

**APPENDIX C**  
**Quality Assurance**

## **APPENDIX C**

### **Quality Assurance Plan**

#### **C.1 Purpose and Scope of the Plan**

The purpose of the quality assurance plan was to establish processes to ensure that:

- Demonstration conditions and operations were planned, communicated, and documented.
- Sufficient measurements were made to assess the effectiveness of the treatment methods.
- Samples taken were representative of the conditions in the demonstration.
- Samples were delivered to the laboratory for analysis without deterioration.
- Samples were processed by the laboratory without deterioration prior to analysis.
- Measurement techniques were sufficiently specific to measure the target compounds.
- Data collected or generated were reliable.

The quality assurance plan applied to all activities, including performing experiments, sampling, and laboratory analysis of samples.

TVA's Analytical Laboratory provided analytical chemistry support for the project by performing analyses for metals, nutrients, and soil characteristics. Procedures for extraction and analysis of EDTA were developed and tested for this project.

#### **C.2 Quality Assurance Responsibilities**

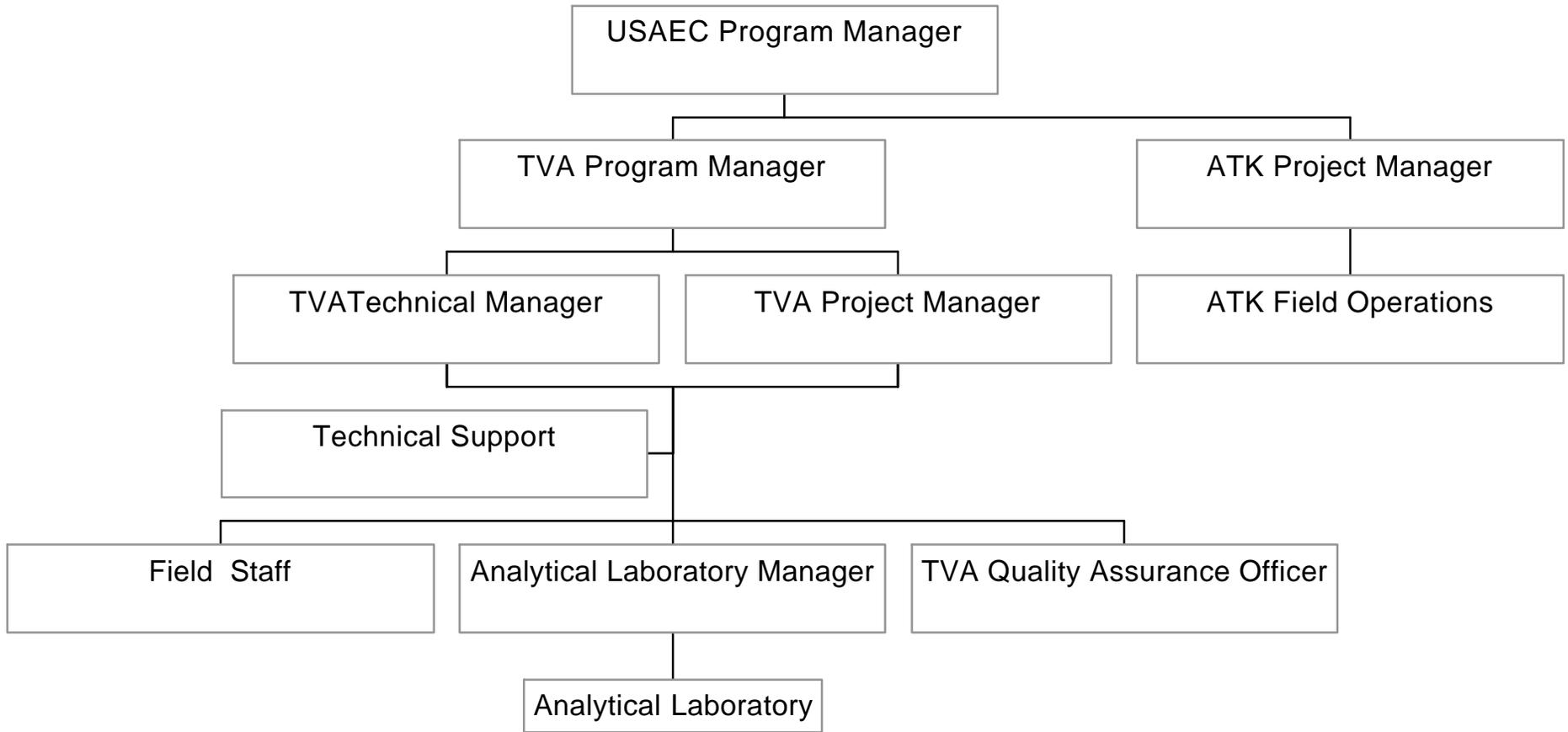
The attached organizational chart (Figure C-1) shows the TVA organizations providing support to the project.

Responsibilities of the USAEC project team were as follows:

- The USAEC Program Manager was responsible for ensuring that the USAEC and ESTCP project and program goals were met.

Responsibilities of the TCAAP project team were as follows:

- The ATK Project Manager was responsible for overall direction of project field operations at TCAAP. These responsibilities included oversight and direction of staffing levels; process design, procurement, construction, and maintenance; field process operations; ATK-directed laboratory work; technical reports; preparation and presentation of technical papers; and conducting tours and briefings. The ATK Project Manager provided direction to ATK team members to ensure that project goals were met, reports were delivered on schedule, and that task schedules and costs were met. The ATK Project Manager ensured that any variances related to ATK areas or responsibility were adequately explained and was the primary interface with TVA.
- The ATK Field Operations Staff provided assistance to the ATK Project Manager to assure that ATK responsibilities were met.



**Figure C-1**  
**Project Organization Chart**

Responsibilities of the TVA project team were as follows:

- The TVA Program Manager was responsible for providing guidance to the project and ensuring that program goals were met. The TVA Program Manager was also responsible for resolving any inconsistencies between USAEC, TCAAP, TVA, and ATK mission objectives and those of the project.
- The TVA Project Manager was responsible for overall direction of the project and was responsible for oversight and direction of staffing levels, process design, equipment installation, maintenance, field process operations, technical reports, preparation and presentation of technical papers, and conducting briefings of USAEC personnel. The TVA Project Manager was responsible for providing direction and executing tasks to ensure that project goals were met, reports were delivered on schedule, and that task schedules and costs were met. The TVA Project Manager ensured that any variances were adequately explained.
- TVA's Technical Manager was responsible for planning and implementing the details of the field studies, including experimental design, field process operations, sampling, documentation, maintaining data integrity, data interpretation, providing technical reports to the TVA Project Manager, and preparation and presentation of technical papers. The TVA Technical Manager was available to assist the TVA Project Manager in conducting briefings to Army personnel. The TVA Technical Manager was also the primary interface with ATK field support staff and provided technical direction for field activities.
- The TVA Field Staff reported to the TVA Technical Manager and was responsible for providing assistance in various field tasks during TVA visits to the site.
- The TVA Analytical Laboratory in Muscle Shoals, Alabama, was responsible for providing analytical measurements on soil, plant, and soil solution samples required in the course of the project and was responsible for review of the data produced, documentation of analytical runs, and ensuring data integrity. The laboratory was managed by the Analytical Laboratory Manager. The Analytical Laboratory Manager reported to the TVA Technical Manager and was responsible for providing project analytical oversight and for final analytical data integrity.
- Technical Support Staff provided technical assistance to the TVA Technical Manager in experimental design, data interpretation, troubleshooting, and report writing.
- The TVA QA Officer was responsible for implementing the QA program and for auditing actions and documentation to ensure adherence to this section. The TVA QA Officer was responsible for providing quarterly QC data reports to the TVA Project Manager.

### **C.3 Quality Program Procedures and Documents**

The Analytical Laboratory activities conducted during this project were carried out in accordance with the laboratory's Quality Assurance Manual which contains the following documents:

- QAPLAN - "Quality Assurance Plan"
- GLP-0001 - "Procedure Format and Style"
- GLP-0002 - "Quality Assurance Records Control"
- GLP-0003 - "Procedure Preparation and Distribution"
- GLP-0004 - "Training"
- GLP-0005 - "Nonconformances and Corrective Actions"
- GLP-0006 - "Control of Reagents and Standards"
- GLP-0007 - "Analysis Work Plan Preparation"
- GLP-0012 - "Treatment of Data"
- GLP-0013 - "Instrument Logbook and Control Chart Maintenance"
- GLP-0016 - "Sample Receipt, Log-in, and Data Handling"
- GLP-0017 - "Control of Changes to Software"
- CP-0001 - "Measurement and Test Equipment Control and Calibration"
- SP-0001 - "Sample Chain of Custody"

Laboratory analyses were conducted in accordance with written procedures. Modifications to procedures found to be necessary to perform the analyses required in this test plan were noted in equipment operation logs or research notebooks until included in revisions to procedures. Two procedures were developed for this project: AP-0047 "EDTA by High Performance Liquid Chromatography" and AP-0057 "Extraction of EDTA from Soil."

The various quality control samples associated with each analytical run were assessed at the time the data were produced by both analytical staff members and the quality assurance officer. Furthermore, project data from all runs were accumulated and assessed for reasonableness and consistency by the researchers. Consequently, research and quality staff members feel that the quality assurance objectives for the analytical measurement processes associated with this project were met.

The experimental portion of this plan was performed in accordance with the project plan. Data, observations, experimental conditions, and minor modifications to planned activities were recorded in research or field notebooks in a complete enough fashion that all actions, results, and conclusions could be reconstructed.

Sampling was conducted in accordance with written work plans, procedures, or instructions to ensure complete samples were taken at correct times and in a manner which did not invalidate conclusions. All actions in sampling were recorded in research or field notebooks or on forms designed to ensure complete documentation of all experimental parameters. Instructions were provided for proper preservation of samples.

#### **C.4 Control of Purchased Items**

Chemicals, equipment, materials, and other items purchased to conduct this project were of suitable quality to meet the project needs as specified in the written procedures. Purchased items were inspected upon receipt to ensure they met the requirements specified in purchase requests. Nonconforming items were not used. Suitable handling activities, storage conditions, and other controls were utilized to ensure quality of purchased items was not degraded after receipt.

#### **C.5 Record Control**

Records of analysis, records of calibration, research notebooks, chromatograms, sampling logs, custody records, work plans, machine printouts, chromatogram traces, logsheets, standard material use records, raw data calculation sheets, and copies of procedures were maintained as quality assurance records as specified in GLP-0003. Records were accumulated in logical arrangement to facilitate retention and review. In-process records and logbooks were stored in the work area in a safe manner to protect against loss, fire, spills, or other damage.

Records of experiments and analyses will be maintained for a three-year period after the end of the project. This includes machine printouts or chromatogram traces, logbooks, notebooks, logsheets, standard material use logs, and raw data calculation sheets. Due to the limited lifetime of computer storage media, any computer media utilized to store analytical file backups or raw data files will be stored for the lifetime of the project plus one year.

#### **C.6 Data Quality Parameters**

##### **C.6.1 Accuracy and Precision**

Percent recovery, relative percent difference, standard deviation, and other commonly used statistical indicators of accuracy and precision were calculated as defined in Chapter 1 of SW-846, 3rd Edition.

##### **C.6.2 Method Detection Limit, Method, Quantitation Limit**

Method Detection Limits were calculated as defined in Title 40, Code of Federal Regulations, Part 136, Appendix A, "Definition and Procedure for the Determination of the Method Detection Limit" - Revision 1.11.

Method Quantitation Limits were defined as five times the Method Detection Limit as in Chapter 1 of SW-846, 3rd Edition, or as the lowest point used in making the calibration curve, whichever was higher.

#### **C.7 Calibration Procedures and Quality Control Checks**

The precision and accuracy of new or revised analytical procedures were investigated before the procedures were used for analysis of samples.

## **C.7.1 Initial Calibration Procedures**

### **C.7.1.1 Laboratory Instrumentation**

The calibration frequencies and quality control tests required in SW-846 for HPLC methods were used in the HPLC method for EDTA. The calibration frequencies and quality control tests required in SW-846 for metals analysis were used for ICP and AA methods. Guidelines for calibration frequencies and tests, as specified by the manufacturer, were used for flow injection analyzer (FIA) methods.

## **C.8 Analytical Laboratory Calibration and Quality Control**

### **C.8.1 General Quality Control Requirements**

The project's analytical data were calculated on vendor-supplied software for the HPLC system, FIA system, and ICP spectrophotometer. These systems typically integrate sample signals, calculate calibration curves automatically, and apply the curves to sample measurements. However, a spreadsheet developed at TVA was used to fit curves and calculate data for the HPLC analysis. Other laboratory calculations were carried out on spreadsheets developed and tested at TVA or on hand-held calculators (e.g., soil moisture). Some devices such as pH meters, give direct readout or printout of analytical data.

### **C.8.2 Batch QC**

With each batch of 20 samples or subset thereof, one method blank, one matrix spike, and one laboratory control sample were run. In addition, one sample duplicate or one matrix spike duplicate was run with each batch. Note: For some analytical techniques, matrix spikes were not possible.

### **C.8.3 Quality Control Requirements for HPLC**

Retention time windows were determined and the device was calibrated during development of the procedure. Five calibration standards were used.

At the beginning of each day that analyses were conducted, the midpoint calibration standard was analyzed. Then, every ten samples and at the end of the run, a midpoint calibration standard was run again in accordance with the quality control requirements for HPLC devices.

### **C.8.4 Quality Control for Automated Laboratory Instrumentation**

Flow Injection Analyzers (FIA) were calibrated before each use following written procedures. For FIA, calibration was performed with standards of five concentrations at the beginning of each day. Concentrations bracketed the range of interest, but were limited to the range of linear response of the device.

For these devices, a midpoint calibration standard was run at least every ten samples and at the end of the run throughout the day. Any group of ten samples preceding and following a midpoint calibration check which fell outside the 15% limit was reanalyzed.

For these devices, a laboratory control sample made from a separate stock than the calibration standards was run with each batch. For any of these devices, samples exhibiting a signal above the linear range of the device were diluted and reanalyzed.

### C.8.5 Definitions

- **Batch** - Usually a group of no more than 20 samples of the same matrix prepared or extracted at the same time with the same reagents.
- **Method Blank** - A sample of clean reagent carried through preparation and extraction in the same manner as samples. One method blank was run with each batch.
- **Matrix Spike** - An aliquot of a sample spiked with a known concentration of all target analytes. Spike concentration was selected to read at five times the Method Quantitation Limit in the sample or about the midpoint of the calibration curve. One matrix spike was run for each batch. Spiking occurred prior to sample preparation and analysis.
- **Matrix Spike Duplicate** - A second aliquot of the same sample treated in the same manner as the matrix spike.
- **Duplicate** - A second aliquot of a sample taken independently through extraction and preparation before analysis.
- **Quality Control Check Sample** - A quality control sample of the same type and matrix as calibration solutions, but made independently from the calibration solutions. This sample is also referred to as a laboratory control sample.

### C.8.6 Data Reduction, Validation, and Reporting

#### C.8.6.1 Data Reduction

The project's analytical data were calculated on vendor-supplied software for the HPLC system, FIA system, and ICP spectrophotometer. These systems typically integrate sample signals, calculate calibration curves automatically, and apply the curves to sample measurements. However, a spreadsheet developed at TVA was used to fit curves and calculate data for the HPLC analysis. Other laboratory calculations were carried out on spreadsheets developed and tested at TVA or on hand-held calculators (e.g. soil moisture). Some devices such as pH meters, give direct readout or printout of analytical data.

The Analytical Laboratory's Chemical Laboratory Analysts were responsible for calculation and reduction of data.

#### C.8.6.2 Data Validation

Analytical measurements were first reviewed by the chemist producing them and then by another chemist before being interfaced with the laboratory database. If quality control samples fell outside limits, the samples were usually scheduled for reanalysis. After questions were resolved, results were passed on to the Laboratory Manager for final review and validation.

Group supervisors or team leaders were responsible for decisions concerning reanalysis of samples and coordinated with the Project Manager when significant problems were discovered or when resampling was required.

### **C.8.6.3 Data Reporting**

Analytical data were reported in units of milligrams per liter for liquid samples. Solid sample results were reported as milligrams per kilogram dry weight unless other units such as percent were more appropriate.

Method Detection Limits and Instrument Detection Limits were reported for each run. Recovery of matrix spikes and recovery of quality control samples were calculated and reported as percentages.

### **C.8.6.4 Corrective Action**

Corrective action in accordance with the requirements of GLP-0005 was not identified in the course of this project.

## **C.9 Performance and System Audits**

### **C.9.1 Performance Audits**

Analytical Laboratory participated in USEPA Water Pollution Studies twice yearly during this project. The Analytical Laboratory investigated any analyte falling outside control limits and reported its findings to the Quality Assurance Officer in writing. Participation in this cross-checking process provides information on Analytical Laboratory's performance as compared to other laboratories in the nation.

### **C.9.2 On-Site System Audits**

The Analytical Laboratory's Quality Assurance (QA) Officer periodically inspected logs, records, printouts, results of quality control checks, documentation, case narratives, research notebooks, and other quality-related aspects of the project to ensure detailed compliance.

## **C.10 Quality Assurance Reports**

### **C.10.1 Status Reports**

TVA's Project Manager provided periodic progress reports to USAEC which contained a summary of accomplishments and a discussion of significant problems and their resolution.

Quarterly quality control data reports were written by the QA Officer addressing:

- Changes in this QA project plan
- Changes in analytical procedures
- Summary of QC program results
- Summary of training
- Results of audits
- Results of performance sample evaluations

- Data quality assessment in terms of precision, accuracy, and MDLs
- Discussion of whether QA objectives were met

## **C.11 Data Management and Analysis**

### **C.11.1 Analytical Data**

Analytical data packages for the project included:

- Sample description or identification information
- Sample analytical results
- Quality control sample results with surrogate recoveries and percent recovery of known compounds

Sufficient data were maintained such that experimental and analytical results could be reconstructed.

Records of all attempts at analysis were maintained whether or not the analysis was successful. However, unusable data were not reported. Data were unusable when quality control samples or quality control checks failed; however, the records for these attempts at analysis were maintained with relevant documentation. Data Qualification Codes in use by the laboratory and which may have been encountered in review of this project's data were as follows:

**NA** - Compound not analyzed

**<MDL** - Compound not detected (value falls less than Method Detection Limit)

**TR or Trace** - Compound present at trace level, indicated but less than MDL

**Q** - "Qualified" - For a sample in which an analyte was quantified, but an associated quality control sample fell outside control limits

## **C.12 Contract Laboratory**

A contract laboratory was used on two instances in October and November 1998 to perform arsenic analysis by ICP when an instrument failed at TVA. The samples were prepared at TVA with inclusion of laboratory duplicates, matrix spikes, method blanks, and laboratory control samples. The total number of samples involved was 104 for the first set and 95 in the second set. Response on the quality control samples was satisfactory.