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QUALITY ASSURANCE PLAN FOR THE K-25 ANALYTICAL CHEMISTRY LABORATORY

May 1990

This document replaces SOP 2300 and incorporates a reformatting of the topics to meet the requirements of ANSI/ASME NQA-1, EPA SW-846, EPA QAMS 005/80, and DOE/EH-0053.

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ACRONYMS

AA	Atomic Absorption
ACD	Analytical Chemistry Department
ADP	Automatic Data Process
ANSI	American National Standards Institute
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CLP	Contract Laboratory Program
DMR	Discharge Monitoring Report
DOE	Department of Energy
EML	Environmental Measurements Laboratory
EMSL	Environmental Monitoring Support Laboratory
EPA	Environmental Protection Agency
GC/MS	Gas Chromatograph Mass Spectrometer
ICP	Inductively Coupled Plasma
IDL	Instrument Detection Limit
MDL	Method Detection Limit
NIOSH	National Institute for Occupational Safety and Health
NPDES	National Pollution Discharge Elimination System
NQA	Nuclear Quality Assurance
ORGDP	Oak Ridge Gaseous Diffusion Plant
OSHA	Occupational Safety and Health Agency
PARCC	Precision, Accuracy, Representativeness, Completeness, Comparability
PAT	Proficiency Analytical Testing
PCB	Polychlorinated Biphenyl
PET	Proficiency Evaluation Testing
PQL	Practical Quantitation Limit
QA/QC	Quality Assurance/Quality Control
QAD	Quality Assessment Division
QAMS	Quality Assurance Management Staff
QAS	Quality Assurance Staff
RCRA	Resource Conservation and Recovery Act
SOP	Standard Operating Procedure
SW	Solid Waste
TD	Technical Division
WP	Water Pollution
WS	Water Study

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1. ORGANIZATION AND PERSONNEL

The Analytical Chemistry Department (ACD) at the Oak Ridge K-25 Plant is a full service analytical laboratory providing sampling, analyses, instrumentation, development, and consulting services supporting a wide range of programs. These programs include environmental, waste management, and health protection. Modern analytical services are provided to meet various regulatory standards including Environmental Protection Agency (EPA), Occupational Safety and Health Agency (OSHA), Department of Energy (DOE), and others. A major portion of the ACD work effort is devoted to environmental and waste programs covered under such acts and agencies as National Pollution Discharge Elimination System (NPDES), Resource Conservation and Recovery Act (RCRA), Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), EPA, DOE, and OSHA.

1.1 ORGANIZATION AND FUNCTIONAL RESPONSIBILITIES

The ACD employs a select staff of highly trained and qualified technical and management personnel. The staff consists of a ratio of approximately one chemist to every two technicians, support personnel, and instrument mechanics. Due to the diversities of many projects, the ACD has established a project and technical organization led by experienced responsive management personnel reporting directly to management. The schematic of the organization is presented in Fig. 1-1. Responsibilities are defined below.

1.1.1 Laboratory Manager

- Overall management of the analytical laboratory.
- Appoints technical managers within department.
- Supervises program managers.
- Coordinates implementation of the quality assurance policy and program as directed by the parent organization.
- Provides the Methodology and Quality Assurance (QA) Manager with the organizational freedom and authority to manage the QA program, to verify that activities affecting quality have been correctly performed, and to take appropriate actions to resolve any quality problems.
- Promptly notifies upper management of potential quality problems or conditions adverse to quality.
- Has overall responsibility for QA in the ACD Laboratory.

K-25 Analytical Chemistry Department

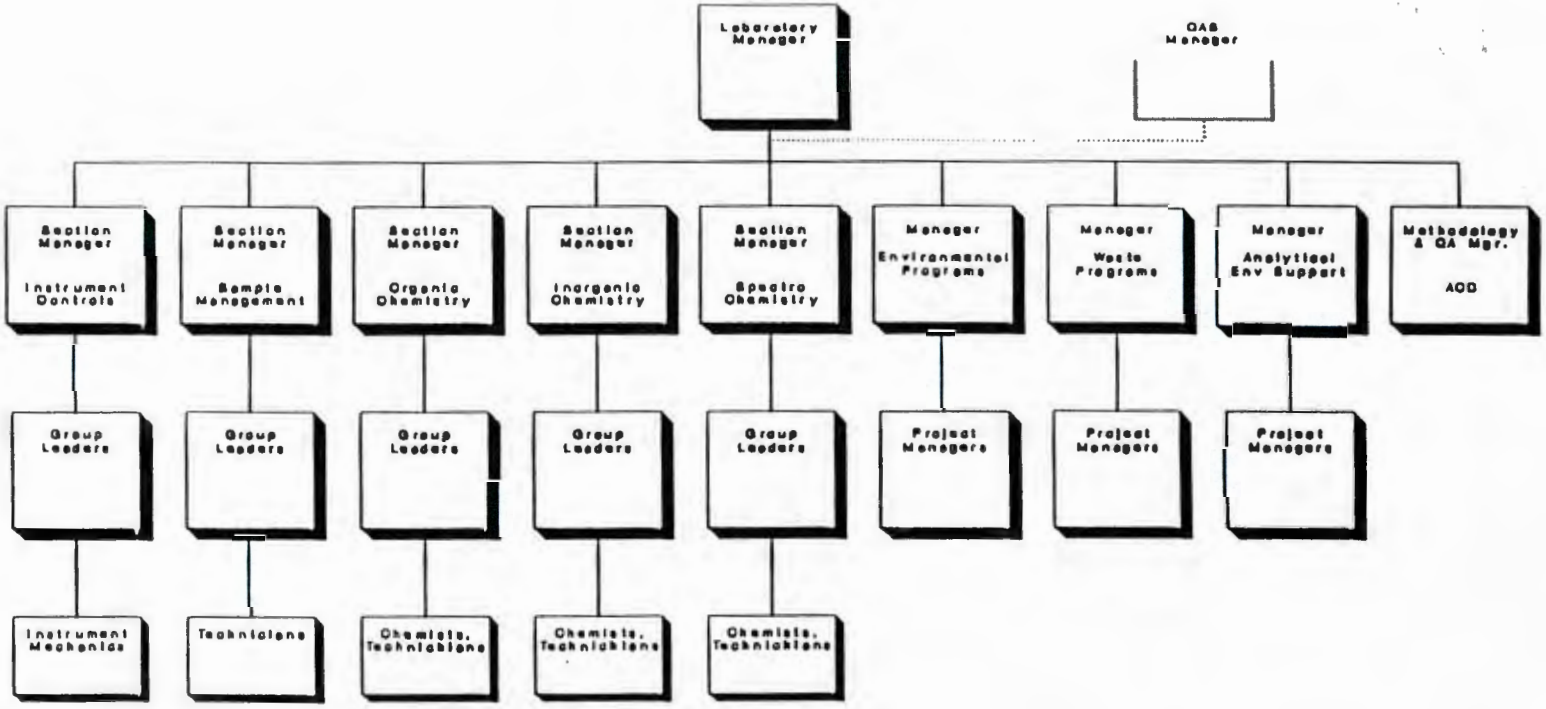

Figure 1-1

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1.1.2 Methodology and Quality Assurance Manager

- Assures adherence to the specified methodology employed by the laboratory.
- Reports directly to the ACD Laboratory Manager and provides QA status reports as requested.
- Assures that quality shall be verified by personnel not directly responsible for performing the work.
- Provides QA oversight to all Laboratory groups and serves as the focal point for the ACD QA program.
- Interprets QA policy and approves the QA practices, standards of performance, and QC procedures used in the ACD.
- Responds to audits.
- Implements corrective action plans.
- Provides guidance and assistance to laboratory personnel in the implementation of the ACD QA program.
- Monitors resolution of conditions adverse to quality and exercises "stop work authority" if a quality problem has escalated beyond the authority of front-line supervision.
- Monitors the processing of nonconforming items.
- Verifies that activities affecting quality have been performed correctly.
- Ensures that training of personnel conforms to the ACD and K-25 Training Programs.

1.1.3 Program Managers

- Supervise project managers who coordinate all phases of laboratory related work and are the principal contact between customers and the ACD.
- Liaison with customers.
- Assure that schedules are met within projects.
- Participate in project planning, budget analysis, scheduling of work, determining proper methodology.

- Final review of quality of the data.
- Ensure compliance with regulatory agencies.

1.1.4 Section Leaders

- Overall management of the section.
- Supervise the group leaders.
- Ensure that schedules are met.
- Ensure that the required QA/QC is applied.
- Provide technical direction.
- Participate in project planning, budget analysis, scheduling of work, determining proper methodology.
- Final review of quality of the data.
- Ensure that procedures pertaining to health and safety, quality assurance, analytical operations, and equipment operations are available and current.
- Ensure that the above procedures are followed.
- Ensure the quality of the data through the proper use of quality tools, such as control charts and other documentation.
- Investigate conditions adverse to quality, and initiate corrective action through appropriate channels.
- Exercise "stop work authority" when a significant condition adverse to quality cannot be resolved satisfactorily.
- Inform Methodology and QA Manager when conditions adverse to quality have not been resolved.

1.1.5 Group Leaders

- Assign duties to technicians and staff and ensure that they are adequately trained to perform their assigned duties.
- Ensure that proper QA/QC is applied.

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- Supervise work within their individual laboratory.
- Review and approve batch folders.
- Approve data for reporting.
- Correct QA deficiencies.
- Schedule work and training.
- Investigate conditions adverse to quality, and initiate corrective action through appropriate channels.

1.1.6 Chemists and Technicians

- Follow the procedures that pertain to their duties, including health and safety, quality assurance, analytical operations, an equipment operations.
- Approve data quality prior to reporting.
- Perform required quality assurance activities and the associated documentation.
- Report nonconformances or defective materials to supervision.
- Provide quality improvement input to supervision.

1.2 LABORATORY FIELDS OF ACTIVITY

The ACD is within the Technical Division (TD) with a Laboratory Manager. The department is divided into a technical organization with five sections and a programmatic organization of program/project managers. The five analytical sections are: Instrumentation Controls, Sample and Waste Management, Organic Analytical Chemistry, Inorganic Analytical Chemistry, and Spectro Analytical Chemistry. The program management organization consists of: Environmental, Waste Management, and Analytical Environmental Support.

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2. QUALITY ASSURANCE PROGRAM

2.1 SCOPE

A major component of the mission of the K-25 ACD is to provide high quality analytical measurements and data that meet the needs of the user by meeting defined standards of quality. As a provider of physical and chemical data, several applicable quality standards must be met to assure that when data are provided to the data users they are of needed quality. Those quality standards are specified by:

(1) Project standards which meet the requirements of DOE, Energy Systems, and site orders and operating procedures.

(2) Project standards which meet the requirements of NQA-1.

(3) Project standards which meet the quality assurance requirements of the customer as defined by regulatory agencies (i.e., EPA, OSHA).

In general, the quality assurance program will be supplemented as necessary to meet any additional requirements, such requirements may be documented in other plans. The purpose of this document is to provide a basic criteria for the quality assurance activities of the ACD. The specific elements of ANSI/ASME NQA-1 (Nuclear Quality Assurance), EPA SW-846, and QAMS 005/80 are addressed and met where applicable in this document. Individual QC SOPs have been combined into an operating manual:

K/QT-1032 ACD QC Manual

Table 2.1 shows the comparison of specific elements between NQA-1, QAMS 005/80, and SW-846.

2.2 PURPOSE

The purpose of this document is to provide a summary of each of the activities relating to the QA/QC activities of the ACD as they apply to NQA-1, QAMS 005/80, and SW-846. In addition, this document outlines those activities the ACD is committed to maintaining in order to ensure conformance to the requirements and responsibilities necessary for being a quality analytical laboratory organization. Standard plant QA procedures which the ACD follows are:

K-25 Standard Practice Procedure 701	Quality Assurance (QA) Organization
K-25 Standard Practice Procedure 705	Quality Assurance (QA) Program
K-25 Standard Practice Procedure 708	Quality Status Reporting
K-25 Standard Practice Procedure 709	Quality Assurance Plans

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Table 2.1 Comparison of NQA-1 to QAMS 005/80 and SW-846

NQA-1 (K-25 QA Plan per NQA-1)	QA Parameters Addressed in the Following Protocols QAMS 005/80/SW-846
1. Organization	4. Project Organization and Responsibility
2. QA Program	3. Project Description /16. QA Reports to Management
3. Design Control	5. QA Objectives/6. Samp Proc/10. Data Validation/11. Int QC/14. PARCC
4. Procurement Document Control	
5. Instructions, Procedures, and Drawings	
6. Document Control	1. Title Page/2. Table of Contents
7. Control of Purch. Items and Services	
8. Identification and Control of Items	7. Sample Custody
9. Control of Process	9. Analytical Procedures
10. Inspection	
11. Test Control	
12. Control of Measuring and Test Equipment	8. Calibration/13. Maintenance
13. Handling, Storage and Shipping	6. Sampling Procedure
14. Inspection, Test and Operations Status	
15. Control of Nonconforming Items	
16. Corrective Action	15. Corrective Action
17. Quality Assurance Records	
18. Audits	12. Performance and System Audits

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K-25 Standard Practice Procedure 710	Quality Assurance Training and Awareness Programs
K-25 Standard Practice Procedure 711	Qualification and Certification of Personnel
K-25 Standard Practice Procedure 731	Control of Purchased Items and Services
K-25 Standard Practice Procedure 758	Handling, Storage, and Shipping
K-25 Standard Practice Procedure 772	Quality Problem and Failure Costs
K-25 Standard Practice Procedure 776	Quality Assurance Records
K-25 Standard Practice Procedure 790	Quality Suggestion Program

Regulatory samples are analyzed using the specified procedures as listed in the Federal Register, SW-846, Contract Laboratory Program (CLP), or other EPA procedures as applicable. The minimum QA/QC applied to any type analysis is given in the procedure below.

Procedure 2303 Analytical Quality Control Implementation

The quality assurance/quality control activities of the laboratory are designed to meet the CLP of EPA for Superfund Activities, and customers may specify CLP criteria for their samples. All GC/MS analyses are performed under the CLP protocol, and the CLP data package is available upon request. Other analyses are performed under CLP protocol upon customer request. All Drinking Water samples are analyzed per the Code of Federal Regulations for Public Water Supply, CFR 40, Part 141.

2.3 ASPECTS OF THE QA PROGRAM

All activities associated with the collection of physical and chemical data are incorporated in this quality assurance program. Such activities include all phases which affect the validity of data, such as sampling, analysis, and data handling. QA terminology used by the ACD is covered in the following procedure:

Procedure 2321 QA Terminology for the ACD

The Quality Assurance Program employed by the ACD consists of three key areas with several elements or activities contributing to the quality standards established by the laboratory.

2.3.1 Responsibility

The responsibility of producing and maintaining high quality data is mandated, supported, and implemented at all management levels relative to the samples. The section leaders are responsible for policy oversight and resources. The group leaders are responsible for implementation, supervision, and the utilization of technical competence.

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2.3.2 Quality Control

Quality control incorporates the following components within the laboratory: (1) protocols incorporating sample measurement and data evaluation, (2) good laboratory practices as specified in SOPs, and (3) educational training which may be formal or informal.

2.3.3 Quality Assessment

Quality assessment exists within the QA program and provides assurance that the quality control is being applied effectively. The laboratory's quality assessment is continually evaluated with internal assessments consisting of control charts; test samples such as duplicates, spikes, or surrogates; audits; and cross checks. External assessment consists of participation in state, federal, and national QC programs.

2.4 SAMPLE TYPES

Samples submitted for analysis by the laboratory have been divided into two categories

2.4.1 Regulatory Samples

All samples where the data are reported to or used by regulatory agencies. These samples are used to evaluate RCRA waste sites, develop clean-up plans, monitor discharges, industrial hygiene, etc. The actual sampling operation is performed by trained technicians using specific approved sampling procedures. A record of the sampling at each locale is made and retained permanently. Regulatory samples also include referee samples from other government agencies and DOE sites.

2.4.2 Nonregulatory Samples

Samples from process control operations, inventory accountability, development activities, etc. The laboratory is not responsible for the sample collection for all of these samples. Upon receipt in the laboratory, proper storage and holding times are maintained for nonregulatory samples.

2.5 TRAINING OF PERSONNEL

Training and qualification of chemists, technicians, and those performing clerical work is an important factor affecting laboratory activities. Training within the ACD varies from direct, on-the-job training by a more experienced person to a formal program involving both classroom and on-the-job training. The extent of training required depends on the complexity of the work, educational background, level of competence, previous experience, the economics involved, and the overall importance of the work in meeting the quality assurance goals of the laboratory. Training within the department is ongoing, and all

laboratory personnel are encouraged to attend seminars, courses, and professional meetings as appropriate. The K-25 training is covered in the following procedure:

K-25 Standard Practice Procedure 322 K-25 Training Procedure

The ACD adheres to SPP 322 for performance-based training. Lesson plans are prepared according to this procedure. Training records are to be recorded by TMIS and by the ACD Training Manager.

The laboratory's technician qualifications system is documented in the following:

Procedure 2333 Qualification of Sampling Personnel
Procedure 2337 Qualification of Personnel
Procedure 2205 ACD Training Plan

Generally, the laboratory group leader, technical support chemists, or analysts experienced in an analytical procedure train other analysts in analytical procedures on a one-on-one basis. Throughout the training period, the trainer is made available to the trainee and reviews the trainee's work on a daily basis. The training may consist of but is not limited to the following:

- Special glassware procedures
- Preparation of standards and reagents
- Operation of instruments and equipment
- Sample analysis
- Calculations
- Quality control requirements and acceptance criteria
- Common interferences and corrective measures

Upon completion of training, the analyst's training record is updated within the laboratory's computer system (AnaLIS) and by hard copy form in the ACD office.

New employees receive quality assurance training as part of their qualification training whereby they are taught the quality control requirements and acceptance criteria. New employees also receive laboratory safety hazards communication as part of their orientation during the first week of employment. Thereafter, safety training is held on a monthly basis for all laboratory personnel through safety meetings.

2.6 QUALITY ASSURANCE REPORTS

Copies of the laboratory performance on the external QC samples are received by the Methodology and QA Manager. If the reports show nonconforming data for the laboratory, the raw data files are reviewed to determine the reason for the unacceptable results.

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A written response is made by the Methodology and QA Manager to the originator of EPA or EPA sponsored control programs. This response documents the reason for the nonconforming laboratory data and the actions to be taken to prevent future failures.

Copies of all laboratory performance data are received by the Methodology and QA Manager and submitted to management with comments where appropriate. All the external QC data are evaluated, condensed, and included as part of the yearly Environmental Report of the Oak Ridge Operations. This report is provided to all levels of upper management and to the state and federal agencies. The reports become a part of the local public library and are given to local news media.

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3. DESIGN CONTROL

As specified in NQA-1, "Scientific investigations shall be defined, controlled, and verified. Process variables affecting scientific investigations shall be measured and controlled." To carry out this requirement, within the laboratory organization is a program management organization which has the responsibility for developing, coordinating, and directing the implementation of programs requiring sampling and analysis as specified by the EPA and other regulatory agencies. To ensure that this responsibility is carried out by the laboratory, each major project requires that a QA Project Plan cover each monitoring or measurement activity to be completed. The K-25 design control is covered in the following procedure.

K-25 Standard Practice Procedure 715 Design Control

3.1 PROJECT MANAGER'S RESPONSIBILITIES

The Project Manager's responsibilities relative to the specific QA within a project includes the following:

(1) Have a good basic understanding of QA practices and the principles within the project QA. Work closely with the Methodology and QA Manager whose job it is to be thoroughly knowledgeable of ways to achieve and document good quality work.

(2) Be knowledgeable of the sampling and analytical procedures used.

(3) Assess if the ACD has adequate resources to accomplish the work at the level of quality desired without taking unjustified shortcuts to hinder the QA.

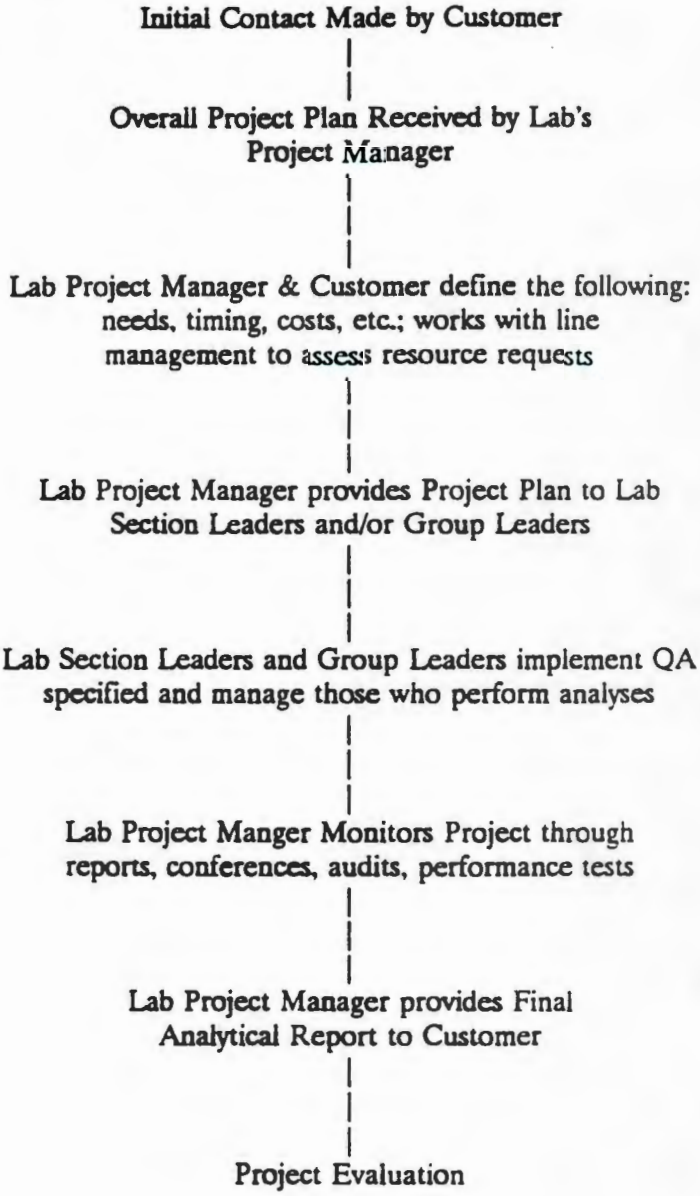
(4) Use well founded QA practices to identify any sampling or analytical problems before effort is lost in producing unreliable data.

3.2 PROJECT DEVELOPMENT

Each project requiring analytical chemistry support starts with the development of a Project Work Plan which describes how the effort will be accomplished. One key component of the Project Plan is a discussion of the sampling and analysis program and the procedures that will be followed to validate the quality of the data. Throughout the course of the project, monitoring of the QA may be carried out by the use of conferences and project reviews, QA audits, performance test samples, split samples, and the review of the project reports and QC charts. ACD services can be obtained following this procedure:

Procedure 2335 Obtaining Laboratory Services

The use of a Project Manager is required to ensure that services are provided. The flow diagram below depicts the flow of work for a project throughout the laboratory:



When the required data quality for a given activity is established, the data user (along with the lab project manager) shall select the appropriate level of analytical support that will supply data of the required quality.

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3.3 PROJECT WORK PLAN

The work plan may be prepared before the start of each project. The plan may include the following:

(1) Project Objectives
(2) Project Organization
(3) Facilities and Equipment needed
(4) Sampling Plan
(5) Analytical Plan
(6) Project QA Plan
(7) Project Schedule

The project QA plan is generally a section within the work plan, specifically addressing quality assurance of the sampling and analysis efforts. The decision to have or not to have a Project QA Plan lies with the project manager or leader with the concurrence of the QAS. In the broader sense, the total work plan encompasses aspects of project quality assurance. The project QA plan presents the policies, organization, objectives, functional activities, and specific QA and quality control activities designed to achieve the data quality goals of the specific projects. The plan must also include a description of the project and the experimental design. Some items included which describe the project are: flow diagrams, tables and charts, anticipated start and completion dates, and the intended use of the acquired data.

Smaller or short-term projects may be coordinated with a project manager. These projects may or may not have formal, written project plans.

3.4 DATA QUALITY OBJECTIVES

Data Quality Objectives are based upon the specific activity pursued and the intended use of the data. They are restricted by several conditions such as field conditions and the goal of the end use of the data. Such tolerances are defined in terms of Precision, Accuracy, Representativeness, Completeness, and Comparability (PARCC). When the data quality produced by a given analytical and sampling operation matches the data quality required, then the PARCC requirements are met. PARCC components are defined as:

- Precision - a measure of the reproducibility of measurements under given set of conditions.

- Accuracy - a measure of the bias that exists in a measurement system.

- Representativeness - the degree to which sample data accurately and precisely represent selected characteristics.

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

- Comparability - the confidence with which one data set can be compared to another.

All the PARCC parameters are interrelated in terms of overall data quality and may be difficult to evaluate separately due to those interrelationships. The relative significance of each of the PARCC parameters depends on the type and intended use of the data being collected and on the project.

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3.5 INTERNAL QUALITY CONTROL

To determine data quality, control samples or checks are used to measure the quality of the sampling and analysis process. Sample blank, method blank, splits, sample and laboratory duplicates/replicates, surrogates, matrix spikes, etc. are used at a planned frequency to accomplish quality control. All QC data are available to the customer. Duplicates, blanks, spikes, surrogates, and replicate data are included as part of the sample data report. QC data are plotted by AnaLIS.

3.6 DATA EVALUATION

The method of calculation of final results from raw data varies from parameter to parameter. The appropriate analytical method shall be referenced for the calculations used to quantitate the amount of analyte in the samples.

In general, results are expressed to two significant figures. Results of aqueous samples are expressed in milligrams per liter or micrograms per liter, and results of nonaqueous samples are expressed on a dry weight basis in milligrams per kilogram, micrograms per kilogram, or micrograms per gram. The moisture content of samples is also reported so that results can be converted to a dry-weight basis. Results obtained for GC/MS analysis are evaluated by the CLP requirements.

The laboratory group leader, on a daily basis, reviews the raw data for meeting specified requirements. Other levels of data validation are covered in the following procedure.

Procedure 2309 Data Validation

The results of quality control checks, such as spikes, duplicates, and blanks, are used for data evaluation as well. The group leader confirms that quality control checks were performed at the required frequency and that the acceptance criteria were met.

Nonconforming internal data result in an automatic mail message to the person entering the data, appropriate supervision, and the Methodology and QA Manager. Immediate corrective action and documentation is required. The requirements for corrective action are given in the following procedure.

Procedure 2314 Documentation of "Out-of-Control" Incident

The approval of the reported result is documented in the AnaLIS. Data packages are available upon request, and in most cases the group leader checks the completed deliverables against the raw data. Raw data are contained in batch folders. This is covered in the following procedure.

Procedure 2309 Data Validation

The group leader or project manager may compile the case narrative addressing problems and observations during the sample analysis. Batch folders may be sent to the section leader for further technical review.

The package may be reviewed by the Methodology and QA Manager who reviews the uniformity of the package relative to QA/QC requirements. This review is always performed on CLP data packages.

3.7 DETECTION LIMITS

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. Practical quantitation limits (PQL) refer to the lowest concentration of analyte that a method can accurately detect in either a sample or blank under ideal conditions. For metals, the instrument detection limit (IDL) refers to the smallest signal above background noise that an instrument can reliably detect.

Detection limit determinations for CLP by ICP and AA are performed by analysis of a standard solution (each analyte in reagent water) at a concentration of three to five times the instrument manufacturer's suggested IDL, with seven consecutive measurements per day on three nonconsecutive days. Each measurement must be performed as though it were a separate analytical sample (i. e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). The standard deviation is calculated each day. The MDL is set at three times the average standard deviation of the measurements. Verification of detection limit studies for metals analysis is performed quarterly.

For non-CLP work by ICP and AA, a reporting limit is used which is derived from another method of IDL determination. This method utilizes the standard deviation of 10

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determinations of a blank solution. Three times this standard deviation is the IDL. The reporting limit used for "ideal" samples is approximately two to four times the IDL.

Detection limit determinations for GC and GC/MS are performed according to CLP requirements.

The following procedure describes detection limits:

Procedure 2326 Limits of Detection

3.8 SAMPLING PROCEDURES

There are specific written procedures for use by the sampling personnel which are based on EPA or Federal Code of Regulations for the routine sampling of monitoring wells and surface water. These procedures are referenced in section 13.1. For specialized and nonroutine sampling, work plans and sampling guidance are provided by a project manager.

3.9 DATA VALIDATION

Different levels of data validation are applied to projects or types of samples depending upon customer requirements and the final use of the data. All data must be reviewed and approved by the analyst or chemist prior to release, and additional levels of validation are made based upon the data quality objectives as defined by the customer. Data validation is covered in two procedures. These are:

Procedure 2309 Data Validation
Procedure 2303 Analytical Quality Control Implementation

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4. PROCUREMENT DOCUMENT CONTROL

Materials procurement for the analytical laboratory on a routine basis is limited to chemicals, reagents, supplies, instruments, and services required to maintain the building and equipment. Procured materials include the standards used for calibration and standardization.

Major items of equipment purchased or leased are procured under government guidelines and utilize engineering or procurement specifications.

ORGDP Standard Practice Procedure 331	Procurement of Classified Items or Services
ORGDP Standard Practice Procedure 719	Procurement Document Control
ORGDP Standard Practice Procedure 731	Control of Purchased Items and Services

Purchases of direct charge items and services are approved by the Laboratory Manager. The requisitioner provides all information needed to complete the procurement document for major capital purchases, including the following: scope of work, technical requirements, required supplier QA program controls, provisions for access for inspection and monitoring and/or performance testing, vendor documentation requirements, conformance requirements, and QA plans for purchase of large dollar items. The requisitioner also identifies technical requirements, drawings, and specifications on the purchase requisition and designate routine or special inspection requirements. Purchase orders are used for expense items.

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5. INSTRUCTION, PROCEDURES, AND DRAWINGS

5.1 ANALYTICAL METHODS

The ACD provides a complete spectrum of environmental testing and analytical services ranging from basic wet chemistry analyses to sophisticated nonroutine organic, inorganic, and radiochemical analyses. Analyses are performed on a wide variety of matrices including water, wastewater, soils, sediments, sludges, hazardous wastes, low-level radioactive wastes, and air sampling media.

The laboratory utilizes nationally approved procedures (if they exist) for the analysis of environmental and industrial hygiene samples. These include EPA procedures, Federal Code of Regulation Procedures, NIOSH procedures for industrial hygiene samples, Standard Method of Analysis for Water and Wastewater, ASTM procedures, and CLP procedures. In lieu of nationally approved procedures, in-house procedures are developed, written, reviewed, and approved by technical personnel. These procedures are reviewed every five years and reapproved.

The K-25 procedure which describes these items is the following:

K-25 Standard Practice 723 Instructions, Procedures, and Drawings

The ACD is able to provide many of the analytical services specified in the following documents: (1) Federal Register, Guidelines Establishing Procedures for the Analysis of Pollutants under the Clean Water Act, 40 CFR, Part 136, July 1987; (2) U.S. EPA Contract Laboratory Programs, Statement of Work for Organic Analysis, Rev. 2/88; (3) U.S. EPA Contract Laboratory Program, Statement of Work for Inorganic Analyses, Rev. 7/87; (4) U.S. EPA Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA/SW-846, Vols. 1A, B, C, and 2, Rev. 3, November 1986, Washington, DC.; and (5) NIOSH Manual, NIOSH, U.S. Dept. of Human Services, Cincinnati, OH. Many other additional analyses are provided upon request.

The program or project managers are responsible for designating the procedures to be employed. The procedures to be used are then specified on the work cards prior to submission to the analyzing groups. Line managers provide copies of all analytical procedures at the laboratory work place.

5.2 STANDARD OPERATING PROCEDURES (SOPs)

To control analyses and avoid misunderstandings leading to erroneous results, procedures are written that provide direction for those performing the work. Written procedures also provide information for training analysts, establish the technical basis of the methods, and document the process used in the analyses. To be effective and to provide

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credibility to the laboratory, procedures must be well written, complete, and correct. Administrative operating procedures provide information for radiation protection handling laboratory wastes, laboratory safety, nuclear material handling, salvaging samples, etc.

SOPs are covered in the following:

Procedure 2203 Writing, Revising, and Maintaining Standard Operating Procedures

The following may be included as elements of the SOP:

(1) Title
(2) Significance and Use
(3) Safety
(4) Responsibilities
(5) Definitions
(6) Interferences
(7) Apparatus and Instrumentation
(8) Reagents
(9) Sampling and Handling
(10) Procedure (step by step instructions for doing the work)
(11) Calculations
(12) Precision and Bias
(13) Safety
(14) References

All SOPs have management and editorial reviews to ensure technical adequacy. Procedures are approved by the Section Leaders and the Lab Manager. Table 5.1 gives an index to the current SOPs in the ACD QC Manual. SOPs relating to a specific section are retained in the section leader's office. SOPs for all sections are retained in the ACD office.

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Table 5.1 Index to individual measurement control and administrative procedures

2300	Description of QA-QC Activities Relating to Analytical Chemistry
2301	Description of Control Programs for the Analytical Chemistry Department
2302	Data Monitoring and Control Chart Plotting
2303	Analytical Quality Control Implementation
2307	Description of Control Samples for PCBs in Oils
2308	Calibration of Laboratory Equipment
2309	Data Validation
2310	Preparation of Air Filter Controls for Metallic Elements
2311	Preparation of Air Sample Controls for Organic Vapors
2312	Preparation of Routine Urine Control Samples
2313	Preparation of Miscellaneous Industrial Hygiene Control Samples
2314	Documentation of "Out-of-Control" Conditions
2316	Controls for Monitoring Discharge Waters
2317	Program for Spiking Samples with Unknown Matrices
2319	Program for Duplicate Samples
2320	Records of Standards
2321	QA-QC Terminology for the Analytical Chemistry Department
2323	Laboratory Notebooks
2324	Laboratory Waste Disposal
2325	Quality Control of Reagent Water for Laboratory Use
2326	Limits of Detection
2327	Segregation of Incoming PCB Samples
2328	Report Preparation for DOE Site Survey Samples
2329	Security for DOE Site Survey Samples
2330	Action Plan for Laboratory Contamination
2331	Internal Chain of Custody
2332	Receipt and Tracking of DOE Site Survey Samples
2333	Qualification Criteria for Field Sampling Personnel

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Table 5.1 (continued)

2335	Obtaining Analytical Chemistry Laboratory Services
2336	Analysis Name Files
2337	Qualifications of Personnel
2021	Ambient Air Sampling for Uranium, Specific Metals, and Particulates
2023A	Well Water Sampling
2024	Sump Sampling
2026	Tank and Drum Sampling
2025	NPDES and Surface Water Sampling
2051	Archiving Designated Samples
2053	Custodian Sample Handling and Receiving
2053B	Radiological Surveying of Received Samples
2053C	Sample Transportation
2054A	Disposal of Non-Uranium Solid Waste
2054B	Disposal of Non-Uranium Liquid Waste and Samples
2054C	Handling and Disposal of Nuclear Accountability Samples and Waste
2054D	K-1004-A Dock and K-1004-E Operation
2054E	Disposal of Vendor Labeled Chemicals
2055	Chain of Custody
2200	Radioactive Contamination Control Policy
2201	Personnel Monitoring for Radioactivity Using Friskers
2203	Writing, Revising, and Maintaining Standard Operating Procedures
2205	Analytical Chemistry Department Training Plan

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6. DOCUMENT CONTROL

Document control for the analytical chemistry laboratory is mainly related to the procedures employed, control and retention of laboratory workbooks, maintaining the QA files, and maintaining the computer database and analytical data.

The section leaders are responsible for ensuring that a copy of the procedure is at the work station where the analysis is performed. The procedures are designated by the line managers, the program manager and the Methodology and QA Manager.

All laboratory workbooks for analytical data are obtained from Plant Records, assigned an accountability number, and must be returned to Plant Records when filled or when employee leaves the company.

All QA/QC data from each analysis is assigned a QA file number for reference purposes and retained as permanent records. Measurement control and QA SOPs are written by the Methodology and QA Manager and approved by the Laboratory Manager. Administrative and technical SOPs are written by the appropriate Section Leader and approved by the Laboratory Manager.

The records on the computer are backed up each day, and these become a permanent record in accordance with DOE and plant policy. The following procedures describe record keeping.

Reference: K-25 Standard Practice Procedure 727 Document Control
ESS.ADP.1 Automatic Data Process (ADP) Software Quality Assurance Program Standard
Procedure 2323 Laboratory Notebooks
Procedure 2309 Data Validation

6.1 LABORATORY RECORDS

The records used to document the work carried out in the laboratory consist of the following: analysis request form, logbook, data records, and the analytical report. Such documents serve to carry out the functions of traceability of analytical results back to raw data and establishment of who did the work and how it was done. The AnaLIS is used to maintain records for managing the sample information. The computer retains sample information received from the customer, provides sample identification, transmits information and data through the laboratory, provides a record of data and information, and reports results of analyses. Analysis names are covered in the following procedure.

Procedure 2336 Analysis Name Files

6.2 DATA REPORTING

Electronic reporting is used for many of the samples from the Oak Ridge area. Hard copy data, including full CLP data packages, are available to the customer upon request.

Reporting of hard copy data, upon request from the program manager, is the responsibility of the Sample Management Section Manager.

DOE Site Survey sample data are reported according to the following procedure.

Procedure 2328 Report Preparation for DOE Site Survey Samples

6.3 PROCEDURES

Specific SOPs and technical procedures are written as required for operation. The format for these are given in the following:

Procedure 2303 Analytical Quality Control Implementation
Procedure 2203 Writing, Revising, and Maintaining Standard Operating Procedures

The procedures issued by the ACD are controlled according to the following:

Procedure 2339 Control of ACD Procedures

This procedure ensures that all procedures in the work areas are the latest revision. It also ensures that all affected personnel have been informed and trained, if necessary, in the application of the new or revised procedure.

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7. CONTROL OF PURCHASED MATERIALS AND EQUIPMENT

Supplies and chemicals used in the sampling and analysis are covered by purchase specifications issued by the Central Procurement Division. Designated lots of solvents and acids are purchased to ensure reliability, and chemicals are labeled with date of receipt and expiration date. This is covered in the following procedure.

Procedure 2320 Records of Standards

The reliability or quality of measurements made in the laboratory depends on the integrity of the equipment and materials purchased and used. An inventory of all hazardous materials within ACD is generated and maintained by ACD and Industrial Hygiene.

When items cost less than $50,000, the requisition is reviewed and approved by the Section Leader and submitted to the Purchasing Department. When items exceed $50,000, the requisition is reviewed and approved by the Section Leader, Laboratory Manager, Division Head, and Plant Manager. Capital equipment requests costing less than $5,000 are reviewed and approved by the Section Leader. Capital equipment requested over $5,000 require request forms for Capital Equipment Funds, Division Manager approval, and submission at a specified time prior to fiscal year budget meetings. An alternative to capital equipment requests that may be denied because of a lack of capital equipment funds is leasing of the equipment.

Procurement control is covered in the following Energy Systems Procurement Procedures:

Reference:	K-25 Standard Practice Procedure 731	Control of Purchased Items and Services
	K/QA - 7.0	Control of Purchased Items and Services
	Procedure - 1	Procurement General
	Procedure - 4	Warranties and Manufacture's Data
	Procedure - 6	Handling of Damaged or Defective Material
	Procedure - 8	Inspection of Material

8. IDENTIFICATION AND CONTROL OF ITEMS

This procedure provides a system for assuring that correct and/or accepted items are used and controlled so that identification of items is established and maintained.

8.1 MANAGEMENT OF SAMPLES

8.1.1 Archiving Samples

Samples may be archived at the customer's request. This is covered by the following:

Procedure 2051 Archiving Designated Samples

The data base consists of the customer's sample number, requested parameters, and deadlines. The parameters and other pertinent information are automatically placed in the responsible section's backlog. Data are retrievable as to be traceable to the sample.

8.1.2 Uranium Samples

Samples containing uranium will be handled and disposed of in accordance with the following procedures:

Procedure 2054C	Handling and Disposal of Nuclear Accountability Samples and Waste
Procedure 2201	Personnel Monitoring for Radioactivity Using Friskers
Procedure 2200	Radioactive Contamination Control Policy
Procedure 2053B	Radioactive Surveying of Received Samples

8.1.3 Sample Receipt and Custody

The primary objective of sample custody is to create an accurate written verified record that can be used to trace the possession and handling of the samples from the moment of collection until receipt by the laboratory. Adequate sample custody will be achieved by means of approved field and analytical documentation.

CLP samples will be maintained under strict chain of custody as specified in the protocol.

All regulatory samples are received by Sample Management under a chain-of-custody procedure. This includes samples obtained by the laboratory samplers and those received from outside sources. The following procedures covering the actual receipt and sample custody activity.

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Procedure 2055	Chain of Custody
Procedure 2053	Custodian Sample Handling and Receiving
Procedure 2054A	Disposal of Non-Uranium Solid Waste
Procedure 2054B	Disposal of Non-Uranium Liquid Waste and Samples
Procedure 2327	Segregation of Incoming PCB Samples
Procedure 2329	Security for DOE Site Survey Samples
Procedure 2331	Internal Chain of Custody and Priority of Analysis
Procedure 2332	Receipt and Tracking of DOE Site Survey Samples
Procedure 2335	Obtaining Analytical Chemistry Laboratory Services

8.2 MANAGEMENT OF EQUIPMENT

Surplus equipment, stored equipment, and in-use equipment will be handled in accordance with Energy Systems procedures.

K-25 Standard Practice Procedure 339	Disposal of Excess or Surplus Equipment or Material
K-25 Standard Practice Procedure 736	Identification and Control of Items

8.3 PROCEDURES

The following procedures identify ORGDP's general QA methods:

K-25 Standard Practice Procedure 705	Quality Assurance Program
K-25 Standard Practice Procedure 701	Quality Assurance Organization

These procedures are used to identify required quality of items and services.

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9. CONTROL OF PROCESS

The analytical process within the laboratory is carried out in a planned, systematic, and controlled manner producing results that are valid and based on sound technology. An analysis involves discrete actions taken in a specific order. Any change in an action or in the order without a valid reason can produce unsatisfactory results. To control the analysis process and avoid errors leading to unsatisfactory results, procedures that are written and followed provide direction for those doing the work. The following describes how the analytical process is controlled beginning with sample receipt and continuing through analysis and disposal.

Procedure 2309	Data Validation
Procedure 2314	Documentation of "Out-of-Control" Incidents
Procedure 2301	Description of Control Programs for ACD
Procedure 2053	Sample Handling and Receiving
Procedure 2053B	Radiological Surveying of Received Samples
Procedure 2317	Procedure for Spiking Samples with Unknown Matrices
Procedure 2319	Program for Duplicate Samples
Procedure 2308	Calibration of Laboratory Equipment
Procedure 2303	Analytical Quality Control Implementation
Procedure 2310	Preparation of Air Filter Controls For Metallic Elements
Procedure 2311	Preparation of Air Sample Controls for Organic Vapors
Procedure 2312	Preparation of Routine Urine Control Samples
Procedure 2313	Preparation of Miscellaneous Industrial Hygiene Control Samples
Procedure 2324	Laboratory Waste Disposal
Procedure 2054A	Disposal of Non-Uranium Solid Waste
Procedure 2054B	Disposal of Non-Uranium Liquid Waste and Samples
Procedure 2054C	Handling and Disposal of Nuclear Accountability Samples and Waste
Procedure 2054D	K-1004-A Dock and K-1004-E Operation
Procedure 2054E	Disposal of Vendor Labeled Chemicals
K-25 Standard Practice Procedure 741	Control of Processes

Figure 9-1 illustrates the procedure change documentation required for modifying a procedure.

Procedure 2203	Writing, Revising, and Maintaining Standard Operating Procedures

The laboratory utilizes nationally approved procedures (if they exist) for the analysis of environmental and industrial hygiene samples. These include EPA procedures, Federal Code of Regulation Procedures, NIOSH procedures for industrial hygiene samples, Standard Method of Analysis for Water and Waste Water, ASTM procedures, and CLP procedures. In lieu of nationally approved procedures, in-house procedures are developed, written, reviewed, and approved by technical personnel. These procedures are reviewed every five years and reapproved.

Analytical Chemistry Department

PROCEDURE MODIFICATION/CHANGE NOTICE

The following changes are approved for Procedure No. _____, Revision No. _____.
(if applicable)
Clearly reference each step in the original procedure that has been changed.

_____

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APPROVALS:

_____ _____
Supervisor Date

_____ _____
Technical or Program Manager Date

_____ _____
Methodology and QA Manager Date

Fig. 9-1

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10. INSPECTION

Deficiencies may be found during the normal performance of work. Other ways are through audits and surveillance activities such as management assessments. External audits may also be performed. An internal audit program is carried out by personnel not directly responsible for work and operations being audited, but who have a good working knowledge of the laboratory's operation.

Surveillance inspections are made for evaluation of projects. Surveillance activities monitor or observe whether an item or activity conforms to specified requirements. QC personnel review the following items as appropriate for the project:

(1) Validity or authenticity of samples, field measurements, and methodology
(2) Proper documentation
(3) Use of standard units
(4) Proper sample identification
(5) Conformance to appropriate sample handling and preservation techniques
(6) Conformance to chain of custody
(7) Appropriateness of required number and types of field QC samples
(8) Logbook protocols and agreement with actual samples
(9) Documentation of equipment calibration
(10) Conformance with appropriate decontamination procedures
(11) Corrective actions and reports associated with variances and nonconformances

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11. TEST CONTROL

11.1 INTERNAL QC CHECKS

All GC/MS analyses are performed using the QA/QC specified in the CLP protocol. All analyses, other than GC/MS, are performed using the QA/QC specified by EPA or the Federal Register for regulatory samples. If there is no specified QC, the criteria given in procedures listed below are applied.

Procedure 2303 Analytical Quality Control Implementation
Procedure 2309 Data Validation

Internal QC checks (or Bench Controls analyzed) are made with each batch of samples processed for an analyte. The results from the internal QC checks are plotted by the computer, and access to the data plots is available to personnel performing the analyses and other appropriate personnel. This is covered in the following procedure.

Procedure 2302 Data Monitoring and Control Chart Plotting

Any nonconforming results cause an automatic mail message to be generated to the person entering the data and appropriate supervision. Immediate corrective action is required. The requirements for corrective action are given in the following procedure.

Procedure 2314 Documentation of "Out-of-Control" Incident

Included in the internal QC check program are:

(1) Duplicate samples obtained on 10% of all sample taken by the laboratory sampling group and submitted as separate samples.

Procedure 2319 Program for Duplicate Samples

(2) One or more spikes in each batch of samples processed where feasible.

Procedure 2317 Program for Spiking Samples

(3) Replicate analysis of prepared samples. These are performed per regulatory requirements, at customer request, or to verify the instrument output.

The following procedures also provide internal QC checks:

Procedure 2307 Description of Control Samples for PCBs in Oils
Procedure 2310 Preparation of Air Filter Controls for Metallic Elements
Procedure 2311 Preparation of Air Sample Controls for Organic Vapors

Procedure 2312	Preparation of Routine Urine Control Samples
Procedure 2313	Preparation of Miscellaneous Industrial Hygiene Control Samples
Procedure 2316	Controls for Monitoring Discharge Waters

11.2 EXTERNAL QC PROGRAM

The laboratory participates in the following external control programs:

(1) CLP-EPA - Performance samples supplied by EPA EMSL-Las Vegas under the Superfund program for laboratories.

(2) DMR-EPA - Waste water QC samples supplied by EPA EMSL-Cincinnati for all NPDES permit holders.

(3) QAD-EPA - Radionuclide QC sample supplied by EPA Las Vegas for laboratories performing radionuclide analyses.

(4) QAD-EML - Environmental radionuclide samples supplied by the DOE Environmental Laboratory in New York.

(5) WP-XXX - Water Pollution QC samples supplied by EPA Las Vegas for the DOE laboratories.

(6) WS-XXX - Water Supply QC samples supplied by EPA for all laboratories qualified to perform analysis of drinking water supply samples.

(7) P.A.T. - Proficiency Analytical Testing QC samples supplied by NIOSH for maintaining certification for Industrial Hygiene related samples.

(8) P.E.T. - Proficiency Evaluation Testing QC samples from a private supplier of QC environmental samples to Martin Marietta Energy Systems.

(9) Asbestos - Round-robin testing per CFR, includes the following labs: ORGDP, PGDP, PORTS, ORNL, Y-12, WMCO, TVA, and Martin Marietta in MD.

(10) P.E.E - Artificial urine sample purchased from a private supplier of QC samples for health physics and industrial hygiene analytes.

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12. CONTROL OF MEASURING AND TEST EQUIPMENT

The reliability or quality of measurements made in the laboratory depends significantly on the integrity of the equipment and materials used. Practices must be followed that provide control over the integrity of items used. Measurements of poor quality will adversely affect programs by causing incorrect decisions, loss of material, rejected analyses, equipment failure, and operational errors. It is important to control the measurement processes so that reported results will be within required tolerances.

12.1 CALIBRATION OF EQUIPMENT

Calibration is the single most important operation in the measurement process. Equipment items that are used generally throughout the laboratory are controlled through a calibration program. Calibration of analytical balances is performed by the plant instrument shop. The GC/MS instruments are calibrated, tuned, and operated in accordance with the CLP criteria which also meets the criteria specified in other EPA documents and the Federal Register. The ICP, AA, GC, and similar instruments are calibrated and operated in accordance with the requirement of the procedure being employed. The record of calibrations for each analysis run is maintained as a permanent record. The calibration of all equipment must be performed or verified with each analysis run.

Equipment calibration is covered in the following:

K-25 Standard Practice Procedure 344	K-25 Calibration/Certification Program
Procedure 2308	Calibration of Laboratory Equipment

Equipment not covered under instrument calibration and maintenance consists of refrigerated storage units for samples. These are continuously monitored and a hard copy of the temperature reading made. Copies consist either of graph or printed paper tapes and are identified by units.

12.2 CALIBRATION OF GLASSWARE

Calibration is the single most important operation in the measurement process. Volumetric glassware is used to prepare calibration standards, bench standards, samples for analysis, etc., thus the glassware used for these preparations must be of known accuracy.

The ACD does not calibrate volumetric glassware. The glassware is purchased with known accuracy per federal and ASTM specifications.

Volumetric glassware is manufactured and sold per the following specifications:

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Class A - with permanent numbers which are traceable to the original calibration by the manufacturer.

Class A - without numbers.

Class B - the tolerance is twice the allowable Class A tolerance.

The allowable tolerance is stated in the appropriate specification.

References:

ASTM E 969	Standard Specification for Volumetric (Transfer) Pipets
NNN-P-395	Federal Specification Volumetric Pipets
ASTM E 288	Standard Specification for Volumetric Flask
NNN-F-288	Federal Specification Volumetric Flask
ASTM E 287	Standard Specification for Burets
NNN-B-789	Federal Specification for Burets

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12.3 SAMPLE CONTAINERS AND GLASSWARE

All sampling bottles used for groundwater monitoring are purchased from I-Chem and are precleaned. The containers, septa, liners, and closures are washed in laboratory-grade nonphosphate detergent. This is followed by a three-times rinse with tap water. The next step is a three-times rinse with American Society for Testing Materials (ASTM) Type I deionized water. The containers, septa or liners, and closures are then oven dried. After they are removed from the oven, the liners are placed in the closures, Teflon side down, and placed on containers. Attendants wear gloves during this process, and the containers are not removed from the preparation room until they are sealed.

All sample containers for NPDES sampling are purchased precleaned from the manufacturer. Currently, the 200 series I-Chem bottles are used. Bottles for biological sampling are sterilized according to *Standard Methods for the Examination of Water and Waste Water* (1985), Method 904.

Glassware used in the laboratory for sample preparation and analysis are cleaned according to best laboratory practices. Cleaning procedures are posted in each laboratory at the glassware cleaning stations.

12.4 PREVENTIVE MAINTENANCE

The most important benefit of a good preventive maintenance program is to increase measurement system availability (proportion of up time) and thus increase data completeness. In addition, the quality of the data should improve as a result of good equipment operation.

Periodic preventive maintenance is required for all sensitive equipment. All major instruments have maintenance contracts with the manufacturer, which cover all repairs and service. Manufacturer's recommended maintenance criteria are followed. Minor items of equipment and the computers are serviced by in-house instrument personnel. Records of service, repairs, etc., are recorded in the maintenance logbook. Records are covered in the procedure:

Procedure 2323	Laboratory Notebooks, Instrument Logbooks, Miscellaneous Logbooks

12.5 REAGENTS AND STANDARDS

Documentation is provided for the standards used for instrument calibration, internal controls, bench standards, spike solution, and for traceability of the analysis of samples to specific standards used. The methods used are covered in the following procedures.

Procedure 2320	Records of Standards
Procedure 2325	Quality Control of Reagent Water for Laboratory Use

12.6 DECONTAMINATION OF FIELD EQUIPMENT

The acceptable decontamination procedure for all equipment used in the field is detailed in the following document:

K/QT-262	NPDES and Surface Water Sampling Procedure

Documentation for all cleaning is recorded. The preferred location for all decontamination of equipment is in the laboratory or in a controlled clean environment. Field decontamination is avoided when possible by having sufficient numbers of sampling devices for the sampling activity.

All nonsampling equipment is decontaminated using high-pressure steam or scrubbing with a laboratory grade detergent and rinsing with reagent water. After decontamination, all equipment is covered with plastic sheeting or aluminum foil.

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13. HANDLING, STORAGE, AND SHIPPING

13.1 SAMPLING PROCEDURES

There are specific written procedures for use by the sampling personnel which are based on EPA or Federal Code of Regulations for the routine sampling of monitoring wells and surface water. These are covered in the following:

Procedure 2025	NPDES and Surface Water Sampling
Procedure 2023A	Well Water Sampling
Procedure 2333	Qualification Criteria for Field Sampling Personnel
K/QT-167	Groundwater Sampling and Analysis Plan
K/QT-168	QA/QC Documentation for Groundwater Monitoring
K/QT-191	QA Plan for NPDES Monitoring
K/QT-262	NPDES and Surface Water Sampling Procedure
K/QT-324	QA Plan for Drinking Water Sampling and Analysis

For specialized and nonroutine sampling, work plans and sampling guidance are provided by a project manager. EPA documents are utilized in the development of these plans. These are also covered in the following:

Procedure 2024	Sump Sampling
Procedure 2026	Tank and Drum Sampling
Procedure 2021	Ambient Air Sampling for Uranium, Specific Metals, and Particulates

13.2 SAMPLE HANDLING, STORAGE, AND SHIPMENT

Shipment is mainly related to samples shipped from the site to other laboratories. Preservation is in accordance with EPA protocol and all handling and storage is in accordance with the K-25 Plant written procedure and policies. The main goal is to ensure the integrity of the samples and ensure that all DOT regulations are followed on shipments leaving the K-25 site. Shipment of samples within the plant are per EPA requirements. These shipments may also be subject to on-site transportation codes. This includes refrigerated coolers.

Samples should be handled and stored in the laboratory in ways that do not adversely affect their composition, which involves preventing contamination from impurities and/or a change in concentration.

Anticipation of reanalysis prescribes proper environmental control. If reanalysis is not anticipated, environmental conditions are not observed, and the samples are stored at room temperature. Disposal of samples will be in accordance with state and federal regulations.

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The following procedures address sample and waste disposal:

Procedure 2054A	Disposal of Non-Uranium Solid Waste
Procedure 2054B	Disposal of Non-Uranium Liquid Waste and Samples
Procedure 2054C	Handling and Disposal of Nuclear Accountability Samples and Waste
Procedure 2054D	K-1004-A Dock and K-1004-E Operation
Procedure 2054E	Disposal of Vendor Labeled Chemicals
K-25 Standard Practice Procedure 346	Material/Equipment Transfer to other Sites
K-25 Standard Practice Procedure 338	Shipment and Off-Site Transportation of Materials and Equipment
K-25 Standard Practice Procedure 336	Management of Nuclear Materials
K-25 Standard Practice Procedure 337	Accounting for Nuclear Materials and Samples
K-25 Standard Practice Procedure 340	Hazardous Materials Management
K-25 Standard Practice Procedure 341	Waste Disposal Management

The procedures which cover sample handling, storage, and shipment are:

Procedure 2051	Archiving Designated Samples
Procedure 2053	Sampling Handling
Procedure 2053B	Radiological Surveying of Received Samples
Procedure 3053C	Sample Transportation
Procedure 2055	Chain of Custody
K-25 Standard Practice Procedure 758	Handling, Shipping, and Storage

13.3 SAMPLE HOLDING TIMES

When samples are taken by the Sample Management group, they are sent to the laboratory for analysis within 24 hr after collection in order to ensure that the most reliable and accurate answers will be obtained as a result of the analysis. The holding time begins from the date of collection in the field. Preservatives are added in the field. Holding times are based on the following protocols: EPA Methods for Chemical Analysis of Water and Wastes, Federal Register, SW-846 3rd ed., and CLP.

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14. INSPECTION, TEST, AND OPERATING STATUS

Data from both the internal and external QC programs are used to determine the performance of the laboratory. The internal QC program provides information for immediate evaluation of the data by the supervisor and permits immediate corrective action. Corrective action on computer-flagged nonconformances requires documentation by the originator.

Data from external controls provide the official performance of the laboratory. Any nonconforming data is investigated and used to take corrective actions where needed.

Technical evaluation of deficiencies in performance consists of two components. The cause of deficiencies is determined and actions are identified for correcting deficiencies, including actions that will minimize recurrence. Responsibilities for action are identified, and assigned actions recorded and reported as appropriate. Emphasis is placed upon actions to be taken that will minimize the probability for subsequent failures or deficiencies.

Measurement control programs have been established to provide for the initiation and maintenance used to estimate the measurement system quality of the laboratory. These programs are designed to provide documented data showing that the operation of the individual laboratory groups and the sample analysis results obtained are in conformance with the QA/QC requirements of EPA, OSHA, State of Tennessee, and customer requirements. Control programs are covered in two procedures. These are:

Procedure 2301	Description of Control Programs for the Analytical Chemistry Department
Procedure 2303	Analytical Quality Control Implementation

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15. CONTROL OF NONCONFORMING ITEMS

Nonconforming equipment, items, activities, conditions, procedures, and unusual incidents that could affect compliance with program requirements will be identified, controlled, and reported in a timely manner. A nonconformance is defined as a malfunction, failure, deficiency, or deviation which renders the quality of an analytical result unacceptable or indeterminate. Each nonconformance will be reviewed and disposition given for the item, activity, or condition. The disposition of a nonconformance will be documented and approved by the section responsible for the issuance of the nonconformance. The Methodology and QA Manager will concur with the disposition of the nonconformance.

The modification, repair, rework, or replacement of nonconforming equipment, items, or activities will require the reverification of acceptability. In certain instances, these actions may require that corrective action be completed and verified before work continues. The equipment, item, or activity which has the deficiency may be temporarily stopped while the nonconformance is being investigated. If the nonconformance does not significantly affect the technical quality or use of the work, the work may continue pending resolution of the nonconformance. The basis for such decision will be documented and submitted to the Methodology and QA Manager for review and approval. The documentation will include the statement that the decision was made prior to continuing with the work. The records of nonconformance and their dispositions will be filed. This is covered in the following procedure:

Procedure 2314 Documentation of "Out-of-Control" Conditions

Nonconformance due to laboratory contamination is covered in the following procedure:

Procedure 2330 Action Plan for Laboratory Contamination

When applicable, nonconforming items will be controlled by the following procedure.

K-25 Standard Practice Procedure 767 Control of Nonconforming Items

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16. CORRECTIVE ACTIONS

16.1 TYPES OF CORRECTIVE ACTIONS

Corrective action is necessary when any measurement system fails to follow this QA plan. In general, items needing corrective action fall into three categories: (1) short-term, (2) long-term, and (3) QC. Each category requires a different action.

Short-term corrective action consists of minor and major problems which can be corrected immediately. Examples may include failure to date or sign a form, incorrectly preserving a sample, and errors in data entry. Long-term corrective action consists of minor and major problems which require a series of actions to resolve the problem. QC corrective action consists of corrective action following a failure to meet QC criteria specified in this QA plan and the analytical methods. Corrective actions may also follow the standard procedure for controlling nonconforming items.

K-25 Standard Practice Procedure 767	Control of Nonconforming Items

16.2 PROCEDURES

The data obtained from QC sample and internal controls are used to evaluate the analytical process and take necessary action to keep the system in control. The Methodology and QA Manager is responsible for assessment of quality control sample information.

There are three procedures which describe the external QC sample, internal QA criteria, actions to be taken, and documentation required. The procedures are:

Procedure 2301	Description of Control Program for the Analytical Chemistry Laboratory
Procedure 2309	Data Validation
Procedure 2314	Documentation of Out-of-Control Incidents

Completion of corrective action should be evident by data returning to prescribed acceptable limits.

16.3 RECURRING QUALITY PROBLEMS

There may be recurring quality problems where corrective actions have not been effective. In these cases the following actions will be initiated:

(1) Determine the events leading to the quality problems occurrence

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(2) Develop an understanding of the technical and work activities associated with the quality problem

(3) Ascertain the quality problem's generic implications

(4) Determine the extent to which similar quality problems, or precursors to the problem, have been recognized by the responsible organization, the effectiveness of any corrective actions that were taken, and recognition of any generic implications and impacts on completed work

(5) Consider stopping work associated with the applicable activity

(6) Recommend remedial actions that can be taken by the responsible organization to preclude recurrence.

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17. QUALITY ASSURANCE RECORDS

17.1 SAMPLES

The records for each batch of samples processed or analyzed are maintained as permanent records. This includes the hard copy data from batch analysis, standardization, calibrations, and associated data which are not entered into the computer. These data may be in the form of logbooks or work sheets. All loose-sheet data are collected and assembled into a folder for file storage purposes. Records are covered in the following:

Procedure 2309	Data Validation
Procedure 2323	Laboratory Notebooks

All data entered into the computer are backed up each day and the tapes stored in accordance with the DOE policy for maintaining a back up for all computer generated data each and every day. All storage of data is in accordance with the DOE and Plant written policies.

17.2 QUALITY CONTROL

To provide a complete record, all QC problems and corrective actions applied must be documented. Historical records assist in identifying long-term problems and enabling management to apply long-term corrective actions such as personnel training, replacement of instrumentation, improvement of sampling procedures, etc. The following procedure is used for QC tracking and recording:

Procedure 2302	Data Monitoring and Control Chart Plotting

17.3 QUALITY ASSURANCE REPORTS

Copies of the laboratory performance on the external QC samples are received by the Methodology and QA Manager. If the reports show nonconforming data for the laboratory, the raw data files are reviewed to determine the reason for the unacceptable results.

A written response is made by the Methodology and QA Manager to the originator of EPA or EPA sponsored control programs. This response documents the reason for the nonconforming laboratory data and the actions to be taken to prevent future failures.

Copies of all laboratory performance data are received by the Methodology and QA Manager and submitted to management with comments where appropriate. All the external QC data are evaluated, condensed, and included as part of the yearly Environmental Report of the Oak Ridge Operations. This report is provided to all levels of upper management and to the state and federal agencies. The reports become a part of the local public library and are given to local news media.

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18. AUDITS

The role of audits in the overall management program is verification. While audits do not improve data quality if all work is correctly performed, they do provide assurance that the work prescribed for the measurement program has been conducted properly.

Audits are a very important item in the QC program for the laboratory. They provide a documented means of detecting missing QC requirements and potential problem areas. Audits form one of the bases for corrective action requirements and constitute a permanent record of the conformance of measurement systems to QA requirements.

Items to be examined during an audit may include the availability and implementation of approved work procedures, calibration, operation of equipment, packaging, storage and shipping of samples, performance documentation, and nonconformance documentation.

The records of operations may be reviewed during an audit to verify that laboratory and field-related activities were performed in accordance with appropriate approved procedures. Items reviewed may include the calibration records of equipment, daily field activity logs, chain-of-custody documentation, and data resulting from field and laboratory operations.

There are three different sources of audits, evaluations, reviews, etc., performed on the laboratory. These are in-house audits, special program audits, and general audits.

18.1 SOURCES OF AUDITS

18.1.1 In-House Audits

In-house audits are performed by technical personnel with expertise in laboratory analysis and EPA requirements. An in-depth audit of both the sampling and analysis sections is scheduled to ensure that all phases are covered within a three-year period. Specific programs or operations may be audited more frequently.

18.1.2 Special Program Audits

Special program audits are associated with a specific activity and are performed by the State of Tennessee, NIOSH, Water Supply personnel, special DOE audits, customers, etc. A written record of the findings are made by the laboratory at the time the audit is performed. This record plus the planned corrective actions are sent to the auditors to ensure that there is complete understanding. The final report is also commented on and corrective actions listed.

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18.1.3 General Audits

General audits are made of the entire laboratory and performed by the central Martin Marietta Environmental staff, DOE personnel, or EPA personnel. The findings on these types of reports are sent for transmittal to DOE from plant management. An action plan is developed for items to be resolved.

18.2 TYPES OF AUDITS

18.2.1 Performance Audits

Performance audits are made to quantitatively evaluate the quality of data produced by the total measurement system (sample collection, sample analysis, and data processing). The individuals performing the audit, their standards, and equipment are different from the regular team (operating the measurement system) and their standards and equipment in order to obtain an independent assessment.

Performance audits provide objective assessments of the accuracy of the data collected by a given measurement system. They also provide identification of out-of-control measurements and of systematic bias. Measurement of improvement in data quality can be based on data from previous and current audits.

18.2.2 System Audits

System audits are qualitative on-site inspections and reviews of the total measurement system. The auditor should have extensive background experience with the measurement system being audited.

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