICS: TOXICITY (1311)

ITEM	YES	COMMENT
Are matrix spikes at the a concentration equivalent to the corresponding regulatory level but not less than five times the MDL?		
When the recovery of matrix spike is below the expected analytical method performance, is the use of internal calibration methods, modification of the analytical methods, or use of alternative analytical methods employed to accurately measure the concentration of the TCLP extract?		
Is the method of standard additions employed as the internal calibration quantitation methods for each metallic contaminant if: (1) Matrix spike recovery from the TCLP extract is less than 50% and the concentration does not exceed the regulatory level, and (2) The contaminant concentration in the TCLP extract is within 20% of the appropriate regulatory level?		
Is the method of standard additions used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed?		
Are four identical aliquots of TCLP solution used for the method of standard additions?		
Is the holding time from TCLP extraction to preparative extraction for semivolatiles less than or equal to seven days?		

CHART I-36

WASTE CHARACTERISTICS: TOXICITY (1311)

ITEM
Additional observations, comments, or problems:

APPENDIX J

SHORT CHECKLISTS
FOR
ON-SITE LABORATORY INSPECTIONS

SHORT CHECKLISTS FOR ON-SITE LABORATORY INSPECTIONS

CHARTS J-1 through **J-10** contains a short version of laboratory inspection checklists. The short checklists are developed to reduce the amount of paper work to be brought to an on-site laboratory inspection. Only the major areas to be examined are listed in the short checklists. They serve as a reminder for an experienced inspector to check the adequacy of laboratory facility, equipment, operation, and QA/QC policy and practice during an on-site inspection. Depending on a laboratory's performance and an inspector's preference, the inspector may choose the long checklists (Appendix I), the short checklists, or a hybrid of both to perform an on-site laboratory inspection.

Short Laboratory Inspection Checklists

CHART J-1 Lab Organization, Personnel, and Management

1. Organization
   a. Organization chart
   b. Management structure
   c. Principal officers
      (1) Lab Director - ten years
      (2) Lab Manager - seven years
      (3) Organic Lab Manager - five years
      (4) Inorganic Lab Manager - five years
      (5) QA Officer - five years
   d. Reporting relationships

2. Personnel
   a. Resumes
   b. Job descriptions
   c. Training program
      (1) Initial training and evaluation
      (2) Continuing training and auditing
      (3) Documentation
   d. Minimum Experience without supervision
      (1) GC supervisor - three years
      (2) GC analysis - one year
      (3) Pesticide residue analysis - two years
      (4) GC/MS supervisor - three years
      (5) GC/MS analysis - one year
      (6) GC/MS spectral interpretation - two years
      (7) HPLC analysis (explosives) - one year
      (8) Organic sample preparation - one year

      (9) AA/ICP supervisor - three years
      (10) AA/ICP analysis - one year
      (11) Metal sample preparation - six months
      (12) Wet chemistry supervisor - three years
      (13) UV/VIS analysis (cyanide) - one year
      (14) IR analysis (TRPH) - one year
      (15) IC analysis (common anions) - one year
      (16) Classical analysis - one year

      (17) Radiochemistry supervisor - five years
      (18) Radionuclides analyst - two years
      (19) Gross alpha/beta analysis - six months

CHART J-2 Lab Facility, Equipment, and Instrumentation

1. Facility
   a. Security
   b. Sample storage
   c. Chemical storage
   d. Bench space
   e. Number of hoods
   f. Ventilation
   g. Document archives

2. Equipment
   a. Reagent water system (Free from interferents at MDL; resistivity ≥16M Ω.)
   b. Conductivity meters (Daily or before-use calibration check; cell constant determined annually.)
   c. pH meters (Scaled to ≤0.1 pH unit; standardized daily at two pH units that bracket the expected pH range and are no more than three to four pH units apart; temperature compensated.)
   d. Analytical balance (Capable of weighing to 0.1 mg; daily or before-use check with a minimum of one Class S weight in the range to be used and monthly with a series of Class S weights; ≤0.1%)
   e. Class S weights (50 mg to 4 kg; calibrated within five years and traceable to NIST.)
   f. Drying ovens (Temperature checked before and after each usage.)
   g. Muffle furnace (Temperature verified annually.)
   h. Hotplates (Capable of temperature control within ±5°C.)
   i. Water bath (Capable of temperature control within ±5°C.)
   j. Refrigerators (Temperature checked twice daily.)
   k. Thermometers (Mercury type: scaled to ≤1°C; checked annually against NIST traceable thermometer at two separate temperatures; Dial-type: calibrated quarterly against NIST traceable thermometer.)
   l. Autopipetors (Daily or before-use check of delivery volume gravimetrically.)
   m. Volumetric glassware (Class A segregated from others.)
   n. Glassware cleaning station (Metals, ammonia, phosphorus, volatiles, and semivolatiles.)
   o. Sonicator (Titanium horn; 475 watts with pulsing capability.)
   p. TCLP (ZHE)
   q. LIMS (Audit trail and security.)
   r. Safety equipment
   s. Waste disposal

CHART J-2 Lab Facility, Equipment, and instrumentation
(continued)

3. Instrumentation
   a. AA: Metals (7000s)
      (1) GFAA with Zeeman correction (As, Pb, Sb, Se, Tl)
      (2) CVAA (Hg)
      (3) FLAA
   b. ICP: Metals (6010A)
   c. GC:
      (1) ECD (8080, 8150A)
      (2) ELCD (8010A, 8140)
      (3) FID (8015A, 8040A, 8100)
      (4) PID (8020)
   d. GC/MS:
      (1) VOA (8240A, 8260)
      (2) BNA (8250, 8270A)
   e. HPLC:
      (1) PAH (8310)
      (2) Explosives (draft 8330)
   f. IC: Common Anions (300s)
   g. IR: TRPH (418.1)
   h. UV/VIS: Cyanide (9010A, 9012)
   i. Autoanalyzers

4. Backup Instrumentation and Preventive Maintenance

CHART J-3 Sample Receipt, Storage, and Preservation

1. SOPs

2. Sample Receipt
   a. Cooler receipt checklist
   b. Ventilation hood
   c. External chain-of-custody
   d. Internal chain-of-custody
   e. Unambiguous sample number
   f. Documentation of problems and resolutions
   g. Coordination with the primary contractor and the USACE

3. Sample Storage
   a. Temperature controlled (4±2°C; thermometer in liquid.)
   b. Security (Locked storage.)
   c. Segregation for volatiles and standards

4. Sample Preservation
   a. Cold storage
   b. pH preservations (Check and adjust.)
      (1) pH<2: Ammonia, COD, hardness, Kjedahl and organic nitrogen, metals, nitrate-nitrite, oil & grease, organic carbon, total phosphorus, TOX, radiological tests, gross alpha and beta, and total radium.
      (2) pH< 4: Phenolics.
      (3) pH> 9: Sulfide.
      (4) pH ≥ 12: Cyanide.

5. Scheduling and Tracking (Sample holding times and client requested suspense dates.)

CHART J-4 Sample Preparation

1. SOPs

2. Chemicals and Reagents:
   a. Reagent-grade chemicals shall meet the current Committee on Analytical Reagents of the ACS specifications or better and with minimum purity >90%.
   b. All chemicals and reagents shall be labelled and signed with the date of receipt or preparation.
   c. All reference materials and measurements shall be traceable to NIST.
   d. All acids shall be reagent grade or better, except high-purity grade or equivalent for ICP work.
   e. All solvent shall be chromatographic grade or better.
   f. All reagent documentation shall indicate:
      (1) Solvent
      (2) Concentration
      (3) Date
      (4) Preparer's name
      (5) Expiration date

3. Definition of Batch: Samples of ≤20 with similar matrix prepared and analyzed with same technique and reagents at same time or time sequence. Each batch should have a complete set of method required laboratory QC samples.

4. Matrix Types:

a. Surface water	b. Groundwater	c. Wastewater
d. Soil	e. Sediment	f. Sludge
g. Incineration ash	h. TCLP extract	i. Leachate
j. Oil	k. Product	m. Waste

   n. Other (Plant, biological, etc.)

5. Field QC Samples (Blind to analysts.)
   a. Trip blanks
   b. Rinsate blanks
   c. Field duplicates

6. Laboratory QC Samples (5% per batch.)
   a. Method blanks
   b. Matrix duplicates
   c. Matrix spikes
   d. Matrix spike duplicates
   e. Lab control samples
   f. Any other method specific QC samples

CHART J-5 Sample Analysis

1. SOPs

2. Method Validation
   a. New method or instrument
      (1) Accuracy
      (2) Precision
      (3) Detection limits
      (4) Linear calibration ranges
   b. Modified method or instrument
      (1) Accuracy
      (2) Precision
      (3) Detection limits
      (4) Linear calibration ranges

3. Instrument Calibration

4. General QA/QC
   a. System performance audit
   b. Analyst's performance audit
   c. Blind QA samples
   d. Documentation

5. Method Specific Laboratory QC Samples
   a. Method blanks
   b. Matrix duplicates
   c. Matrix spikes
   d. Matrix spike duplicates
   e. Laboratory control samples (LCS)
   f. Other method specific QC samples (ICS, CCS, etc.)

6. Method References: (Promulgated.)
   a. USEPA SW-846, Revision 0, September 1986:
      7000s, 7040/7041, 8020, 8080, 9060.
   b. USEPA SW-846, Revision 1, July 1992:
      6010A, 8010A, 8150A, 8240A, 8270A, 9010A.
   c. USEPA-600/44-79-020, March 1983:
      418.1.

CHART J-5.1 Halogenated Volatile Organic Compounds by GC (8010A)

1. Number of Analytes: 34
2. Preservation/Storage Conditions: $Na_2S_2O_3$ if chlorine present (aqueous); stored at 4°C.
3. Holding Time: 14 days.
4. Amount for Extraction: 5 mL (aqueous) and 5 grams (solid) by 5030A.
5. Method of Validation:
   (1) Extract and analyze four replicates of QC check standard. Compare results with Table 3.
   (2) MDL (Table 1) shall be empirically established and verified semiannually for each matrix.
   (3) Linear calibration range shall be established and verified semiannually.
6. Standards:
   (1) Standard Solution Expiration: Stock standards (except gases): six months; stock gas standards: two months; calibration standards: 24 hours if no headspace
   (2) Internal Standards: Optional; no internal standards specified.
   (3) Surrogate Standards: Add surrogates (bromochloromethane, 2-bromo-l-chloropropane, and 1,4-dichlorobutane) to encompass range of temperature program. Results within lab established control limits.
   (4) QC Check Standards: If MS/MSD results fall outside control limits, a QC check standard must be analyzed and fall within those ranges designated in Table 3.
7. Calibration:
   (1) Initial Calibration: Minimum of five levels with the lowest near but above MDL; linear correlation coefficient ≥ 0.995. If %RSD<20, linearity is assumed and average RF may be used.
   (2) Continuing Calibration: Mid-level calibration standard run every ten samples and at the end of the analytical run. If not within ±15% of predicted response, recalibrate.
8. Analysis: An example of run log is listed below.
   (1) Initial Batch: (≤20 samples of similar matrix.)
      - CCV (≤ ±15%)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 3 or manufacturer/lab established limits.)
      - Samples (≤10)
      - CCV (≤ ±15%)
      - Samples (≤7)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with lab established limits.)
      - MSD (Compare results with lab established limits.)
   (2) Middle Batch: (≤20 samples of similar matrix.)
      - CCV (≤ ±15%)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 3 or manufacturer/lab established limits.)
      - Samples (≤10)
      - CCV (≤± 15%)
      - Samples (≤7)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with lab established limits.)
      - MSD (Compare results with lab established limits.)
   (3) Final Batch: (≤20 samples of similar matrix.)
      - BFB
      - CCV (≤ ±15%)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 3 or manufacturer/lab established limits.)
      - Samples (≤10)
      - CCV (≤± 15%)
      - Samples (≤7)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with lab established limits.)
      - MSD (Compare results with lab established limits.)
      - CCV (≤± 15%)
9. Other Criteria:
   (1) When doubt exists in compound identification, second column or GC/MS confirmation should be used.
   (2) Establish retention time windows at $\pm 3\sigma$ with three injections throughout 72 hours.
   (3) Establish %R for surrogates, LCS, BS, and MS, and RPD for BD, MD, and MSD.

CHART J-5.2 Aromatic Volatile Organic Compounds by GC (8020)

1. Number of Analytes: 8
2. Preservation/Storage Conditions: pH<2 with HCl, H_2SO_4, or $NaHSO_4$ (aqueous). $Na_2S_2O_3$ if chlorine present (aqueous); stored at 4°C.
3. Holding Time: 14 days.
4. Amount for Extraction: 5 mL (aqueous) and 5 grams (solid) by 5030A.
5. Method of Validation:
   (1) Extract and analyze four replicates of QC check standard. Compare results with Table 3.
   (2) MDL (Table 1) shall be empirically established and verified semiannually for each matrix.
   (3) Linear calibration range shall be established and verified semiannually.
6. Standards:
   (1) Standard Solution Expiration: Stock standards: six months; calibration standards: 24 hours if no headspace
   (2) Internal Standards: Optional. If used, α,α,α-trifluorotoluene is recommended.
   (3) Surrogate Standards: Add surrogates (bromochlorobenzene, bromofluorobenzene, fluorobenzene, difluorobenzene, and α,α,α-trifluorotoluene are recommended) to encompass range of temperature program. Results within lab established control limits.
   (4) QC Check Standards: If MS/MSD results fall outside control limits, a QC check standard must be analyzed and fall within those ranges designated in Table 3.
7. Calibration:
   (1) Initial Calibration: Minimum of five levels with the lowest near but above MDL; linear correlation coefficient ≥0.995. If %RSD<20, linearity is assumed and average RF may be used.
   (2) Continuing Calibration: Mid-level calibration standard run every ten samples and at the end of the analytical run. If not within ±15% of predicted response, recalibrate.
8. Analysis: An example of run log is listed below.
   (1) Initial Batch: (≤20 samples of similar matrix.)
      - CCV (≤±l5%)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 3 or manufacturer/lab established limits.)
      - Samples (≤10)
      - CCV (≤±15%)
      - Samples (≤7)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with lab established limits.)
      - MSD (Compare results with lab established limits.)
   (2) Middle Batch: (≤20 samples of similar matrix.)
      - CCV (≤±15%)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 3 or manufacturer/lab established limits.)
      - Samples (≤10)
      - CCV (≤±15%)
      - Samples (≤7)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with lab established limits.)
      - MSD (Compare results with lab established limits.)
   (3) Final Batch: (≤20 samples of similar matrix.)
      - CCV (≤±15%)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 3 or manufacturer/lab established limits.)
      - Samples (≤10)
      - CCV (≤±15%)
      - Samples (≤7)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with lab established limits.)
      - MSD (Compare results with lab established limits.)
      - CCV (≤±15%)
9. Other Criteria:
   (1) When doubt exists in compound identification, second column or GC/MS confirmation should be used.
   (2) Establish retention time windows at ±3σ with three injections throughout 72 hours.
   (3) Establish %R for surrogates, LCS, BS, and MS, and RPD for BD, MD, and MSD.

CHART J-5.3 Organochlorine Pesticides and PCBS by GC (8080)

1. Number of Analytes: 26
2. Preservation/Storage Conditions: $Na_2S_2O_3$ if chlorine present (aqueous); stored at 4°C.
3. Holding Time: Extraction: seven days (aqueous) and 14 days (solid). Analysis: 40 days after extraction.
4. Amount for Extraction: One liter (aqueous) by 351OA or 3520A. 30 grams (low level solid) or two grams (medium level solid) by 3540A or 3550.
5. Method of Validation:
   (1) Extract and analyze four replicates of QC check standard. Compare results with Table 3.
   (2) MDL (Table 1) shall be empirically established and verified semiannually for each matrix.
   (3) Linear calibration range shall be established and verified semiannually.
6. Standards:
   (1) Standard Solution Expiration: Stock standards: one year; calibration standards: six months.
   (2) Internal Standards: Optional; no internal standards specified.
   (3) Surrogate Standards: Two surrogates, decachlorobiphenyl (DCBP) and 2,4,5,6-tetrachloro-m-xylene (TCMX). Results must fall within laboratory established limits.
   (4) QC Check Standards: If MS/MSD results fall outside control limits, a QC check standard must be analyzed and fall within those ranges designated in Table 3.
7. Calibration:
   (1) Initial Calibration: Minimum of five levels with the lowest near but above MDL; linear correlation coefficient ≥0.995. If %RSD<20, linearity is assumed and average RF may be used.
   (2) Continuing Calibration: Mid-level calibration standard run every ten samples and at the end of the analytical run. If not within ±15% of predicted response, recalibrate.
8. Analysis: An example of run log is listed below.
   (1) Initial Batch: (≤ 20 samples of similar matrix.)
      GC Column deactivation (GC not used for one day or more; primed at 20x mid-level standard.)
      Instrument blank
      DDT and Endrin degradation check standard (Breakdown <20%.)
      CCV (≤ ±15%)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 3 or manufacturer/lab established limits.)
      - Samples (≤10)
      - CCV (≤±15%)
      - Samples (≤7)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with Tables ONE-39, 40 of SW-846, Rev. O or lab established limits.)
      - MSD (Compare results with Tables ONE-39, 40 of SW-846, Rev. O or lab established limits.)
   (2) Middle Batch: (≤20 samples of similar matrix.)
      - CCV (≤±15%)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 3 or manufacturer/lab established limits.)
      - Samples (≤10)
      - CCV (≤±15%)
      - Samples (≤7)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with Tables ONE-39, 40 of SW-846, Rev. O or lab established limits.)
      - MSD (Compare results with Tables ONE-39, 40 of SW-846, Rev. O or lab established limits.)
   (3) Final Batch: (≤20 samples of similar matrix.)
      - CCV (≤±15%)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 3 or manufacturer/lab established limits.)
      - Samples (≤10)
      - CCV (≤±15%)
      - Samples (≤7)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with Tables ONE-39, 40 of SW-846, Rev. O or lab established limits.)
      - MSD (Compare results with Tables ONE-39, 40 of SW-846, Rev. O or lab established limits.)
      - CCV (≤±15 %)
9. Other Criteria:
   (1) Check for DDT and Endrin degradation. Breakdown must be <20% for packed column GC or <15% for capillary GC.
   (2) Second column confirmation is required for all hits. If compound concentration in the extract >10 ng/mL, GC/MS confirmation could be used.
   (3) Establish retention time windows at $\pm 3\sigma$ with three injections throughout 72 hours.
   (4) Establish %R for surrogates, LCS, BS, and MS, and RPD for BD, MD, and MSD.

CHART J-5.4 Chlorinated Herbicides by GC (8150A)

1. Number of Analytes: 10
2. Preservation/Storage Conditions: $Na_2S_2O_3$ if chlorine present (aqueous); stored at 4°C.
3. Holding Time: Extraction: seven days (aqueous) and 14 days (solid). Analysis: 40 days after extraction.
4. Amount for Extraction: One liter (aqueous) and 50 grams (solid) by 8150A.
5. Method of Validation:
   (1) Extract and analyze four replicates of QC check standard. Compare results with Table 3.
   (2) MDL (Table 1) shall be empirically established and verified semiannually for each matrix.
   (3) Linear calibration range shall be established and verified semiannually.
6. Standards:
   (1) Standard Solution Expiration: Stock standards: one year; calibration standards: six months.
   (2) Internal Standards: Optional; no internal standards specified.
   (3) Surrogate Standards: One/two surrogates added to each sample (avoid use of deuterated analogs.) Results must fall within laboratory established limits.
   (4) QC Check Standards: If MS/MSD results fall outside control limits, a QC check standard must be analyzed and fall within those ranges designated in Table 3.
7. Calibration:
   (1) Initial Calibration: Minimum of five levels with the lowest near but above MDL; linear correlation coefficient ≥ 0.995. If %RSD<20, linearity is assumed and average RF may be used.
   (2) Continuing Calibration: Mid-level calibration standard run every ten samples and at the end of the analytical run. If not within ±15% of predicted response, recalibrate.
8. Analysis: An example of run log is listed below.
   (1) Initial Batch: (≤20 samples of similar matrix.)
      - CCV (≤±15%)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 3 or manufacturer/lab established limits.)
      - Samples (≤10)
      - CCV (≤±15%)
      - Samples (≤7)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with lab established limits.)
      - MSD (Compare results with lab established limits.)
   (2) Middle Batch: (≤20 samples of similar matrix.)
      - CCV (≤±15 %)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 3 or manufacturer/lab established limits.)
      - Samples (≤10)
      - CCV (≤±15%)
      - Samples (≤7)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with lab established limits.)
      - MSD (Compare results with lab established limits.)
   (3) Final Batch: (≤20 samples of similar matrix.)
      - CCV (≤±l5%)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 3 or manufacturer/lab established limits.)
      - Samples (≤10)
      - CCV (≤±15%)
      - Samples (≤7)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with lab established limits.)
      - MSD (Compare results with lab established limits.)
      - CCV (≤±15%)
9. Other Criteria:
   (1) When doubt exists in compound identification, GC/MS or second column confirmation should be used.
   (2) Establish retention time windows at $\pm 3\sigma$ with three injections throughout 72 hours.
   (3) Establish %R for surrogates, LCS, BS, and MS, and RPD for BD, MD, and MSD.

CHART J-5.5 Volatile Organic Compounds by GC/MS (8240A)

1. Number of Analytes: 74 (Minimum: 35 in Table 2 of 8240, Rev. 0, 1986
2. Preservation/Storage Conditions: pH<2 with HC1, H_2S0_4, or $NaHS0_4$ (aqueous). $Na_2S_20_3$ if chlorine present (aqueous); stored at 4°C.
3. Holding Time: 14 days.
4. Amount for Extraction: 5 mL (aqueous) and 5 grams (solid) by 5030A.
5. Method of Validation:
   (1) Extract and analyze four replicates of QC check standard. Compare results with Table 6.
   (2) EQLs (Table 2) shall be empirically established and verified semiannually for each matrix.
   (3) Linear calibration range shall be established and verified semiannually.
6. Standards:
   (1) Standard Solution Expiration: Stock standards (except gases): six months; stock gas standards: two months; calibration standards: daily.
   (2) Internal Standards: Bromochloromethane, l,4-difluorobenzene, and chlorobenzene-d_5. RT must be within ±30 seconds from last calibration; area must be -50 to +100%.
   (3) Surrogate Standards: 4-Bromofluorobenzene, 1,2-dichloroethane-d_4, and toluene-d_8. Recover limits in Table 8.
   (4) QC Check Standards: If MS/MSD results fall outside control limits, a QC check standard must be analyzed and fall within those ranges designated in Table 6.
7. Calibration:
   (1) GC/MS Tuning: 50 ng of 4-bromofluorobenzene (BFB) which meets the criteria given in Table 3.
   (2) Initial Calibration: Minimum of five levels with the lowest near but above MDL. %RSD should be <30% for each CCC (1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene, and vinyl chloride). RF>O.30 for SPCCs (chloromethane, l,l-dichloroethane, chlorobenzene, and 1,1,2,2-tetrachloro-ethane) except 0.25 for bromoform.
   (3) Continuing Calibration: Mid-level calibration standard run every 12 hours. RF>O.30 for SPCCs except 0.25 for bromoform. RF for each CCC must be <25% difference from initial calibration.
8. Analysis: An example of run log is listed below.
   (1) Initial Batch: (≤20 samples of similar matrix.)
      - BFB tuning to meet criteria in Table 3
      - CCV (Mid-concentration calibration standard every 12 hours.)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 6 or manufacturer/lab established limits.)
      - Samples
      - BFB (Table 3; every 12 hours.)
      - CCV (Mid-concentration calibration standard every 12 hours.)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with Tables ONE-39, 40 of SW-846, Rev. O or lab established limits.)
      - MSD (Compare results with Tables ONE-39, 40 of SW-846, Rev. O or lab established limits.)
   (2) Middle or Final Batch: (≤20 samples of similar matrix.)
      - BFB (Table 3; every 12 hours.)
      - CCV (Mid-concentration calibration standard every 12 hours.)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 6 or manufacturer/lab established limits.)
      - Samples
      - BFB (Table 3; every 12 hours.)
      - CCV (Mid-concentration calibration standard every 12 hours.)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with Tables ONE-39, 40 of SW-846, Rev. O or lab established limits.)
      - MSD (Compare results with Tables ONE-39, 40 of SW-846, Rev. O or lab established limits.)
9. Other Criteria:
   (1) Compound ID: All ions >10% intensity must be ±20% of standard; ±0.06 RRT units of standard RRT.
   (2) Establish %R for surrogates, LCS, BS, and MS, and RPD for BD, MD, and MSD.
   (3) The most recent version of the EPA/NIST Mass Spectral Library or equivalent should be available.

CHART J-5.6 Semivolatile Organic Compounds by GC/MS (8270A)

1. Nunber of Analytes: 233 (Minimun: 65 in Table 2, 8270, Rev. O, 1986.)
2. Preservation/Storage Conditions: $Na_2S_2O_3$ if chlorine present (aqueous); stored at 4°C.
3. Holding Time: Extraction: seven days (aqueous) and 14 days (solid). Analysis: 40 days after extraction.
4. Amount for Extraction: One liter (aqueous) by 351OA or 3520A at pH>11 and pH<2. 30 grams (low level solid) or two grams (medium level solid) by 3540A or 3550.
5. Method of Validation:
   (1) Extract and analyze four replicates of QC check standard. Compare results with Table 6.
   (2) EQLs (Table 2) shall be empirically established and verified semiannually for each matrix.
   (3) Linear calibration range shall be established and verified semiannually.
6. Standards:
   (1) Standard Solution Expiration: Stock standards: one year; calibration standards: one year; daily continuing calibration standards: one week.
   (2) Internal Standards: 1,4-Dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, crysene-d_{12}, and perylene-d_{12}. RT must be within ±30 seconds from last cal bration; area must be -50 to
   (3) Surrogate Standards: Nitrobenzene-d_5, 2-fluorobiphenyl, p-terphenyl-d_{14}, phenol-d_5, 2-fluorophenol, and 2,4,6-tribromophenol. Recover limits in Table 8.
   (4) QC Check Standards: If MS/MSD results fall outside control limits, a QC check standard must be analyzed and fall within those ranges designated in Table 6.
7. Calibration:
   (1) GC/MS Tuning: 50 ng of decafluorotriphenylphosphine (DFTPP) which meets the criteria given in Table 3. The standard should also contain 4,4'-DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance. (Degradation of DDT <20%. No peak tailing.)
   (2) Initial Calibration: Minimum of five levels with the lowest near but above MDL. %RSD should be <30% for each compound and must be <30% for each CCC (Table 4). Retention time for each compound agrees within 0.06 relative retention time unit. RF>O.05 for SPCCs (N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitro-phenol, and 4-nitrophenol.)
   (3) Continuing Calibration: Mid-level calibration standard run every 12 hours. RF>O.05 for SPCCs. RF for each CCC must be <30% difference from initial calibration.
8. Analysis: An example of run log is listed below.
   (1) Initial Batch: (≤ 20 samples of similar matrix.)
      - DFTPP tuning to meet-criteria in Table 3
      - CCV (Mid-concentration calibration standard.)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 6 or manufacturer/lab established limits.)
      - Samples
      - DFTPP (Table 3; every 12 hours.)
      - CCV (Mid-concentration calibration standard every 12 hours.)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with Tables ONE-39, 40 of SW-846, Rev. O or lab established limits.)
      - MSD (Compare results with Tables ONE-39, 40 of SW-846, Rev. O or lab established limits.)
   (2) Middle or Final Batch: (≤ 20 samples of similar matrix.)
      - DFTPP (Table 3; every 12 hours.)
      - CCV (Mid-concentration calibration standard every 12 hours.)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Table 6 or manufacturer/lab established limits.)
      - Samples
      - DFTPP (Table 3; every 12 hours.)
      - CCV (Mid-concentration calibration standard every 12 hours.)
      - MD (Compare results with lab established limits.)
      - MS (Compare results with Tables ONE-39, 40 of SW-846, Rev. O or lab established limits.)
      - MSD (Compare results with Tables ONE-39, 40 of SW-846, Rev. O or lab established limits.)
9. Other Criteria:
   (1) Compound ID: All ions >1O% intensity must be ±20% of standard; ±O.06 RRT units of standard RRT.
   (2) Establish %R for surrogates, LCS, BS, and MS, and RPD for BD, MD, and MSD.
   (3) The most recent Version of the EPA/NIST Mass Spectral Library or equivalent should be available.

CHART J-5.7 Metals by ICP (6010A)

1. Number of Analytes: 26 metals
2. Preservation/Storage Conditions: pH<2 with $HN0_3$; stored at 4°C (solid).
3. Holding Time: Six months.
4. Amount for Digestion: 100 mL (aqueous) by 3005A (aqueous total recoverable or dissolved metals), 3010A (aqueous total metals), 3040 (dissolution procedures); and 1.00-2.00 grams (solid) by 3050A (solid total metals).
5. Method of Validation: IDL (listed in Table 1 of Method 6010A) shall be empirically established and verified for each matrix.
6. Standards:
   (1) Standard Solution Expiration: Stock standards: specified by manufacturer; must be monitored weekly; calibration standards: prepare fresh at time of use.
7. Calibration:
   (1) Initial Calibration: Per instrument manufacturer's specifications (should consist of a daily minimum of three levels plus a calibration blank.) Before beginning the sample run, reanalyze the highest mixed calibration standard. Concentration values should be ≤±5% of the true values or the established control limits, whichever is lower.
   (2) Continuing Calibration: A mid-level, second source CCV run every ten samples and at the end of the analytical run; %R=9O-11O.
   (3) Interference check solution (ICS): Used to spike sample with the element of interest at concentrations of 10x IDL. Run at the beginning and the end of an analytical run or twice during every 8-hour work shift, whichever is more frequent.
8. Analysis: An example of run log is listed below.
   (1) Initial Batch: (≤20 samples of similar matrix.)
      - Minimum of three level calibration plus a calibration blank
      - Highest mixed standard (<±5% of true value)
      - ICS (<±20% of true value)
      - LCS (Based on control chart or <±20% prior to establishment of control chart.)
      - Samples (≤10)
      - Calibration blank (<3σ of the mean blank value.)
      - CCV (<±l0%)
      - Samples (≤6)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - MD (RPD<20)
      - MS (%R=80-120)
      - MSD (RPD<20, %R=80-120.)
   (2) Middle Batch: (≤20 samples of similar matrix.)
      - Calibration blank (<3σ of the mean blank value.)
      - CCV (<±1O%)
      - LCS (Based on control chart or <±20% prior to establishment of control chart.)
      - Samples (≤10)
      - Calibration blank (<3σ of the mean blank value.)
      - CCV (<±l0%)
      - Samples (≤6)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - MD (RPD<20)
      - MS (%R=80-120)
      - MSD (RPD<20, %R=80-120.)
   (3) Final Batch: (20 samples of similar matrix.)
      - Calibration blank (<3σ of the mean blank value.)
      - CCV (<±1O%)
      - LCS (Based on control chart or <±20% prior to establishment of control chart.)
      - Samples (≤10)
      - Calibration blank (<3σ of the mean blank value.)
      - CCV (<±1O%)
      - ICS (Additional ICS, if more than eight hours.)
      - Samples (≤6)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - MD (RPD<20)
      - MS (%R=80-120)
      - MSD (RPD<20, %R=80-120)
      - Calibration blank (<3σ of the mean blank value.)
      - ICS (<±20%)
      - CCV (<±10%)

CHART J-5.7 Metals by ICP (6010A) (continued)

9. Other Criteria:
   (1) Test for matrix interference with each matrix using a 5-fold serial dilution test (if >50x IDL), Percent difference <±10%; or a (10-10Ox IDL) post-digestion spike test, %R=75-125.
   (2) Use MSA to compensate for matrix interferences.
   (3) Use multiple exposures for both calibration and sample analysis.
   (4) Establish %R for LCS, BS, and MS, and RPD for BD, MD, and MSD.

CHART J-5.8 Metals by Flame and Graphite Furnace AA (7000s)

1. Number of Analytes: 27 metals
2. Preservation/Storage Conditions: pH<2 with $HN0_3$; stored at 4°C (solid).
3. Holding Time: Six months.
4. Amount for Digestion: 100 mL (aqueous) by 3005A (aqueous total recoverable metals or dissolved metals by FLAA), 301OA (aqueous total metals by FLAA), 3020 (aqueous total metals by GFAA, except As by 7060 and Se by 7740), 3040 (dissolution procedures for AA); and 1.00-2.00 grams (solid) by 3050A (solid total metals by FLAA and GFAA).
5. Method of Validation: MDL (listed in Table 1 of Method 7000A) shall be empirically established and verified semiannually for each matrix.
6. Standards:
   (1) Standard Solution Expiration: Stock standards: specified by manufacturer; must be monitored weekly; calibration standards: prepare fresh at time of use.
7. Calibration:
   (1) Initial Calibration: Minimum of a daily three level calibration plus a calibration blank. Verify with a calibration blank and a mid-level ICV from a second source; %R=90-110.
   (2) Continuing Calibration: A mid-level, second source CCV or QC check standard run every ten samples and at the end of the analytical run; %R=80-120.
8. Analysis: An example of run log is listed below.
   (1) Initial Batch: (≤20 samples of similar matrix.)
      - Minimun of a three level calibration plus a calibration blank
      - Calibration blank (<IDL)
      - ICV (<±10%)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Based on control chart or <±20% prior to establishment of control chart.)
      - Samples (≤10)
      - CCV (<±20%)
      - Samples (≤7)
      - MD (RPD<20)
      - MS (%R=75-125)
      - MSD (RPD<20, %R=75-125.)
   (2) Middle Batch: (≤20 samples of similar matrix.)
      - CCV (<±20%)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Based on control chart or <±20% prior to establishment of control chart.)
      - Samples (≤10)
      - CCV (<±20%)
      - Samples (≤7)
      - MD (RPD<20)
      - MS (%R=75-125)
      - MSD (RPD<20, %R=75-125.)
   (3) Final Batch: (≤20 samples of similar matrix.)
      - CCV (<±20%)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Based on control chart or <±20% prior to establishment of control chart.)
      - Samples (≤10)
      - CCV (<±20%)
      - Samples (≤7)
      - MD (RPD<20)
      - MS (%R=75-125)
      - MSD (RPD<20, %R=75-125.)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - CCV (<±20%)
9. Other Criteria:
   (1) Test for matrix interference with each batch using a 5-fold (1+4) dilution test (if sample >25x MDL); percent difference <10%. If dilution test fails or all samples in the batch <10x MDL, perform a (2-5x sample or 20x MDL) spike recovery test; %R=85-115.
   (2) Use MSA to compensate for multiplicative interferences, i.e., matrix or physical interferences.
   (3) Use Zeeman background correction for additive interferences, i.e., nonspecific absorption and scattering.
   (4) Establish %R for LCS, BS, and MS, and RPD for BD, MD, and MSD.

CHART J-5.9 Mercury by Cold Vapor AA (7470/7471)

1. Number of Analytes: Mercury (Hg)
2. Preservation/Storage Conditions: pH<2 with $HN0_3$; stored at 4°C (solid).
3. Holding Time: 28 days.
4. Amount for Digestion: 100 mL (aqueous) and 0.2 grams (solid) by 7470 (aqueous) and 7471 (solid).
5. Method of Validation: MDL (0.0002 mg/L listed in Section 1.0) shall be empirically established and verified semiannually for each matrix.
6. Standards:
   (1) Standard Solution Expiration: Stock standards: specified by manufacturer; must be monitored weekly; calibration standards: prepare fresh at time of use.
7. Calibration:
   (1) Initial Calibration: Minimum of a daily five level calibration plus a calibration blank. Verify with a calibration blank and a mid-level ICV from a second source; %R=90-110.
   (2) Continuing Calibration: A mid-level, second source CCV or QC check standard run every ten samples and at the end of the analytical run; %R=80-120.
8. Analysis: An example of run log is listed below.
   (1) Initial Batch: (≤20 samples of similar matrix.)
   - Minimum of a five level calibration plus a calibration blank
   - Calibration blank (<IDL)
   - ICV (<±10%)
   - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
   - LCS (Based on control chart or <±20% prior to establishment of control chart.)
   - Samples (≤10)
   - CCV (<±20%)
   - Samples (≤7)
   - MD (RPD<20)
   - MS (%R=75-125)
   - MSD (RPD<20, %R=75-125.)

   (2) Middle Batch: (≤20 samples of similar matrix.)
   - CCV (<±20%)
   - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
   - LCS (Based on control chart or <±20% prior to establishment of control chart.)
   - Samples (≤10)
   - CCV (<±20%)
   - SampLes (≤7)
   - MD (RPD<20)
   - MS (%R=75-125)
   - MSD (RPD<20, %R=75-125.)

   (3) Final Batch: (≤20 samples of similar matrix.)
   CCV (<±20%)
   MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
   LCS (Based on control chart or <±20% prior to establishment of control chart.)
   Samples (≤10)
   CCV (<±20%)
   Samples (≤7)
   MD (RPD<20)
   MS (%R=75-125)
   MSD (RPDx20, %R=75-125.)
   MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
   CCV (<±20%)
9. Other Criteria:
   (1) Test for matrix interference with each batch using a 5-fold (1+4) dilution test (if >25x MDL); percent difference <10%. If dilution test fails or all samples in the batch <1OX MDL, perform a (2-5x sample or 20x MDL) spike recovery test; %R=85-115.
   (2) Use MSA to compensate for matrix interferences.
   (3) Establish %R for LCS, BS, and MS, and RPD for BD, MD, and MSD.

CHART J-5.10 Total and Amenable Cyanide by Calorimetry (9010A)

1. Number of Analytes: Total CN and CN amenable to chlorination
2. Preservation/Storage Conditions: pH ≥12 with NaOH. $NaAsO_2$ or ascorbic acid if oxidizing agents present; stored at 4°C.
3. Holding Time: 14 days.
4. Amount for Preparation: 500 mL (1,000 mL if both total and amenable CN) (aqueous) and 1-5 grams (2-10 grams if both total and amenable CN) (solid) by distillation procedures in 9010.
5. Method of Validation: MDL (0.02 mg/L listed in Section 1.0) shall be empirically established and verified semiannually for each matrix.
6. Standards: Stock standards expiration: not specified; calibration standards expiration: daily.
7. Calibration:
   (1) Samples contain no sulfides:
      (a) Initial Calibration: Daily minimum of six levels and a calibration blank, plus a minimum of two of the above standards (high and low) distilled. The distilled ones should be <±lO% of undistilled ones.
      (b) Continuing Calibration: Mid-level calibration standard run every batch and should be <±15% of expected value.
   (2) Samples contain sulfides:
      (a) Initial calibration: Daily six standards and calibration blank. All standards are distilled as the samples using the method of standard additions.
      (b) Continuing Calibration: Mid-level calibration standard run every batch and should be <±15% of expected value.
8. Analysis: An example of run log is listed below.
   (1) Samples contain no sulfides:
      (a) Initial Batch: (≤20 samples of similar matrix.)
         - Minimum of six level plus blank calibration
         - Check standards (Second source, middle level, no distillation, <±15%.)
         - MB (Distilled; <MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
         - LCS (Distilled; compare results with manufacturer/lab established limits.)
         - Samples (Distilled.)
         - MS (Distilled; compare results with lab established limits.)
         - MD (Distilled, <±20%)
      (b) Middle Batch: (≤20 samples of similar matrix.)
         - Check standards (Second source, middle level, no distillation, <±15%.)
         - MB (Distilled; <MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
         - LCS (distilled; compare results with manufacturer/lab established limits.)
         - Samples (Distilled.)
         - MS (Distilled; compare results with lab established limits.)
         - MD (Distilled, <±20%)
      (c) Final Batch: (≤20 samples of similar matrix.)
         - Check standards (Second source, middle level, no distillation, <±15%.)
         - MB (Distilled; <MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
         - LCS (Distilled; compare results with manufacturer/lab established limits.)
         - Samples (Distilled.)
         - MS (Distilled; compare results with lab established limits.)
         - MD (Distilled, <±20%)
         - Check standards (Second source, middle level, no distillation, <±15%.)
   (2) Samples contain sulfides:
      (a) Initial Batch: (≤20 samples of similar matrix.)
         - Minimum of six level plus blank calibration using MSD
         - Check standards (Second source, middle level, no distillation, <±15%.)
         - MB (Distilled; <MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
         - LCS (Distilled; compare results with manufacturer/lab established limits.)
         - Samples (Distilled.)
         - MS (Distilled; compare results with lab established limits.)
         - MD (Distilled, <±20%)
      (b) Middle Batch: (≤20 samples of similar matrix.)
         - Check standards (Second source, middle level, no distillation, <±15%.)
         - MB (Distilled; <MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
         - LCS (Distilled; compare results with manufacturer/lab established limits.)
         - Samples (Distilled.)
         - MS (Distilled; compare results with lab established limits.)
         - MD (Distilled, <±20%)

CHART J-5.10 Total and Amenable Cyanide by Calorimetry (9010A) (continued)

(c) Final Batch: (≤20 samples of similar matrix.)
- Check standards (Second source, middle level, no distillation, <±15%.)
- MB (Distilled; <MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
- LCS (Distilled; compare results with manufacturer/lab established limits.)
- Samples (Distilled.)
- MS (Distilled; compare results with lab established limits.)
- MD (Distilled, <±20%)
- Check standards (Second source, middle level, no distillation, <±15%.)

9. Other Criteria:
   (1) Use MSA to compensate for matrix interferences.
   (2) Establish %R for check standards, LCS, BS, and MS, and RPD for BD and MD.

CHART J-5.11 Total Organic Carbon by a Carbonaceous Analyzer (9060)

1. Number of Analytes: No specific compounds.
2. Preservation/Storage Conditions: pH<2 with HCL or H_2SO_4. Protect from light and atmospheric O_2; stored at 4°C.
3. Holding Time: 28 days.
4. Amount for Extraction: 50 mL.
5. Method of Validation: MDL (1 mg/L listed in Section 1.0) shall be empirically established and verified semiannually for each matrix.
6. Standards: Standard Solution Expiration: Not specified.
7. Calibration:
   (1) Initial Calibration: Per instrument manufacturer's specifications, correlation coefficient ≥0 .995.
   (2) Continuing calibration: Percent difference <10% of initial calibration.
8. Analysis: An example of run log is listed below.
   (1) Initial Batch: (≤20 samples of similar matrix.)
      - Initial calibration
      - CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Based on lab established control limits and should be <10% difference.)
      - Samples
      - MS (Based on lab established control limits.)
      - MD (Based on lab established control limits.)
   (2) Middle Batch: (≤20 samples of similar matrix.)
      - CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Based on lab established control limits and should be <10% difference.)
      - Samples
      - MS (Based on lab established control limits.)
      - MD (Based on lab established control limits.)
   (3) Final Batch: (≤20 samples of similar matrix.)
      - CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Based on lab established control limits and should be <10% difference.)
      - Samples
      - MS (Based on lab established control limits.)
      - MD (Based on lab established control limits.)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
9. Other Criteria: Establish %R for LCS, BS, and MS, and RPD for BD and MD.

CHART J-5.12 Total Recoverable Petroleum Hydrocarbons by IR (418.1)

1. Nunber of Analytes: Non-polar petroleum hydrocarbons. No specific compounds.
2. Preservation/Storage Conditions: pH<2 with HCL (aqueous); stored at 4@C.
3. Holding Time: 28 days.
4. Amount for Extraction: 1,000 mL (aqueous) by 418.1 and 20 grams (solid) by 9071 steps 7.1 thru 7.11 (Soxhlet extraction, 3540A.)
5. Method of Validation: MDL (1 mg/L listed in Section 1.0) shall be empirically established and verified annually for each matrix.
6. Standards:
   (1) Reference oil: Mixture of 15.0 mL n-hexadecane, 15.0 mL isooctane, and 10.0 mL chlorobenzene.
   (2) Standard Solution Expiration: Stock Standards: six months; working standards: one week.
7. Calibration:
   (1) Initial Calibration: Daily minimum of four levels plus a calibration blank; correlation coefficient >0.995.
   (2) Continuing calibration: Percent difference <10% of initial calibration.
8. Analysis: An example of run log is listed below.
   (1) Initial Batch: (≤20 samples of similar matrix.)
      - Initial calibration
        CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Based on lab established control limits and should be <10% difference.)
      - Samples
      - MS (Based on lab established limits.)
      - MD (Based on lab established limits.)
   (2) Middle Batch: (≤20 samples of similar matrix.)
      - CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Based on lab established control limits and should be <10% difference.)
      - Samples
      - MS (Based on lab established limits.)
      - MD (Based on lab established limits.)
   (3) Final Batch: (≤20 samples of similar matrix.)
      - CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - LCS (Based on lab established control limits and should be <10% difference.)
      - Samples
      - MS (Based on lab established limits.)
      - MD (Based on lab established limits.)
      - MB (<MDL, <5% of regulatory limits, or <5% of measured sample concentration.)
      - CCV (Mid-level; independently prepared; percent difference <10% of initial calibration.)
9. Other Criteria:
   (1) Acidify solid samples to pH=2 with HCL.
   (2) Use $MgSO_6 \cdot H_2O$ for solid samples.
   (3) Establish %R for LCS, BS, and MS, and RPD for BD and MD.

CHART J-6 Data Reduction, Validation, and Reporting

1. SOPs

2. Computerized Data Reduction (Manually checked.)

3. Multiple Levels of Data Review
   a. Analyst or peer (100%)
   b. Supervisor (≥10%)
   c. QA Officer (≥10%)
   d. Lab Manager/Director (≥10%)

4. Data Qualifier Flags

5. Report Generation and Archives
   a. Prenumbered, permanently bound notebooks.
   b. Corrections do not obliterate original data.
   c. Revised entry is signed or initialed and dated.
   d. Records are traceable, retrievable, legible, and complete.
   e. All data and reports stored in a secured area for a minimum of three years after final reports.

6. Corrective Actions and Documentation

CHART J-7 Performance and System Audits

1. SOPs

2. Designated Internal Auditor

3. Performance Audit
   a. External QA
   b. Internal QA
      (1) Initial evaluation of new analysts
      (2) Periodical audit of experienced analysts
      (3) Single blind PE samples
      (4) Double blind PE samples
   c. Round robin testing
   d. Corrective actions
   e. Documentation

4. System Audit
   a. Methodologies
      (1) New method
      (2) Modified method
      (3) New instrument
   b. Documentation

5. Control Charts: Established for each type of QC parameters, methodologies, and matrices; updated quarterly or when 20 new data points are obtained.
   a. MB
   b. LCS
   c. MD
   d. MS
   e. MSD
   f. Surrogate
   g. Others

CHART J-8 Laboratory Safety

1. Safety and Chemical Hygiene Plan
   a. Safety meeting
   b. Safety inspection
   c. Fire drill

2. Safety Equipment
   a. Eyewash fountain
   b. Emergency shower
   c. Safety glasses and gloves
   d. Fire alarm
   e. Fire Extinguisher
   f. Emergency light
   g. Flammable material storage
   h. Hazardous area escape
   i. OSHA signs
   j. First aid kit

CHART J-9 Waste Management

1. SOPs
   a. Waste stream analysis
   b. Waste segregation program
   c. Waste recycle program

2. Are residual USACE samples properly disposed of?

3. Does the lab have a Hazardous Waste Coordinator? (Federal RCRA Compliance Checklist, Appendix E, Section 7.)

4. Is the lab a conditionally exempted small quantity generator?
   a. The lab generates less than 100 kg per month of hazardous waste or less than 1 kg per month of acute hazardous waste.
   b. There is never more than 1,000 kg stored on site.
   c. Waste is sent to a TSDF, a facility that beneficially reuse the waste, or a state permitted facility.

5. Are there records to substantiate the above claims?

6. Does the lab use a manifest when shipping hazardous waste?

7. Is aqueous waste disposed of into a sanitary sewer only if it is neutralized and approved in writing by the sewer authority?

8. Does the lab have the following documents for review?
   a. USEPA Notification Form 8700-12
   b. USEPA Identification Number
   c. Small Quantity Generator Permit
   d. RCRA Part A Permit
   e. RCRA Part B permit
   f. NPDES Permit
   g. Manifests
   h. Waste Analysis Records
   i. Land Ban Records
   j. Exception Reports
   k. Biennial Reports
   l. Annual Reports
   m. Training and Personnel Files
   n. Contingency Plan/SPCC Plan
   o. Agreements with Local Emergency Authorities
   p. Used Oil Records
   q. Hazardous Waste Management Plan

CHART J-10 Government QA Functions (Applicable to government QA labs only.)

1. Project Coordination
   a. Designated coordinator
   b. Review CDAPS
   c. DQO clarification

2. QA Activities
   a. Review/comment on project documents
   b. Attend project meeting
   c. Perform site visits
   d. Receive/review government QA samples
   e. Analyze government QA samples
   f. Evaluate contractor QC data
   g. Prepare CQARS
   h. Other activities

3. CQAR Preparation
   a. Evaluation Parameters
      (1) Precision (RPD based on MD, MSD, and BSD/LCSD if not enough samples.)
      (2) Accuracy (Spike recovery based on LCS, MS/MSD, surrogates, and BS/BSD if not enough samples.)
      (3) Representativeness (Holding time, MB, and MQ/MSD.)
      (4) Comparability (Analytical method, MDL, precision, accuracy, and reporting unit.)
      (5) Completeness (COC, holding times, MDL, MB, LCS, MS, MD/MSD, and surrogates.)
      (6) Others
   b. Evaluation Criteria
   c. Agreement between contractor and government data
   d. Timely release (Within 20 working days after receipt of contractor's final QC data, but before the completion of contractor's final engineering report.)

4. Contract Management (Appendix L)
   a. Prepare SOW for new contract
   b. Evaluate and select contractor
   c. Assess contractor's data quality
   d. Request corrective actions
   e. Suspend/terminate contract

APPENDIX K

SAMPLE RECORD

OF

THE LABORATORY VALIDATION DATABASE

Window 1

LABORATORY FILE

GENERAL

VIEW SCREEN: 1/10
UPDATING DATE: 02/25/92

Record No: 222 ___
Lab Name: ABC alytical Laboratory _____
POC Name: Joe Frank, Mary George _____
Address: 123 Main Street _____
Suite 4 _____
City: Anytown _____
State: MD
Zip: 56789
Phone: (111) 222-3333
FAX: (111) 222-4444

Window 1
[ABC A.MD]

LABORATORY FILE

Project 1

FUNDS 1

VIEW SCREEN: 2/10

Request Date: 06/04/91
Project Name: Elmwood County Landfill_____ State: NJ
Type: SF ___ Phase: _____ Contract No: DACW01-91-C-2345
A-E/Contractor: DEF, Inc. _____ State: PA

USACE TM: John Dow _____ Office: CEMRO-ED-EZ _____
Phone: (222) 333-4444 Sampling Date: 09/01/91

HTW Analyses: VOA, BNA, PCB, PEST, TAL METALS, TRPH, CN._____

PE Sample Cost: $1100.00 Travel Cost: $275.00_ Labor Cost: $420.00_
Funds Billed: $300.00_ Date: 06/10/91
Funds Received: $300.00_ Date: 07/01/91
Funds Remarks: Billed ABC Lab $300 for non-project PEs.___

Figure K-1 Sample Record of the Laboratory Validation Database

```
Window 1
[ABC A.MD]                    LABORATORY FILE
                                                    VIEW SCREEN: 3/10
       Project 2                  FUNDS 2

   Request Date: 01/20/92
   Project Name: Any AFB; Fire Fighting Training 2A_ State: AZ
           Type: ACC _ Phase: PA/SI_______Contract  No:DACAO1-91-B-1234
 A-E/Contractor: Any Environmental Services, Inc._____ State: CA

       USACE TM: Paula Smith __________   Office: CEMRK-ED-EZ __________
          Phone: (333) 444-5555   Sampling Date: 04/15/92

   HTW Analyses: RCRA METALS, TRPH, AVO, TPH (Mod. 8015).____________________
                 ____________________________________________________________

 PE Sample Cost: $400.00_  Travel Cost: $0.00     Labor Cost: $0.00___
   Funds Billed: $0.00____  Date: 00/00/00
Funds Received: $0.00____  Date: 00/00/00
  Funds Remarks: ____________________________________________
```

```
Window 1
[ABC A.MD]                    LABORATORY FILE
                                                    VIEW SCREEN: 4/10
                                  LQMM

  Request Date: 06/05/91 Receiving Date: 06/07/91
  Title: Laboratory Quality Management Manual_______________ Date: 05/01/91
  Reviewer: S. Smith_______  Date: 07/16/91

    EPA-CLP VOA: _ Exp.Date: 00/00/00  Organics: x  Exp.Date: 05/01/92
     Inorganics: x Exp.Date: 03/01/91    Dioxin: _  Exp.Date: 00/00/00

         Dioxin: _    Radiochemical: x      TCLP: x
         EPA Compendium Air: x     AIHA/NIOSH Air: x

    # of Chemist: 35_, GC: 10, GC/MS: 4_, ICP: 2, AA: 2_, IR: 2,
          UV/VIS: 2, HPLC: 2, Ion Chromatography: 2.
  Remarks: TCLP w/ZHE; AIHA accredited for analysis of organic solvents and m
           etals; EPA Compendium method T01 only._____________________
           ____________________________________________________________
```

Figure K-1 Sample Record of the Laboratory Validation Database (continued)

Window 1
[ABC A.MD]

LABORATORY FILE

PE SAMPLE 1

VIEW SCREEN: 5/10

	PARAMETER	MATRIX	METHOD	DATE SUPPLIED	DATE RECEIVED	PASS	
1	VOA__	H2O_	8240__	06/10/91	07/03/91	GOOD	1
2	BNA__	H2O_	8270__	06/10/91	07/03/91	GOOD	2
3	BNA__	SED_	8270__	07/15/91	07/27/91	PASS	3
4	PEST_	H2O_	8080__	06/10/91	07/15/91	PASS	4
5	PCB__	H2O_	8080__	06/10/91	07/03/91	GOOD	5
6	PCB__	SED_	8080__	06/10/91	07/03/91	GOOD	6
7	META3	H2O_	SW846_	06/10/91	07/03/91	GOOD	7
8	META3	SED_	SW846_	06/10/91	07/15/91	PASS	8
9	TRPH_	H2O_	418.l_	06/10/91	07/03/91	GOOD	9
10	TRPH_	SOIL	418.1_	06/10/91	07/03/91	GOOD	10

Window 1
[ABC A.MD]

LABORATORY FILE

PE SAMPLE 2

VIEW SCREEN: 6/10

	PARAMETER	MATRIX	METHOD	DATE SUPPLIED	DATE RECEIVED	PASS	
11	HVO__	H2O_	8010__	00/00/00	00/00/00	____	11
12	AVO__	H2O_	8020__	01/26/92	02/19/92	GOOD	12
13	HERB_	H2O_	8150__	07/15/91	07/27/91	FAIL	13
14	TOC__	H2O_	9060__	06/10/91	07/03/91	GOOD	14
15	CN___	H2O_	9010__	06/10/91	07/03/91	GOOD	15
16	ANION	H2O_	300s__	06/10/91	07/03/91	GOOD	16
17	TPH__	H2O_	8015M_	01/26/92	02/19/92	GOOD	17
18	TPH__	SOIL	8015M_	01/26/92	02/25/92	PASS	18
19	_____	_____	_____	00/00/00	00/00/00	____	19
20	_____	_____	_____	00/00/00	00/00/00	____	20

Figure K-1 Sample Record of the Laboratory Validation Database (continued)

Window 1
[ABC A.MD]

LABORATORY FILE

PE SAMPLE 3

VIEW SCREEN: 7/10

PE Sample Remarks:

07/11/91: BNA/s: Several compunds were diluted out and not detected, PEST: Degradation products were too high and reported as false positives, METAL/s: Due to interelement interferences, Sb and Cd were high outside 3 σ, Se was not detected. Failed to identify and quantify HERB. 07/15/91: Revised data for PEST and METAL/s acceptable; BNA/s and HERB notacceptable. Order 2nd BNA/s and HERB. 07/27/92: Pass 2nd BNA/s but fail 2nd HERB. 02/25/92: TPH/s low outside 2ó. Revised results acceptable._____

Window 1
[ABC A.MD)

LABORATORY FILE

INSPECTION

VIEW SCREEN: 8/10

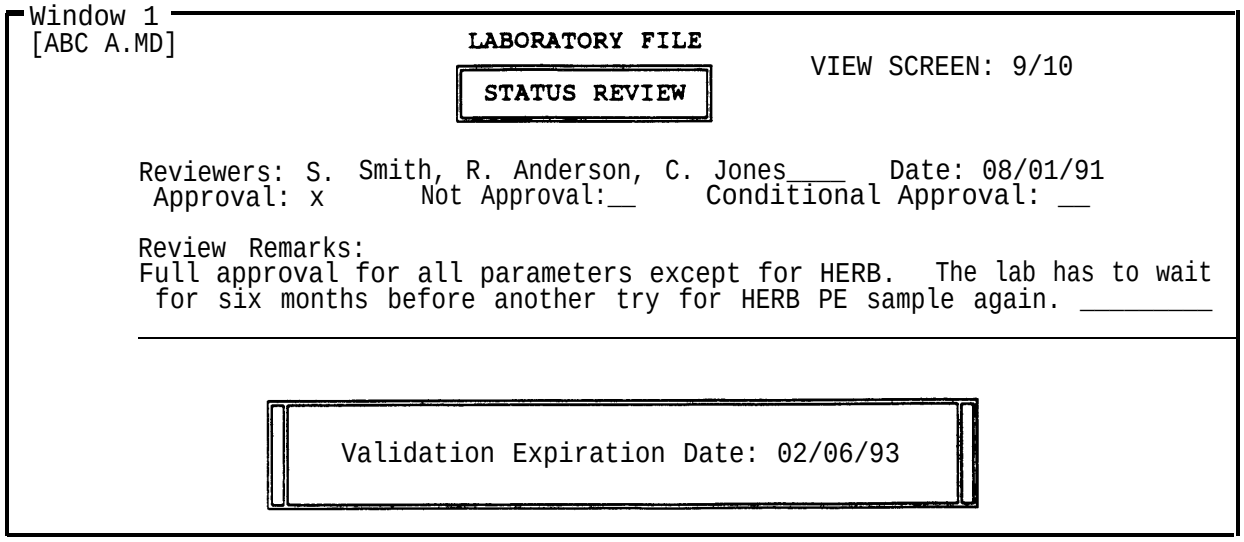
Inspector: S. Smith _____ Inspection Date: 07/25/91

Date Inspection Report To TM 1: 08/02/91 TM 2: 00/00/00 Lab: 08/02/91

Inspection Remarks:
An excellent lab. No major deficiency except for failed HERB PE. Two inor deficiencies: Needs segregated storage area for volatile standards and improved documentation for standard preparation in metal section.__

Lab Responses: Response Date: 07/30/91
Satisfactory. Will have segregated storage for volatile stadards and improved logbook for metal standard._____

Figure K-1 Sample Record of the Laboratory Validation Database (continued)



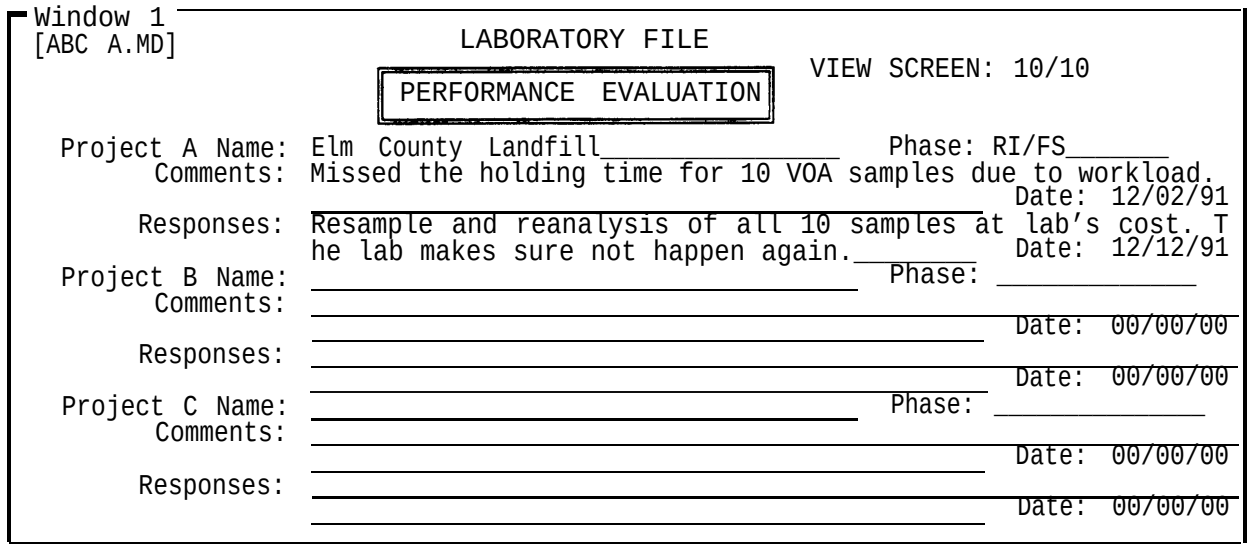


Figure K-1 Sample Record of the Laboratory Validation Database (continued)

APPENDIX L

SUPPLEMENTAL QUESTIONNAIRE

FOR

VALIDATION OF USACE DIVISION LABORATORIES

SUPPLEMENTAL QUESTIONNAIRE FOR VALIDATION OF USACE DIVISION LABORATORIES

This supplemental questionnaire is designed to elicit additional information from USACE division laboratories that serve as government Quality Assurance (QA) laboratories for the USACE HTRW programs. Please make a concerted effort to furnish the information as accurately and concisely as possible.

The preliminary questionnaire (Appendix E) is divided into seven sections. This supplemental questionnaire adds the eighth section to address the QA role of the government QA laboratories.

SECTION 8. QUALITY ASSURANCE CONTROLS

1. Are procedures documented for performing all of the required QA activities per ER 1110-2-263? Yes [ ] No [ ] If yes, please attach copies of any available SOPs to this questionnaire.

2. What kinds of QA activities does your laboratory engage in?

[ ] Review and Comment on Project Documents
[ ] Attend Project Meetings
[ ] Perform Site Visits
[ ] Receive/Review Incoming QA Samples

[ ] Analyze QA Samples In-House
[ ] Evaluate Contractor's Data
[ ] Write Chemical QA Reports
[ ] Other Activities (specify)

_____

_____

_____

_____

3. Summary of QA Staff. The primary duties of the QA staff should be the performance of QA duties as described in Item 2 above.

Name	Title	Education	Yrs Exp	QA & Other Duties Performed

4. For the complete last fiscal year, how much chemistry work (in dollars) has your lab completed?

In-House		Contracted Out	
HTRW Testing	_____	HTRW Testing	_____
Other Testing	_____	Other Testing	_____

5. For the HTRW work described above, please check the chemical analyses that are routinely conducted in-house at your laboratory at the present time and those that will be conducted at your laboratory within the next couple years:

Method	Parameters	Run Now	Run Later
8240	Volatile Organics		
8010	Halogenated Volatile Organics		
8020	Aromatic Volatile Organics		
8250/8270	Semivolatile Organics		
8080	Organochlorine Pesticides		
8080	Polychlorinated Biphenyls		
8150	Chlorinated Herbicides		
6010	Trace Metals (ICP)		
7000s	Trace Metals (Flame AA)		
7000s	Trace Metals (GFAA)		
7470/7471	Trace Metals (Hg by CVAA)		
418.1	Petroleum Hydrocarbons		
8015M	Petroleum Hydrocarbons		
8100/8310	Polynuclear Aromatic Hydrocarbons		
8330	Nitroaromatics and Nitramines		
9010/9012	Cyanide		
9060	Total Organic Carbon		
300s	Common Anions		
1311	TCLP		
Other analyses (Please list.)			

6. For the above HTRW work, how much work (in dollars) is conventional analytical work (or QC work) and how much work (in dollars) is used to perform the QA duties?

In-House		Contracted Out	
QC Work		QC Work	
QA Work		QA Work	

7. Does your laboratory write Chemical Quality Assurance Reports? Yes [ ] No [ ] If yes, how many reports has your laboratory completed during last fiscal year? _____ Please attach copies of some representative reports to this questionnaire.

8. Please complete CHART L-1 (Page L-6) for all HTRW projects performed by your laboratory during last fiscal year. Make additional copies of CHART L-1 if needed.

9. Please complete the following chart for all commercial analytical chemistry laboratories that are under current contracts to support your laboratory. For each laboratory, please list the size and duration of the contract and the type of work each laboratory is performing for you.

Lab Name/State	Contract Size/Duration	Type of Work

CHART L-1

Please circle all applicable QA functions for each HTRW project performed by your laboratory during last fiscal year.

Project Name and Phase	QA Activities
	1 2 3 4 5 6 7 8 9
	1 2 3 4 5 6 7 8 9
	1 2 3 4 5 6 7 8 9
	1 2 3 4 5 6 7 8 9
	1 2 3 4 5 6 7 8 9
	1 2 3 4 5 6 7 8 9
	1 2 3 4 5 6 7 8 9
	1 2 3 4 5 6 7 8 9
	1 2 3 4 5 6 7 8 9
	1 2 3 4 5 6 7 8 9
	1 2 3 4 5 6 7 8 9
	1 2 3 4 5 6 7 8 9
	1 2 3 4 5 6 7 8 9
	1 2 3 4 5 6 7 8 9
	1 2 3 4 5 6 7 8 9
	1 2 3 4 5 6 7 8 9

Type of QA Functions:
(1) Document Review, (2) Attend Project Meetings, (3) Perform Site Visits, (4) Receive/Review Incoming QA Samples, (5) Analyze QA Samples In-House, (6) Evaluate Contractor's Data, (7) Write Chemical QA Reports, (8) Other Activities as listed on Page L-3, (9) All of the above.

APPENDIX M

LIST OF ABBREVIATIONS, ACRONYMS, FORMULAS, SYMBOLS, NUMBERS, AND TERMS

LIST OF ABBREVIATIONS, ACRONYMS, FORMULAS, SYMBOLS, NUMBERS, AND TERMS

The following list defines the abbreviations, acronyms, formulas, symbols, numbers, and terms used in this manual. Some of these entries are common throughout the environmental industry and government offices; many are unique to the U.S. Army Corps of Engineers. They are listed here as an aid to the reader of this manual.

Term	Definition
AA	Atomic absorption spectrophotometer
AAR	Asbestos Analyst Registry
ACC	Air Combat Command
ACS	American Chemical Society
AF	Air Force
Ag	Silver
Al	Aluminum
ALKAL	Alkalinity (Methods 310s)
ANION	Common anions (Methods 300.0/300s)
APHA	American Public Health Association
API	American Petroleum Institute
ARCS	Alternative Remedial Contracts Strategy
As	Arsenic
AST	Aboveground storage tank
ASTM	American Society for Testing and Materials
AVO	Aromatic volatile organic compounds (Method 8020)
AWWA	American Water Works Association
Ba	Barium
BD	Blank duplicate
BFB	4-Bromofluorobenzene
Be	Beryllium
BNA	Base neutral, and acid extractable organic compound
BRAC	Base Realignment and Closure
BS	Blank spike
BSD	Blank spike duplicate
Ca	Calcium
CCC	Calibration check compound
CCS	Continual calibration standard
CCV	Continual calibration verification
Cd	Cadmium
CDAP	Chemical Data Acquisition Plan
CEMRD	Corps of Engineers, Missouri River Division

Term	Definition
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
CMS	Corrective measures study
CN	Total and amenable cyanide (Methods 9010A/9012)
Co	Cobalt
COC	Chain-of-custody
COD	Chemical oxygen demand (Methods 410s)
COR	Contracting officer representative
CQAR	Chemical Quality Assurance Report
Cr	Chromium
Cr (VI)	Hexavalent chromium
Cu	Copper
CVAA	Cold vapor atomic absorption
Dalapon	A trade name for a herbicide
DCBP	Decachlorobiphenyl
DDD	Dichlorodiphenyl-dichloroethane
DDT	Dichlorodiphenyl-trichloroethane
DERP	Defense Environmental Restoration Program
DFTPP	Decafluorotriphenylphosphine
DOE	Department of Energy
DOT	Department of Transportation
DQO	Data quality objective
DRO	Diesel range organics
ECD	Electron capture detector
ELCD	Electrolytic conductivity detector
EM	Engineering manual
EMSL	Environmental Monitoring Systems Laboratory
Endrin	A trade name for an insecticide
ENG	Engineering
EP	Extraction procedure
EQL	Estimated quantitation limit
ER	Engineering regulation
ERDEC	Edgewood Research, Development and Engineering Center
EXPLO	Nitroaromatics and nitramines explosives (draft Method 8330)
FAX	Facsimile
Fe	Iron
FID	Flame ionization detector
FLAA	Flame atomic absorption
FOA	Field operating activities
FPD	Flame photometric detector
FR	Federal Register
FUDS	Formerly Used Defense Site
FS	Feasibility study
GC	Gas chromatography

Term	Definition
GC/MS	Gas chromatograph/mass spectrometer
GFAA	Graphite furnace atomic absorption
GHAA	Gaseous hydride atomic absorption
GPC	Gel permeation chromatography
GRO	Gasoline range organics
HARD	Total hardness (Methods 130s)
HAZWRAP	Hazardous waste remedial action program
HCl	Hydrochloric acid
HERB	Chlorinated herbicides (Method 8150A)
Hg	Mercury
HNO_3	Nitric acid
HPLC	High performance liquid chromatography
H_3PO_4	ortho-Phosphoric acid
HRGC/LRMS	High resolution gas chromatograph/low resolution mass spectrometer
HRGC/HRMS	High resolution gas chromatograph/high resolution mass spectrometer
HQUSACE/OCE	Headquarters, U.S. Army Corps of Engineers/ Office of the Chief of Engineers
H_2SO_4	Sulfuric acid
HTRW	Hazardous, toxic, and radioactive waste
HTRW MCX	HTRW Mandatory Center of Expertise
HVO	Halogenated volatile organic compound
IC	Ion chromatography
ICP	Inductively coupled plasma-atomic emission spectrometer
ICP/MS	Inductively coupled plasma/mass spectrometer
ICS	Interference check standard
ICV	Initial calibration verification
ID	Inside diameter; or identification
IDL	Instrument detection limit
IR	Infrared
IRP	Installation Restoration Program
JP-4	An engine fuel for jet-propelled aircraft
K	Potassium
KCN	Potassium cyanide
LC/MS	Liquid chromatograph/mass spectrometer
LCS	Laboratory control samples
LCSD	Laboratory control sample duplicate
LIMS	Laboratory information management system
LVC	Laboratory validation coordinator
LQMM	Laboratory quality management manual
MB	Method blank
MCX	Mandatory Center of Expertise
MD	Matrix duplicate
MDL	Method detection limit

Term	Definition
METAL	Trace metals (Methods 6010A/7000s)
Mg	Magnesium
Mn	Manganese
Mo	Molybdenum
MRD	Missouri River Division
MRDL	Missouri River Division Laboratory
MS	Matrix spike; or mass spectrometer
MSA	Method of standard additions
MSD	Matrix spike duplicate
Na	Sodium
NaOH	Sodium hydroxide
$Na_2S_2O_3$	Sodium thiosulfate
NACIP	Naval Assessment and Control of Installation Pollutants
NEESA	U.S. Navy Energy and Environmental Support Activity
Ni	Nickel
NIOSH	National Institute for occupational Safety and Health
NIST	National Institute of Standards and Technology
NPDES	National Pollution Discharge Elimination System
NRC	Nuclear Regulatory Commission
NVLAP	National Voluntary Laboratory Accreditation Program
O&G	Oil and grease
OSHA	Occupational Safety and Health Administration
OSW	Office of Solid Waste
PA	Preliminary assessment
PAH	Polynuclear aromatic hydrocarbons (Methods 8100/8310)
PARCCS	Precision, accuracy, representativeness, comparability, completeness, and sensitivity
PAT	Proficiency Analytical Testing Program
Pb	Lead
PCB	Polychlorinated biphenyls (Method 8080)
PCDD	Polychlorinated dibenzo-p-dioxins
PCDF	Polychlorinated dibenzofurans
PE	Performance evaluation
PES	PE sample supplier
PEST	Organochlorine pesticides (Method 8080)
PFB	Pentafluorobenzylbromide
pH	Potential of hydrogen
PHENO	Phenols (Method 8040A)
PHENL	Phenolics (Methods 9065/9066/9067)
PID	Photoionization detector
POC	Point of contact

Term	Definition
POL	Petroleum, oils, or lubricant
PP	Priority pollutant
ppb	Part per billion
ppm	Part per million
PQL	Practical quantitation limit
QA	Quality assurance
QC	Quality control
%R	Percent recovery
RCRA	Resource Conservation and Recovery Act
RFA	RCRA facility assessment
RFI	RCRA facility investigation
RA	Remedial action
RADCHEM	Radiochemistry
RAS	Routine Analytical Service
RD	Remedial design
REM	Remedial Engineering Management
RF	Response factor
RI	Remedial investigation
RPD	Relative percent difference
RRT	Relative retention time
SAE	Society of Automotive Engineers
SAS	Special Analytical Service
Sb	Antimony
SBA	Small Business Administration
SDWA	Safe Drinking Water Act
Se	Selenium
SEM	Scanning electron microscope
SF	Superfund
SI	Site investigation
SIM	Selected ion monitoring
SOP	Standard operating procedure
SOW	Scope of work
SPCC	System performance check compound; or spill prevention control and countermeasure
SQG	Small quality generator
SS	Stainless steel
SW-846	The USEPA document, "Test Method for Evaluating Solid Waste"
TAL	Target Analyte List
TBA	Tetrabutylammonium
TCL	Target Compound List
TCD	Thermal conductivity detector
TCDD	Tetrachlorodibenzo-p-dioxins
TCLP	Toxicity characteristic leaching procedure
TCMX	2,4,5,6-Tetrachloro-m-xylene
TDS	Total dissolved solids (Method 160.1)

Term	Definition
TIC	Tentatively identified compound
Tl	Thallium
TO	Toxic organics
TOC	Total organic carbon (Method 9060)
TOX	Total organic halides (Methods 9020A/9022)
TM	Technical manager
TM/COR	Technical manager/contracting officer representative
TPH	Total petroleum hydrocarbons (modified Method 8015)
TRPH	Total recoverable petroleum hydrocarbons (Method 418.1)
TSS	Total suspended solids (Method 160.2)
USACE	U.S. Army Corps of Engineers
USAEC	U.S. Army Environmental Center
USAFOEHL	U.S. Army Force Occupational and Environmental Health Laboratory
USDA	U.S. Department of Agriculture
USEPA	U.S. Environmental Protection Agency
USGS	United States Geological Survey
UST	Underground storage tank
UV/VIS	Ultraviolet/Visible
V	Vanadium
VOA	Volatile organic analyte (Method 8240A)
VOC	Volatile organic compound
v/v	Volume to volume
WES	Waterways Experiment Station
WP	Water pollution
WPCF	Water Pollution Control Federation
WS	Water supply
ZHE	Zero-headspace extractor
Zn	Zinc
130s	USEPA methods for total hardness
300s	USEPA methods for common anions
300.0	USEPA method for common anions by ion chromatography
310s	USEPA methods for alkalinity
410s	USEPA methods for chemical oxygen demand
413.1	USEPA method for oil and grease by gravimetric method
413.2	USEPA method for oil and grease by IR
418.1	USEPA method for total recoverable petroleum hydrocarbons by IR
601	USEPA method for purgeable halocarbons by GC
602	USEPA method for purgeable aromatics by GC

Term	Definition
603	USEPA method for acrolein and acrylonitrile by GC
604	USEPA method for phenols by GC
606	USEPA method for phthalate esters by GC
607	USEPA method for nitrosamines by GC
608	USEPA method for organochlorine pesticides and PCBS by GC
609	USEPA method for nitroaromatics and isophorone by GC
610	USEPA method for polynuclear aromatic hydrocarbons by GC
611	USEPA method for haloethers by GC
612	USEPA method for chlorinated hydrocarbons by GC
624	USEPA method for purgeables by GC/MS
625	USEPA method for base/neutrals and acids by GC/MS
1010	USEPA method for ignitability by Pensky-Martens closed-cup method
1020A	USEPA method for ignitability by Setaflash closed-cup method
1110	USEPA method for corrosivity toward steel
1310A	USEPA method for extraction procedure toxicity test method and structural integrity test
1311	USEPA method for toxicity characteristic leaching procedure
3005A	USEPA method for acid digestion of water for total recoverable or dissolved metals for analysis by FLAA or ICP
3010A	USEPA method for acid digestion of aqueous samples and extracts for total metals for analysis by FLAA or ICP
3020A	USEPA method for acid digestion of aqueous samples and extracts for total metals for analysis by GFAA
3040	USEPA method of dissolution products for oils, greases, or waxes
3050A	USEPA method for acid digestion of sediments, sludges, and soils
3060	USEPA method for alkaline digestion of hexavalent chromium in solid waste
3510A	USEPA method for separator funnel liquid-liquid extraction
3520A	USEPA method for continuous liquid-liquid extraction
3540A	USEPA method for Soxhlet extraction
3550	USEPA method for sonication extraction

Term	Definition
3580A	USEPA method for waste dilution
3610A	USEPA method for alumina column cleanup
3620A	USEPA method for Florisil column cleanup
3630A	USEPA method for silica gel cleanup
3640	USEPA method for gel-permeation cleanup
3650A	USEPA method for acid-base partition cleanup
3660A	USEPA method for sulfur cleanup
3810	USEPA method for headspace extraction and screening of purgeable organics
5030A	USEPA method for purge-and-trap
6010A	USEPA method for metal analysis by ICP
7000s	USEPA methods for metal analysis by FLAA, GFAA, or CVAA
7060	USEPA method for analysis of aqueous arsenic samples by GFAA
7061A	USEPA method for analysis of aqueous arsenic samples by GHAA
7470	USEPA method for analysis of mercury in liquid waste by CVAA
7471	USEPA method for analysis of mercury in solid or semisolid waste by CVAA
7740	USEPA method for analysis of aqueous selenium samples by GFAA
7741	USEPA method for analysis of aqueous selenium samples by GHAA
8010A	USEPA method for halogenated volatile organics by GC
8015A	USEPA method for nonhalogenated volatile organics by GC
8015M	Modified USEPA Method 8015 for total petroleum hydrocarbons by GC
8020	USEPA method for aromatic volatile organics by GC
8040A	USEPA method for phenols by GC
8080	USEPA method for organochlorine pesticides and PCBs by GC
8100	USEPA method for polynuclear aromatic hydrocarbons by GC
8140	USEPA method for organophosphorus pesticides by GC
8150A	USEPA method for chlorinated herbicides by GC
8240A	USEPA method for volatile organics by GC/MS
8250	USEPA method for semivolatile organics by packed column GC/MS
8270A	USEPA method for semivolatile organics by capillary GC/MS

Term	Definition
8280	USEPA method for PCDD and PCDF by GC/MS
8310	USEPA method for polynuclear aromatic hydrocarbons by HPLC
8330	Draft USEPA method for nitroaromatics and nitramines by HPLC
9010A	USEPA method for total and amenable cyanide by manual calorimetric method
9012	USEPA method for total and amenable cyanide by automated calorimetric method
9060	USEPA method for total organic carbon
9065	USEPA method for phenolics by manual spectrophotometric method
9066	USEPA method for phenolics by automated calorimetric method
9067	USEPA method for phenolics by spectrophotometric method
9071	USEPA method for oil and grease extraction of sludge samples

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