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Quality Assurance Program Plan

Environmental Sampling and Nonradioactive/Radioactive Analysis

by

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APPENDIX A - Radioactive Material License

1. IDENTIFICATION OF SUBMITTAL LABORATORY
(Effectivity and Approval)

Document Title:	Quality Assurance Project Plan for Environmental Sampling and Nonradioactive/Radioactive Analysis Laboratory in San Antonio
Organization Title:	Southwest Research Institute Chemistry and Chemical Engineering Division Department of Environmental Chemistry
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Plan Coverage:	Division 01 Department of Environmental Chemistry Laboratory in San Antonio

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Approval for Division

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Title	Vice President, Chemistry and Chemical Engineering Division
Signature	Donald E Johnson
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Title	SwRI Quality Assurance Manager
Signature	Thomas C. Trbovich

Date: 11/30/93

INTRODUCTION

REV 9

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Approvals:

Division 01 Quality Assurance		Date: 11/30/93
Division 01 Management		Date: 11/30/93
SwRI QA Management		Date: 12/1/93

2.0 INTRODUCTION

2.1 Commitment to Quality Assurance (QA)

The Southwest Research Institute (SwRI) Division 01 Environmental Laboratory shall devote the necessary personnel and equipment to fulfill the quality assurance/quality control (QA/QC) requirements of all projects. The SwRI Environmental Laboratory has implemented the QA/QC program described in this document in all laboratories on the main campus in San Antonio to assure generation of monitoring and analytical measurement data of known acceptable quality to meet the requirements of each project's statement of work.

2.2 Purpose and Scope of Quality Assurance Project Plan (QAPP)

The purpose of this QAPP is to convey the philosophy, organizational structure and procedures governing the QA/QC program for environmental sampling and analysis services performed by the Environmental Laboratory in San Antonio. This plan has been prepared in accordance with "Guidelines and Specifications for Preparing Quality Assurance Program Plans, "QAMS-004/80, September 20, 1980", " QAMS-005/80, December 29, 1980; "Quality Assurance", Office of Nuclear Energy, DOE 5700.6C, August 21, 1991; ASME/NQA-1, "Quality Assurance Program Reguirements for Nuclear Facilities. The Quality Assurance Department at Southwest Research Institute is an independent organizational element that reports directly to the President of SwRI. The QA support for projects has been tailored to meet the quality requirements imposed by organizations including the Department of Energy (DOE), the Department of Defense (DOD), the Nuclear Regulatory Commission (NRC), the Environmental Protection Agency (EPA), and many industrial firms. These quality programs have been designed to satisfy applicable requirements of quality documents such as MIL-Q-9858A, MIL-I-45208A, MIL-STD-45662A, and DOE Order 5700.6C, Federal Acquisition Regulations (FAR) paragraphs 52.246-1 through 52.246-8, the Food and Drug Administration (FDA) Good Laboratory Practices and Good Manufacturing Practices, and the Environmental Protection Agency (EPA) Good Laboratory Practices.

SwRI's Nuclear Quality Assurance Program Manual (NQAPM) describes an effective and economical Quality Assurance (QA) Program that complies with the requirements of 10 CFR 50, Appendix B, ASME NQA-1, and DOE 5700.6C. The procedures within this manual establish the basic quality requirements for activities such as Engineering, Design, Fabrication, Inspection, and Testing performed by applicable division personnel.

2.3 Quality Operating Plans

The written quality plan for each contract will be separate from the QAPP but will ensure procedures within this QAPP for documentation and reporting requirements are satisfied. For nuclear requirements immediately after contract award, a Project Quality Plan will be prepared that will specifically invoke sections of the Institute NQAPM and applicable Division Operating Procedures that will be used to conduct the quality activity for this project.

All environmental sampling and analytical services performed within the laboratory in San Antonio, will follow a QA workplan to assure compliance to the clients' requirements and validity of the data. This QA workplan will be established prior to the initiation of the project by the Project Manager, Analytical Supervisor, and Division 01 QAU. The client will be notified of any modifications to the QA workplan during the project. Smaller projects requiring minimal QA/QC effort will be established upon initiation of the project and submission in writing from the Project Manager that the client requests to minimize QC.

In the event that no QA workplan is specified by the client, all environmental sampling and analytical services preformed within the laboratory at San Antonio shall be conducted in accordance with the provisions of this QAPP.

QUALITY ASSURANCE POLICY

REV 7

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Approvals:

Division 01 Quality Assurance		Date: 11/30/93
Division 01 Management		Date: 11/30/93
SwRI QA Management		Date: 12/1/93

3.0 QUALITY ASSURANCE POLICY

The following paragraphs are quoted from Southwest Research Institute Operating Policies and Procedures 10.1.1 and present the quality assurance policy of the Institute.

> *It is the policy of Southwest Research Institute (SwRI) to ensure that the equipment and items supplied to, or services performed for, clients shall conform to the specified requirements of the applicable codes, regulations, standards, specifications, and contracts.*
>
> *The responsibility for developing, conducting, and auditing the Quality Assurance (QA) Program to ensure the implementation and enforcement at SwRI of the policy stated above, is assigned to the Quality Assurance Department. In recognition of the importance of this policy, the Director of the Quality Assurance Department will keep the Office of the President of SwRI informed on QA program performance and recommend action as necessary to ensure the required quality assurance.*
>
> *Although quality assurance programs are administered by the QA Department with oversight provided by the Office of the President of SwRI, quality assurance is a responsibility of all SwRI employees performing project activities.*
>
> *Each Division Vice President (DVP) is responsible for (1) ensuring that for each project, the need for a formal QA program is identified and (2) if a QA program is required, ensuring that the Quality Assurance Department is made aware of that requirement. In the case of programs with no formal QA requirements, the DVP or his delegate will determine the extent and application of any QA program and will request QA support as appropriate from the Manager of the QA Department. In either case, the Director of the QA Department will ensure that the QA requirements and documentation necessary to implement those requirements are identified. The Manager of the QA Department is responsible for ensuring that those activities to be performed by QA personnel are documented. The DVP is responsible for ensuring that those activities to be performed by project personnel are documented. QA personnel shall review and approve all procedures governing quality-related activities and will maintain a record copy of each procedure*

It is the policy of the Chemistry and Chemical Engineering Division (C&CED) to implement a quality assurance program in all environmental projects to ensure that all measurement data generated shall be scientifically sound, of known quality, and adequately documented.

QUALITY ASSURANCE MANAGEMENT

REV 8

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Approvals:

Division 01 Quality Assurance	[signature]	Date:	11/30/93
Division 01 Management	[signature]	Date:	11/30/93
SwRI QA Management	S.C. Silverio	Date:	12/1/93

4.0 QUALITY ASSURANCE MANAGEMENT

4.1 *SwRI Quality Assurance Management Structure*

The Director of Institute Quality Assurance has been given the responsibility of operating quality assurance programs for the Institute. The President of SwRI has given the Director of QA the authority and organizational freedom to ensure that quality activities shall be accomplished. The organization chart of the Quality Assurance Department is shown in Figure 4.1.

The Institute Quality Assurance Manager is responsible for the activities of the QA personnel and for providing direction to his staff. The QA Manager is responsible for helping to evaluate and assisting in the resolution of deficiencies and quality problems.

The Assistant Manager of Institute Quality Assurance plans, coordinates and directs the efforts of QA Engineers and Technicians to ensure that quality assurance program activities satisfy contractual quality requirements.

The Quality Assurance Engineer (QAE) is responsible for ensuring that contract quality activities for projects to which he/she is assigned shall be properly implemented or reported as deficiencies. A QAE will be assigned to implement the requirements of this QAPP.

The Quality Assurance Technician (QAT) inspects, surveys, monitors, and audits products and services for compliance with contractual requirements. The QAT assigned will perform these functions under the guidance of the responsible QAE.

This plan defines the QA/QC program for all sampling, analytical services, and environmental measurements performed in the San Antonio laboratory (i.e., Environmental Engineering, Environmental Chemistry, Fire Technology, Applied Chemistry, and Chemical Demilitarization sections) within the C&CED Environmental Monitoring and Analysis,. The organization charts of the C&CED and the analysis and sampling group are presented in Figures 4.2, 4.3, and 4.4, respectively.

4.2 *Assignment of Quality Responsibilities within the Chemistry and Chemical Engineering Division*

4.2.1 *Organization Independence of Quality Function*

The manager of SwRI Quality Assurance maintains responsibility to oversee the quality assurance within the C&CED. He then delegates the coordination of quality activities within the division to a quality officer. This designee will supervise all quality control (QC) activities, and submit QA program assessment reports (see Section 4.5) to the manager of Quality Assurance for review. Thus, the Institute Quality Assurance Department will provide independent overview of the QA/QC effort. In addition, as shown in Figure 4.3, the quality officer reports directly to the director(s) of the division on quality matters. Giving the quality officer the necessary independence to supervise quality activities within each project. The Institute Quality Assurance Department will conduct system audits and surveillances as necessary to confirm conformance to requirements or to investigate chronic deficiencies in the QA/QC program.

4.2.2 *Responsibilities in Large Projects with a Quality Assurance Project Plan*

4.2.2.1 Typical Positions

Large projects for which a quality assurance project plan is prepared will typically have the following positions:

Project Manager
Study Director
Analytical Supervisor
Analyst
Sampling Coordinator (if applicable)
Quality Assurance/Quality Control Officer (Institute QAE)
Quality Assurance/Quality Control Officer (Division)
Quality Assurance Auditor

Figure 4.1 Department of Quality Assurance and Administration

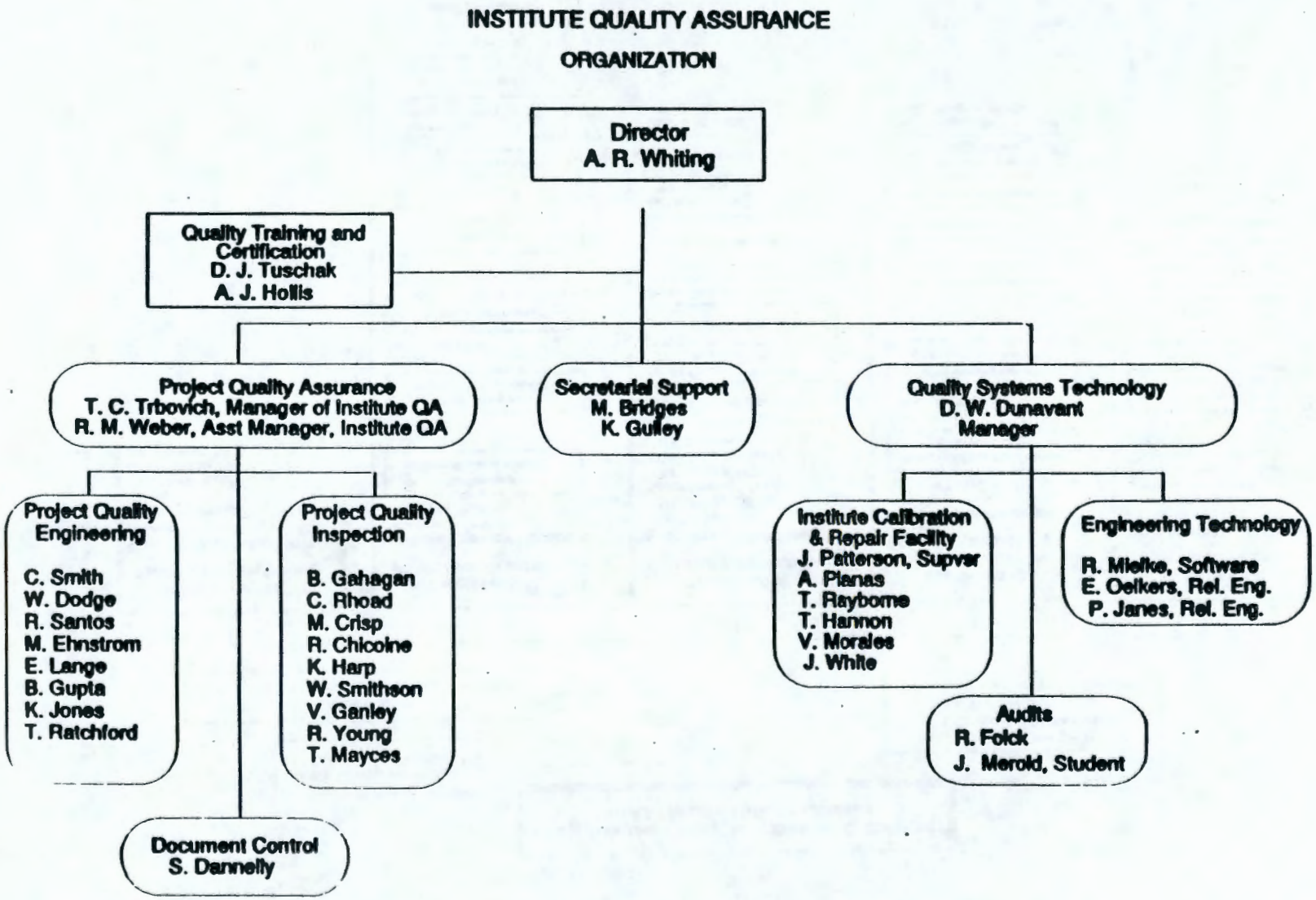

Figure 4.2 Chemistry and Chemical Engineering Division

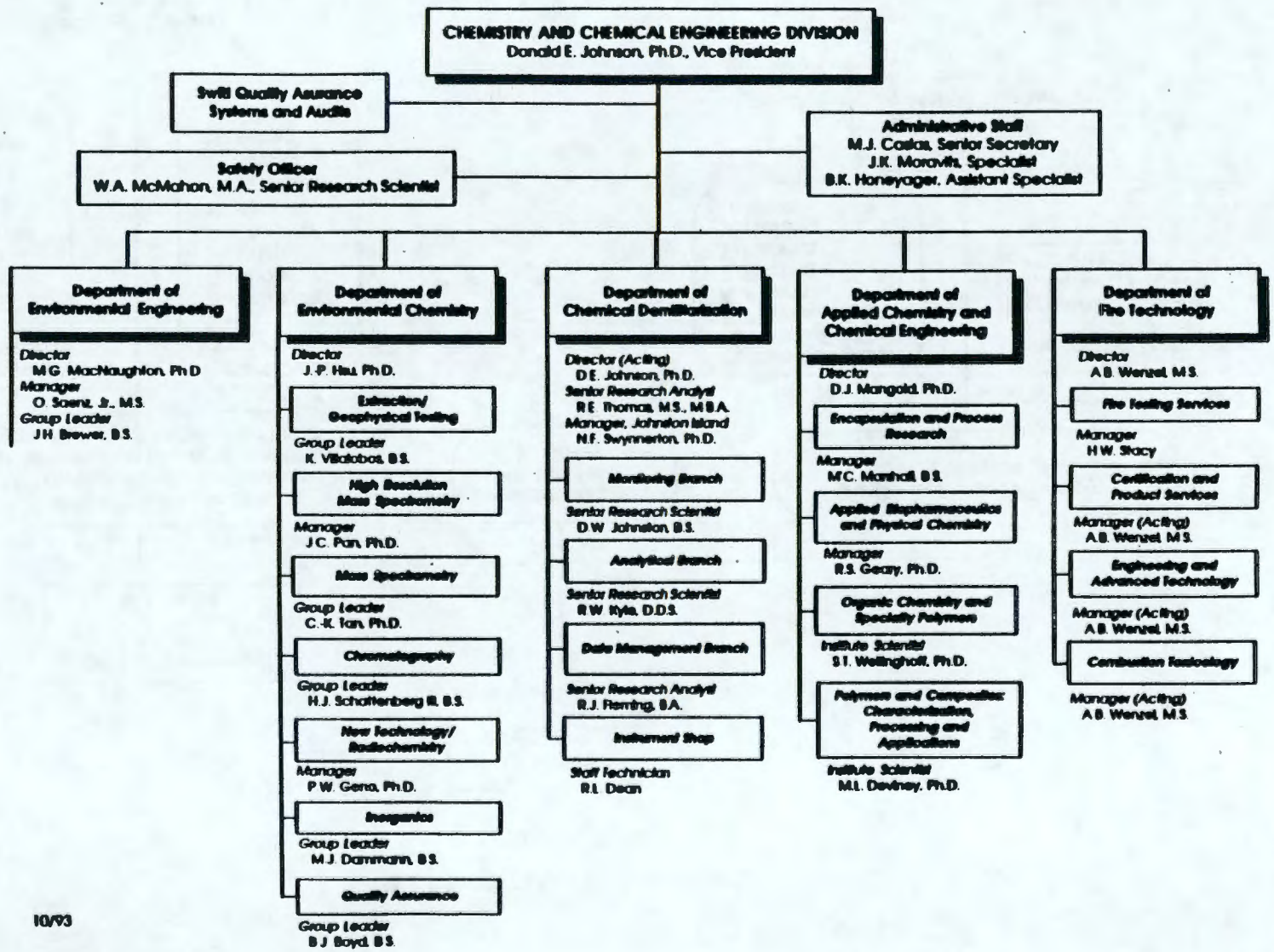

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Figure 4.3 Department of Environmental Chemistry

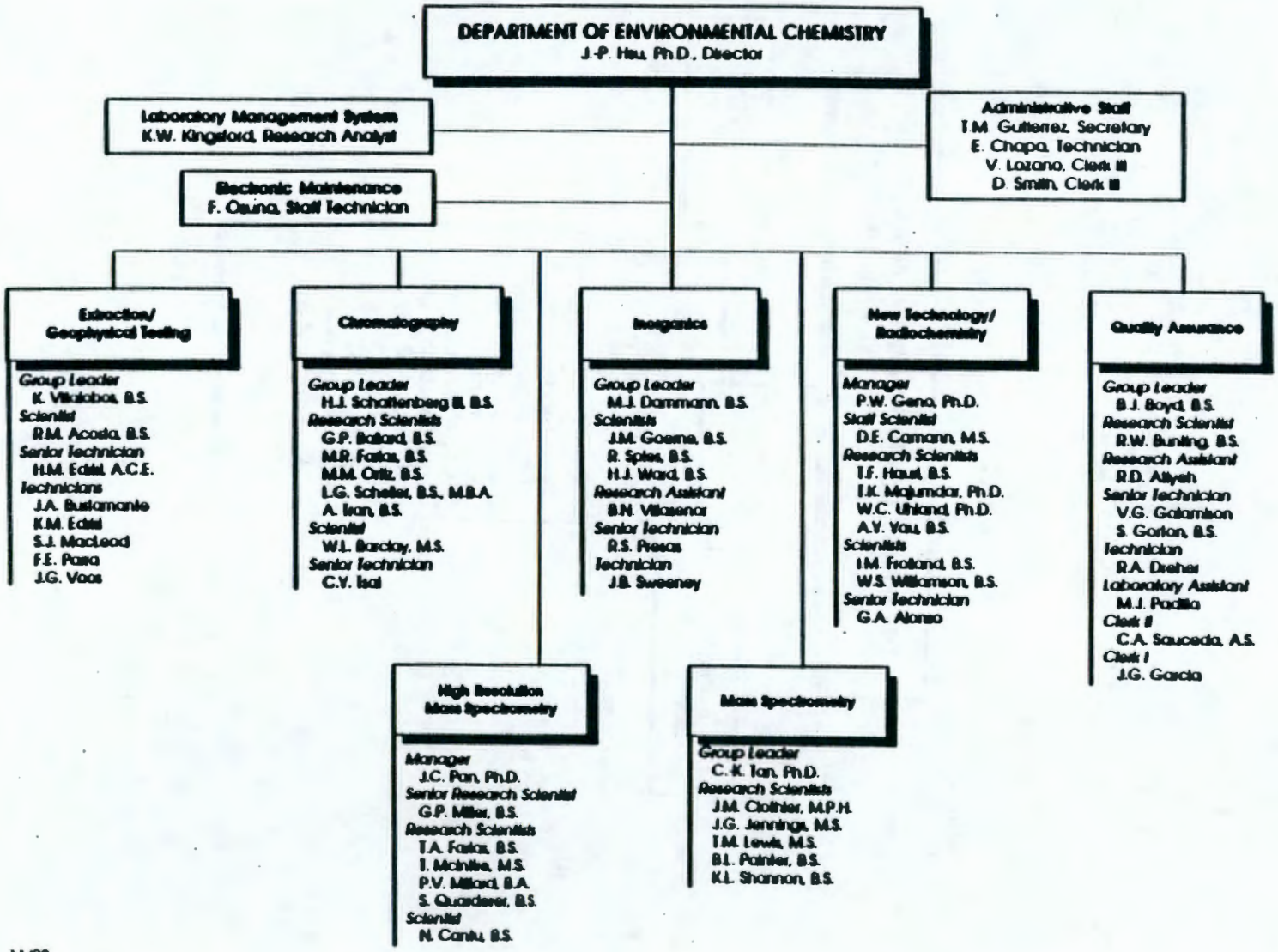
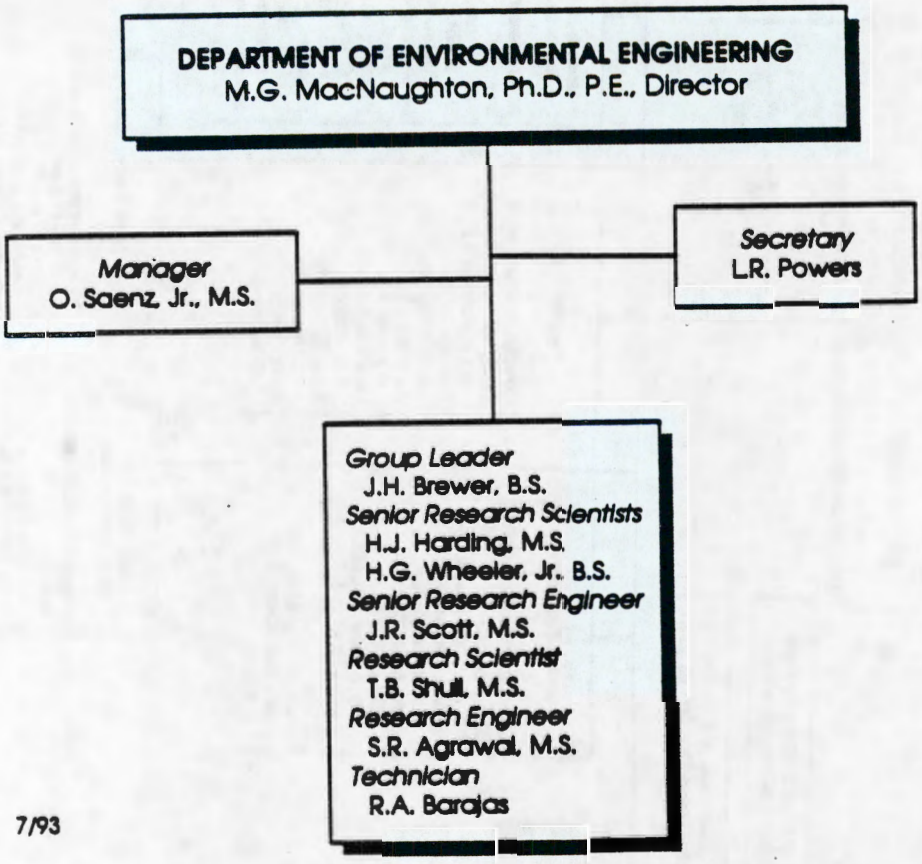


Figure 4.4 Department of Environmental Engineering



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The typical duties of these positions are given below:

Project Manager

- design the work plan/technical proposal
- general technical supervision of project personnel
- financial management
- sponsor communications
- resolve major problems and issues

Study Director

- manages projects requiring GLP regulations
- design the work plan/technical proposal
- general technical supervision of project personnel
- financial management
- sponsor communications
- resolve major problems and issues

Analytical Supervisor

- assist in developing the analytical plan/technical proposal
- assist in preparing the Quality Assurance Project Plan for analytical activities
- develop necessary analytical protocols and analytical standard operating procedures, including control charts
- review analytical results for questionable data and initiate necessary repeat analyses
- report problems to the Project Manager and the QA/QC Officer.

Analyst

- perform analyses
- calculate analytical results
- calculate quality control results
- maintain control charts
- report problems to the Analytical Supervisor for resolution

Sampling Coordinator

- develop necessary sampling protocols, standard operating procedures, and equipment requirements related to sample collection
- prepare the Quality Assurance Project Plan for sampling activities
- train and supervise field sampling personnel
- maintain and calibrate field sampling equipment
- arrange for transport of equipment and supplies to field locations
- determine logistic and custody requirements for sample transport
- document all activities associated with sample collection
- report problems to the Project Manager and the QA/QC Officer

Quality Assurance Officer (Institute QAE)

- schedules internal system audits and performance audits
- report results of quality activities to project line and quality assurance management
- assist in problem resolution

Quality Assurance/Quality Control Officer (Division)

- assist in preparing the Quality Assurance Project Plan
- schedules internal system audits, blind spiked samples, and performance audits
- report results of quality activities to project line and quality assurance management
- establish and set up procedures for log in, documentation of samples, and data deliverable of analytical results
- coordinate sample shipments from the field to the lab
- establish QC requirement, holding times, and deliverable required by the client and communicate to the analyst through a work order
- report analytical and QC data to the sponsor
- report results of Division QA/QC activities to Institute QA/QC Officer and Division 01 Management

Quality Assurance Auditor (Division)

- periodic review of control charts
- check instrument conditions and calibrations
- report results of quality activities to Division QA/QC Officer
- conducts internal inspections/audits
- check holding times of sample extraction and analysis and track status of analytical results
- spot check QC on analytical results and report any problems to the QA/QC Officer
- provide finished data package to QA/QC Officer and Project Manager for review

All assigned project personnel shall remain diligent to identify, report, and promptly rectify problems affecting data quality as they occur. Unresolved problems must be reported to a member of the quality team.

4.2.2.2 Quality and Project Organizations

A generic organization chart for large projects which have a quality assurance project plan is presented in Figure 4.5. This chart indicates the typical reporting relationships among project management, the quality team, and senior C&CED management.

The quality team consists of the QA/QC Officer, and the QA Auditor. The QC auditor shall perform system audits, prepare the blind spiked samples and summarize their results, and monitor control charts.

4.2.3 *Responsibilities in Other Laboratory Projects*

4.2.3.1 Typical Positions

The functions of the QA/QC Officer and QA Auditor remain as specified in 4.2.2.1. However, the QA/QC scope now encompasses all instrumentation and methods employed. Any projects that require deviation from the QAPP shall have such deviations documented in memorandum form with the work order.

4.2.3.2 Quality and Lab Organizations

A generic organization chart showing the Chemistry and Chemical Engineering Department labs in San Antonio and their relationship to the quality team is presented in Figure 4.5. The QA/QC Officer shall supervise the quality team. The QA Auditor shall conduct all audit activities as scheduled and submit audit reports to the Division QA/QC Officer. He/she shall then interact with the section managers and report to senior management through the SwRI QA Officer regarding quality issues. The QA Auditor shall coordinate internal system audits, prepare needed blind spiked samples and summarize their results, and monitor control charts. The same QA Auditor will perform this function in Division 01 environmental laboratories in San Antonio.

4.3 Communication

The organization charts presented in Figures 4.5 and 4.6 indicate the internal and external flow of QA information and reports. Figure 4.5 illustrates typical QA information flow for projects with a QA project plan. Figure 4.6 shows the QA information flow for projects lacking a QA project plan. Reporting responsibilities are discussed in Sections 4.2 and 4.4.

4.4 Document Control

The Division QA/QC Officer and QA Auditor will maintain files of QA program assessment reports, other QA reports to management, and other vital information, plans, and directives. The pagination of every section of the QAPP shall include the number and date of all revisions. The QA Auditor will maintain distribution lists of personnel who need to receive QA reports and information.

Figure 4.5 **Generic Organization Chart for Large Projects with a QA Project Plan Showing Relationships Among Project Management, Senior Management, and Quality Team**

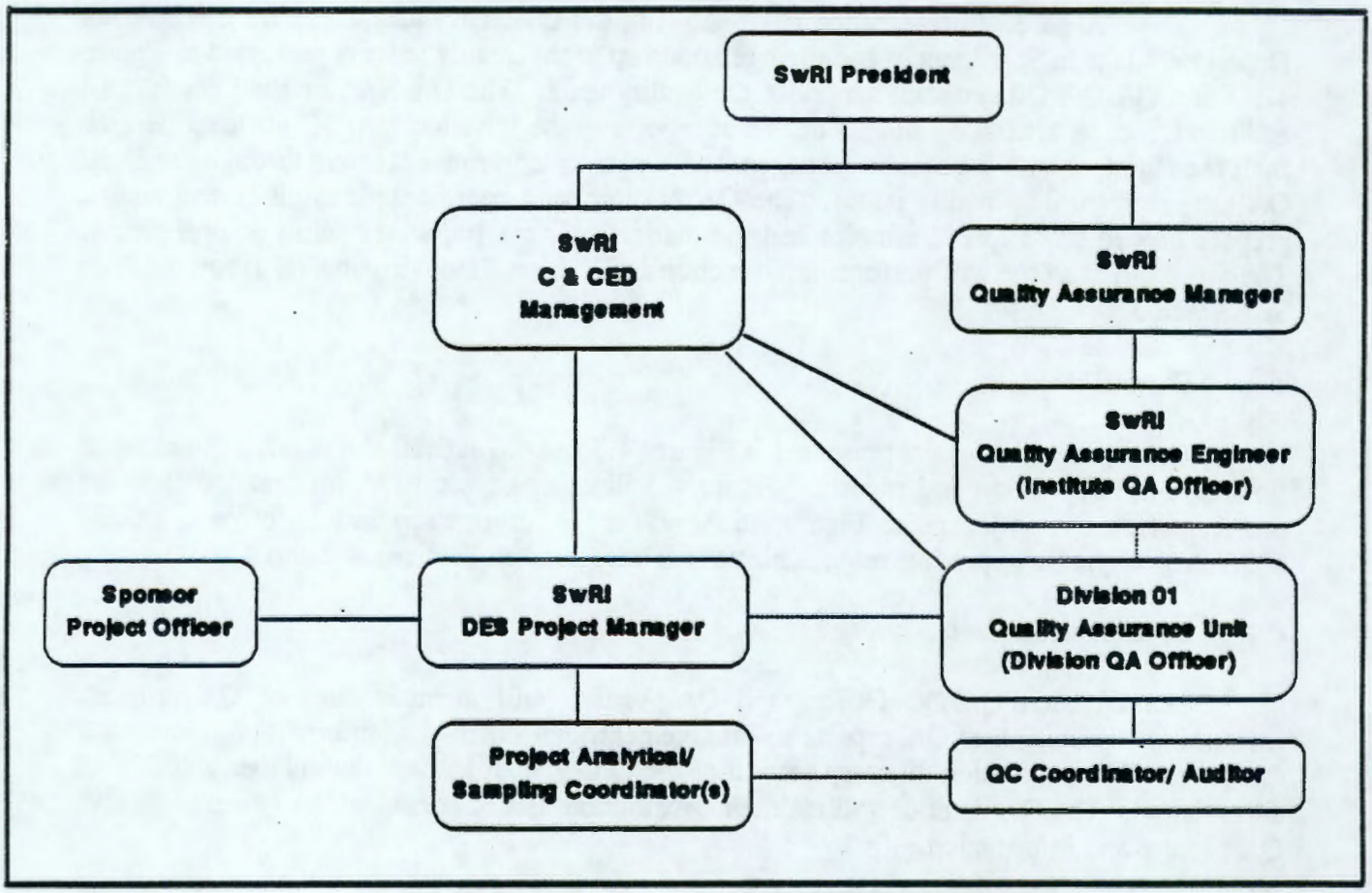
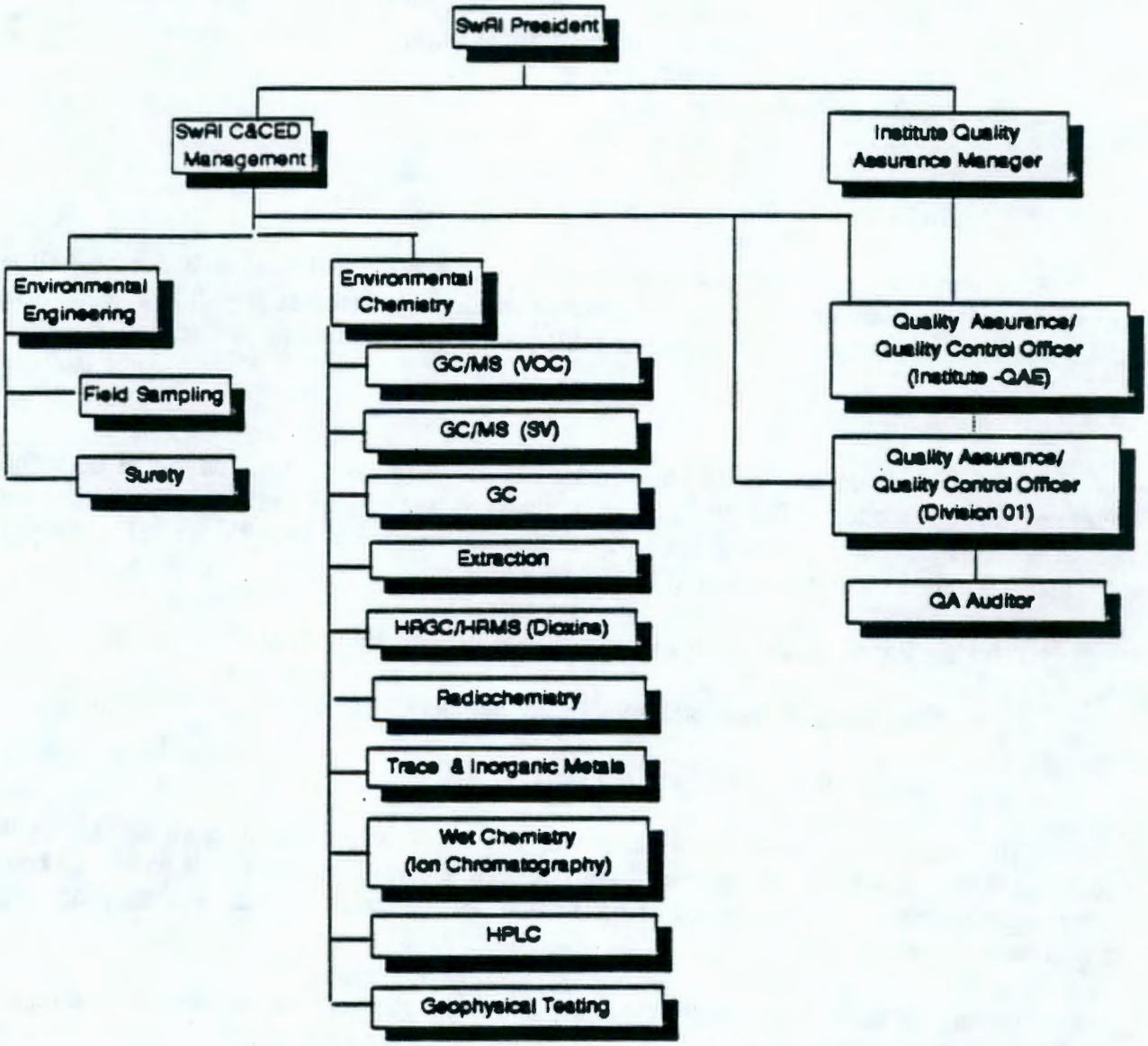


Figure 4.6 Generic Organization Chart for Laboratories in San Antonio Showing Relationships to Laboratory Supervisors, Management and Quality Team



4.5 QA Program Assessment

The QA/QC Officer shall conduct reviews of the total QA/QC program to evaluate its benefits and effectiveness. Based on these assessments, the QA/QC Officer shall prepare a written annual status report of QA activities and progress each year. This annual program assessment report will be submitted to the SwRI quality assurance manager, the director and the C&CED vice president. This assessment report will include such information as:

1. Status of or changes to the QA/QC Project Plan
2. Significant QA problems, accomplishments, and recommendations
3. Results of Performance Audits
4. Results of Systems Audits
5. Summary of QA Training
6. External Audit Findings and Corrective Actions
7. SwRI Internal QA Audit and Surveillance Activities

The Institute QAE will conduct nuclear surveillance on analytical activities as well as inspection of documentation of files and assure duplicate file archives for all raw data. The QAE will also conduct periodic reviews of GLP projects and routine project review.

4.6 Tracking

Work orders are sent to all analytical areas involved, as well as individual personnel assigned to each section. The work order is reviewed against the contract or solicitation to confirm the quality control requirements. Holding times are adhered to as shown in Figures 4.7, 4.8 and 4.9 unless otherwise stated in the contract.

Tracking Forms as shown in Figure 4.10 are used as follows on every sample:

1. The case/SDG and each sample, by number, will be listed on the form.

2. Actual holding times and deliverables will be listed.

The personnel assigned to the area will use the Tracking Form to check against the logbook each morning. If the samples have not been analyzed (or extracted) within 48 hours prior to holding time, the supervisor of the section will be notified. This will enable priority adjustment, if necessary.

During the tracking the proper methods, sample preparations, equipment calibration, and quality control requirements are checked. If the samples have been analyzed and the case and quality control has met requirements, i.e., surrogates, CRQL, MS/MSD and all reanalyses, a request will be made for data transfer to the Division QA/QC Officer.

The individual assigned to the section will notify quality control of any discrepancies and non-compliance items. A correction action is submitted. Follow-up review will be done to confirm the problem has been corrected and provide narrative response.

The sample control records will remain at the storage area until the appropriate sample disposal. At this time the samples are evaluated for each specific hazard to determine the method of disposal. All samples categorized as Hazardous Waste are disposed of through the Institute's Hazardous Waste Disposal System as described in Division 01 SOP-01-01-09 and in the Institute Safety Manual. Documentation of Hazardous Waste Disposal including Waste Data Sheet (Figures 4.12 and 4.13) and a Sample Disposal Log (Figure 4.11). The Sample Control Record is documented with the Sample Disposal data and is filed. These files are kept for a period of 5 years.

Figure 4.7 Required Containers, Preservation Techniques and Holding Time

Required Containers, Preservation Techniques and Holding Times (40 CFR, Part 136, July 1, 1990)			
Parameter No/Name	Container[1]	Preservation[2,3]	Maximum Holding Time[4]
Table 1A-Bacterial Tests			
1-4. Coliform, fecal and total	P, G	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	6 hours
5. Fecal streptococci	P, G	do	Do
Table 1B-Inorganic Tests			
1. Acidity	P, G	Cool, 4°C	14 days
2. Alkalinity	P, G	do	Do
4. Ammonia	P, G	Cool, 4°C, H_2SO_4 to pH <2	28 days
9. Biochemical oxygen demand	P, G	Cool, 4°C	48 hours
11. Bromide	P, G	None required	28 days
14. Biochemical oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours
15. Chemical oxygen demand	P, G	Cool, 4°C, H_2SO_4 to pH <2	28 days
16. Chloride	P, G	None required	Do
17. Chlorine, total residual	P, G	do	Analyze immediately
21. Color	P, G	Cool, 4°C	48 hours
23-24. Cyanide, total and amenable to chlorination	P, G	Cool, 4°C, NaOH to pH >12. 0.6 g ascorbic acid[6]	14 days[6]
25. Fluoride	P	None required	28 days
27. Hardness	P, G	HNO_3 to pH <2, H_2SO_4 to pH <2	6 months
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately
31, 43. Kjeldahl and organic nitrogen	P, G	Cool, 4°C, H_2SO_4 to pH <2	28 days
Metals[7]			
18. Chromium VI	P, G	Cool, 4°C	24 hours
35. Mercury	P, G	HNO_3 to pH <2	28 days
3, 5-8, 10, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75. Metals, except chromium VI and mercury	P, G	do	6 months
38. Nitrate	P, G	Cool, 4°C	48 hours
39. Nitrate-nitrite	P, G	Cool, 4°C, H_2SO_4 to pH <2	28 days
40. Nitrite	P, G	Cool, 4°C	48 hours
41. Oil and grease	G	Cool, 4°C, H_2SO_4 to pH <2	28 days
42. Organic carbon	P, G	Cool, 4°C, HCl or H_2SO_4 to pH <2	Do
44. Orthophosphate	P, G	Filter immediately, Cool, 4°C	48 hours
46. Oxygen, Dissolved Probe	G, Bottle and top	None required	Analyze immediately
47. Winkler	do	Fix on site and store in dark	8 hours
48. Phenols	G only	Cool, 4°C, H_2SO_4 to pH <2	28 days
49. Phosphorus (elemental)	G	Cool, 4°C	48 hours

Required Containers, Preservation Techniques and Holding Times (40 CFR, Part 136, July 1, 1990)			
Parameter No/Name	Container[1]	Preservation[2,3]	Maximum Holding Time[4]
50. Phosphorus, total	P, G	Cool, 4°C, H_2SO_4 to pH <2	28 days
53. Residue, total	P, G	Cool, 4°C	7 days
54. Residue, Filterable	P, G	do	7 days
55. Residue, Nonfilterable (TSS)	P, G	do	7 days
56. Residue, Settleable	P, G	do	48 hours
57. Residue, Volatile	P, G	do	7 days
61. Silica	P	do	28 days
64. Specific conductance	P, G	do	Do
65. Sulfate	P, G	do	Do
66. Sulfide	P, G	Cool, 4°C add zinc acetate plus sodium hydroxide to pH >9	7 days
67. Sulfite	P, G	None required	Analyze immediately
68. Surfactants	P, G	Cool, 4°C	48 hours
69. Temperature	P, G	None required	Analyze
73. Turbidity	P, G	Cool, 4°C	48 hours
Table 1C-Organic Tests[8]			
13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 56, 66, 88, 89, 92-95, 97. Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C, 0.008% $Na_2SO_3O_3$[5]	14 days
6, 57, 90. Purgeable aromatic hydrocarbons	do	Cool, 4°C, 0.008% $Na_2S_2O_3$[5], HCl to pH 2[9]	Do
3, 4. Acrolein and acrylonitrile	do	Cool, 4°C, 0.008% $Na_2S_2O_3$[5], adjust pH to 4-5[10]	Do
23, 30, 44, 49, 53, 67, 70, 71, 83, 85, 96. Phenols[11]	G, Teflon-lined cap	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction, 40 days after extraction
7, 38. Benzidines[11]	do	do	7 days until extraction[12]
14, 17, 48, 50-52. Phthalate esters[11]	do	Cool, 4°C	7 days until extraction; 40 days after extraction
72-74. Nitrosamines[11,14]	do	Cool, 4°C, store in dark, 0.008% $Na_2S_2O_3$[5]	Do
76-82. PCBs[11] acrylonitrile	do	Cool, 4°C	Do
54, 55, 65, 69. Nitroaromatics and isophorone[11]	do	Cool, 4°C, 0.008% $Na_2S_2O_3$[5] store in dark	Do
1, 2, 5, 8-12, 32, 33, 58, 59, 64, 68, 84, 86. Polynuclear aromatic hydrocarbons[11]	do	do	Do
15, 16, 21, 31, 75. Haloethers[11]	do	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	Do
29, 35-37, 60-63, 91. Chlorinated hydrocarbons[11]	do	Cool, 4°C	Do
87. TCDD[11]	do	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	Do
Table ID-Pesticides Tests:			
1-70. Pesticides[11]	do	Cool, 4°C, pH 5-9[15]	Do

Required Containers, Preservation Techniques and Holding Times (40 CFR, Part 136, July 1, 1990)			
Parameter No/Name	Container[1]	Preservation[2,3]	Maximum Holding Time[4]
Table IE-Radiological Tests			
1-5. Alpha, beta and radium	P, G	HNO_3 to pH <2	6 months

1. Polyethylene (P) or Glass (G).

2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

3. When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of this table, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See § 136.3(e) for details.

5. Should only be used in the presence of residual chlorine.

6. Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

7. Samples should be filtered immediately on site before adding preservative for dissolved metals.

8. Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

9. Sample receiving no pH adjustment must be analyzed within 7 days of sampling.

10. The pH adjustment is not required if acrolein will not be measured. Samples of acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

11. When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12 and 13 (re the analysis of benzidine).

12. If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.

13. Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

14. For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

15. The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

Figure 4.8 Preservative and Holding Times for the Contract Laboratory Protocol

Preservative and Holding Times for the Contract Laboratory Protocol				
			Holding Times	
Parameter	Container	Preservative	Soil	Water
Volatiles by gas chromatography /mass spectroscopy (GC/MS)	Water: 40-mL glass vial with Teflon-lined septa Soil-gas with Teflon-lined septa	Cool, 4°C	10 days	10 days
PCB/Pesticides	G, Teflon-lined lid	Cool, 4°C	Extract within 10 days, analyze 40 days	Extract within 5 days, analyze 40 days
Extractable organics	G, Teflon-lined lid	Cool, 4°C	Extract within 10 days, analyze 40 days	Extract within 5 days, analyze 40 days
Metals	P, G	HNO_3 to pH <2	6 months	6 months
Mercury	P, G	HNO_3 to pH <2	26 days	26 days
Cyanide	P, G	NaOH to pH >12, cool 4°C, add 0.6 g ascorbic acid if residual chlorine present	14 days	14 days
Chromium VI	P, G	HNO_3 to pH <2	24 h	24 h

Figure 4.9 Preservative and Holding Times for the EPA Document - SW-846, 3rd Edition

Preservative and Holding Times for the Contract Laboratory Protocol				
			Holding Times	
Parameter	Container	Preservative	Soil	Water
Volatiles by gas chromatography/ mass spectroscopy (GC/MS) and GC	Water: 40-mL glass vial with Teflon-lined septa Soil-gas with Teflon-lined septa	Cool, 4°C	14 days	14 days
PCB/Pesticides	G, Teflon-lined lid	Cool, 4°C	Extract within 7 days, analyze 40 days	Extract within 7 days, analyze 40 days
Extractable organics	G, Teflon-lined lid	Cool, 4°C	Extract within 7 days, analyze 40 days	Extract within 7 days, analyze 40 days
Metals	P, G	HNO_3 to pH <2	6 months	6 months
Mercury	P, G	HNO_3 to pH <2	28 days	28 days
Cyanide	P, G	NaOH to pH >12, cool 4°C, add 0.6 g ascorbic acid if residual chlorine present	14 days	14 days
Chromium VI	P, G	HNO_3 to pH <2	24 h	24 h

Project No.	Case	SDG	Client	Work Order

SOUTHWEST RESEARCH INSTITUTE

ORGANIC TRACKING SHEET

SAMPLE NO.	CED	VTSR	EXTRACTION DATE		ANALYSIS DATA												COMMENTS
					VOLATILES		SEMIVOLATILES		PESTICIDES								
			REQ	ACT	REQ	ACT	REQ	ACT	REQ	ACT	REQ	ACT	REQ	ACT	REQ	ACT	

Figure 4.10 Tracking Forms

Project No.	Case	SDG	Client	Work Order

SOUTHWEST RESEARCH INSTITUTE

INORGANIC TRACKING SHEET

SAMPLE NO	VTSR	ANALYSIS DATE																Comments
		WET CHEM		WET CHEM		METALS		METALS		INORGANIC		INORGANIC						
		Neg	Act	Neg	Act	Neg	Act	Neg	Act	Neg	Act	Neg	Act	Neg	Act	Neg	Act	

Figure 4.11 Hazardous Waste Log

Attachment # 1

Southwest Research Institute

HAZARDOUS WASTE LOG				
DATE	COMPOUND	QUANTITY (mL)	NAME	PROJECT NO.

Figure 4.12 Mixed Waste Data Sheet

Attachment # 2

Southwest Research Institute
MIXED WASTE DATA SHEET

Item No.	ID Number	Material Description	Material Quantity	Project No.
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				

I hereby certify that the above information is complete and accurate.

Printed Name _____ Signature _____ Date _____

Figure 4.13 Bulk Waste Data Sheet

Attachment # 3
Southwest Research Institute
BULK WASTE DATA SHEET

Container ID Number (Assigned at Central Storage) _____

Division Hazardous Waste Representative _____ Ext. _____

General Waste Description _____

_____

Quantity in Container(s) _____ kg. lb. cc. oz.

Chemical Constituents	Concentration
1.	
2.	
3.	
4.	
5.	
6.	
7.	
8.	

EPA Characterization Codes (D Nos.) _____ _____ _____

OSHA: Contain listed compounds? __ Yes __ No PHS: Infectious Wastes? __ Yes __ No

Is the waste reactive with water? __ Yes __ No with air? __ Yes __ No

Viscosity ___ Low ___ Medium ___ High

Layers at 70°F ___ None ___ Two ___ Multilayers

Give any additional information on the hazards of the waste: pH _____ Flash Pt. _____

BTU: _____ Specific Wt. _____ Color _____

NOTICE: THIS DATA SHEET WILL NOT BE ACCEPTED WITHOUT SIGNATURE AND DATE:

I hereby certify that the above information is complete and accurate.

_____ _____ _____
Signature Title Date

NOTE: PLEASE WRITE YOUR NAME AND BUILDING NUMBER ON EACH DRUM.

4.7 Analytical Procedures

The laboratory currently handles all EPA, ASTM, SW-846, and other methods (see Figure 4.14) used for Routine Analytical Services and Special Analytical Services. These methods shall be readily available to all analysts with detection limits and matrix requirements by method. If variances will be needed the client is contacted in writing regarding these modifications and the purpose for any changes. A notation is made in the analytical report identifying the approval of modification.

Figure 4.14 Laboratory Analytical Methods

CONVENTIONAL PARAMETERS AND BACTERIOLOGICAL ANALYSES ON WATER AND WASTEWATER

Parameter	Methods	
	Std Mtds	EPA
Acidity	402	305.2
Alkalinity	403	310.1
Bicarbonate (only)	403	310.1
Calcium	314B	130.2
Carbonate (only)	403	310.2
Carbon dioxide	410A	
Chloride	407B	325.3
Chlorine demand	409	
Chlorine residual	408F	330.2
Color	204	110.3
Cyanide	412	335.2
Hardness	314B	130.2
Nitrate	418D	352.1
Nitrite	419	354,1
Nitrogen		
Ammonia with distillation	417A	350.2
Ammonia without distillation	417B	350.2
TKN	420B	351.3
Organic (only)		
Oil and Grease		
Separatory funnel technique	503A	413.1
Infrared	503B	413.2
Soxhlet extraction	503C	
Oxygen Demand		
Biochemical	507	405.1
Chemical	508	410.1
Immediate		

CONVENTIONAL PARAMETERS AND BACTERIOLOGICAL ANALYSES ON WATER AND WASTEWATER

Parameter	Methods	
	Std Mtds	EPA
pH Value	423	150.1
Phenol with distillation	410A	420.1
Phenol without distillation	510B	420.1
Phosphorus		
Ortho	424E	365.2
Total	424C	365.2
Residue		
Total	209A	160.3
Total volatile and/or fixed	209F	160.4
Settleable matter	209E	160.5
Total dissolved solids	209B	160.1
Total Suspended solids	209C	160.2
Fixed and volatile solids	209D	160.4
Silica	525	370.1
Specific conductance	205	120.1
Sulfate	426	375.4
Sulfide	427	376.1
Sulfite	428	377.1
Surfactants	412B	425.1
Total carbon	505	415.2
Total organic carbon	505A	415.2
Turbidity	214	180.1

QC requirements specified in the methods will be followed

METAL ANALYSES

Metal	AAS/Furnance			AAS/Flame			ICAP/AES		
	SW-846	EPA	CLP-M	SW-846	EPA	CLP-M	SW-846	EPA	CLP-M
Aluminum (Al)		202.2		7020	202.1		6010	200.7	200.7
Antimony(Sb)	7041	204.2	204.2	7040	204.1		6010	200.7	200.7
Arsenic(As)	7060	206.2	206.2	7061	206.1		6010	200.7	200.7
Barium(Ba)		208.2		7080	208.1		6010	200.7	200.7
Beryllium(Be)	7091	210.2	210.2	7090	210.1		6010	200.7	200.7
Boron(B)							6010	200.7	200.7
Cadmium(Cd)	7131	213.2	213.2	7130	213.1		6010	200.7	200.7
Calcium(Ca)				7140	215.1	215.1	6010	200.7	200.7
Chromium(Cr)	7191	218.2	218.2	7190	218.1		6010	200.7	200.7
Cobalt(Co)	7201	219.2		7200	219.1		6010	200.7	200.7
Copper(Cu)		220.2		7210	220.1		6010	200.7	200.7
Gold(Au)		231.2			231.1				
Iron(Fe)		236.2		7380	236.1		6010	200.7	200.7
Lead(Pb)	7421	239.2	239.2	7420	239.1		6010	200.7	200.7
Lithium(Li)									
Magnesium(Mg)	7450				242.1	241.1	6010	200.7	200.7
Manganese(Mn)		243.2		7460	243.1		6010	200.7	200.7
Mercury (Hg)	7470‡ 7471‡	245.1‡ 245.1‡	245.1‡						
Molybdenum(Mo)	7481	246.2		7480	246.1		6010	200.7	200.7
Nickel(Ni)		249.2		7520	249.1		6010	200.7	200.7
Potassium(K)		258.1		7610		258.1	6010	200.7	200.7
Phosphorus(P)									
Selenium(Se)	7740	270.2	270.2	7741	270.3		6010	200.7	200.7
Silicon(Si)							6010	200.7	200.7
Silver(Ag)		272.2	272.2	7760	272.1		6010	200.7	200.7
Sodium(Na)		273.2		7770	273.1	273.1	6010	200.7	200.7
Thallium(Tl)	7841	279.2	279.2	7840	279.1		6010	200.7	200.7
Tin(Sn)		282.2		7870	282.1				
Titanium(Ti)		283.2			283.1				200.7
Vanadium(V)	7911	286.2		7910	286.1		6010	200.7	200.7
Zinc(Zn)		286.2		7950	289.1		6010	200.7	200.7

* Gaseous hydride
† On request
‡ Manual cold vapor method

ORGANIC ANALYSIS

Parameter	Methods		
	CFR	SW-846	EPA
GAS CHROMATOGRAPHY(GC)			
Halogenated volatile organics	601	8010	502.1
Nonhalogenated volatile organics		8015	
Aromatic volatile organics	602	8029	
Acrolein, acrylonitrile, acetonitrile	603	8030	
Organochlorine pesticides and PCBs	608	8080	505/508
PCBs only	608	8080	508
Chlorinated hydrocarbons	612	8120	
Organophosphorus pesticides	614	8140	507
Chlorinated herbicides	615	8150	515.1
Other analyses not listed			
GAS CHROMATOGRAPHY/MASS SPECTROMETRY(GC/MS)			
Priority pollutants			
Volatile organics (VOAs)	624	8240	524.2
Semivolatile organics using fused silica capillary columns	625	8270	525.1
Up to 10 additional peaks for VOAs and semivolatiles	625	8270	
Polynuclear aromatic hydrocarbons (PAH) using the PAH retention index system	675		
2,3,7,8-Tetrachlorodibenzo-p-dioxin(TCDD)or total TCDD	613		
2,3,7,8-TCDD and 2,3,7,8-TCDF		8280	
Tetra through octa polychlorinated dibenzo-p-dioxins(PCDD) and dibenzofurans (PCDF)		8280	
Tetra through octa PCDD and PCDF		8290	
Special analysis including chemical ionization (pulsed positive ion and negative chemical ionization), PCB isomers, pyrolysis, head space, characterization, etc.			
CONTRACT LABORATORY PROGRAM (CLP) FOR ORGANICS including hazardous substances and additional compounds by GC/MS and GC/EC	Methods listed above		
EPA APPENDIX IX COMPOUNDS (Organics and Inorganics)	Part 261		
FOURIER TRANSFORM INFRARED SPECTROMETER (FTIR) Qualitative and/or quantitative characterization of various organic and inorganic compounds in aqueous and nonaqueous matrices			

ORGANIC ANALYSIS

Parameter	Methods		
	CFR	SW-846	EPA
COMPUTERIZED ULTRAVIOLET-NEAR INFRARED AND ULTRAVIOLET-VISIBLE ARRAY PROCESSOR This array processing system observes the evolution of the UV-visible spectrum at 0.1 second intervals. Data handling is similar to the FTIR (storage, subtraction, etc.). The spectrophotometer has a very large dynamic range and small quantities of chromophoric impurities can be detected.			
NUCLEAR MAGNETIC RESONANCE (NMR) • Structure identification of organic compounds in aqueous or organic solutions • High temperature polymer characterization • Purity analysis of organic materials • Analysis of ^{1}H, ^{13}C, ^{15}N, ^{17}O, ^{31}P and a number of other nuclei in organic molecules.			
HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)			
Polynuclear aromatic hydrocarbons (PAH)		8310	525.1
Explosives		8330	
Carbamate pesticides	531		
LIQUID CHROMATOGRAPHY/MASS SPECTROMETRY (LC/MS) Analysis of high molecular weight polar and nonpolar organic compounds in aqueous and nonaqueous liquids			

MISCELLANEOUS ANALYSIS

Parameter	Method(s)
ION CHROMATOGRAPHY	
Major anions (individual)	
Chloride	EPA 300.0
Fluoride	"
Nitrate-N	"
Mitrite-N	"
Ortho phosphate-P	"
Sulfate	"
Major cations (individual)	
Calcium	Dionex (IC)
Magnesium	"
Sodium	"
Potassium	"
Major anions or cations (complete)	"
Other ions Phenols	Dionex (IC)
Formate	"
Acetate	"
Cyanides	"
Sulfides	"
Others not listed	
BIOTREATABILITY - Lab Study	ASTM D2667-82
X-RAY FLUORESCENCE Metal scan of liquids and solids for elements heavier than sodium	NA
COULTER COUNTER (COMPUTERIZED) Particle size distribution of soils, sediments, dust, etc., from 1 μm to 1 mm. Instrument is capable of performing volume and/or weight distributions.	

RCRA ANALYSIS OF GROUND WATER

Parameter	Methods
INDICATORS OF DRINKING WATER SUITABILITY (IDWS)	
Metals by AAS/Furnace	
Arsenic	EPA 206.2
Barium	EPA 208.2
Cadmium	EPA 213.2
Chromium	EPA 218.2
Lead	EPA 239.2
Mercury*	EPA 245.1
Selenium	EPA 270.2
Silver	EPA 272.2
Sample Preparation	
TOTAL METALS	
PESTICIDES BY GC/ECD	
Endrin Lindane Methoxychlor Toxaphene	CFR 608
2,4-D 2,4-TP (Silvex)	CFR 615
TOTAL PESTICIDES	
RADIOACTIVITY	
Radium 226 and 288	STD 707; 708
Gross Alpha	STD 703
Gross Beta	STD 703
Strontium	STD 704
Tritium	STD 708
TOTAL RADIOACTIVITY	
OTHERS	
Nitrate as N	EPA 300.0 (IC)
Fluorides	EPA 300.0 (IC)
Turbidity	EPA 180.1;STD 214
Total coliform bacteria	STD 909
TOTAL OTHERS	
TOTAL IDWS	

RCRA ANALYSIS OF GROUND WATER

Parameter	Methods
INDICATORS OF GROUND WATER QUALITY (IGWQ)	
Chloride	EPA 300.0 (IC)
Sulfate	EPA 300.0 (IC)
Iron	EPA 200.7 (ICP)
Manganese	EPA 200.7 (ICP)
Sodium	EPA 200.7 ICP)
Phenol	EPA 420.1
TOTAL IGWQ QUALITY	
INDICATORS OF GROUND WATER CONTAMINATION (IGWC) (Quadruplicate Analysis)	
pH	EPA 150.1;STD 423
Specific conductance	EPA 120.1;STD 205
Total organic carbon	EPA 415.2; STD 5-05A
Total organic halides	SW-846 9020
TOTAL COST FOR IGWC	
TOTAL FOR COMPLETE RCRA ANALYSIS	

• Mercury is analyzed by the cold vapor method

† These isotopes are analyzed only if the gross alpha exceed 5 pCi/L and gross beta exceeds 8 pCi/L

WASTE EVALUATION BY SW-846 PROCEDURES

Parameter	SW-846 Method
IGNITABILITY Pensky-Martens Closed Cup	1010
CORROSIVITY (toward steel)	1110
REACTIVITY	Gas evolution
EXTRACTION PROCEDURE TOXICITY (EP TOX)	
Leachate preparation	1310
Analysis of Leachate	
Metals*	
Arsenic	7060 (furn)
Barium	7080 (flame)
Cadmium	7130 "
Chromium	7190 "
Lead	7420 "
Mercury	7470 (cold vapor)
Selenium	7740 (furn)
Silver	7760 (flame)
Sample preparation	
Pesticides by GC/EC	
Endrin Lindane Methoxychlor Toxaphene	8080
2,4-D 2,4,5-TP (Silvex)	8150
TOTAL EP TOX	
TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) includes volatile and semivolatile organics and metals	40 CFR 261.3

* Analysis by method of additions

AIR SAMPLES

Parameter
AMBIENT AIR**
Vost Tube (TO 1) or CMS Tube (TO 2) Analysis
Preparation
PUF (TO 10)
Analysis (GC/ECD)
Preparation
2.2 cm ID x 7.6 cm length
6.5 cm ID x 7.6 cm length
8.9 cm ID x 7.6 cm length
Canister (TO 14) Analysis
Canister cleaning
Canister sampler cleaning
Fixed Gases (O_2, N_2, CO_2) Percent
Trace
Formaldehyde† (NIOSH 3500)
Nitrogen oxides† (NO_2, NO_3) by IC
Sulfur dioxide† (total sulfur by ICAP)
Suspended particulate matter† (TSS)
Carbon monoxide
Hydrogen sulfide† (total sulfide by titration)
Asbestos (bulk identification or fiber count)
Others
INDUSTRIAL HYGIENE (PERSONAL SAMPLES)
Chemical substances in the work environment adopted by ACGIH

* The range of adsorbents used to collect contaminants for these types of analysis is broad and includes polyurethane foam (PUF), XAD resins, florisil, charcoal, Tenax and bag sampling. Preparation includes media clean-up and quality control check by an approved and analytical method. Methodology is also varied. Specific sampling and analytical plans are designed on an individual basis to accomplish the goals of each task. Methods are those sepcified by EPA and ACGIH

** All other TO methods are also performed. Prices upon request.

† From impinger

GEOPHYSICAL TESTING

Parameter	Method
Atterberg Limits	ASTM-4318
Calcium Carbonate	ASTM-4373
Hydrometer	ASTM-D422
Moisture Retention	ASTM-D2325
Moisture Content	ASTM-D2216
Permeability	ASTM-D2434
Procter	ASTM-D69/D1557
Wet Sieve	ASTM-C117/C136
Dry Sieve	ASTM-D422
Specific Gravity	ASTM-C127/C128/C854
Unconfined Compressive Strength	ASTM-C39/C109/D2166
One-Dimensional Consolidation Properties	ASTM-D4186

PERSONNEL QUALIFICATIONS

REV 9

Copy No. 33

Approvals:

Division 01 Quality Assurance	[illegible]	Date:	11/30/93
Division 01 Management	[illegible]	Date:	11/30/93
SwRI QA Management	T.C. Silovich	Date:	12/1/93

5. PERSONNEL QUALIFICATIONS

All professional staff members in the laboratory shall have a complete understanding of the requirements of the quality assurance program. A control copy of each revision of the plan and a briefing on the plan will be provided by the quality officer or his/her designee.

The QA/QC Officer and QA Auditor will review the QA requirements specific to the project with the Analytical Supervisor and Project Manager prior to starting project. The quality assurance plan for the project will be prepared in accordance with the project requirements. Upon project start-up, the remaining project assignments will be made. All personnel assignments to tasks affecting data quality shall be made on the basis of adequate education, training, and experience. Inexperienced personnel will be closely supervised and provided with standard operating procedures. All project personnel will be trained in QA/QC responsibilities upon assignment to the project.

5.1 Training

New technicians shall be shown location of controlled copies of the QAPP and standard operating procedures. They will be formally trained by the QA Auditor as well as the Analytical Supervisor. Documentation of any training is maintained in a training file for each employee. All revisions to procedures shall be given to staff maintaining controlled copies (in written form). A new employee training session and annual training are offered to all employees by Division Personnel. Prior to any actual work procedures requiring GLP or Nuclear procedures, employees will be trained by the QA/QC Officer for Good Laboratory Practice Procedures and by the Institute QA for Nuclear Training. Training files will be updated to show the latest training and will be re-evaluated annually.

A quarterly training seminar will be held for all new personnel and all personnel will review on an annual basis. This training will cover the following information:

- Copy of Quality Assurance Program Plan
- Copy of Standard Operating Procedures
- Documentation requirements*
- Custody procedures*
- Review of current projects*
- Special analytical request procedures*
- Contact personnel for questions/problems*

* A handout will also be provided to all personnel.

At the end of each training session, a Certificate of Completion will be given to the employee and a copy will be put in his/her training file. The personnel training manual will be updated annually with new training information.

5.2 Personnel Safety

The purpose of a laboratory safety program is to provide convenient information for hazard recognition and control. Fires, explosions and exposure to chemical, biological agents, and other hazards can usually be anticipated and therefore prevented.

For these reasons, Southwest Research Institute has developed a Safety Manual which is distributed to every employee. In addition to the Safety Manual, every employee is familiarized and instructed with the proper use of safety equipment in his/her laboratory. According to the Federal (OSHA) Hazard Communication standard each laboratory has a continually updated notebook of Material Safety Data Sheets.

FACILITIES, EQUIPMENT AND SERVICES

REV 7

Copy No. 33

Approvals:

Division 01 Quality Assurance	Lo Ann Boyd	Date:	11/30/93
Division 01 Management	[illegible]	Date:	11/30/93
SwRI QA Management	S.C. Silovich	Date:	12/1/93

6. FACILITIES, EQUIPMENT, AND SERVICES

6.1 Cleaning of Sample Preparation Containers

In analyzing for trace amounts of organic and inorganic substances, it is imperative to ensure that all materials that may come in contact with the sample will be free from contamination. Specific instructions for preparation of material will be incorporated into the project work plan, and the instructions in the laboratory SOP will be followed. All glassware used for inorganic and organic analyses shall be cleaned in accordance with the standard operating procedures. The quality of glassware cleaning procedure is checked through the use of method blank preparations with each sample set. Should the blank preparation show the presence of contamination, then the source of contamination is sought by examination of the various components of sample preparation including glassware, reagents, and sample storage containers.

6.1.1 Organic Analysis

All solvents used will distilled in glass and glassware will be cleaned as specified in SOP-01-08-01.

6.1.2 Inorganic Analysis

During the sample preparation, contamination from the laboratory can also affect the analytes content of the samples. For this reason, all operations which require the same or some stage of its processing to be exposed to the laboratory atmosphere (i.e., weighing operations, opening digestion bombs, etc.) will be performed on clean benches. To reduce the quantity of contamination in the laboratories, routine cleaning will be scheduled into the work week. This is to consist of 15 to 20 minutes of house cleaning duties at the end of each day and on the last work day of the week the last 30 to 40 minutes will be utilized in very thorough cleaning of the laboratories and equipment. This ensures a minimum of contaminants in the laboratory atmosphere at any particular time.

6.2 Instrument Calibration

6.2.1 Gas Chromatographs shall be calibrated in accordance with SOP 01-05-01.

The analytical coordinator or the analyst will prepare calibration standards that contain the compound(s) of interest. The standards will normally be prepared at concentrations covering two or more orders of magnitude that completely bracket the working range of the chromatographic system.

The analyst will assemble the chromatographic apparatus delineated in the work plan and establish the proper operating parameters for the compound(s) under study. By injecting calibration standards, the analyst will establish the sensitivity limit of the detector and the linear range of the analytical system for each compound.

Prior to using a cleanup procedure, e.g., Florisil column, the analyst will process a set of calibration standards through the system to validate the elution pattern given in the analytical method and the absence of interferences from the reagents used.

A list of equipment and capabilities is listed in Table 1.

6.2.2 *Gas Chromatography/Mass Spectrometry shall be calibrated in accordance with SOP 01-05-01.*

Daily calibration checks will be made using perfluorotributylamine (FC-43) as the calibration standard. When the calibration is complete, performance evaluation will be made.

For volatile organic analysis, 4-bromofluorobenzene (BFB) is used to tune the mass spectrometer. The key ion abundance criteria are shown in Table 2.

6.2.3 *Dioxin shall be calibrated in accordance with SOP-01-13-01 and SOP-01-13-03.*

Minimum calibration shall be performed using Native CDDs and CDFs requirements given in the above-referenced SOPs.

6.2.4 *Atomic Absorption Spectrophotometry shall be calibrated in accordance with SOPs 01-07-05 and 01-07-13.*

This calibration procedure is patterned after "Monitoring Industrial Wastewater," USEPA Technology Transfer, August 1973, Section 8, Preparation of Standards and Calibration. Stock solutions of the metal(s) under study will be prepared using high purity standards and reagents at concentrations of 1000 mg of the metal per liter of water. These stock solutions will be used to prepare the calibration standards.

Prepare a blank acid and calibration standard using the same type of acid as will be used for sample processing. The calibration standards should cover the range of the samples, where this is known or can be reasonably determined. Aspirate the blank, followed by the standards from highest to lowest concentration. To establish that the calibration has not changed, blanks and standards will be run not less frequently than after every 20 samples, but more frequently if in the judgment of the analyst a system change seems likely. Flameless analysis generally requires more frequent continuous calibration verification (after every 10 to 15 samples).

If the sample matrix is so complex that viscosity, surface tension, and components cannot be accurately matched with standards, the method of standard additions will be used. Standard additions will be made to portions of the sample, and the calibration curve will be prepared from the responses to these aspirations. These standards will be analyzed in the same manner and with the same frequency as the reagent standards discussed above. A complete description of the method of additions technique is given in Section 8.5 of "Monitoring Industrial Wastewater."

6.2.5 Ion Chromatograph(s) shall be calibrated in accordance with SOP-11-02

At the beginning of each work day or upon each alteration to the analysis being performed, the analyst will establish the calibration by running a series of standards at a minimum of three concentration levels: low, medium and high. The medium standard will be run after every ten samples to verify the calibration. If the response differs from the calibration response by more than 10%, the analyst will:

- repeat the standard injection to determine if the previous result was correct,
- if correct, repeat the calibration using the three standards,
- rerun all samples after the last consistent calibration check.

6.2.6 Total Organic Carbon (TOC) Analyzer shall be calibrated in accordance with SOP 01-11-01.

The TOC analyzer is internally calibrated to give a direct readout of micrograms per milliliter (μg/mL) of organic carbon. At the beginning of each work day, the analyst will prepare and inject the standard for the appropriate range:

- 10 ppm for 1 mL
- 400 ppm for 250 μl
- 2000 ppm for 40 μl

A second injection of the standard should be performed. If the two are within 5% of each other, the instrument is calibrated as such. A third injection is performed. The output should read within 5% of the standards true value. It this is so, analysis is started.

If this is not true, then calibration is repeated until the standard is adequate. A CCV (standard of original concentration) will be injected after every six sample "sets" of injections, or after six soil sample combustions. If the response differs from the true value by more than 10%, or per client, the analyst will repeat the injection to determine if previous result was correct. If correct, repeat the calibration and rerun all samples after the last consistent calibration check.

Table 1 ANALYTICAL INSTRUMENTATION

Description	Number	Year(s) Purchased
Gas Chromatographs/High Resolution Mass Spectrometer (GC/HRMS)		
■ Fisons Autospec Ultima with GTC A200S autosampler	1	1993
■ Fisons Autospec with CTC A200S autosampler	1	1990
■ Finnigan 8200	1	1989
Gas Chromatographs/Mass Spectrometers/Mass Spectrometers (GC/MS/MS)		
■ Fisons TRIO-III with Waters 600-MS HPLC connected to a Vestec particle beam interface (Universal Interface, Module B)	1	1988
■ Finnigan TSQ-4600 with CTC A200S autosampler	1	1986
Gas Chromatographs (GC); Total: 22		
■ Hewlett-Packard 5890 GC with NP/FP detectors/a HP 7673A autosampler	5	1986-91
■ Hewlett-Packard 5890 GC with a FP detector/a HP 7673A autosampler	1	1991
■ Hewlett-Packard 5890 GC with dual EC detectors/a HP 7673A autosampler	10	1986-91
■ Fisons CE 800 GC with dual EC detectors/a CTC A200S autosampler	3	1992
■ Hewlett-Packard 5890 GC with FI/TC detectors/a HP 7673A autosampler	1	1987
■ Hewlett-Packard 5890 GC with FI detector/a HP 7673A autosampler	1	1987
■ Hewlett-Packard 5890 GC with dual pairs of OI PI/ELC detectors/Tekmar LCS-2000 purge and trap unit/Dynatec Precision Sampling PTA-30 autosampler	1	1989
Gas Chromatographs/Mass Spectrometers (GC/MS); Total: 15		
■ Finnigan 4600 with CTC A200S autosampler	2	1986
■ Finnigan 4500 with CTC A200S autosampler	1	1985
■ Finnigan 4500 with Tekmar 2000/Dynatec Precision Sampling PTA-30 autosampler	2	1985
■ Finnigan 3200 with CTC 120 pyroprobe unit	1	1981
■ Fisons TRIO-1000 with Tekmar LSC-2000 purge and trap unit/Dynatec Precision Sampling PTA-30 autosampler	2	1990-91
■ Fisons TRIO-1000 with Entech ELA Model 2000 canister autosampler	1	1992
■ Fisons MD-800 with CTC A200S autosampler	3	1992
■ Fisons TRIO-II with Tekmar LSC-2000 purge and trap unit/Dynatec Precision Sampling PTA-30 autosampler	2	1988-89
■ Finnigan with ion trap	1	1990

Description	Number	Year(s) Purchased
High Performance Liquid Chromatographs (HPLC); Total: 9		
■ Waters HPLC with Waters 600E multisolvent delivery system capable of generating gradients using up to four solvents, postcolumn derivatization, Waters 420-AC fluorescence, and Waters 700 Satellite Waters Intelligent Sample Processor (WISP) autosampler	2	1989
■ Waters HPLC with 600E multisolvent delivery system, 990 photodiode array detector capable of continuous spectral monitoring and acquisition from 190 to 800 nm, 410 differential refractometer, 700 Satellite WISP autosampler, and U6K manual injector	1	1988
■ Waters HPLC with 600E multisolvent delivery system, 486 tunable absorbance detector, and 600 satellite WISP autosampler	3	1991
■ Waters HPLC with 486 tunable absorbance detector, 470 scanning fluorescence detector, and 717 autosampler	3	1992
Air Sampling and Analysis (Method TO-14 and PM-10)		
■ Anderson or Scientific Instrumentation Specialist (SIS) cansiters (6. Liters)	182	1988-1993
■ SIS Canister Sampler for Pressurized Canister Sampling	2	1990
■ SwRI Canister Cleaning System, Consisting Sixteen Port Manifold, Canister Heating Unit and two Diffusion Pumps (Capable to Reach 10^{-6} torr with 16 Canisters on Manifold)	1	1993
■ Wedding & Associates, Inc. Critical Flow High Volume Sampler	1	1991
Gel Permeation Chromatographs (GPC); Total: 5		
■ Waters GPC with fraction collector, 501 HPLC pump, 440 absorbance detector, and 712 WISP autosampler	4	1990-91
■ Analytical Bio-Chemistry Laboratories with Autoprep 1002A with a fixed wavelength ISCO UH-5 UV detector	1	1986
Radiochemistry		
■ Beckman LS6000TA liquid scintillation counter	1	1993
■ Canberra 2400F proportional counter	1	1993
■ Canberra CWSLC-D gamma and alpha spectroscopy system	1	1993
Ion Chromatograph (IC)		
■ Dionex 2020i dual-channel with conductivity, electrochemical, UV-Vis detectors and Dionex automated sampler	1	1985

Description	Number	Year(s) Purchased
Atomic Absorption Spectrophotometers (AAS)		
■ Perkin-Elmer 4100 Zeeman with Perkin-Elmer AS-60 autosampler	1	1993
■ Perkin-Elmer 5100 Zeeman with Perkin-Elmer AS-60 autosampler	1	1989
Inductively Coupled Argon Plasma Atomic Emission Spectrometer (ICP)		
■ Spectro FME-50 with Spectro ultrasonic nebulizer and a microprocessor controlled random access autosampler	1	1992
■ Perkin-Elmer 6500 including a Perkin-Elmer AS-3 automatic microsampler system	1	1984
Mercury Analyzer		
■ PS200 Leeman Lab Automated Mercury Analyzer	1	1992
■ AP200 Leeman Lab Automated Mercury Digestor	1	1992
Supercritical Fluid Chromatograph (SFC)		
■ Lee Scientific 622 with pneumatically operated valve system for split/direct SFC injection, split/splitless GC injection, and two FIDs	1	1988
Supercritical Fluid Extractor (SFE)		
■ Suplex SFE-50	1	1990
Microwave Digestor		
■ GT Instruments Milestone MLS 1200 digestor/drying module with GT Fam 35 fumes absorbing module, Em 5 exhaust module and Acm 100 automated capping module	1	1991
Total Organic Halide Analyzer		
■ Xertex Dohrman	1	1982
Total Organic Carbon Analyzer		
■ Xertex Dohrman	1	1982
Geotechnical Testing		
■ Soil Test CN-4235 Mechanical Soil Compactor	1	1992
■ Soil Test CL-278 Hydrometer Jar Bath	1	1992
■ Soil Test USA Standard Sieves with Sieve Size from 3.5" to 53 μm	1 each	1989
■ Soil Test CL-305A Motorized Sieve Shaver	1	1989
■ Soil Test G-27-325-340 Specific Gravity Set	1	1986
■ Soil Test CN-989 Sand Density Cone	1	1989
■ Soil Test A-86A 5-Bar Pressure Plate Extractor	1	1991
■ Soil Test CN-170 Electromagnetic Vibrator Table	1	1991
■ Soil Test K-605 and K-610 Liquid Limit device with Removable Cup	1	1991
■ Soil Test U-580 Unconfined Compressor Apparatus	1	1991
■ Soil Test Cl-204 Liquid Limit Device with Counter	1	1989

Description	Number	Year(s) Purchased
Wet Chemistry		
■ Technicon TRAACS 800	1	
■ Kjeltec System 1026 distilling unit	1	1991
■ Milton Roy Spectronic 1201	1	1990
■ YSI dissolved oxygen analyzer	1	1989

Table 2 Ion Abundance Criteria

For volatile organic analysis, 4-bromofluorobenzene (BFB) is used to tune the mass spectrometer. The key ion abundance criteria are as follows:

Mass	Ion Abundance Criteria
50	8.0 - 40 % of mass 95
75	30 - 66.0 % of mass 95
95	Base Peak, 100 % of relative abundance
96	5.0 - 9.0 % of mass 95
173	< 2.0 % of mass 174
174	50.0 - 120.0 % of mass 95
175	4.0 - 9.0 % of mass 174
176	93.0 - 101.0 % of mass 174
177	5.0 - 9.0 % of mass 176

For extractable organic analysis, performance evaluation will be performed using decafluorotriphenylphosphine (DFTPP). The key ion abundance criteria are as follows.

Mass	Ion Abundance Criteria
51	30.0 - 80.0 % of mass 198
68	< 2 % of mass 69
69	Mass 69 relative abundance
70	< 2 % of mass 198
127	25.0 - 75.0 % of mass 198
197	<1 % of mass 198
198	Base peak, 100 % of relative abundance
199	5.0 - 9.0 % of mass 198
275	10.0 - 30.0 % of mass 198
365	> 0.75 % of mass 198
441	Present, but less than mass 443
442	40.0 - 110.0 % of mass 198
443	15.0 - 24.0 % of mass 442

Method blanks shall be run to determine background daily and when changing columns to avoid contamination. For quantitation, inject one or more of the appropriate standards at the initiation of each analytical exercise to ensure that the column is performing properly.

There are 10 GC/MS instruments available. The Finnigan 3000 series is 12 years old; the Finnigan 4000 series is less than 6 years old; and all VG GC/MS are less than 1 year old.

6.2.7 Atomic Emission Spectrometry shall be calibrated in accordance with SOPs 01-07-12 and 01-07-25.

Calibration Standards - This calibration procedure is patterned after Test Methods for Evaluating Solid Waste, Method 6010, Paragraph 7.2, 7.3. Stock solutions of the metal(s) under study will be prepared using high purity standards and reagents at concentrations of 1000 mg/per liter. These stock solutions will be used to prepare the calibration standards.

Daily Calibration - At the beginning of each day the instrument will be profiled and calibrated as per the manufacturer specifications. The sample introductory system is optimized before calibration begins. Prepare a blank and calibration standards using the same type of acid as will be used for sample processing. The calibration standards should cover the range of the samples where this is known or can be reasonably determined. Aspirate the blank followed by the standards from highest to lowest.

Initial Calibration Blank (ICB), Initial Calibration Verification (ICV), Continuing Calibration Blank, (CCB) and Continuing Calibration Verification (CCV) - To establish that the calibration has not changed a blank and a continuing calibration verification standard will be run immediately after calibration, then after every 10 samples, and at the end of the analytical run. The standard material for making up the continuing calibration verification standard shall be from a source different than that used to make up the calibration standards. Preferably the use of NBS Standard material shall be employed for this purpose. If one is not available then a source from a different vendor than the one from which the calibration standards were obtained shall be used.

6.2.8 Mercury Analyzer shall be calibrated in accordance with SOP-01-07-24.

The PS200 Mercury Analyzer uses a program called a MACRO. Depending on the MACRO used this instrument will run a 5 point or 6 point calibration curve.

If the curve falls out (r-value of less than .995), the PS200 will automatically re-run the curve.

The check standard must be within ±20% of the true value or the PS200 will automatically recalibrate and rerun the Initial Calibration Blank (ICB) and Initial Calibration Verification (ICV).

The PS200 runs a Continuing Calibration Blank (CCB) and Continuing Calibration Verification (CCV) every 10 samples. If the CCV is not within 20% of true value the instrument will re-calibrate and rerun the last 10 samples before the last CCV run and continue with the sample run.

6.2.9 Radio Chemistry

6.2.9.1 Liquid Scintillation Counter:

The calibration of the liquid scintillation counter shall be checked each day that it is to be used. This shall be performed in accordance with SOP-01-16-05, which is patterned from the procedure in the counter's manual for checking the system's calibration.

During analysis of samples, backgrounds shall be run at the beginning of a procedure and automatically subtracted from the sample's values, or shall be intermixed with the samples and manually subtracted. One background will be run for every twenty samples, and more frequently if necessary. Standards shall be run as necessary, with a minimum of one per run, and more frequently, if necessary.

6.2.9.2 Proportional Counter:

The calibration of the proportional counter shall be checked each day that it is to be used. This will be performed in accordance with SOP 01-16-06, which is patterned from the procedure in the Los Alamos publication *Health and Environmental Chemistry: Analytical Techniques, Data Management, and Quality Assurance*, in the section referencing proportional counter calibration.

One background shall be run for every twenty samples, and more frequently, if necessary. Standards shall be run as necessary, with a minimum of one per run, and more frequently, if necessary.

6.2.9.3 Gamma Spectroscopy System:

The calibration of each gamma detector shall be checked each day that it is to be used. This shall be performed in accordance with SOP 01-16-07, depending on the type of analysis performed. Both methods are patterned from the procedure in the Los Alamos publication *Health and Environmental Chemistry: Analytical Techniques, Data Management, and Quality Assurance*, from the section referencing gamma ray spectroscopy.

While running samples, backgrounds shall be taken initially, and additional ones shall be taken later, if necessary. Standards shall be measured with the detector in question, as necessary.

6.2.9.4 Alpha Spectroscopy System:

The calibration of each alpha detector shall be checked each day that it is to be used. This shall be performed in accordance with SOP 01-16-08. This method is patterned from the procedure in the Los Alamos publication *Health and Environmental Chemistry: Analytical Techniques, Data Management, and Quality Assurance*, from the section referencing alpha ray spectroscopy.

While running samples, backgrounds shall be taken initially, and additional ones shall be taken later if necessary. Standards shall be measured with the detector in question, as necessary.

6.2.10 Other Instrumentation

Other instrumentation including Fourier transform infrared spectrophotometer, nuclear magnetic resonance analyzer, and UV/VIS spectrophotometers will be calibrated according to manufacturer's specifications. Performance checks will be run to assure that the instruments will be operating properly and that operational checks shall be run to verify proper system performance.

6.3 Geophysical Testing Calibration

6.3.1 Attenberg Limits - Method ASTM-4318:

The calibration will be accomplished in accordance with SOP 01-20-15.

6.3.2 Calcium Carbonate - Method ASTM-4373:

The calibration will be accomplished in accordance with SOP 01-20-16.

6.3.3 Hydrometer - Method ASTM-D422:

The calibration will be accomplished in accordance with SOP 01-20-09.

6.3.4 Moisture Retention - Method ASTM-D2325:

The calibration will be accomplished in accordance with SOP 01-20-13.

6.3.5 Moisture Content - Method ASTM-2216:

The calibration will be accomplished in accordance with SOP 01-20-17.

6.3.6 Permeability - Method ASTM-D2434:

The calibration will be accomplished in accordance with SOP 01-20-18.

6.3.7 Proctor - Method ASTM-D69/D1557

The calibration will be accomplished in accordance with SOP 01-20-11.

6.3.8 Wet Sieve - Method ASTM-C117/C136:

The calibration will be accomplished in accordance with SOP 01-20-03 and SOP 01-20-06.

6.3.9 Dry Seive - Method ASTM-D422:

The calibration will be accomplished in accordance with SOP 01-20-09.

6.3.10 Specific Gravity - Method ASTM-C127/C128/C136:

The calibration will be accomplished in accordance with SOP 01-20-04, SOP 01-20-05 and SOP 01-20-07.

6.3.11 Unconfined Compressive Strength - Method ASTM-C39/C109/D2166:

The calibration will be accomplished in accordance with SOP 01-20-01, SOP 01-20-02 and SOP 01-20-12.

6.3.12 One Dimensional Consolidation Properties - Method ASTM-D4186:

The calibration will be accomplished in accordance with SOP 01-20-14.

6.4 Material Procurement and Control

All standards used for analysis of samples shall be made up from neat standards or solutions traceable to standards. All neat standards shall be kept in a refrigerator specifically assigned to the storage of such standards.

All standards used for each analytical area shall be stored in a refrigerator assigned exclusively to the storage of such standards. Analytical standards shall be prepared under a designated laboratory hood.

The preparation of all standards shall be documented in a laboratory notebook. An example of an entry may be sequentially coded with a seven digit number aaa-bb-cc-(L23-STD1), where:

aaa	=	Lab location
bb	=	Notebook number
cc	=	Page number

The preparation of all standards shall be documented in a notebook reserved for this purpose.

All standards labels shall contain as a minimum the following information:

1. standard identification information (i.e., components, concentration, etc.)
2. date prepared
3. expiration date
3. initials of person preparing standard
4. solvent(s) used in preparation

Solvents shall be ordered in lots and marked with lot and date upon receipt at the laboratory. Solvents for the organic analytical area shall be monitored by the extraction laboratory. Chemicals for trace metals shall be monitored by the metals preparation laboratory. Each time an individual solvent or chemical is used the date opened and lot number shall be marked on the solvent bottle.

6.5 Maintenance

All instruments will be maintained in accordance with manufacturer's recommendations and normal laboratory practice. All maintenance will be documented and maintained in permanent records by the individual responsible for each instrument. This will include both routine, scheduled maintenance and unscheduled maintenance required by operational failures.

Each instrument shall be assigned a specific log book in which the following information is recorded:

1. samples analyzed
2. operating conditions
3. routine maintenance
4. date and signature of person(s) making entry
5. supervisory review

Major maintenance for all HP gas chromatographs shall be recorded in a separate logbook. Likewise, all major maintenance for the GC/MS and other analytical systems shall be recorded in a separate logbook.

All documentation in logbooks and other documents shall be in black water-resistent ink. If an error is made, corrections shall be made by crossing a line through the error and entering the correct information. Changes shall be dated and initialed. Inserts shall be taped on page with initials and date of insert written over both insert and logbook page. No information shall be obliterated or rendered unreadable. A copy of the instrument logbook covering all the samples of a project shall be filed in the project file.

6.6 Balances, Refrigerators, and Ovens

Calibration of all laboratory balances shall be verified and the results documented in a logbook on a weekly basis with a complete set of "S" weights by grams. There are top balances located in several of the laboratories. Calibration of balances located in the laboratories shall be verified upon use by the analyst and documented in a separate bound logbook. Balances shall be checked every six months by the Institute Calibration Group with traceable standards to the NIST. An inspection sticker shall be placed on the balance dated and initialed as well as an inspection report being filed.

The temperatures of all refrigerators, freezers, and drying ovens shall be monitored daily and the results documented in a bound logbook. Logbooks shall contain the required temperature for each refrigerator and oven with tolerance ranges. When the tolerances are exceeded, documentation shall be made and the discrepancy reported and repaired. If the ovens are being used for drying glassware, constituting a deviation from analytical temperature, this will be documented in the log.

DATA GENERATION

REV 7

Copy No. 33

Approvals:

Division 01 Quality Assurance	JoAnn Boyd	Date:	11/30/93
Division 01 Management	Tom Ford	Date:	11/30/93
SwRI QA Management	T.C. Silovich	Date:	12/1/93

7. DATA GENERATION

To provide high quality data to the sponsor, certain quality control measures must be incorporated into every scope of work and implemented. The numbers and types of quality control samples will be specified by the sponsor in the statement of work. The Proposal/Project Manager and Analytical/Sampling Coordinator shall review these requirements with the QA/QC Officer and QA Auditor and incorporate them along with any additional requirements felt necessary into the work order, the work plan, or the technical or QA project plans as they shall be developed. The QA/QC Officer will be consulted to ensure consistency with organizational goals.

7.1 Quality Assurance Project Plans

The laboratory participates in the following series of blind spiked performance evaluation samples (shipment at the stated frequency) as the external portion of its performance audit program:

- EPA Water Supply (WS) series—semiannual
- EPA Water Pollution (WP) series—semiannual
- EPA Contract Laboratory Program (CLP) Organics Performance Evaluation series—quarterly
- NIOSH Proficiency Analytical Testing (PAT) Program
- Performance Evaluation Samples for proposals
- U.S. Army Corp of Engineers Missouri River Division - 18 months
- EPA/EMSL Radiochemistry Intercomparison Study
- AASHTO Physical Testing - Round Robin

The nature and frequency of additional internal blind spiked samples needed to assure the accuracy of the project data shall be specified by the analytical coordination in the work plan, after consultation with the project manager and the quality officer.

The depth and frequency of internal system audits should depend on the familiarity of project personnel with the instrumentation, methods, and operating procedures to be employed and the frequency of sampling and analysis. The QA/QC Officer or QA Auditor will schedule the audits on an annual basis with changes made for new contracts. Typically the QA/QC Officer or a designated auditor who is not in a production capacity will conduct an initial evaluation of proposed methods and standard operating protocols. The initial systems audit will be performed by the QA/QC Officer or his designated auditor. Follow-up audits will normally be performed by the QA/QC Officer or QA Auditor.

7.2 Quality Control Samples

In each work order or plan, the numbers and types of blank, duplicate, matrix spike and matrix spike duplicate samples will be specified. The frequency of these analyses must be related to the purpose of the study, but the following serve as general guidelines for the development of the quality control portion of the work plan.

At the initiation of an analytical procedure for trace organics and inorganics, the apparatus and the reagents necessary for analysis of samples will be assembled and a series of blank determinations will be performed. These blank determinations will involve all reagent solvents and glassware as required in a standard method of analysis and will determine when background peaks are sufficiently low (or absent) to permit the analysis of the sample to proceed. If satisfactory blanks are not obtained in these initial samples, then additional steps will be taken to determine the cause and eliminate it. Once the initial methodology has been established, then blank determinations will be performed as a routine procedure throughout the program as samples are analyzed. A blank will normally be analyzed to verify non-contamination of the samples.

The following tests will normally be run with each designated batch of samples for analysis: (REsults and raw dara will be avialable to the client upon request.

- one method blank,
- one standard at a median concentration, as a continuing calibration verification
- one matrix spike sample for determination of recovery,
- one matrix spike duplicate sample for determination of recovery for organics/
- one duplicate analyses of one sample from the batch for repeatability for inorganics

Other Quality Control sample types and their uses are given in the following table.

Type	Purpose
Field Blank	Evaluate handling and shipping procedures
Field Spike	Evaluate laboratory performance
Laboratory Control Sample	Evaluate the laboratory performance (no matrix interferences)

If appropriate to the purpose of the study, these will be incorporated into the work plan and implemented, as required by the client.

7.3 Method Validation

If a method is to be used for which no accuracy and precision statements are available and with which this laboratory has no familiarity, then a series of samples will typically be run to determine these characteristics. The following suggested program is patterned after *"Analytical Quality Control."* (see Bibliography)

The study will include concentrations at three different levels: near the sensitivity limit, near the upper bound of expected values, and in between the two. Seven replicates of each concentration should be made, with all the normal procedural steps involved. The recommended order of analysis is seven sequences of low, medium, and high concentrations, which allows for the maximum interferences during operation.

Calculations of average percent recovery and associated standard deviations, will be available from the EPA Quality Control Manual or the quality officer.

7.4 Documentation and Sample Custody

The maintenance of records on the samples collected, received, and analyzed is a critical part of the laboratory's performance. The following records are normally kept for a sampling and analysis program:

- Field log
- Custody transfer sheet
- Laboratory log
- Data form

The uses of these forms are specified below.

7.4.1 Field Log for Samples Collected by SwRI

All samples taken in the field should be recorded in a book prepared specifically for that program. Entries should include the date and time of sampling, intended analysis, preservation, if required, date of shipment to the analytical laboratory, and sample designation. In many programs conducted in this laboratory, the field sampling crew is provided with preprinted labels for attaching to the sample containers as the samples shall be collected. All pertinent information can be placed on the sample label and a code assigned for future identification.

7.4.2 Field Chain-of-Custody Documents

The sampling coordinator will be responsible for documenting the samples collected. Documentation will consist of sample labels, field sampling log books, and chain-of-custody forms. Samples will be collected in prelabeled bottles with the appropriate information in waterproof ink. If the sampling is performed by SwRI, the Field Sampling Crew Chief will collect the samples and record the identifying numbers and other pertinent information in the field sampling log book. If the samples are collected by the client, the custody transfer form will serve as permanent documentation.

The Field Sampling Crew Chief will execute the chain-of-custody documentation utilizing the four-part SwRI custody transfer form (Figure 7.1). The disposition of the copies will be as follows:

White and Yellow	Placed in shipping containers and sent with samples to laboratory
Pink	Retained by sampling crew chief
Gold	Supplied to responsible individual at sampling site as record of samples collected.

7.4.3 Laboratory Custody Procedures

Upon arrival in the laboratory, the Sample Custodian will note the condition of the samples, verify that all sample labels correspond to the identifying numbers on the custody transfer form, and sign and date the transfer form to acknowledge receipt. The sample custodian will retain the yellow copy of the form and place the white (original) in the permanent project file.

Unless another log-in procedure is specified in the QA Project Plan, the Sample Custodian will ensure that the samples will be registered in the laboratory log book. The following information is entered into the sample log: code number, sample source, sample description, matrix type, date of receipt, SwRI project number, and consignee. The samples will be assigned a code number as they are received unless the client code is requested and simplified. The code number is a six-digit number composed as follows:

Place 1 - last digit of year
Place 2 - month
Place 3 - consignee
Places 4-6 - sequence number.

Consignee codes shall be assigned by the sample custodian. The client ID or SwRI control number will be carried through the analytical procedures and all results will have the control number on them as a means of verifying the origin of the result.

Samples with critical holding time requirements will be transferred to the analutical lab immediately by use of the Custody Transfer Sheet (Figure 7.2).

Samples shall be delivered to the Shipping/Receiving Department at SwRI. The Sample Custodian is responsible for transferring samples to the laboratory and for obtaining the original airbill which is kept in the project file.

In the event of Saturday delivery, the Sample Custodian or his designated representative (only Sample custodian is mentioned hereafter) will pick up the samples from the guardhouse at the front gate of SwRI and transfer them to the laboratory.

At the laboratory sample log-in, the Sample Custodian initiates an **SwRI Sample Log-In Form** (Figure 7.3) for each project, or for each set of concurrently arriving samples.

- The Sample Custodian first examines the shipping container and:
  - Records any evidence of tampering or other damage on the Sample Log-In sheet (called the Sheet herein),
  - Records the presence/absence of custody seal(s) on the shipping container,
  - Records the condition of the custody seal(s) (i.e., intact, broken)
  - Removes the custody seal(s), which will be incorporated into the project file
- He/she opens the shipping container, removes the enclosed sample documents and records the following information on the sheet:

  - Log-in date
  - Presence/absence of the chain-of-custody records
  - Receipt of airbill and record airbill number
  - Project or batch number

- The Sample Custodian removes sample containers and sorts them by client sample number sequence.

- Records the condition of samples (intact, broken, leaking, etc.) presence/absence of sample tags on sheet.

- If sample tags are present, will compare them with chain of custody record(s)

- Documents whether these numbers agree, or if there is a discrepancy between tag numbers received and those listed on the chain-of-custody record.

- If sample tags are not listed on the chain-of-custody record, he/she will record this fact.

Figure 7.1 Custody Transfer Form

Southwest Research Institute CUSTODY TRANSFER FORM		
Sampling Site	Sample Type (Air, Water, etc.)	Ship To:
Sampling Personnel:	Sample Condition (ph, Temp, etc.)	Name of Shipper:
Name:		Address:
Phone		Telephone:
Date:		Date Shipped:
Time:		Air Bill No.
Sample ID	Description	Sample Condition on Receipt
Additional Information		Received By:
		Date:
		Time

Figure 7.2 Custody Transfer Sheet

SwRI Sample Custody Transfer Sheet

CLIENT: _____ PROJECT NUMBER: _____
DATE: _____ VTSR: _____ TIME: _____
SAMPLE ID(s): _____ _____
_____ _____
_____ _____
_____ _____

RELINQUISHED BY: _____ OF _____
DATE: _____ TIME: _____

RECEIVED BY: _____ OF _____
DATE: _____ TIME: _____

RELINQUISHED BY: _____ OF _____
DATE: _____ TIME: _____

RECEIVED BY: _____ OF _____
DATE: _____ TIME: _____

RELINQUISHED BY: _____ OF _____
DATE: _____ TIME: _____

RECEIVED BY: _____ OF _____
DATE: _____ TIME: _____

RELINQUISHED BY: _____ OF _____
DATE: _____ TIME: _____

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DATE: _____ TIME: _____

RECEIVED BY: _____ OF _____
DATE: _____ TIME: _____

RELINQUISHED BY: _____ OF _____
DATE: _____ TIME: _____

RECEIVED BY: _____ OF _____
DATE: _____ TIME: _____

RELINQUISHED BY: _____ OF _____
DATE: _____ TIME: _____

RECEIVED BY: _____ OF _____
DATE: _____ TIME: _____

Figure 7.3 SwRI Sample Log-In Sheet

SAMPLE LOG-IN SHEET

Lab Name: Southwest Research Institute — Page ___ of ___

Received By (Print Name): _____ — Log-In Date: _____

Received By (Signature): _____

Case Number: _____ Sample Delivery Group No.: _____ SAS Number: _____		EPA SAMPLE #	CORRESPONDING SAMPLE TAG #	CORRESPONDING ASSIGNED LAB #	REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.
REMARKS:					
1. Custody Seal(s)	Present/Absent* Intact/Broken				
2. Custody Seal No.:	_____ _____				
3. Chain-of-Custody Records	Present/Absent*				
4. Traffic Reports or Packing List	Present/Absent*				
5. Airbill	Airbill Sticker Present/Absent*				
6. Airbill No.	_____ _____				
7. Sample Tags	Present/Absent*				
Sample Tag Numbers	Listed/Not Listed on Chain of Custody				
8. Sample Condition	Intact/Broken*/Leaking				
9. Does information on custody records, traffic reports and sample tags agree?	Yes/No*				
10. Date Received at Lab:	_____				
11. Time Received:	_____				
Sample Transfer					
Possession: _____	_____				
Acceptance: _____	_____				
By: _____	_____				
On: _____	_____				

* Contact SMO and attach record of resolution

Reviewed By: _____ — Logbook No.: _____

Date: _____ — Logbook Page No.: _____

FORM DC-1

- Compares the following documents to verify agreement of the information contained in them:
  - Chain-of-custody record(s)
  - Sample tags
  - Packing lists
  - Airbill

- Documents both agreement among the forms and any discrepancies found.
- Completes the custody sheets for each sample:

  - Condition of sample containers (good, broken, leaking, headspace on volatile water samples, etc.)
  - Custodian signature and date received
  - Sample delivery group identification

If all samples recorded on the chain-of-custody record were received by the laboratory and there are no problems observed with the sample shipment, the custodian signs the chain-of-custody record in the "received for laboratory by:" box on the document. If problems are noted, signs for shipment and notes problems in remarks box of the sheet detailing the problems. The client is contacted for direction, and the appropriate lab personnel shall be notified. The problems and client directions shall be detailed in a telephone conversation record stored in the project file and described in the narrative.

The information on the date and time of sample receipt, chain-of-custody record number, sample numbers, SwRI laboratory numbers, and sample tag numbers is recorded on the table of the sample log-in sheet and the inter-laboratory work order (Figure 7.4) Samples will be logged into a bound logbook documenting information needed for tracking. (Figure 7.5)

The Sample Custodian then removes sample tags from sample containers and adds them to the project file. A Sample Control Record (Figure 7.6) is filled out for each location at which samples will be stored (Volatiles in the locked refrigerators for volatiles, pesticides/semivolatiles/inorganics in the locked walk-in refrigerator). Samples will be stored in the appropriate location, and the Sample Control Record remains on a notebook by the correct storage location.

The Sample Custodian then fills out the Laboratory Work Order Form (which will be incorporated into the appropriate sample tracking file) with the holding dates, QC requirements, and deliverables deadlines, attaches a copy of the custody sheets and distributes a copy of the entire set to the appropriate personnel.

Finally, the Sample Custodian assembles all paperwork in the project file in the sequence specified in the standard project file inventory. Typically, these documents will include the airbill, chain-of-custody record, UPS records of sample cooler return, the sample tags and cooler seals, and the photocopy and carbon copies of the custody sheets.

Figure 7.4 Laboratory Work Order

Southwest Research Institute

Laboratory Work Order

Serial Number 4733

VTSR: 24 November 93 Time: 08:00

Project: 01-0000-000
Case #: 12345
SDG: XX111
Client: SOUTHWEST RESEARCH

Manager: ANYONE
Logged in by: MORIN
Checked by: _____

System ID	Customer ID	CED	Matrix	Required Analyses
38125	XX111	11/23/93	LOW WATER	BN00, EX00, PE00, VO00
38126	XX222	11/23/93	LOW WATER	BN00, EX00, PE00, VO00
38127	XX333	11/23/93	LOW WATER	BN00, EX00, PE00, VO00

Samples: 3

CASE NOT COMPLETE AS PER CHAIN OF CUSTODY.

COOLER TEMP IS 4.0°C.

SAMPLE ID XX111 IS THE SAMPLER SPECIFIED MISMATCH.

TAT IS 35 DAYS FROM LAST SAMPLE RECEIVED AT LAB.

Date must be to QA..... 24 December 93
Absolute Latest Date..... 28 December 93

Samples Received to Date: 003
Turnaround time is 35 Days from VTSR.

Date Printed: 24 November 93 Time Printed: 09:12:08

Work Orders in this SDG:

4733

* - Indicates work group contains an MS/MSD

Page Number: 001

Figure 7.5 Sample Log-in Book

SAMPLE LOG-IN BOOK

REF	SOURCE	MATRIX	SAMPLE ID	DATE/TIME	FOL	ASSY
311 P	EPA	L SOIL	SMO 167	11-06-93 / 1100	01-4657-070	PADO
			SMO 168			
			SMO 169			
			SMO 170			
			SMO 171			
			SMO 172			
		L WASTE	SMO 173			
		L SOIL	SMO 174			
			SMO 175			
			SMO 176			
		L WASTE	SMO 177			
		L SOIL	SMO 178			
			SMO 179			
			SMO 180			
			SMO 181			
311 P	EPA	L SOIL	SMO 182	11-06-93 / 1100	01-4657-076	PADO
			SMO 183			
			SMO 184			
			SMO 185			
			SMO 186			
			SMO 187			
			SMO 188			
			SMO 189			
			SMO 190			
			SMO 191			
311 P	EPA	L WASTE	SV 9173	11-06-93 / 1100	01-4657-072	PADO
			SV 9175			
			SV 9176			
			SV 9177			
			SV 9178			
			SV 9179			
			SV 9180			
			SV 9181			
311 P	HEB	PRODUCE	BE-A-1359	11-08-93 / 1115	01-2357-[illegible]	PCTT
			EW-A-1359			
			CD-A-1359			
			CC-A-1359			
			PE-A-1359			
			RO-A-1359			
			RP-A-1359			

Figure 7.6 Sample Control Record

SwRI Sample Control Record (BNA, EXT, PEST)

Required Analysis: BN00, EX00, PE00 Refrigerator: 13

Project: 01-0000-000
Case: 12345
SDG: XX111
Work Order #: 4733
Logged in: 11/24/93 MORIN

				DATE							
				TIME							
				BY							
				Analysis							Disposal
System ID	Customer ID	Matrix	Cont. Desc	Quantity	Lab Out	In	Lab Out	In	Lab Out	In	OUT
36125	XX111	LOW WATER									
36126	XX222	LOW WATER									
36127	XX333	LOW WATER									

For Transfer of Custody....note below on this form the date, from whom, to whom, and reason for the CoC.

7.5 Sample and Extract Storage

7.5.1 The following procedures shall be used to control the storage of samples:

1. Immediately after sample receipt and log-in, samples shall be stored in a secure area designated for samples. The samples shall be stored in their original containers unless damaged. The client is contacted regarding any problems. Damaged samples shall be disposed of in an appropriate manner and this disposal is documented.

   The following areas are designated for storage of samples:

   - Volatile soil and water samples shall be stored in a locked refrigerator/freezer located in the volatiles area.
   - Soil and water samples for semivolatiles/pesticide/inorganics analysis shall be kept in locked walk-in coolers, as designated.

   Under no circumstances will samples be stored in the same refrigerator/freezer as standards or quality control samples. The storage areas shall be kept secure at all times. The sample custodian shall control access to the storage area through custody documentation. Control of keys is documented by a signature list.

2. Whenever samples are transferred into or out of storage, the transfer shall be documented on a SwRI Sample Control Record sheet. (Ordinarily, samples will only be removed once for the purpose of extraction, digestion, or analysis.)

3. The location of stored samples shall be recorded on the Sample Control Record.

4. Volatile samples shall be stored separately from other samples.

5. The temperatures of the refrigerators and freezers shall be recorded on regular work days.

6. Samples shall be disposed of according to Standard Disposal Procedures. Samples may be disposed of 60 days after data submission of the data package. (See Standard Operating Procedures for sample and extract storage.)

7.5.2 The following procedures shall be used to control the storage of sample extracts:

1. Sample extracts shall be preserved in bottles/vials with Teflon-lined septa and maintained at 4°C. Pesticide and semivolatile extracts will be stored in a locked refrigerator devoted exclusively to the storage of sample extracts. Sample extracts shall not be stored with standards or extracts from industrial clients.

2. Immediately upon generating the sample extract, it is stored in the appropriate location and a Sample Control Record is initiated for the extracts for that project. Each Sample Control Record is used to record only samples from that particular project. Extracts remain in locked sample storage until removed for analysis.

3. Whenever sample extracts are removed from the storage area, this is documented on a Sample Control Record. After an extract has been removed for storage by an analyst, the analyst is responsible for the custody of the sample. Each analyst must return the extracts to the storage area before the end of the working day. In the event that samples or sample extracts shall be transferred from one analyst to another, this fact must be documented on a Sample Control Record documenting change of custody. If the extract is used in entirety, this shall be documented on the Sample Control Record.

4. After final submission of analytical data for the particular project, all sample extracts for the project are boxed (along with the VOA samples), and stored in a locked refrigerator. This fact is also documented on the appropriate Sample Control Records.

5. Within 60 days after data submission, SwRI will dispose of samples and sample extracts.

7.6 Preventing Sample Contamination

Preventing contamination of samples, sample extracts, and standards is a continuous process starting with the log-in and storage and continuing throughout all stages of analysis.

7.6.1 Sample Log-in and Storage

Sample containers shall be checked for damage or leakage upon arrival. Any sample showing signs of damage or contamination is disposed of in an appropriate manner. This is documented, the client is contacted, and the issue is discussed in the project narrative or report.

Soil and water samples shall be stored in a locked walk-in refrigerator devoted exclusively for the storage of these samples.

Standards shall not stored with samples.

7.6.2 Sample Extraction for Organic Analysis

To keep sample contamination to a minimum during all processes, the following laboratory procedures are followed:

1. Extraction glassware used for extraction of low level samples is kept separately from glassware used for extraction of higher level samples.

2. Phthalate contamination is minimized by avoiding the use of plastics in the organic extraction laboratory.

3. All extraction solvents will be pesticide grade or equivalent.

4. Cleaning of laboratory glassware is done according to standard operating procedures.

7.6.3 Storage of Sample Extracts

Pesticide and semivolatile extracts generated from the extraction of samples shall be kept in a locked refrigerator specifically assigned to the storage of these samples. Separate locations shall be assigned to the storage of standards and of extracts from other industrial clients.

7.7 Security for Laboratory and Samples

The National Enforcement Investigations Center (NEIC) of EPA defines custody of evidence in the following ways:

1. It is in your actual possession, or

2. It is in your view, after being in your physical possession, or

3. It was in your possession and then you locked it up or sealed it up to prevent tampering, or

4. It is in a secure area.

To satisfy these custody provisions, the following standard operating procedures are implemented by SwRI Chemistry and Chemical Engineering Division:

1. Samples and extracts shall be stored in a secure area.

2. Access to the laboratory in the main building (Bldg 70) is through a monitored reception area, and all exit doors shall be auto-locking. All other buildings follow the same security procedures. All SwRI employees must wear a picture identification badge. Any person in the lab without an employee badge or a visitor identification badge is escorted to the reception area.

3. All personnel entering or leaving the building outside of regular working hours will be required to sign-in, documenting time of arrival and/or departure.

4. Visitors must sign-in the reception area and be escorted while in the laboratory.

5. Refrigerators, freezers, and other sample storage areas will be kept locked.

6. Only the designated sample custodian and designated personnel have keys to the sample preparation or analysis.

7. Samples and extracts remain in locked sample storage until removed for sample preparation or analysis.

8. All transfers of samples and extracts into and out of storage or from one analyst to another shall be documented on a Sample Control Sheet.

9. These internal custody records will be maintained by the Sample Custodian.

10. After a sample or extract has been removed from storage by the analyst, the analyst is responsible for the custody of the sample. Each analyst must return the samples or extract to the storage area before the end of the working day. If the extract is used in entirety, this shall be documented on the Sample Control Record.

7.8 Traceability of Standards

All standards used for analysis of samples will be made up from neat standards or solutions provided by an outside source traceable to the NIST.

All neat standards shall be kept in a refrigerator specifically assigned to the storage of such standards. Only pesticide grade or equivalent solvents will be used to prepare working standards.

All standards will be stored in designated refrigerators assigned exclusively to the storage of such standards. If indicated, analytical standards shall be prepared within the laboratory hood.

The preparation of all standards shall be documented in a laboratory notebook.

All standards labels must contain as a minimum the following information:

1. Standard identification information (i.e., components, concentration, etc)
2. Date prepared
3. Expiration date
4. Initials of person preparing standard
5. Solvent(s) used in preparation

If standards will be stored in noncalibrated glassware, volumes shall be marked and monitored.

7.9 Maintaining Instrument Records and Logbooks

Each instrument is assigned a specific log book in which the information shall be recorded as shown in section 6.4.

Major maintenance for all instruments is recorded in a separate logbook.

All documentation in logbooks and other documents shall be as shown in Section 6.4.

7.10 Analysis Work Order Form

An analysis work order form (Figure 7.4) will be provided to each analyst which indicates the type of analysis and the specific parameters to be sought. The sample identifier is placed on the form to minimize the risk of the analyst assigning an analytical result to the wrong sample. Other items noted on the work order form will be the date of receipt, collection end date, holding time requirements, deliverables requirements, and the QC required for the project.

7.10.1 Data Reporting Forms

Analytical results shall be reported by the analyst who is assigned responsibility for these specific parameter(s). Included on the data will be sample identification, analytical results for blanks and samples, quality control results including matrix spikes, matrix spike duplicates, date of analysis, and signature of analyst. All supporting material for the analytical results such as strip charts, computer printouts, and copies of laboratory notebooks will be submitted with data for archive requirements.

The analytical coordinator or his/her designee reviews the data reporting form for the quality of the data. The data package then is submitted to the QA/QC officer for required QA/QC validation, narrative construction identifying the problems incurred and construction of the data package. The data is then submitted to the analytical coordinator for final review. After this review, any remaining deficiencies shall be corrected, the project manager is notified for a review of the data to be submitted, and the data is made a part of the permanent project file.

7.10.2 *Maintenance of Records*

All records will be kept on file for the duration of the project in the central files at the San Antonio laboratory. Upon completion of the project, the files shall be transferred to inactive status and maintained for a minimum of two years.

7.10.3 *Data Evaluation*

All data generated by this laboratory will undergo a thorough review and validation prior to their release to the sponsor. The review process will be specified in the work plan and will follow the guidelines delineated in Section 8 of this document.

7.11 Standard Operating Procedures

The standard procedures listed in this document and any procedures supplied by the sponsor will be incorporated into the work plan quality assurance program by reference. Additional standard operating procedures shall be prepared by the analytical coordinator to describe routine tasks which shall be delegated and/or performed frequently.

7.12 Study Planning and Design

If the data are to be useful to the client, then the studies must be designed in a manner that will provide valid defensible results. Considerations in this regard include:

- selection of numbers of samples, locations of samples and acceptable conditions for sample collection,
- sampling methodology and analytical methodology used,
- selection of parameters for study and of data to be measured concurrently,
- inclusion of quality control samples, including field blanks and spikes, samples for analysis by an alternative laboratory, and others as appropriate,
- frequency and depth of performance and systems audits,
- statistical soundness in terms of the ability of the plan to address the questions under study.

All of these factors should be considered in developing the work plan in order to allow the underlying questions to be answered in a timely and efficient manner.

DATA PROCESSING

REV 6

Copy No. 33

Approvals:

Division 01 Quality Assurance		Date:	11/30/93
Division 01 Management		Date:	11/30/93
SwRI QA Management	L.C. Silovich	Date:	12/1/93

8. DATA PROCESSING

Each work plan will describe procedures to collect, validate, store, transfer, and reduce all data generated. General procedures are given below.

8.1 Collection

The Analytical Supervisor will prepare the data for the recording of data generated on each program. Each analyst will provide the analytical data pertinent to the analysis being performed and will execute these upon completion of the analysis. The analyst will be responsible for reviewing the data as it is generated to ensure that the results shall be reasonable and interpretable and that all information required for recalculation of the results is available. The analytical data sheets, chromatograms, absorbance traces, or spectra, and supporting information will be supplied to the analytical coordinator for review.

8.2 Validation

The Analytical Supervisor for each analytical task will perform a review of all data. This review will consist of the following elements: review of analytical program, examination of results, verification of results to insure the QC requested by the client. The data is then validated by the QC validator who is independent of the actual production of the analytical results.

8.2.1 Review of Analytical Program

The Analytical Supervisor will review each set of results to ensure that the required program elements, including method blanks, surrogate spikes, and QC samples, have been accomplished according to the project workplan. The supporting data will be reviewed to ensure that analyses were performed under the correct conditions and that all required procedural steps were performed.

8.2.2 Examination of Results

The Analytical Supervisor will examine the results obtained along with the pertinent chromatograms, spectra, or absorption traces to ensure that the results shall be reasonable and that the analyst has interpreted the results correctly. Any unusual or unexpected results will be reviewed and a resolution will be made as to whether the analysis should be repeated or the result recalculated.

8.2.3 Recalculation of Results

The Analytical Supervisor will selectively recalculate results to ensure that the calculation was performed properly. Emphasis will be placed on those results showing a significant concentration of one or more of the analytes of interest. As a minimum, one set of calculations will be checked for each batch of samples analyzed.

8.3 Data Storage

The QA/QC officer will have the responsibility for the control and protection of archived data. Where the project deals with material of a sensitive or confidential nature, measures will be included in the work plan to ensure that access is limited to those individuals who have agreed to be bound by terms of the confidentiality agreement and who have a need for the information.

8.4 Data Transfer

Following the analyst review, the verified data will be transferred from the analytical data sheets to reporting forms. The analyst will be responsible for ensuring that all required information is provided for validation purposes. All data will be verified against the analytical data sheets, and the completed forms will be reviewed by the Project Manager or his designee prior to submission of the results to the sponsor.

8.5 Data Reduction

Data reduction will be carried out using prescribed documented techniques. Any statistical summaries of the data will be presented in such a manner that the reviewer can easily judge the validity of the procedure and of any conclusions drawn. Assumptions made in performing any statistical analyses will be clearly stated, as will confidence/ significance levels used for any tests of hypotheses. Data summaries will be checked against the raw data for consistency and summary statistics recalculated in the event of doubt.

DATA QUALITY ASSESSMENT

REV 6

Copy No. 33

Approvals:

Division 01 Quality Assurance	Lo Ann Bazzel	Date: 11/30/93
Division 01 Management	[illegible]	Date: 11/30/93
SwRI QA Management	L.C. Silovich	Date: 12/1/93

9. DATA QUALITY ASSESSMENT

The data report shall include the accuracy and precision data designed during the project. Upon request, accuracy and precision will be performed from analysis of spiked samples, replicate samples, or other designated QC requests. If the samples will be collected by SwRI, a statement will be included assessing the degree to which the samples represent the entity being sampled. (e.g. 10% of sampling).

CORRECTIVE ACTION

REV 7

Copy No. 33

Approvals:

Division 01 Quality Assurance		Date: 11/30/93
Division 01 Management		Date: 11/30/93
SwRI QA Management	S.C. Silovich	Date: 12/1/93

10. CORRECTIVE ACTION

The ultimate responsibility for maintaining quality of the analyses and sampling lies with the management of the department and division. The routine operation of the quality assurance/quality control program, however, falls upon the analysts, Laboratory Supervisors, Analytical, and Sampling Coordinators, QA Auditor, Project Managers, and the QA/QC Officer. Major responsibilities are discussed in Section 4.2 Evaluation of the QA/QC Program is discussed in Section 4.5. QA/QC reporting is also discussed in Section 4.

It is the responsibility of the project personnel to identify perceived problems which may affect data quality and to report these problems both to their project supervisor and to their quality team contact. If the supervisor regards the problem as serious enough to potentially impact data quality, he/she will initiate appropriate corrective action. Corrective action relates to the overall QA management scheme: who is responsible for taking corrective actions; when are corrective actions to be taken; and who follows-up to see that corrective actions have been taken and that they have produced the desired results. Results from the following QA activities may indicate a need for corrective action:

1. Performance audits
2. Systems audits
3. Inter-laboratory comparison studies
4. Failure to adhere to a QA Program or Project Plan or to a Standard Operating Procedure

Non-compliances will be minimized through specification (in the work plan or operating protocols) and implementation of internal controls prior to an adverse project impact. Each measurement system shall have limits to establish when corrective action must be taken. A procedure shall be established for each measurement system to show what corrective action will be followed when established limits for this procedures have been exceeded. The level within the organization that is responsible for taking corrective action shall also be specified.

The process involves corrective actions to the areas involved and management. Responses to corrective actions indicating the cause of the problem and corrective actions to prevent recurrence shall be submitted for inclusion in the final report. A copy of the corrective action form is presented in Figure 10.1

Figure 10.1 Corrective Action Form

CAR #: _____

Southwest Research Institute
Division 01

CORRECTIVE ACTION REQUEST

Discrepancy:

Suggested Corrective Action to Prevent Recurrence:

Initiated by: _____ **Date:** _____

Area Supervisor Responsible: _____

Response Due Date: _____

(To be filled out by Area Supervisor)

Corrective Action Taken:

Date for Completion of Corrective Action: _____

Supervisor: _____ **Date:** _____

QAU Verified: _____ **Date:** _____

BIBLIOGRAPHY

REV 6

Copy No. 33

Approvals:

Division 01 Quality Assurance		Date: 11/30/93
Division 01 Management		Date: 11/30/93
SwRI QA Management	L.C. Silhavil	Date: 12/1/93

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RADIOACTIVE MATERIAL LICENSE

Texas Department of Health
BUREAU OF RADIATION CONTROL

032997

RADIOACTIVE MATERIAL LICENSE

Pursuant to the Texas Radiation Control Act and Texas Health Department regulations on radiation, and in reliance on statements and representations heretofore made by the licensee, a license is hereby issued authorizing the licensee to receive, acquire, possess and transfer radioactive material listed below; and to use such radioactive material for the purpose(s) and at the place(s) designated below. This license is subject to all applicable rules, regulations and orders of the Texas Department of Health now or hereafter in effect and to any conditions specified below.

LICENSEE

1. Name: Southwest Research Institute
   Attn: Frank A. Iddings
2. Address: P. O. Drawer 28510
   San Antonio, Texas 78284

This license is issued pursuant to and in accordance with a letter
Dated: February 4, 1993
Signed by: Frank A. Iddings

3. License Number	Amendment Number
L00775	45

PREVIOUS AMENDMENTS ARE VOID

4. Expiration Date: December 31, 1993

RADIOACTIVE MATERIAL AUTHORIZED

5. Radioisotope	6. Form of Material	7. Maximum Activity*	8. Authorized Use
A. Any radioactive material with Atomic number less than 84.	A. Any.	A. 500 mCi of any single radioisotope. Total: 25 Ci	A. Research and Development.
B. H-3	B. Any.	B. 5 Ci	B. Research and Development.
C. Cr-51	C. Any.	C. 500 Ci	C. Research and Development.
D. Mn-54	D. Any.	D. 200 Ci	D. Research and Development.
E. Fe-55/ Fe-59	E. Any.	E. 20 Ci total.	E. Research and Development.
F. Co-58	F. Any.	F. 600 Ci	F. Research and Development.
G. Co-60	G. Any.	G. 100 Ci	G. Research and Development.
H. Ni-63	H. Any.	H. 1 Ci	H. Research and Development.
I. Po-210	I. Sealed sources.	I. 500 Ci	I. Research and Development.
J. Ra-226	J. Sealed sources.	J. 100 μCi	J. Research and Development.
K. Am-241	K. Any.	K. 1 Ci	K. Research and Development.
L. U-238 and its activation products.	L. Any.	L. 50 mgms	L. Research and Development.

*Ci-Curie mCi-Millicurie μCi-Microcurie

RADIOACTIVE MATERIAL LICENSE

LICENSE NUMBER	AMENDMENT NUMBER
L00775	45

5. Radioisotope	6. Form of Material	7. Maximum Activity	8. Authorized Use
M. Np-237	M. Any.	M. 50 mgms	M. Research and Development.
N. U-238	N. Any.	N. 100 kgms	N. Research and Development.
O. Cs-137	O. Sealed source (NC Model 850233).	O. 1 Ci	O. Survey instrument calibration.
P. Co-60	P. Sealed sources.	P. 100,000 Ci	P. Irradiation of materials and lower animals for research and development. For receipt and storage in the licensee's Radiation Effects Facility and for transfer to authorized recipients.
Q. Am-241	Q. Sealed source (GI Model VD[HP]).	Q. 1 source of 25 mCi	Q. Research and development of well logging instrumentation.
R. Co-60	R. Sealed source (GI Model R-6-A).	R. 2 sources of 100 Ci	R. Industrial radiography using Radionics Model P-60-100-2 exposure device and source exchange using Gamma Industries Model C-8 source changer.
S. Ir-192	S. Sealed source (GI Model R-6-A).	S. 3 sources of 50 Ci each.	S. Industrial radiography using Radionics Model P-60-100-2 exposure device and source exchange using Gamma Industries Model C-10 source changer.
T. Ir-192	T. Sealed source (GI Model A-2-A).	T. 2 sources of 100 Ci each.	T. Industrial radiography using Gamma Industries Model Gamma Century SA exposure device and source exchange using Gamma Industries Model C-10 source changer.
U. Cs-137	U. Sealed source (TN Model 57157C).	U. 2 sources of 500 mCi each.	U. Density measurement using Texas Nuclear Model 5202 source holder.
V. Co-60	V. Sealed sources.	V. 75,000 Ci	V. Storage in DOT approved shipping containers.

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5. Radioisotope	6. Form of Material	7. Maximum Activity	8. Authorized Use
W. Pu-238	W. Sealed source (A/S Model PPC2, PPC4, PPC5, or PPC-X).	W. 1 source of 30 mCi	W. X-ray fluorescence analysis in Columbia Scientific Model 700 Minilab analyzer.
X. Cf-252	X. Sealed source (IPL foil)	X. 1 source of 100 µCi	X. Research and development of a time-of-flight mass spectrometer.
Y. Any radioactive material.	Y. Any.	Y. 1 µCi/gm of total activity per sample.	Y. Possession as a consequence of laboratory analysis of hazardous material samples.
Z. Cs-137	Z. Sealed sources.	Z. 60,000 Ci	Z. For receipt and storage in storage in the licensee's Radiation Effects Facility and for transfer to authorized recipients.
AA. Any radioactive material.	AA. Any.	AA. 1 µCi/gm of total activity per sample.	AA. Alpha and gamma ray spectroscopy of soils.
AB. Any radioactive material.	AB. Any.	AB. As acquired incident to decontamination.	AB. Possession incident to decontamination of sealed sources.
AC. Ir-192	AC. Any.	AC. 50 Ci total.	AC. Possession incident to failure and metallurgical analysis of sealed sources.

9. Radioactive material shall be stored and used only at:

Site Number	Location
000	San Antonio - 6220 Culebra Road

10. Radioactive material authorized in Parts Q, R, S, T and Y of Conditions 5, 6, 7 and 8 shall also be used at temporary sites throughout Texas.

11. The licensee shall comply with the provisions of Parts 11, 12, 13, 21, 22, 31 and 41 of the Texas Regulations for Control of Radiation (TRCR).

12. Radioactive material shall only be used by, or under the supervision of, individuals designated by the Radiological Health and Safety Committee, David H. Wood, Ph.D., Chairman.

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13. The licensee is hereby exempted from TRCR Part 36, provided that all downhole operations authorized by this license are accomplished in cased wells and at depths of 200 feet or less. Notwithstanding the above exemption, the licensee shall comply with the provisions of TRCR 36.4 and 36.501.

14. The individual designated to perform the functions of Radiation Safety Officer (RSO) for activities covered by this license is Frank A. Iddings.

15. Radioactive material used for industrial radiography shall only be used by David G. Cadena, Jr. (#1054), Edward H. Ruescher (#0680), James J. McArdle III (#1064), Simon S. Marin (#1065) and/or Frank A. Iddings (#1263) or by radiographer trainee(s) under the direct supervision and in the physical presence of the radiographer trainer(s) designated in Condition 16 below.

16. On the job training for industrial radiographer trainees shall be under the direct personal supervision of David G. Cadena, Jr., Edward H. Ruescher, Simon S. Marin and/or Frank A. Iddings.

17. Sealed sources containing radioactive material shall not be opened.

18. Sealed sources of radioactive material, nickel-63 foil, and/or plated alpha emitting sources shall be tested for leakage and/or contamination in accordance with the provisions of TRCR 11.7.

19. Notwithstanding TRCR 11.7(c), the interval of testing for leakage of the sources described in Part U of Conditions 5, 6, 7 and 8 shall not exceed three years.

20. Radiation survey instruments shall be calibrated at intervals not to exceed three months by persons licensed by the Agency, another Agreement State, or by the United States Nuclear Regulatory Commission (NRC).

21. The licensee is authorized to perform survey instrument calibration in accordance with procedures dated November 1, 1982.

22. The licensee is authorized to perform tests for leakage and/or contamination on teletherapy sources for customers and on their own sealed sources. Such tests shall be capable of detecting 0.005 μCi of contamination on the test sample and the results of such tests shall be provided to the customer in terms of μCi.

23. In lieu of using the conventional radiation caution colors (magenta or purple on yellow background) as provided in the TRCR 21.203(a), the licensee is hereby authorized to label detector cells and cell baths, containing radioactive material and used in gas chromatography devices, with conspicuously etched or stamped radiation caution symbols without a color requirement.

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24. The licensee is authorized to receive, possess and use sealed sources of iridium-192 (Ir-192) or cobalt-60 (Co-60) where the radioactivity exceeds the maximum amount of radioactivity specified in Condition 7 of this license provided:

    A. Such possession does not exceed the quantity per source specified in Condition 7 by more than 20% for Ir-192 or 10% for Co-60.
    B. records of the licensee show that no more than the maximum amount of radioactivity per source specified in Condition 7 of the license was ordered from the supplier or transferor of the radioactive material.
    C. the levels of radiation from radiographic exposure devices and storage containers do not exceed those specified in the TRCR 31.11.

25. Individuals involved in operations which use, during any 24 hour period, more than 50 mCi of iodine-125 (I-125) and/or iodine-131 (I-131) or unvented laboratory operations involving 10 mCi of I-125 and/or I-131 in a noncontained form shall have bioassays performed within one week or if the use of I-125 and/or I-131 is on a continual basis, bioassays shall be performed once every two weeks. Records of the bioassays shall be maintained for inspection by the Agency and the action points listed below shall be observed.

    A. Whenever the thyroid burden at the time of measurement exceeds 0.12 µCi of I-125 or 0.04 µCi of I-131, the following actions shall be taken:
        (1) An investigation of the operations involved, including ventilation surveys shall be carried out to determine the causes of exposure and to evaluate the potential for further exposures.
        (2) If the investigation indicates that further work in the area might result in the exposure of a worker to concentrations that are excessive, the licensee shall restrict the worker from further exposure until the source of exposure is discovered and corrected.
        (3) Corrective actions that will eliminate or lower the potential for further exposures shall be implemented.
        (4) A repeat bioassay shall be taken within one week of the previous measurement in order to confirm the effectiveness of the corrective action taken or to verify internal radioiodines present.
        (5) Reports or notification shall be provided as required by TRCR 21.405 and 21.408.

    B. If the thyroid burden at any time exceeds 0.5 µCi of I-125 or 0.14 µCi of I-131, the following actions shall be taken:
        (1) Prevent the individual from any further handling of I-125 or I-131 until the thyroid burden is below the above limits.
        (2) Carry out all steps described above.
        (3) As soon as possible, refer the case to appropriate medical consultation for recommendations regarding therapeutic procedures that may be carried out to accelerate removal of radioactive iodine from the body. This should be done within two to three hours after exposure when the time of exposure is known so that any prescribed thyroid blocking agent would be effective.
        (4) Carry out repeated measurements at approximately one-week intervals at least until the thyroid burden is less than 0.12 µCi of I-125 or 0.04 µCi of I-131.

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26. Individuals involved in operations which utilize, at any one time, more than 100 mCi of hydrogen-3 (tritium) in a noncontained form, other than metallic foil, shall have bioassays performed within one week following a single operation and at weekly intervals for continuing operations.

27. The licensee shall not use radioactive material in or on human beings or in any field application where such activity is released except as provided otherwise by specific condition of this license.

28. Each person using radioactive material shall be provided with a copy of the licensee's Radiological Health and Safety Manual. Proposed changes in or additions to the licensee's manual shall be submitted to the Agency for approval before being incorporated into this document.

29. The licensee is authorized to receive and load teletherapy sources into customer's teletherapy source holders.

30. A. Tritium shall not be used in such a manner as to cause any individual to receive a radiation exposure such that urinary excretion rates exceed 28 µCi of tritium per liter when averaged over a calendar quarter.
    B. Urinalysis shall be performed at weekly intervals on all individuals who work in the restricted areas of facilities in which tritium is used. If the average concentration of tritium in urine for any single individual during a calendar quarter is less than 10 µCi per liter, urinalysis may be performed on that individual at monthly intervals for the following calendar quarter and may continue at monthly intervals so long as the average concentration in the calendar quarter remains below 10 µCi per liter. The urine specimen shall be collected on the same day of the week if possible.
    C. A report of an average concentration in excess of the limit specified in (A) above for any individual shall be filed, in writing, within 30 days of the end of the calendar quarter with the Agency. The report shall contain the results of all urinalyses for the individual during the calendar quarter, the cause of the excessive concentrations, and the corrective steps taken or planned to assure against a recurrence.
    D. Any single urinalysis which discloses a concentration of greater than 50 µCi per liter shall be reported, in writing, within seven days of the licensee's receipt of the results.

31. Notwithstanding TRCR 31.30(a), the licensee is hereby exempted from conducting a quarterly audit of each radiographer listed in Condition 15 provided that an annual review of the conditions of this license, the licensee's operating and emergency procedures and the TRCR is conducted with each radiographer. Records documenting this review shall be maintained for inspection by the Agency.

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32. Notwithstanding TRCR 31.18(b), the licensee is hereby exempted from a quarterly inspection and maintenance program provided the inspections required under TRCR 31.18(a) are conducted prior to each use of radiographic equipment. Records documenting such inspections shall be maintained for inspection by the Agency.

33. The licensee is authorized to disassemble and clean the licensee's nickel-63 electron capture detector cells in accordance with procedures submitted February 1, 1993.

34. Except as specifically provided otherwise by this license, the licensee shall possess and use the radioactive material authorized by this license in accordance with statements, representations, and procedures contained in the following:

    application dated November 16, 1983,
    Radiological Health and Safety Manual as revised November 1, 1982,
    letters dated February 8, 1984, June 21, 1985, July 19, 1985,
    April 8, 1986, December 1, 1988, December 22, 1988,
    February 27, 1989, January 12, 1990, May 8, 1990,
    October 30, 1990, June 18, 1991, August 21, 1991,
    February 14, 1992, April 22, 1992, December 7, 1992,
    December 17, 1992, January 13, 1993, January 14, 1993,
    January 28, 1993 and February 4, 1993, and
    information received February 1, 1993.

    The TRCR shall prevail over statements contained in the above documents unless such statements are more restrictive than the regulations.

FRH:mr

FOR THE TEXAS DEPARTMENT OF HEALTH

Date February 8, 1993

Administrator, Licensing Branch