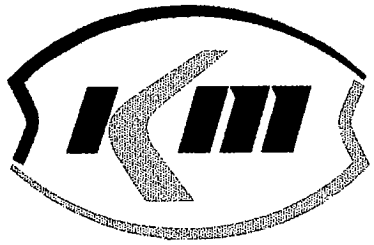
Kerr McGee Chemical Corporation



Henderson
Phase II

Work Plan &
Health and Safety Plan

October 10, 1996

KERR-McGEE CHEMICAL CORPORATION
POST OFFICE BOX 55 • HENDERSON, NEVADA 89009

May 10, 1996

Mr. Allan Biaggi
Nevada Division of Environmental Protection
333 West Nye Lane
Carson City, Nevada 89710

Subject: Phase II Work Plan

Dear Mr. Biaggi:

Attached for your review are four copies of Kerr-McGee Chemical Corporation's (KMCC's) Phase II Work Plan, which supplements KMCC's response to the August 15, 1994, Letter of Understanding (LOU), between KMCC and the Nevada Division of Environmental Protection (NDEP). This attachment contains information related to field sampling requested by NDEP in the LOU. KMCC's response to the LOU is submitted under separate cover, concurrently with this document.

It is KMCC's intent that the Common Areas Public Involvement Plan, included in the BMI Common Areas Phase II Work Plan, suffice for the KMCC public involvement requirements of KMCC's Phase II Work Plan. However, KMCC recognizes its responsibility for placing KMCC specific documents in the document repository and will do this.

KMCC would appreciate having NDEP review this Work Plan prior to our finalizing the Consent Agreement. We believe that this will help expedite the overall process by focusing the Consent Agreement on an already defined scope of work. Your assistance in this matter is appreciated.

If you have any questions, please feel free to contact me at (702) 651-2234.

Sincerely,

Susan Crowley
Staff Environmental Specialist

smc\WPCOV.WPD
cc: PSCorbett
RANapier
RHJones
PRDemps

THE KERR-McGEE CHEMICAL CORPORATION
HENDERSON, NEVADA FACILITY

PHASE II
WORK PLAN

October 14, 1996

Prepared for:
Nevada Division of Environmental Protection

Prepared by:
Kerr-McGee Chemical Corporation

EXECUTIVE SUMMARY

Kerr-McGee Chemical Corporation (KMCC) plans to conduct field and analytical work at KMCC's Henderson, Nevada facility (Facility) as part of the Phase II Environmental Conditions Assessment at the Basic Management Inc. Complex (BMI Complex). This Work Plan sets out the objectives and scope of the fieldwork and analyses to be performed. A report summarizing the results of the field work and analyses will be prepared and submitted to the Nevada Division of Environmental Protection (NDEP).

In April 1993, KMCC submitted to the NDEP, a Phase I Environmental Conditions Assessment report prepared by Kleinfelder, Inc. (Report) on behalf of KMCC. In June 1993, and again in August 1993, representatives of NDEP and KMCC met to discuss additional information and data needs identified by NDEP based on their review of the Report. A Letter of Understanding (Attachment A) summarizes the agreements reached at those meetings.

Three types of action items are included in the Letter of Understanding: (1) items that can be addressed by KMCC by providing additional existing information in a Written Response, (2) items requiring further field work by KMCC, and (3) items requiring further field work by the Henderson Industrial Site Steering Committee (HISSC). This Work Plan addresses only category (2). Category (1) and (3) will be addressed in separate documents.

CONTENTS

TABLE OF CONTENTS

ATTACHMENTS

TABLE OF CONTENTS (continued)

LISTS OF FIGURES, TABLES AND PLATES

Figures

Tables

TABLE OF CONTENTS (continued)

Plates

PHASE II

WORK PLAN

Section 1

Overview

1. OVERVIEW

The KMCC Site at Henderson, Nevada, occupies part of an industrial area known as the Basic Management Incorporated (BMI) complex (Complex). Figure 1 shows the location of the Facility. Plate 1 is a Facility map showing topographic features and the location of groundwater and recovery wells.

1.1 Site History

The Complex, including KMCC's property, is located in an unincorporated portion of Clark County, Nevada, and is completely surrounded by the City of Henderson. Originally sited and operated by the U.S. Government as a magnesium production facility, the Complex operated from August 1942 to November 1944 to support the war effort.

Following cessation of magnesium operations, a portion of the Complex was leased from the Government by Western Electrochemical Co. (WECCO) in 1945. By 1952, WECCO had purchased various portions of the Complex. In 1955, WECCO merged with American Potash and Chemical Company (AP & CC). In 1962, AP & CC purchased the current ammonium perchlorate plant, sodium perchlorate plant, and half of the sodium chlorate plant from the Government. KMCC acquired AP & CC by merger in 1967. Later, KMCC acquired the remainder of the sodium chlorate plant. In addition to the production of chemical oxidizers, the Facility also began production of manganese dioxide and boron-based products.

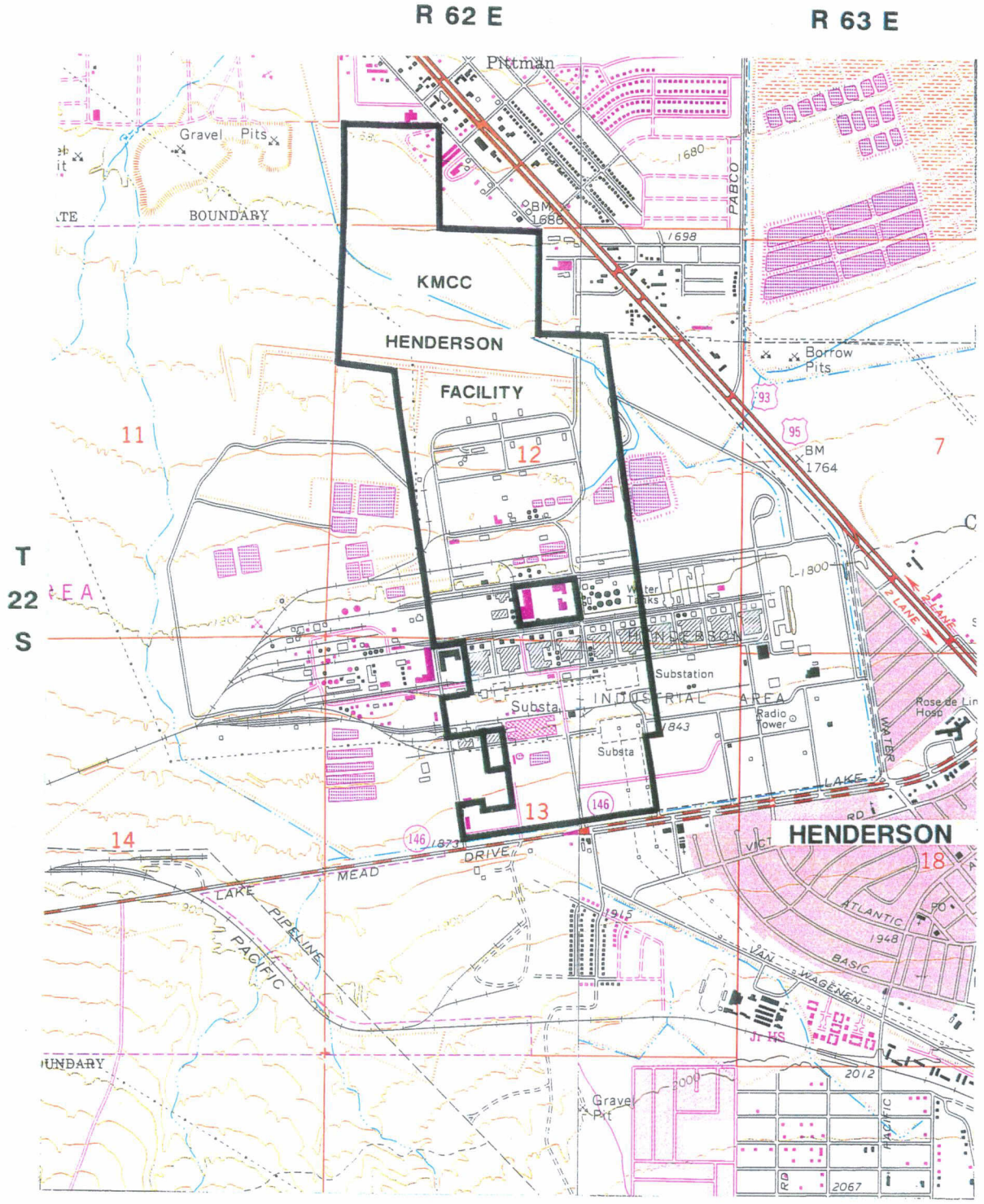
1.2 Environmental Conditions Assessment

On April 25, 1991, representatives from KMCC, Timet, Montrose, Chemstar and Stauffer/Pioneer (commonly known as the Henderson Industrial Site Steering Committee (HISSC)) entered into a consent agreement with the Nevada Department of Environmental Protection (NDEP) to conduct a Phase I Environmental Conditions Assessment (ECA).

On behalf of KMCC, Kleinfelder, Inc., a Las Vegas environmental contractor, prepared an ECA report (Report) covering current and historical waste management practices on KMCC property and submitted it to the NDEP in April 1993. NDEP reviewed the Report and subsequently met with representatives of KMCC on June 9 and August 20, 1993, to discuss issues which needed clarification or further study. The NDEP issued a Letter of Understanding (LOU) on August 15, 1994, summarizing the agreements reached

at the meeting (see Attachment A). The action items included in the LOU can be divided into three types: (1) items that can be addressed by KMCC by providing additional information, (2) items requiring further fieldwork by KMCC, and (3) items requiring further fieldwork by the HISSC.

Responses to (1) above are covered in a separate document titled "Responses to Letter of Understanding, Henderson, Nevada Facility, May 1996" with its subsequent revisions. Responses to (2) are contained in this Work Plan, and responses to (3) are included in the BMI Common Areas Work Plan.



SOURCE: U.S.G.S. LAS VEGAS SE / HENDERSON, NEVADA 7.5 MINUTE QUADRANGLES: 1967, 1970

N

SCALE

0 2000 FEET

NEVADA

QUADRANGLE LOCATION

FIGURE 1
Location of the KMCC Henderson, Nevada Facility

ECI

PHASE II

WORK PLAN

Section 2

Environmental Conditions

Investigation

2. ENVIRONMENTAL CONDITIONS INVESTIGATION

This Work Plan defines fieldwork at KMCC's Henderson, Nevada, facility to determine the nature and extent of soil and groundwater impacts related to past and present operations. This Work Plan has been specifically prepared to respond to items identified in the LOU that require field sampling to be done by KMCC.

2.1 Data Objectives

The objective of this Work Plan is to define the sample collection and analysis work as specified by the LOU. The results will be used to determine whether the locations discussed below require further assessment.

2.2 Sampling & Analysis

KMCC and the NDEP agreed that a Work Plan should be prepared to cover sampling work done at the Facility and submitted to NDEP as an Attachment to the Phase II Consent Agreement. The Data Collection and Quality Assurance Plan (DCQAP) portion of the Work Plan (Section 3) sets out the specific protocols that will be used for field data collection and analytical work related to this Work Plan.

2.2.1 Common Procedures and Laboratory Analysis

Soil samples analyzed for "soil pH" will be extracted with water and the pH of the resulting solution measured using SW-846 method 9045.

KMCC will analyze some soil samples for total metals by EPA methods 6010/7000 series. Tables 1b and 1c lists the compounds included in the 6010/7000 analytical slate. If specific metals for individual locations are selected, this will be listed in Table 2.

KMCC plans to use EPA Method 8240 (and possibly other EPA methods) when analysis for volatile organic compounds (VOC's) is specified. Table 1a lists the compounds included in the 8240 analytical slate.

KMCC plans to use EPA Method 8270 (and possibly other EPA methods) when analysis for semi-volatile organic compounds (SVOC's) is specified. Table 1f lists the compounds included in the 8270 analytical slate.

EPA Method 8015 will be used to look for the less volatile constituents of diesel. Table 1d lists compounds include in the 8015 analytical slate.

KMCC plans to use EPA Method 300 when analysis for nitrate is specified. Table 1e lists the compounds included in the 300 analytical slate.

2.2.2 Location and Number of Samples

The location and number of samples in each of the areas to be investigated was determined based on:

- the historical use of the area,
- the possible sources of contamination,
- the anticipated distribution of any contaminants possibly present in the areas,
- any visual indications of possible contamination,
- and KMCC's experience with environmental investigations in other areas of the facility.

Past environmental investigations at the site have shown that this method of selecting sampling locations and numbers has been successful in determining the presence or absence of contaminants.

For five of the eight areas to be investigated, visual indications or historical knowledge will be used to select sampling locations from the areas with the highest potential for contamination. For three areas, concentrations are expected to be fairly homogeneous throughout the areas and stratified random sampling approach is used. The use of stratified random sampling assures a more complete coverage of the area than would likely be obtained with a simple random sampling method. Once the number of samples to be collected was determined for the three areas, each of the relevant areas requiring sampling was divided into a number of blocks of approximately equal surface area. The number of blocks of each area was equal to the number of samples to be collected in the area. Each block was overlaid by a grid of 100 cells. Each cell from the grid was identified by a unique number pair (numbers between 0 and 99). The random cell location for the boring location within each block were selected using a random number generator. If the cell coordinates were outside the area to be sampled (since not all the

blocks were perfectly square), the cell coordinates were discarded and a next set of coordinates was generated.

Each of the selected locations for the eight areas are illustrated in Figures 2 through 5. The number of soil and water samples along with analyses are listed in Tables 2 and 3. Following total metal analysis (and other specified tests, see §2.3), the laboratory will retain the unused portion of the sample until instructed by KMCC to perform TCLP or samples disposal.

2.3 Specific Sampling Locations

2.3.1 "Trade Effluent" Settling Ponds, Open Area South of Ponds (LOU Items #1, 2)

The Trade Effluent (TE) Settling Ponds Area is located north of the ammonium perchlorate (AP) storage area and west of the existing ponds "East-WC" and "West-WC" ponds (see Figure 2, note that the TE Ponds are labeled "A" and the Open Area is labeled "B"). The TE ponds were operated by the U.S. Government from the Fall of 1942 to November 1944, as unlined storage impoundments for acid waste neutralized with caustic liquor. The waste was apparently evenly distributed in the ponds with no segregation of materials in different areas. Each TE pond had an area of approximately 20 acres and an average liquid depth of 7.5 feet. The TE ponds received some solid wastes between 1945 and 1979.

Portions of the TE Ponds Area have been utilized for other activities. KMCC constructed and operated a hazardous waste landfill in the western portion of the area between 1980 and 1983. This landfill was closed and capped in 1985 in accordance with RCRA interim status requirements and is currently under a post-closure monitoring program. In October 1988, lined surface impoundments "East-WC" and "West-WC" wereconstructed in the northeastern portion of the Trade Effluent Ponds area. These were permitted with NDEP and are currently in operation.

Accordingly, KMCC proposes to drill seven soil borings in the area unaffected by the closed landfill and ??? impoundments. Since little variation is expected in soil concentration over the pond area, the seven borings should provide an indication of the presence of possible contamination. The boring locations were selected based upon

criteria explained in §2.2.2. These locations are shown on Figure 2 (five in Area A and two in Area B). Each soil boring will be drilled to a total depth of ten feet. To characterize possible remnants of the neutralized aqueous waste conveyed to this area, soil samples will be collected from 0 - 1, 4 - 5, and 9 - 10 feet, and analyzed for total metals and soil pH (21 samples total). Based on EP-Tox results from the borings collected for "East WC" and "West WC," KMCC believes that this depth and these sampling intervals are adequate to characterize this area. Because there is no indication of organic material placement in the trade effluent pond area, organics will not be sampled or analyzed for.

2.3.2 Old P-2, P-3 Ponds (LOU Items #7, 8)

As part of the sodium chlorate (SC) process, surface impoundments were used to collect and concentrate dilute SC solutions. The concentrated solution was recycled from the ponds back into the process. Old P-2 and P-3 were lined ponds, used from 1978 to 1990 (see Figure 3) for this purpose. Old P-2 covered approximately 0.1 acres (4,400 sq. feet) and P-3 covered approximately 0.3 acres (13,000 sq. ft). Both ponds had single-layer synthetic liners. After the ponds were taken out of service, the liner, solids, and underlying soils were removed and disposed of at U. S. Ecology.

Eight shallow soil borings will be advanced with samples collected at a depth of 24-36 inches from P-3. Five shallow soil borings will be advanced with samples collected at the same depth from Old P-2 (locations are shown in Figure 4). A higher number of sampling locations per surface area were selected at Old P-2 and P-3 (when compared to the Trade Effluent Ponds) to take into account a possible failure of either pond liner which would have resulted in a localized area of contamination. Soil boring/sampling locations were selected based upon criteria explained in §2.2.2. Based upon the constituents in the aqueous stream conveyed to the pond, the soil samples from P-3 and Old P-2 will be analyzed for total chromium and soil pH. Because organics were not placed into the ponds, no sampling or analysis will be performed for organics.

2.3.3 AP-1, AP-2 and AP-3 Ponds (LOU Items #16, 17)

Five synthetically-lined surface impoundments (designated as AP-1 to AP-6, excepting AP-2) are a part of the AP process.

AP-1, AP-2 and AP-3 were placed in service in May 1974. AP-1 (double-lined) has a surface area of 0.32 acres and an approximate volume of 370,000 gallons (average depth of 3.5 feet). AP-1 is used to store AP laden filter cake and to recycle AP solution back to the process. AP-2 (single-lined) had a surface area of 0.32 acres and a volume of 400,000 gallons (average depth, 3.8 feet). AP-2 was also used to store filter cake and recycle AP solution. AP-2 was emptied and closed in late 1995. AP-3 (double-lined) has a surface area of 0.05 acres and an approximate volume of 65,000 gallons (average depth, 4.3 feet). AP-3 serves as a pump basin for liquids from AP-1. AP-4 (double-lined) has a surface area of 0.32 acres and an approximate volume of 411,000 gallons (average depth, 4.3 feet). AP-4 is used to concentrate a salt and AP solution which is subsequently returned to the AP process. AP-5 (double-lined) has a surface area of approximately 0.8 acres and an approximate volume of 1,810,000 gallons (average depth, 9 feet). AP-6 (double-lined) was built in 1995. It has a surface area of 1.5 acres and an approximate volume of 2,500,000 gallons (average depth, 7.2 feet). AP-5 and AP-6 receive water from AP-1 and AP-4 as needed.

As specifically required in the LOU, KMCC proposes to collect water samples from three existing groundwater wells to determine the presence of nitrates in the groundwater in the vicinity of the AP ponds. These wells include M-17 which is immediately up-gradient from the ponds, and M-89 and M-25 which are down-gradient from the ponds (see Figure 3). Both the up-gradient and the down-gradient wells are screened in the shallow aquifer. These samples will be analyzed for nitrates using EPA method 300.

2.3.4 Truck Unloading Area (LOU Item #35)[1]

This location is an open area where truckers periodically discarded remnants of various substances (such as soda ash and lime) which they had hauled. Earth berms have been placed on the east, west and south sides of the area to prevent truck access, thereby preventing further use. The area is 160 feet wide north-south by 315 feet long east-west (1.15 acres).

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[1] *This location was associated with the J.B. Kelley Trucking Company.*

KMCC will advance eight soil borings (see Figure 5, Area D) and collect samples from a depth of 24-36 inches. KMCC has no reason to believe that soil in the area is not homogeneous, and therefore eight borings would provide an indication of the presence of possible contamination. The boring/sampling locations were selected as described in §2.2.2. Based upon information provided by a previous terminal manager, inorganic type materials were hauled by the truckers. As a result, sampling and analysis will only be conducted for metals and pH.

2.3.5 Diesel Fuel Storage Tank (LOU Item #45)

KMCC has removed an above ground diesel fuel storage tank located south of Old P-2 (see Figure 3).

KMCC will advance three soil borings to 10 feet with samples collected from each vertical foot. The boring locations will be field selected to sample those areas where soil staining is present to determine the vertical extent of possible contamination. The 0-1, 4-5, and the 9-10 foot samples will be submitted for analysis. The remainder will be extracted, and the extract retained for later analysis if necessary. In addition to soil samples, water samples will be collected from existing monitor wells: M-21, a down-gradient well, and M-10 which is substantially up-gradient (see Plate 1, M-10's location is approximately 200 feet south and upgradient of Unit 5). Because the storage tank contained diesel fuel, the soil and water samples will be analyzed for diesel components using EPA Method 8015.

2.3.6 AP Plant Area Change House/Lab Septic Tank (LOU Item #54)

The AP plant change house and laboratory is located in the west central portion of the KMCC facility. The change house was constructed in the early 1950's and a chemistry laboratory was added in 1980. Waste water effluent from the change house showers, rest rooms, and laboratory sinks discharged to a septic system with an associated leach field (Septic System). The location of the Septic System (Area C) and proposed sample locations are shown in Figure 2.

Laboratory operations included rinsing laboratory equipment, preparing standards, analyzing inorganic samples, preparing analytical solutions and preparing dilute titrants. Hazardous solutions were collected and shipped to an appropriate disposal

facility. Rinse water from the laboratory entered the Septic System until August 1992. In August 1992, use of the Septic System was discontinued. The change house showers, restrooms and laboratory sinks now discharge to a pump station which transfers the water to the City of Henderson sanitary drains.

KMCC proposes to conduct a soil assessment in the area of the old Septic System. This assessment will involve drilling and sampling two soil borings to a depth of 15 feet (see Figure 2, Area C). Locations were selected near the outfall of the septic system to collect information in the area with the maximum potential for contamination. Soil samples will be collected at 4 - 5 feet, 9 - 10 feet, and 14 - 15 feet (for a total of 6 samples). Based upon the substances utilized in the quality control laboratory, these samples will be analyzed for total metals, soil pH, volatile organics and semi-volatile organics.

2.3.7 J.B. Kelley, Inc. Trucking Site (LOU Item #63)

J.B. Kelley leased property from KMCC immediately south and east of the truck unloading area and operated a trucking operation from the site. The company hauled commodities such as lime and soda ash. The points of interest on the J.B. Kelly Site are the open concrete vaults which formerly served as foundations for storage buildings. KMCC proposes to drill one shallow soil boring through a crack in the concrete floor of the south-most vault (see Figure 5, Area E). Precipitation or leaks onto the vault floor would probably escape from the vault through cracks. Therefore, if contamination exists in the underlying soil, it is likely to be found near one of the concrete cracks in the floor. Because of this, the boring location will be chosen in the field, based upon the location of the floor cracks and not selected randomly. A soil sample will be collected from the underlying soil immediately below the concrete floor.

Additionally, based upon visual inspection, there appears to be sand accumulated in the vaults. Samples of materials on the concrete floor of the vaults will be collected from each of the eight vaults and composited into a single "soil" sample. Based upon the inorganic materials hauled by the trucking operation, the samples, both the boring and the composite, will be analyzed for total metals and soil pH. Because there was no

indication of organic materials present at the trucking operation, sampling and analysis for organics will not be performed.

2.3.8 Satellite Accumulation Point - AP Maintenance Shop

A parts washer is used in the AP maintenance shop to clean oily debris from parts undergoing repair. Non-hazardous cleaning fluids are now used for parts washing, and the use of 1,1,1-TCA has been eliminated. Waste from the parts washer, if containing 1,1,1-TCA, was sent to a hazardous waste disposal site for proper disposal.

A small amount of staining near the 1,1,1-TCA satellite accumulation area was observed during the Phase I field investigation. The staining was the result of minor spillage from a used oil drum, also in the area. Visibly stained soil was picked up and disposed of at Environmental Technology's soil/oil treatment farm. Subsequent tests indicate that TPH is still present.

KMCC proposes to remove visibly affected soil. After this soil is removed, one (1) surface sample will be collected from a randomly selected location, shown in Figure 5, to confirm the removal of TPH effected soil. This sample will be analyzed for TPH by method 8015.

2.3.9 Unit 1 Tenant Stains

Visibly stained soil immediately north of Unit 1 was removed and disposed of at Environmental Technology's soil/oil treatment farm. KMCC proposes to remove visibly affected soil. As the soil is removed, two (2) samples will be collected from randomly selected locations, shown in Figure 5, to confirm the removal of the TPH contaminated soil. The samples will be analyzed for TPH by Method 8015.

2.3.10 Hardesty Chemical Site

A review of the records indicate that Hardesty did make some production. Drawings of the facility show that there were two underground storage tanks located to the north of Unit 2, one for kerosene and one for benzene. A tank farm was also located north of Unit 2 on the north side of the railroad tracks. None of these tanks are present today. KMCC proposes to install a well down gradient from the tank farm and underground storage tanks.

The proposed monitor well location will be drilled to a depth sufficient to encounter the water table (45-50'). Drilling will be accomplished with a hollow stem auger rig. Split-spoon samples will be collected at every 5-foot depth interval for lithologic logging and control.

Following completion of the drilling, a groundwater monitor well will be installed in the borehole. This well, designated M-97, will be constructed of 2-inch diameter, screw-coupled PVC with a 10-foot section of 0.010-inch factory-slotted PVC screen. The entire annulus surrounding the screen will be filled with a filter pack composed of 10-40 washed silica sand. The filter pack sand will extend upward to not more than two feet above the top of the screen. An annular seal composed of bentonite pellets or chips will be placed above the filter pack to a thickness of at least two feet. The remaining well annulus from the top of the bentonite seal to approximately 0.5 feet below grade will be filled with a Portland cement/bentonite grout. An above-ground locking steel casing protector will be installed over the well extending approximately 1.5 feet into the grout.

A concrete pad will be poured around the well, filling the remaining 0.5 feet of annulus and extending outward to form a three-foot square pad. A survey pin will be installed in the pad, and a weep hole will be drilled into the base of the steel casing protector.

After the annular grout seal has cured, the well will be developed. Development will be accomplished utilizing either an airlift system or bailing. Development will continue until clear groundwater is produced, or until development volumes are deemed sufficient by the project hydrologist.

The monitor well will be sampled for volatiles, semi-volatiles, pH and specific conductance. These analytes were selected based upon the substances used at the Hardesty site.

2.4 Summary of Soil and Water Sampling Programs

Tables 2 and 3 summarize the soil and water sampling programs. They reference the number of samples to be collected, the analyses to be performed, and the figure which shows the sample collection points.

TABLE 1a
EPA Method 8240: List of Analytes

ANALYTE	Target Reporting Limits - Water, mg/L	Target Reporting Limits - Soil, mg/L
acetone	0.01	0.010
benzene	0.005 (0.5)*	0.005
bromodichloromethane	0.005	0.005
bromoform	0.005	0.005
bromomethane	0.01	0.005
2-butanone (MEK)	0.01	0.010
carbon disulfide	0.005	0.005
carbon tetrachloride	0.005 (0.5)	0.005
chlorobenzene	0.005 (100)	0.005
chloroethane	0.01	0.010
chloroform	0.005 (6)	0.005
chloromethane	0.01	0.005
dibromochloromethane	0.005	0.005
1,1-dichloroethane	0.005	0.005
1,2-dichloroethane	0.005	0.005
1,1-dichloroethene	0.005	0.005
1,2-dichloroethene (total)	0.005 (0.7)	0.005
1,2-dichloropropane	0.005	0.005
cis-1,3-dichloropropene	0.005	0.005
trans-1,3-dichloropropene	0.005	0.005
ethylbenzene	0.005	0.005
2-hexanone	0.01	0.010
methelene chloride	0.005	0.005
4-methyl-2-pentanone (MIBK)	0.01	0.010
styrene	0.005	0.005
1,1,2,2-tetrachloroethane	0.005	0.005
tetrachloroethene	0.005	0.005
toluene	0.005	0.005
1,1,1-trichloroethane	0.005	0.005
1,1,2-trichloroethane	0.005	0.005
trichloroethene	0.005 (0.5)	0.005
vinyl acetate	0.01	0.010
vinyl chloride	0.01 (0.2)	0.005
xylenes (total)	0.005	0.005

* Numbers in parenthesis are the maximum concentrations allowed by 40 CFR §261.24 Table 1 (governing the toxicity characteristic for the Resource Conservation and Recovery Act (RCRA)).

Table 1b
Method 6010/6010A: List of Analytes

ANALYTE	Target Reporting Limit, mg/kg[a]
Arsenic	2.0
Barium	40.0
Cadmium	1.0
Chromium	2.0
Lead	20.0
Selenium	1.0
Silver	2.0

[a] Reported in soil matrix by Lockheed Laboratories for 1996 second quarter.

Table 1c
Method 7470/7471: List of Analytes

ANALYTE	Target Reporting Limits, mg/kg[a]
Mercury	0.1

[a] Reported in soil matrix by Lockheed Laboratories for 1996 second quarter.

Table 1d
Method 8015 M - Diesel Range: List of Analytes

ANALYTE	Target Reporting Limit	
	mg/L (water) [a]	mg/kg (soil) [a]
Diesel Range Organics	1	30

[a] Determined by Lockheed on 6/26/95 (water) and 7/18/95 (soil)

Table 1e
Method 300: List of Analytes

ANALYTE	Target Reporting Limit, mg/L [a]
Nitrate as Nitrogen	0.02

[a] Reported in water matrix by Lockheed Laboratories for 1996 second quarter.

Table 1f
Method 8270: List of Analytes

Analytical Parameters	Target Reporting Limit*	
	Soil (µg/kg)	Water (µg/L)
Acenaphthene	< 660	< 10
Acenaphthylene	< 660	< 10
Anthracene	< 660	< 10
Benzo (a) anthracene	< 660	< 10
Benzo (a) pyrene	< 660	< 10
Benzo (b) fluoranthene	< 660	< 10
Benzo (g,h,i) perylene	< 660	< 10
Benzo (k) fluoranthene	< 660	< 10
Benzoic Acid	< 3300	< 50
Benzyl Alcohol	< 1300	< 20
Bis (2-chloroethoxyl) methane	< 660	< 10
Bis (2-chloroethyl) ether	< 660	< 10
Bis (2-chloroisopropyl) ether	< 660	< 10
Bis (2-ethylhexyl) phthalate	< 660	< 10
4-Bromophenyl-phenylether	< 660	< 10
Butylbenzylphthalate	< 660	< 10
Carbazole	< 660	< 10
2-Chloronaphthalene	< 660	< 10
2-Chlorophenol	< 660	< 10
2,4-Dichlorophenol	< 660	< 10
4-Chloro-3-methylphenol	< 1300	< 20
4-Chloroaniline	< 1300	< 20
4-Chlorophenylphenyl ether	< 660	< 10
Chrysene	< 660	< 10
Di-n-butylphthalate	< 660	< 10
Di-n-octylphthalate	< 660	< 10
Dibenzo (a,h) anthracene	< 660	< 10
Dibenzofuran	< 660	< 10
1,2-Dichlorobenzene	< 660	< 10
1,3-Dichlorobenzene	< 660	< 10
1,4-Dichlorobenzene	< 660	< 10
3,3-Dichlorobenzidine	< 1300	< 20
2,4-Dichlorophenol	< 660	< 10
2,4-Dimethylphenol	< 660	< 10
2,4-Dinitrophenol	< 3300	< 50
2,4-Dinitrotoluene	< 660	< 10
2,6-Dinitrotoluene	< 3300	< 10
4,6-Dinitro-2-methylphenol	< 3300	< 50
Diethylphthalate	< 660	< 10
Dimethylphthalate	< 660	< 10
Fluoroanthene	< 660	< 10
Fluorene	< 660	< 10
Hexachlorobenzene	< 660	< 10
Hexachlorobutadiene	< 660	< 10
Hexachloro cyclopentadiene	< 660	< 10
Hexachloroethane	< 660	< 10
Indeno (1,2,3-cd) pyrene	< 660	< 10
Isophorone	< 660	< 10
2-Methylnaphthalene	< 660	< 10
2-methylphenol	< 660	< 10
4-methylphenol	< 660	< 10
2-Nitroaniline	< 3300	< 50
3-nitroaniline	< 3300	< 50
4-nitroaniline	< 3300	< 20
2-Nitrophenol	< 660	< 10
4-nitrophenol	< 3300	< 50
n-Nitroso-di-n-propylamine	< 660	< 10
n-Nitroso-diphenylamine	< 660	< 10
Naphthalene	< 660	< 10
Nitrobenzene	< 660	< 10
Pentachlorophenol	< 3300	< 50
Phenanthrene	< 660	< 10
Phenol	< 660	< 10
Pyrene	< 660	< 10
1,2,4-trichlorobenzene	< 660	< 10
2,4,5-trichlorophenol	< 660	< 10
2,4,6-trichlorophenol	< 660	< 10

* Practical quantitation limits (PQLs) are obtained from Lockheed Analytical Services for standard soil and water composition. PQLs are subject to change as a result of sample matrix interference.

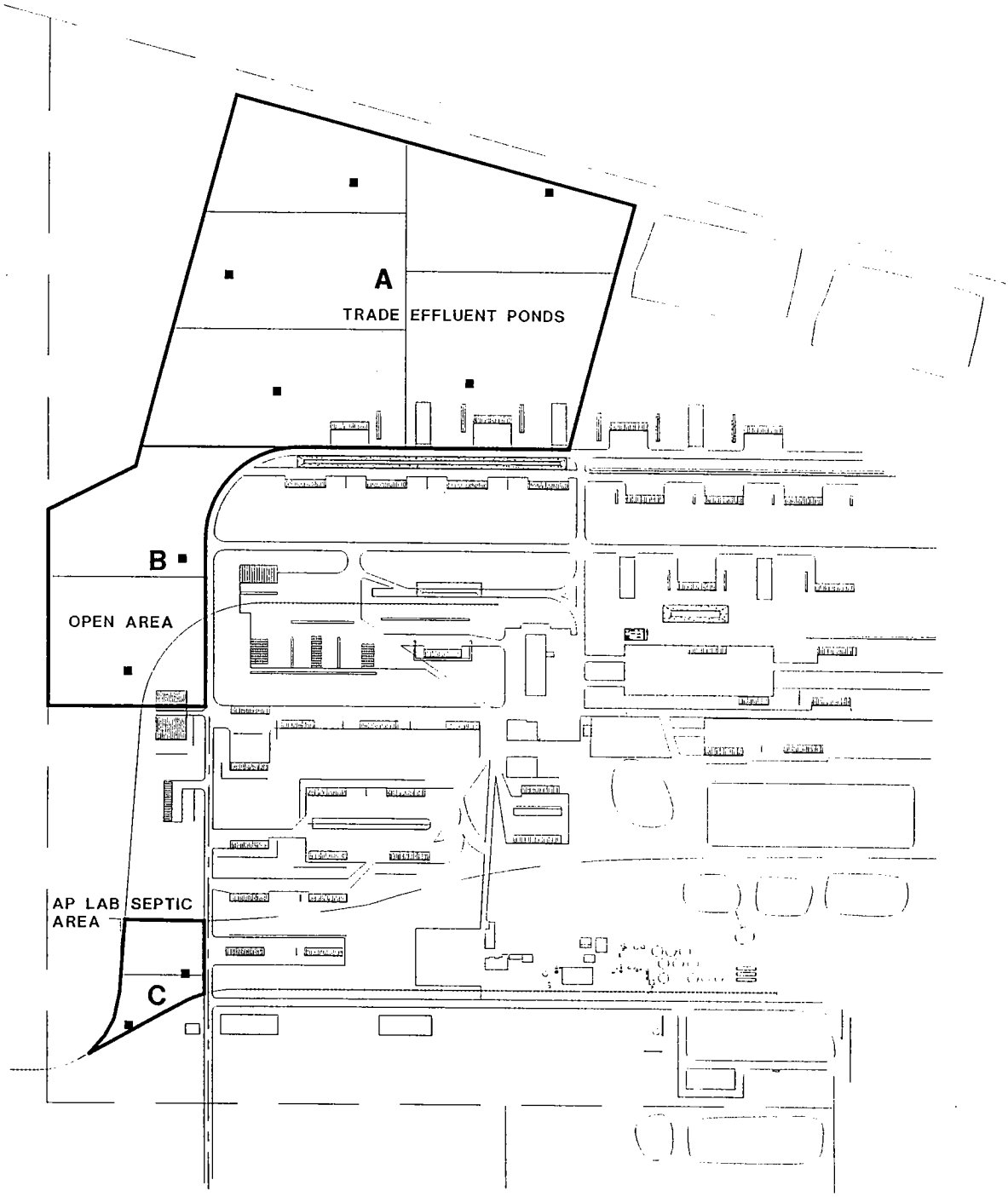
TABLE 2
Summary of Soil Sampling Program

§	Location	Analyses [1]	See..	No. of Borings	Samp Per Boring	Total Depth (ft)
2.3.1	Trade Effluent Ponds and Open Area, Items No. 1, 2	Metals, Soil pH	Fig.2, (Areas A & B)	7	3	10
2.3.2	Old P-2, P-3 Ponds, Items No. 7, 8	Total Chromium, Soil pH	Fig.4	13	1	3
2.3.4	Truck Unloading Area, Item No. 35	Metals, Soil pH	Fig.5 (Area D)	8	1	3
2.3.5	Diesel Storage Tank, Item No. 45	Method 8015 (for diesel components)	Fig.3 (in Area F)	3	3	10
2.3.6	AP Change House/Lab Septic Field, Item No. 54	Metals, Soil pH, Volatile Organics by 8240, semi-volatile by 8270	Fig.2 (Area C)	2	3	15
2.3.7	J.B. Kelley Trucking, Item No. 63	Metals, Soil pH	Fig.5 (Area E)	1 (+1 surface soil)	1	Soil Surface
2.3.8	Satellite Accumulation Point-AP Maintenance Shop, Item No. 39	Method 8015	Fig. 5 (Area F)	(1 surface soil)	1	Soil Surface
2.3.9	Unit 1 Tenant Stains, Item No. 41	Method 8015	Fig. 5 (Area G)	(2 surface soil)	1	Soil Surface

[1] Metals will include arsenic, barium, cadmium, chromium, lead, mercury, selenium and silver.

TABLE 3
Summary of Water Sampling Program

§	Location	Wells to Be Sampled	Analyses	See...
2.3.3	AP Ponds, Items No. 16, 17	M-17, M-89 and M-25	Nitrate	Plate 1, and Fig.3
2.3.5	Diesel Storage Tank, Item No. 45	M-10, M-21	Method 8015 (for diesel components)	Plate 1, and Fig.3
2.3.10	Hardesty Chemical Site, Item No. 4	Hardesty Assessment Monitor Well	Volatiles by 8240, Semi-volatiles by 8270, pH and specific conductance	Plate 1, and Fig. 5



■ RANDOMLY SELECTED SOIL BORING LOCATION

0 200' 400' 800'

SCALE IN FEET

N

FIGURE 2

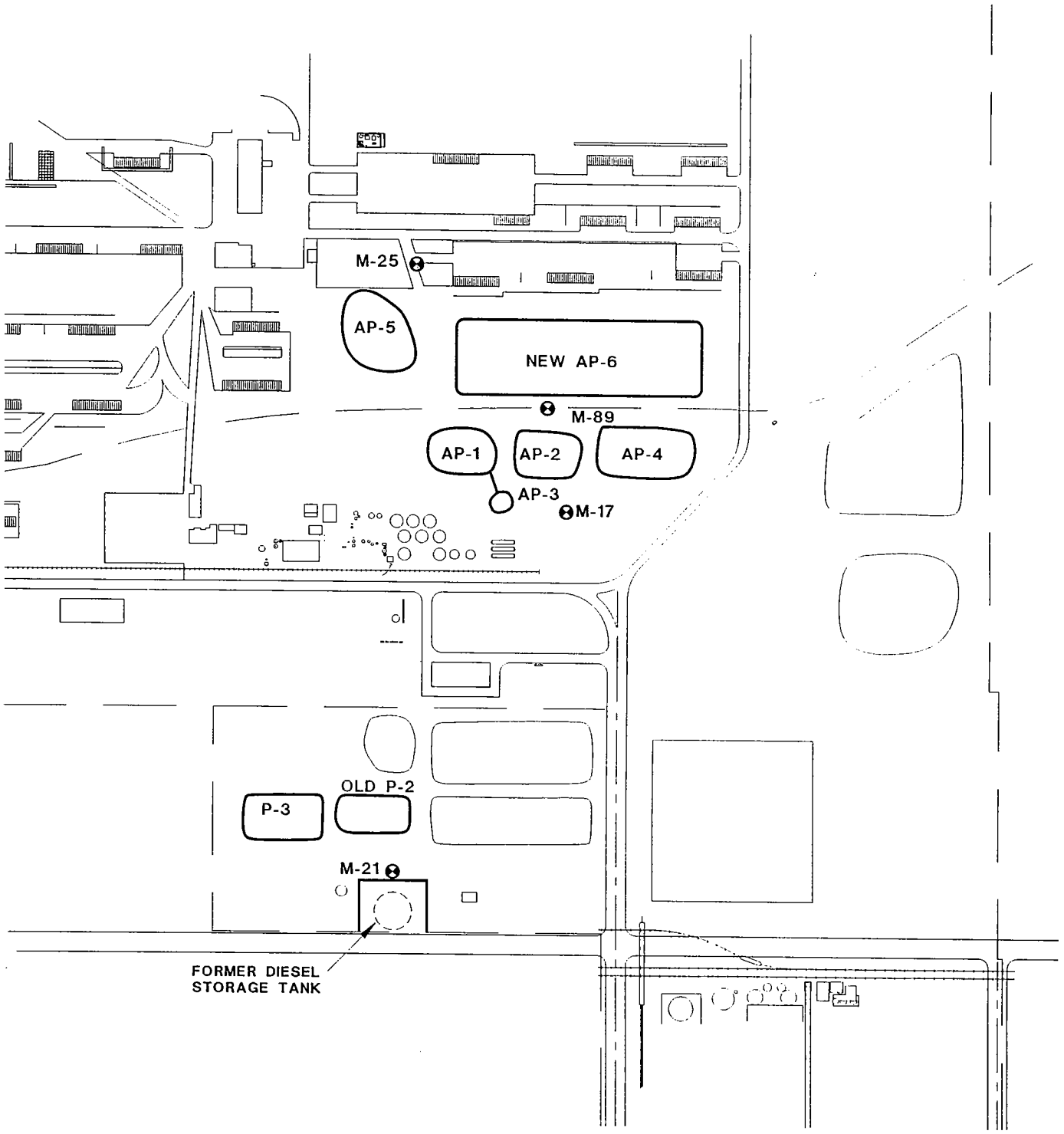
SOIL BORING LOCATIONS

Trade Effluent Ponds and AP Lab Septic Areas

Kerr McGee Chemical Corp.

Henderson, NV

DRAWN: M. SCOP	DATE: 5/2/96	PROJECT NO. 4020-004-300	REV.
FILE NO. 4020004E	CHK BY:		



LOCATION OF MONITORING WELL TO BE SAMPLED

0 150' 300' 600'

SCALE IN FEET

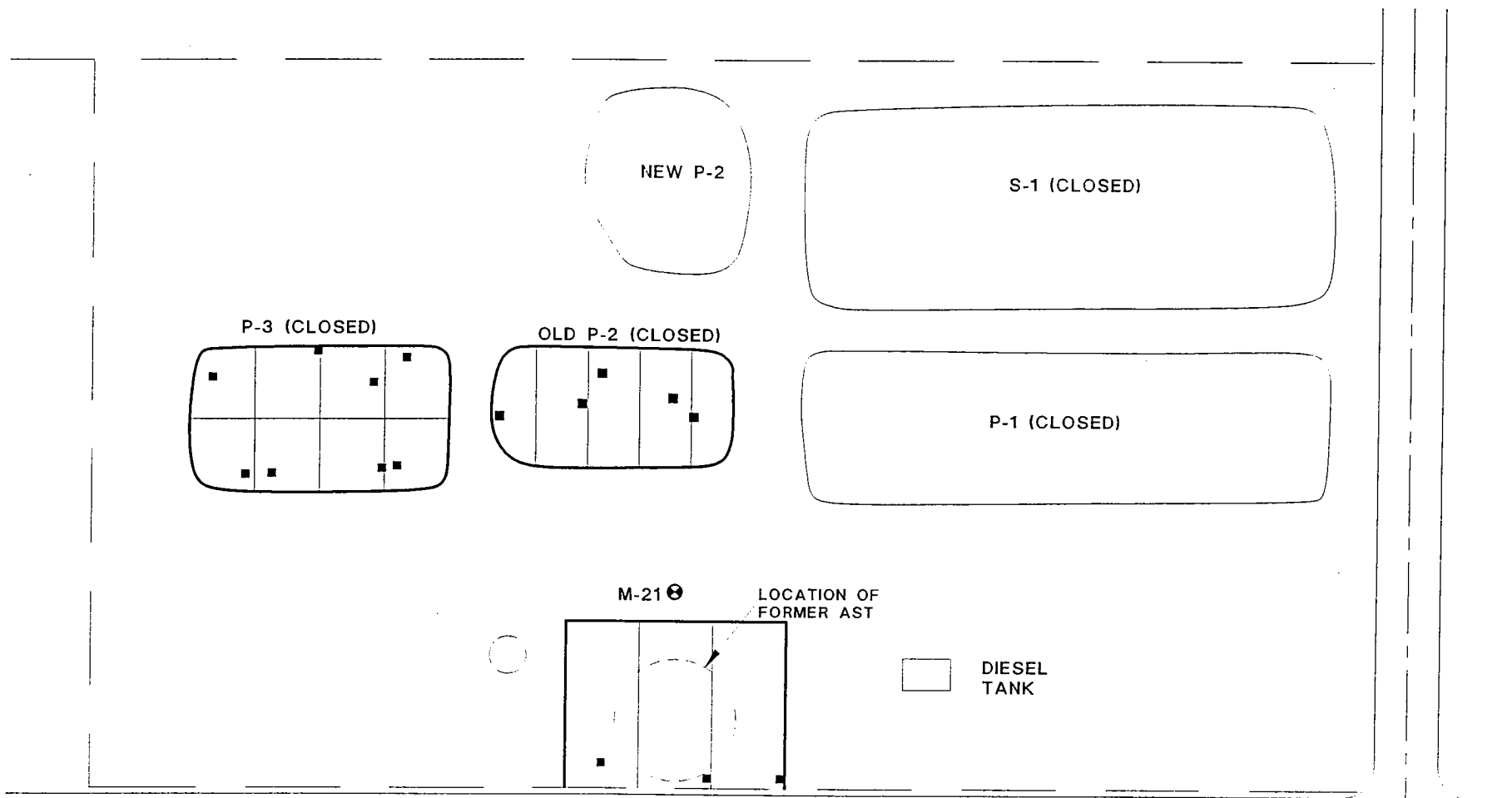
N

FIGURE 3

MONITORING WELL LOCATIONS

Sodium Chlorate and AP Pond Locations
Kerr McGee Chemical Corp.
Henderson, NV

DRAWN: M. SCOP	DATE: 5/1/96	PROJECT NO. 4020-004-300	REV.
FILE NO. 4020004G	CHK BY:		



■ RANDOMLY SELECTED SOIL BORING LOCATION

0 50' 100' 200'

SCALE IN FEET

FIGURE 4

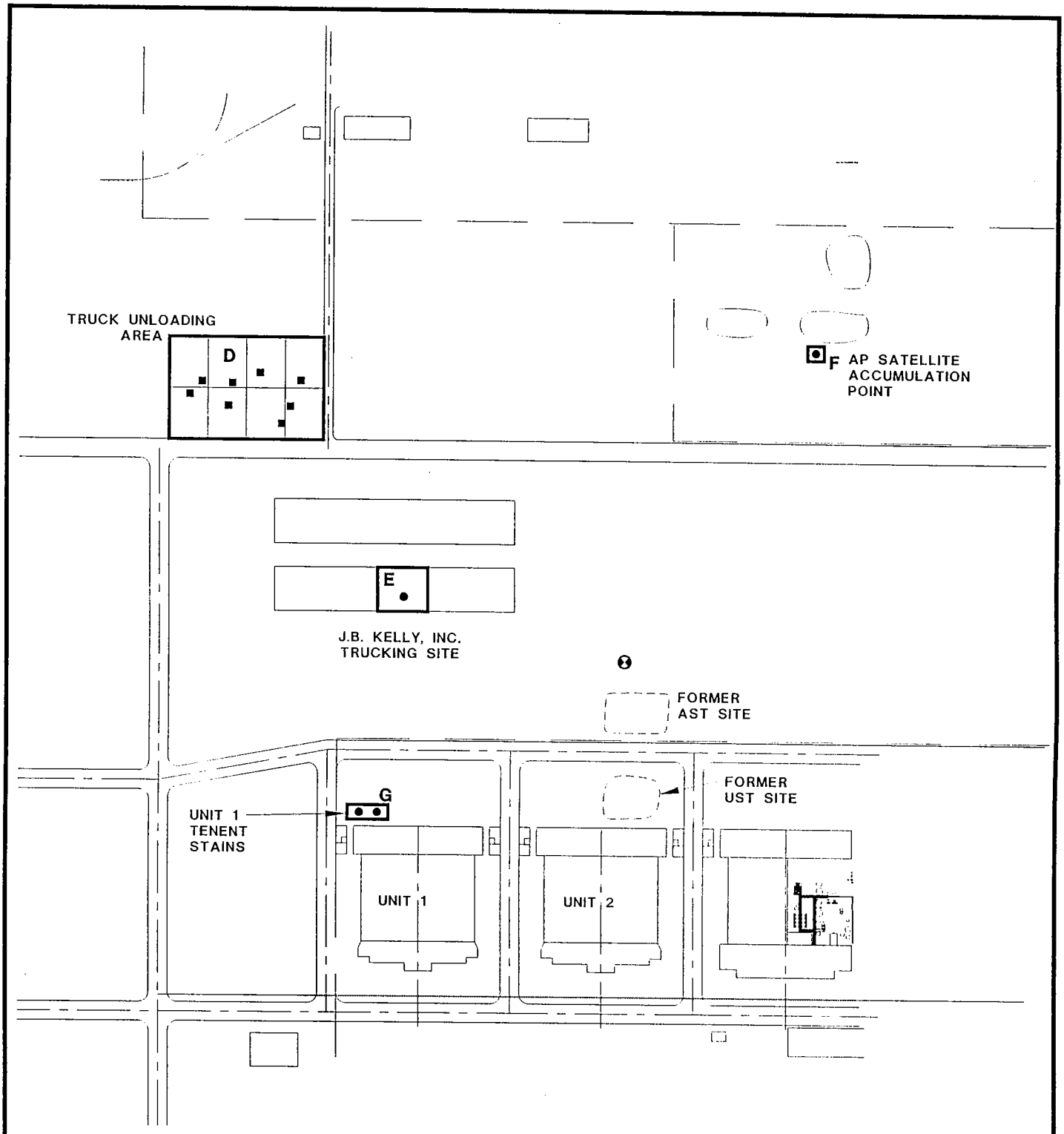
SOIL BORING LOCATIONS

Old P-2 & P-3 Ponds

Kerr McGee Chemical Corp.

Henderson, HV

DRAWN: M. SCOP	DATE: 5/1/96	PROJECT NO.	REV.
FILE NO. 4020004H	CHK BY:	4020-004-300	



PROPOSED HARDESTY ASSESSMENT MONITOR WELL LOCATION

RANDOMLY SELECTED SOIL BORING LOCATION

EXACT SAMPLING LOCATION TO BE DETERMINED IN THE FIELD

0 150' 300' 600'

SCALE IN FEET

N

FIGURE 5

SOIL BORING LOCATIONS

Phase II Work Plan

DRAWN: M. SCOP	DATE: 5/2/96	PROJECT NO.	REV.
FILE NO. 4020004F	CHK BY:	4020-004-300	

PHASE II
WORK PLAN

Section 3
Data Collection and Quality Assurance Plan

3. DATA COLLECTION AND QUALITY ASSURANCE PLAN

Kerr-McGee Chemical Corporation (KMCC) has prepared this Work Plan for the production facility in Henderson, Nevada. The Work Plan describes in detail collection protocols for groundwater samples and surface and sub-surface soil samples. Also included are details on field and laboratory quality assurance/quality control programs, laboratory analyses, and monitor well installation and abandonment procedures.

This document is to be used by facility personnel and consultants responsible for obtaining the types of samples described above or who are responsible for monitor well installation/abandonment. A copy of this plan will be kept on file at the facility for the appropriate personnel to review and reference.

3.1 Groundwater Sampling Protocol

Groundwater sampling from wells is required to define and document potential impacts to native groundwater quality resulting from Facility operations. In order to ensure that the analytical data developed for the groundwater is accurate, the sampling personnel must be certain that the collected sample is representative of the groundwater system that exists around the monitoring well.

3.1.1 Sampling Team Members

It is the responsibility of Kerr-McGee Chemical Corporation's environmental personnel to collect or supervise the collection of all routine groundwater monitor well samples at the Facility. For the purposes of this Work Plan, ENSR will accomplish the sampling under the supervision of KMCC environmental personnel.

3.1.2 Well Locations and Sampling Frequency

All monitoring wells are readily accessible and are clearly visible. Keys for the off-site wells are kept at the facility and are under the control of facility environmental personnel. Most on-site monitor wells are not locked but are within the fenced are of the Facility.

Both the frequency of sampling and analytical parameters list are per agreement between KMCC and the regulatory agency. Please see Section 2 of this Work Plan for specific information.

3.1.3 Steps Taken Prior to Sample Withdrawal

3.1.3.1 Initial Observations

Upon arrival, field personnel who sample KMCC groundwater monitor wells make note of the general well and site conditions arrival on an appropriate field sampling log (Figure 6). An inspection of monitor wells is made to determine general well condition and the evidence of any damage. For example, general comments about the weather conditions for each sampling date are recorded. Specific comments on each of the wells are then made with typical notations to include, but not be limited to, whether a well is in a flooded area, ground subsidence, the presence of nearby desiccation cracks, etc.

A notation concerning missing well locks or caps is made if appropriate. Observations related to the physical condition of the well are recorded if it appears the well has been damaged (for example, from vandalism, being struck by a vehicle, etc.). It is the responsibility of the Facility environmental personnel to maintain the monitor wells in good repair.

3.1.3.2 Measurement of Static Water Level and Well Depth

After all general and pertinent observations are recorded, the field technician will measure a static water level in all facility groundwater monitor wells that are sampled. The well cap is removed and laid upside down on the ground or placed in the field vehicle. An electrical water level indicator is tested to make sure it is operational. After cleaning, the probe is lowered downhole until water level is detected. Three successful measurements are taken and averaged to obtain the depth to water. The depth to water is then recorded (field sampling log) to the nearest 0.01 foot with respect to the reference point (either a mark or a notch) at the top of the casing. All reference points have been measured in relation to mean sea level by a licensed surveyor.

Total well depth is measured with the water level indicator, or a similar device, by lowering the probe downhole until it touches the bottom of the well. Total depth measurement is then recorded on the field sampling log (Figure 6) to the nearest 0.01 foot with respect to the same measuring point from which depth to water measurements are made.

Following depth to water and total well depth measurements, the probe and submerged tape are cleaned and rinsed in distilled/deionized water and wiped with a disposable towel.

3.1.4 Withdrawing a Water Sample

3.1.4.1 Calculating Volume of Water to be Evacuated

It is standard practice to evacuate a minimum of three casing volumes from each well before obtaining a sample. There are instances, however, where well recovery is extremely slow and three casing volumes cannot be removed. When this occurs, the well is evacuated to near dryness and sampled after sufficient volume of water is available to obtain the necessary volume of water required for analyses. Regardless of well capacity, samples for volatile organic analysis (VOA) will be withdrawn from the well within one (1) hour of purging. The volume of water purged from each well before obtaining a sample is recorded (field sampling log) by the field technician.

KMCC has prepared a table (Table 4) used by sampling personnel in determining the amount of fluid to be evacuated from various size monitor wells. In instances where the table is not useful, the field technician will calculate the volume of fluid to be removed by using the conversion factors of 0.16, 0.37, 0.65, 1.02 and 1.47 gallons per foot of water in well for 2, 3, 4, 5 and 6 inch diameter wells, respectively. For example, given a 2 inch well with 40 feet of standing fluid:

40 ft. x 0.16 gal/ft = 6.4 gal. per casing volume

Therefore, 19.2 gallons of fluid would need to be evacuated before a sample is taken (3 casing volumes).

3.1.4.2 Evacuation of Well Water

Evacuation of well water is performed with the use of Teflon or stainless steel bailers and attached polyethylene rope or with dedicated pneumatic bladder pumping systems (i.e, Well Wizard by QED Groundwater Specialists). Purging/sampling equipment is inspected for damage prior to each use and either repaired (if possible) or replaced.

When well purging is performed with bailers, a clean plastic sheet or a 30 gallon trash can lined with clean plastic is placed adjacent to the monitor well to keep the bailer and rope from coming in contact with the ground. This procedure is done to prevent the bailer and rope from possibly becoming contaminated from touching the ground before, during and after the bailing and sampling process.

Well evacuation with a bailer is performed by gently lowering the bailer downhole to prevent de-gassing of the fluid column. The bailer is allowed to fill and is then retrieved from the well. Purged water is collected and disposed of in accordance with applicable federal, state and local regulations.

In case of poorly yielding wells where three casing evacuations cannot be made, the well is allowed to recover before collecting samples.

For wells equipped with a dedicated pneumatic bladder pumping system, purging is performed according to the manufacturer's recommendations, using the supplied pump controller panel and power source.

3.1.4.3 Field Measurements and Field QA/QC Procedures

A minimum of four (4) separate groundwater samples will be collected in inert containers (i.e, plastic beakers or glass bottles) during the purging process for the field determination of pH, specific conductivity, and temperature. The data will be recorded on the field sampling log. It is possible, for low-yeilding wells, that the well will be purged dry prior to collection of the four samples. In this case, less than four samples might be collected.

TABLE 4: VOLUME OF WATER TO BE PURGED

FROM VARIOUS SIZE MONITOR WELLS

	# Gal. to Evacuate One Casing Volume					# Gal. to Evacuate Two Casing Volumes					# Gal. to Evacuate Three Casing Volumes					AMOUNT OF WATER IN WELL (IN FEET)
	2"	3"	4"	5"	6"	2"	3"	4"	5"	6"	2"	3"	4"	5"	6"	
1.0	0.16	0.37	0.65	1.02	1.47	0.32	0.74	1.30	2.04	2.94	0.48	1.11	1.95	3.06	4.41	
2.0	0.32	0.74	1.30	2.04	2.94	0.64	1.48	2.60	4.08	5.88	0.96	2.22	3.90	6.12	8.82	
3.0	0.48	1.11	1.95	3.06	4.41	0.96	2.22	3.90	6.12	8.82	1.44	3.33	5.85	9.18	13.23	
4.0	0.64	1.48	2.60	4.08	5.88	1.28	2.96	5.20	8.16	11.76	1.92	4.44	7.80	12.24	17.64	
5.0	0.80	1.85	3.25	5.10	7.35	1.60	3.70	6.50	10.20	14.70	2.40	5.55	9.75	15.30	22.05	
6.0	0.96	2.22	3.90	6.12	8.82	1.92	4.44	7.80	12.24	17.64	2.88	6.66	11.70	18.36	26.46	
7.0	1.12	2.59	4.55	7.14	10.29	2.24	5.18	9.10	14.28	20.58	3.36	7.77	13.65	21.42	30.87	
8.0	1.28	2.96	5.20	8.16	11.76	2.56	5.92	10.40	16.32	23.52	3.84	8.88	15.60	24.48	35.28	
9.0	1.44	3.33	5.85	9.18	13.23	2.88	6.66	11.70	18.36	26.46	4.32	9.99	17.55	27.54	39.69	
10.	1.60	3.70	6.50	10.20	14.70	3.20	7.40	13.00	20.40	29.40	4.80	11.10	19.50	30.60	44.10	
15.	2.40	5.55	9.75	15.30	22.05	4.80	11.10	19.50	30.60	44.10	7.20	16.65	29.25	45.90	66.15	
20.	3.20	7.40	13.00	20.40	29.40	6.40	14.80	26.00	40.80	58.80	9.60	22.20	39.00	61.20	88.20	
25.	4.00	9.25	16.25	25.50	36.75	8.00	18.50	32.50	51.00	73.50	12.00	27.75	48.75	76.50	110.00	
30.	4.80	11.10	19.50	30.60	44.10	9.60	22.20	39.00	61.20	88.20	14.40	33.30	58.50	91.80	132.00	

Before the pH measurements are performed, the pH meter is calibrated following manufacturer's recommendation in pH 7 and pH 4, or pH 7 and pH 10 buffer solutions. Calibration notes are recorded in a field calibration notebook.

Following standardization, the pH probe is rinsed in distilled or deionized water and then immersed in the first field sample for a pH determination. The pH of the sample is allowed to equilibrate before the reading is recorded (field sampling log).

Following equilibration and recording, the probe is rinsed with deionized water and placed in the second field sample. This method of equilibration, recording and rinsing is followed until the four pH measurements are made. The samples are then combined with the purging water, and the containers are rinsed with deionized water.

Conductivity measurements are made on a minimum of four (4) samples following calibration of the meter according to manufacturer's recommendations. Following calibration, the meter is checked in a solution of 10,000 umhos/cm^3. Again, calibration notes are recorded in a field calibration notebook.

The conductivity probe is then rinsed with deionized water and placed in one of the field samples. Allowing adequate time for equilibration, the conductivity and temperature of the samples is then recorded (field sampling log) followed by a thorough rinsing of the probe in deionized water. The remaining three samples are treated in the same manner until the four conductivity measurements have been recorded. The samples are then combined with the purging water, and the containers are rinsed with deionized water.

3.1.4.3.1 Equipment Rinsate Samples - When equipment rinsate samples are collected, they are prepared by contacting deionized water with the sampling equipment, followed by filling the appropriate bottles and submitting them to the laboratory with the rest of the samples. Equipment rinsate samples are taken at the end of the sampling day and serve as a measure of decontamination effectiveness.

3.1.4.3.2 Trip Blanks - Trip blanks are submitted to the laboratory only when sampling events include analysis for volatile organic compounds. Trip blanks are samples used to identify possible contamination during sample transport. The trip blank samples will be prepared in the laboratory with organic-free water. They will be shipped

with the sample containers to the field, stored with the field samples, and returned to the laboratory for analysis with the field samples.

3.1.4.4 Sample Collection

Following well purging and field measurement determinations, sample containers are filled in the following order: 1) extractable organics, 2) nitrate. KMCC has contracted with Lockheed Laboratories of Las Vegas, Nevada, to do the laboratory analyses. Each sample will be labeled with a unique ID # which will allow the sample to be tracked to its corresponding field location.

All sample bottles, "blue ice" and labels are shipped or delivered to the KMCC facility by Lockheed. The bottles provided have been pre-cleaned according to SW-846 methodology, and if requested by KMCC, may already contain preservatives upon arrival at the Facility.

Water samples for laboratory analyses are taken from the dedicated pump discharge line or poured from the stainless steel or Teflon bailer into appropriate clean containers. Sample containers for organic analyses (other than volatiles) are amber glass bottles with Teflon-lined caps. Samples for inorganic analyses are collected in either glass or plastic containers. All sample containers, particularly those which are to be used for organic analyses are filled to the top to minimize headspace and therefore the possible change in certain volatile constituents. Preservation methods and holding times for each analytical parameter, as well as the sample container type are included in Table 5.

3.1.4.5 Split Sampling Events

In the event an authorized regulatory agency wishes to obtain split samples, it is the agency's responsibility to supply their own collection containers and preservatives. Well purging will be done according to this DCQAP, and sample containers will be filled alternately between KMCC and the regulatory agency.

3.1.4.6 Sample Preservation and Shipment

Immediately after collection, all samples are stored in an insulated sample shuttle (chest) and cooled to approximately 4°C with ice. Please refer to Table 5 for recommended containers, preservation, storage and holding time requirements for the collected water samples.

Samples are packed in ice and shipped to the contract laboratory within 24 hours of collection. Chain of custody forms (see Figure 7) accompany all samples.

3.1.4.7 Contaminated Equipment Disposal

All contaminated field equipment will be disposed of in accordance with applicable local, state and federal regulations.

3.1.5 Field Chain of Custody

Following sample collection, the field technician will prepare the shuttles for delivery to Lockheed. The shuttles are packed with bottles, ice (or "blue ice"), and chain of custody forms (Figure 7). The shuttles are shipped to Lockheed where they are inspected on arrival for evidence of breakage/tampering. The shuttle is then opened, the chain of custody form signed as to the date and time opening occurred, and the samples are logged into the Lockheed sample management system.

3.1.6 Data Reporting

All analytical data is reported to the facility contact designated on the chain of custody form.

TABLE 5. RECOMMENDED CONTAINERS, PRESERVATION, STORAGE AND HOLDING TIMES FOR WATER AND WASTEWATER

DESCRIPTION	METHOD	MATRIX	SAMPLE CONTAINER[1]	PRESERVATION[5]	PREP/ANALYSIS HOLDING TIME*	MINIMUM VOLUME
TPH as Diesel	8015 M	H_2O	G (a) TefCap	Cool 4° C	14/40 days[2,3]	1L
Nitrate	353.2, 9200	H_2O	P or G	Cool 4° C	48 hrs.[4]	500 mL
pH	150.1	H_2O	P or G	None	Analyze immediately	500 mL
Specific conductance	120.1	H_2O	P or G	Cool 4° C	28 days	

TABLE 5: FOOTNOTES

1 G(x) = glass; AG(x) = amber glass; P(x) = plastic; TefSep = Teflon Septum; TefCap = Teflon lined cap; x= cleaning protocol as follows: a = acid wash + solvent wash + oven dry (for semi volatiles or metals); b = oven dry (for volatiles); c = acid wash (for metals only). The procedures are described in more detail in Section 4.

2 7 days from sampling date for extraction, 40 days from extraction date for analysis of the extract. 14 days until extraction if listed as 14/40.

3 EPA does not control TPH holding times. The holding time given is the laboratory practice by analogy with Oil and Grease.

4 Certain anions require special handling. Holding times and preservation for a particular sample will be determined by the requirement for the anion of interest with the shortest holding time, e.g. nitrate and nitrite - 48 hours; orthophosphate - filter and 48 hours.

5 Sample preservation should be performed immediately upon sample collection except when specifically allowed by the method. Documentation is inspected at the time of sample receipt to assure samples are properly preserved (See Section 5).

"*" Indicates internal guidance rather than a binding regulatory preservation requirement or holding time. Exceptions may occur.

FIELD CHAIN OF CUSTODY - Environmental Samples

Kerr-McGee Chemical Corporation	Laboratory:	Project Name:
P. O. Box 55		Shipping Document / PO #:
8000 West Lake Mead Dr.		Sampled By:
Henderson, NV 89009	Atten: Sample Receiving	Requested Turnaround:

Samples represented on this chain-of-custody are expected to be collected and analyzed using EPA protocol. Note any discrepancies.

Sample Id	Collection		Matrix Code	Sample Type	Field Preserved (Y or N)	Container - # and Volume								Comments
	Date	Time												

Relinquished By:			**Received By:**		
(Print)	(Signature)	Date/Time	(Print)	(Signature)	Date/Time
1.					
2.					
3.					
4.					

Requested Turnaround:	Matrix Code:			Sample Type:
24 Hour Normal lab turn around	SW - Surface Water	SL - Soil	WW - Waste Water	G - Grab
48 Hour	GW - Groundwater	SD - Solid	AR - Air	C - Composite
5 Day	DW - Drinking Water	SG - Sludge	O - Other	O - Other

Distribution: Original to accompany samples / Copy to be retained by sampler

FIGURE 7

Chain of Custody

3.2 Surface and Near Surface Soil Sampling Protocol

Surface and near surface soil samples (depth: 0 to 1 foot) will be collected following procedures described in "Preparation of Soil Sampling Protocol: Techniques and Strategies," an EPA document prepared by Benjamin J. Mason (May 1983). Generally, the procedures allow for the use of soil punches, scoops and shovels, soil probes and augers, and power augers.

3.2.1 Initial Observations

Soil sampling locations will be marked in the field and referenced on field sample log and chain of custody forms with a unique sample ID #, so as to enable re-sampling of that exact location at a later date, if necessary. A hand held G.P.S. instrument will be used to determine the longitude, latitude, and elevation of the sample locations. Weather conditions on the sampling date will be described as will any unusual weather events (for example, drought) just prior to the sampling. Other appropriate notes will be made as deemed necessary.

3.2.2 Obtaining Soil Samples

Depending upon the required laboratory analyses, soil samples will be collected using various methods. Surface and near surface samples that require chemical testing will be collected with soil punches, scoops, shovels, probes or augers and stored in glass jars.

Soil samples collected with scoops, shovels, probes and augers will be described according to ASTM Method D2488, "Description and Identification of Soils (Visual Manual Procedures)."

3.2.2.1 Field Compositing

Where composite samples must be taken, compositing will be done with large dedicated plastic sheets (one time use only) or with stainless steel mixing bowls. In each case, clods of soil will be broken up before being mixed with hand tools. Following mixing, the soil will be placed in a pile, sectioned into four quarters, and a small sample from each quarter will be taken and mixed together to form the composite. The composite will then be placed in a glass jar and shipped with the rest of the samples to the laboratory. The excess soil will be discarded.

3.2.2.2 Sample Preservation

Soil samples collected in jars for chemical analyses will be stored at approximately 4°C in the sample shuttle pack and shipped to the laboratory within 24 hours of sampling.

Table 6 has been prepared to show the recommended containers, preservation, storage, and holding times for soils, solids and waste.

3.2.2.3 Decontamination Procedures

All sampling equipment will be decontaminated between use with a steam cleaner or in a non-phosphate detergent solution (i.e., Alconox or similar) followed by rinsing with diionized water. All decontamination rinses will be containerized until analytical information related to the samples is received. The decontamination rinses will be disposed of according to federal, state and local regulatory requirements.

3.2.3 Field Chain of Custody

Chain of custody forms will accompany all soil samples collected and shipped for analyses. (See Section 3.1.5 and Figure 7).

3.2.4 Data Reporting

All analytical data is reported to the facility contact designated on the chain of custody form.

3.3 Subsurface Soil Sampling Protocol

Subsurface soil sampling refers to those procedures used to obtain soil samples from a depth of greater than 1 foot. In some instances, small portable power augers may be useful to slightly greater depths, but generally the procedures require the use of a drill rig and a split-barrel (split-spoon) sampler with brass sleeves.

3.3.1 Initial Observations

Borehole locations will be marked in the field and referenced on field sample log and chain of custody forms with a unique sample ID # and referenced to permanent/semi-permanent structures and located on facility site drawings, so as to enable re-sampling of that exact location at a later date if necessary. A hand held G.P.S. instrument will be used to determine the longitude, latitude, and elevation of the borehole and/or sample locations. Weather conditions on the sampling date will be described as will

any unusual weather events (for example, drought) just prior to the sampling. Other appropriate notes will be made as deemed necessary.

3.3.2 Obtaining Sub-Surface Soil Samples

Some borings will require either continuous or intermittent sampling from grade level to total depth. Such samples will be collected by either the use of split-barrel (split-spoon) samplers. Split-barrel samples will be collected following ASTM Method D1586.

All boreholes will be visually logged from grade level to the target depth. Soil and formation description will follow ASTM Method D2488 with notes also made as to water level and visual or olfactory evidence of contamination. A photoionization detector or comparable instrument will be used in the field to screen logged samples for volatile constituents.

Borings not completed as monitor wells will be grouted back to the surface using a tremie line and Type I Portland cement with 5% bentonite. Those bore holes which collapse on themselves leaving less than eight inchs of depression will not be grouted back to the surface.

TABLE 6: RECOMMENDED CONTAINERS, PRESERVATION, STORAGE AND HOLDING TIMES FOR SOILS, SOLIDS AND WASTE

DESCRIPTION	METHOD	MATRIX	SAMPLE CONTAINER[1]	PRESERVATION	PREP/ANALYSIS HOLDING TIME*	MINIMUM VOLUME
GC/MS-Purgeables	8240	Soil/Waste	G (b) TefSep or TefCap	Cool 4°C[2]	14 days	100g or 4 oz. Jar
TPH as Diesel	8015 M	Soil	G (a) TefCap	Cool 4° C	14/40 days [3,4]	100 g or 4 oz. Jar
Metals-AA	7000	Soil	P or G (c)	Cool 4°C	6 mos.	100g or 8 oz. Jar
ICP Metals	6010	Soil	P or G (c)	Cool 4°C	6 mos.	100 g or 8 oz. Jar
Mercury	7471	Soil	P or G (c)	Cool 4°C	28 days	
pH	EPA 9045	Soil	P or G	Cool 4°C	ASAP	100g or 8 oz. Jar

TABLE 6: FOOTNOTES

[1] G(x) = glass; AG(x) = amber glass; P(x) = plastic; TefSep = Teflon septum; TefCap = Teflon lined cap; x = cleaning, protocol as follows: a = acid wash + solvent wash + oven dry; b = oven dry; c = acid wash

[2] Sample containers not filled completely, thus risking volatile loss, will be noted as a nonconformance at the time of inspection for log-in.

[3] 14 days from sampling, 40 days from extraction date for analysis of extract.

[4] EPA does not specifically mention *soil* oil-and-grease, TPH or EDB holding times. The aqueous holding times are 28 days.

"*" Indicates internal guidance rather than a binding regulatory preservation requirement or holding time. Exceptions may occur in particular states.

3.3.2.1 Field Compositing

Where composite samples must be taken, compositing will be done with large dedicated plastic sheets (one time use only) or with stainless steel mixing bowls. In each case, clods of soil will be broken up before being mixed with hand tools. Following mixing, the soil will be placed in a pile, sectioned into four quarters, and a small sample from each quarter will be taken and mixed together to form the composite. The composite will then be placed in a glass jar and shipped with the rest of the samples to the laboratory. The excess soil will be stored and disposed of in accordance with federal, state and local regulatory requirements.

3.3.2.2 Sample Preservation

Soil samples collected off auger flights or from the split barrel for wet chemistry analyses will be stored in glass jars and kept at approximately 4 $^{\circ}$C until delivered to the laboratory by the next day.

3.3.2.3 Decontamination Procedures

All sampling equipment will be decontaminated prior to and between use with a steam cleaner or in a detergent solution (i.e. Alconox or similar), followed by rinsing with distilled or deionized water. All decontamination rinses will be containerized until analytical information related to the samples is received. If no substances of concern are found, the decontamination rinses will be disposed of per federal, state and local regulatory requirements.

3.3.3 Field Chain of Custody

Chain of custody forms will accompany all soil samples collected and shipped for analyses (see Section 3.1.5 and Figure 7).

3.3.4 Data Reporting

All analytical data is reported to the facility contact designated on the chain of custody form.

3.4 Laboratory QA/QC Program

All water and soil samples submitted to the contract analytical laboratory for analyses will be handled and analyzed according to industry standards. Most analytical methodologies originate from USEPA SW-846 or other published EPA analytical

resources. Attachment B contains the contract laboratory Quality Assurance/Quality Control Manual.

3.5 Well Installation/Abandonment Procedures

All groundwater monitor wells have been installed so as to yield representative groundwater quality data. Installation methods will include the use of hollow stem auger or rotary wash drilling techniques with minimal introduction of drilling fluid into the borehole.

Temporary wells may remain open-hole or PVC completed if all that is required is a water level elevation measurement or a grab sample for chemical analysis. Temporary wells will be plugged within two days after being drilled by cementing them with a tremie line from total depth to grade using a neat Portland Type I cement with 5% bentonite.

Any permanent monitor wells needing to be abandoned or replaced will be drilled out or casing pulled out in its entirety. If borehole collapse occurs or casing/screen remains in the hole, the borehole will be drilled out before plugging with a tremie line and neat cement/bentonite grout. If the casing or screen cannot be removed or drilled out, the casing is cut at ground surface and is filled using a tremie line with cement mixed with 5% bentonite. Well abandonment/plugging report forms will be filed, as appropriate, with the regulatory agency within 30 days of decommissioning.

Wells installed for longer term monitoring will be 2 inches or larger in diameter and constructed of PVC, stainless steel, or Teflon material as agreed upon between KMCC and the regulatory agency. All joints will be flush threaded without the use of cementing compounds.

Monitor well completion will be in accordance with the guidelines contained in the "RCRA Groundwater Monitoring Technical Enforcement Guidance Document" (US EPA, 1986). Specifically, the annular space between the screen and borehole will be filled with a filter pack of proper gradation to provide mechanical retention of the formation sand and silt. The filter pack will extend no more than two feet above the top of the well screen. At the bottom of the screen will be a 0.5 foot or longer dense phase sampling cup.

A minimum of two feet of bentonite pellets will be placed immediately above the filter pack in the annular space between the well casing and borehole. Above the bentonite seal will be a cement/bentonite grout mixture consisting of 3 to 5 pounds of bentonite per 94 pound sack of cement with approximately 6.5 gallons of water. A tremie line will be used to place the grout from a depth to three feet below grade level. Following a suitable amount of time to allow grout settlement, the annular space from three feet below grade to grade level will be sealed with concrete, blending into a cement apron extending three feet from the outer edge of the borehole. Figure 8 has been reproduced and modified from the "RCRA Groundwater Monitoring Technical Enforcement Guidance Document" (US EPA, 1986) to show how the wells will be constructed.

Following well completion, well development will be initiated to remove any fluids used during drilling and to remove fines from the natural formation to provide a particulate free discharge.

Development will be done by reversing flow direction or surging the well. No fluids other than natural formation water will be added during development, and any collected water remaining from the development process will be put into the facility wastewater treatment system. A locking cap will be placed on all wells.

A record of drilling (Figures 9 and 10) and well construction details (Figure 11) will be completed and be kept on site. The record will include:

- date/time of construction
- drilling method/fluid use
- well location ($\pm$0.5 ft.)
- borehole diameter and well casing diameter
- well depth ($\pm$0.1 ft.)
- drilling and lithologic logs
- depth to first saturated zone
- casing material
- screen material and design
- casing and screen joint type
- screen slot size length

- filter pack material/size
- filter pack volume
- filter pack placement method
- sealant materials
- sealant volume
- sealant placement method
- surface seal design/construction
- well development procedure
- type of protective well cap
- ground surface elevation
- top of casing elevation (to 0.01 ft MSL)
- detailed drawing of well (including dimensions)

Following well construction, a certification report will be prepared by a qualified geologist or geotechnical engineer which includes an accurate log of the soil boring and depicts the location, elevations, material specifications, construction details and soil conditions encountered in the boring of the well.

All wells will be permanently numbered and surveyed by a licensed surveyor as to location ($\pm$0.5 ft) and elevation (MSL) of the top of each well casing ($\pm$0.01 ft). Well locations will be plotted on the facility base map.

Kerr McGee Chemical Corporation
Field Sampling Log

Well ID _____ Personel _____
Date _____ Involved
Time _____ _____

Weather : _____

Well Condition : _____

Well Information :
Depth to Water _____
Depth of Well _____ (see completion data)
Calculated 3 casing Volumes _____
Well Evacuation Method _____
Final Evacution Volume _____
Well Water Appearance _____

Equipment Information :
pH meter model # _____ Serial # _____
pH Calib Std _____ Lot # _____ Exp _____
_____ Lot # _____ Exp _____

SC meter model # _____ Serial # _____
SC Calib Std _____ Lot # _____ Exp _____

Field Measurements :

Time	Vol Evac	pH	Temp	SC	Observtions
pH Std					
SC Std					
pH Std					
SC Std					

Sample Collection:
Time Started _____ Method _____
Time Finished _____ Bottles _____

FIGURE 6 -

Field Sampling Log

FIELD CHAIN OF CUSTODY - Environmental Samples

Kerr-McGee Chemical Corporation	Laboratory:	Project Name:
P. O. Box 55		Shipping Document / PO #:
8000 West Lake Mead Dr.		Sampled By:
Henderson, NV 89009	Atten: Sample Receiving	Requested Turnaround:

Samples represented on this chain-of-custody are expected to be collected and analyzed using EPA protocol.
Note any discrepancies.

Sample Id	Collection		Matrix Code	Sample Type	Field Preserved (Y or N)	Container - # and Volume								Comments
	Date	Time												

Relinquished By:			**Received By:**		
(Print)	(Signature)	Date/Time	(Print)	(Signature)	Date/Time
1.					
2.					
3.					

Requested Turnaround:	Matrix Code:			Sample Type:
24 Hour Normal lab turn around	SW - Surface Water	SL - Soil	WW - Waste Water	G - Grab
48 Hour	GW - Groundwater	SD - Solid	AR - Air	C - Composite
5 Day	DW - Drinking Water	SG - Sludge	O - Other	O - Other

Distribution: Original to accompany samples / Copy to be retained by sampler

FIGURE 7

Chain of Custody

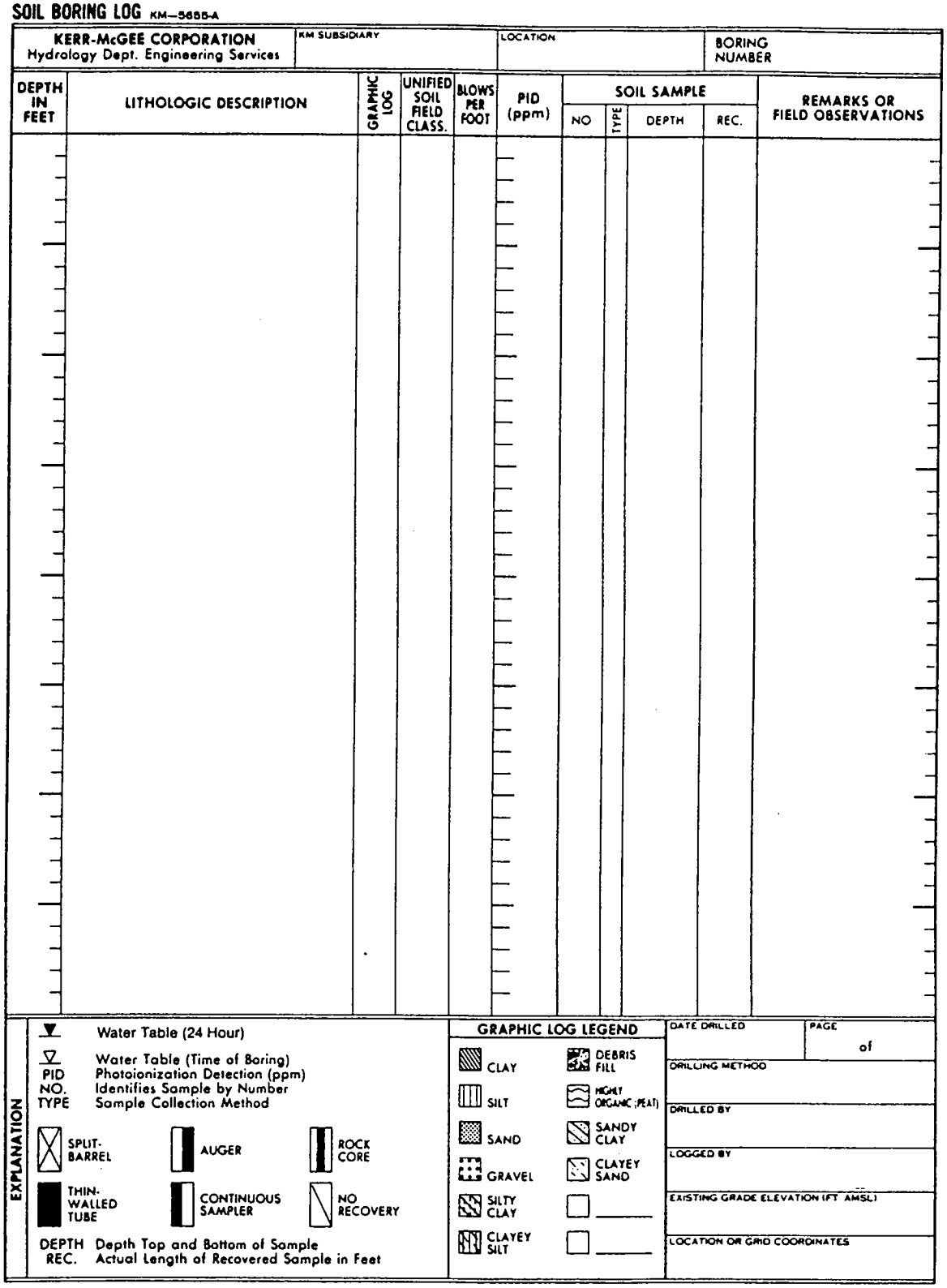


FIGURE 8

Soil Boring Log - KM-5655-A

KERR-McGEE CORPORATION
HYDROLOGY DEPARTMENT

DRILLER'S LOG SUMMARY

Bore Hole Number: _____ Rig Number: _____

Date Bore Hole Started: _____ Ground Elevation: _____

Time Bore Hole Started: _____ Weather: _____

Date Bore Hole Completed: _____

Time Bore Hole Completed: _____

Driller's Name: _____

Helper's Name: _____

Technician's Name: _____

Auger Drilling: From _____ feet to _____ feet, and

Rotary Wash Drilling: From _____ feet to _____ feet, and

Completed Bore Hole Depth: _____

Bottom Sample Depth: From _____ to _____ feet

Groundwater First Noted: _____ feet @ _____ hours (date: _____)

Depth to Groundwater _____ hours/minutes after bore hole completion: _____ (date: _____)

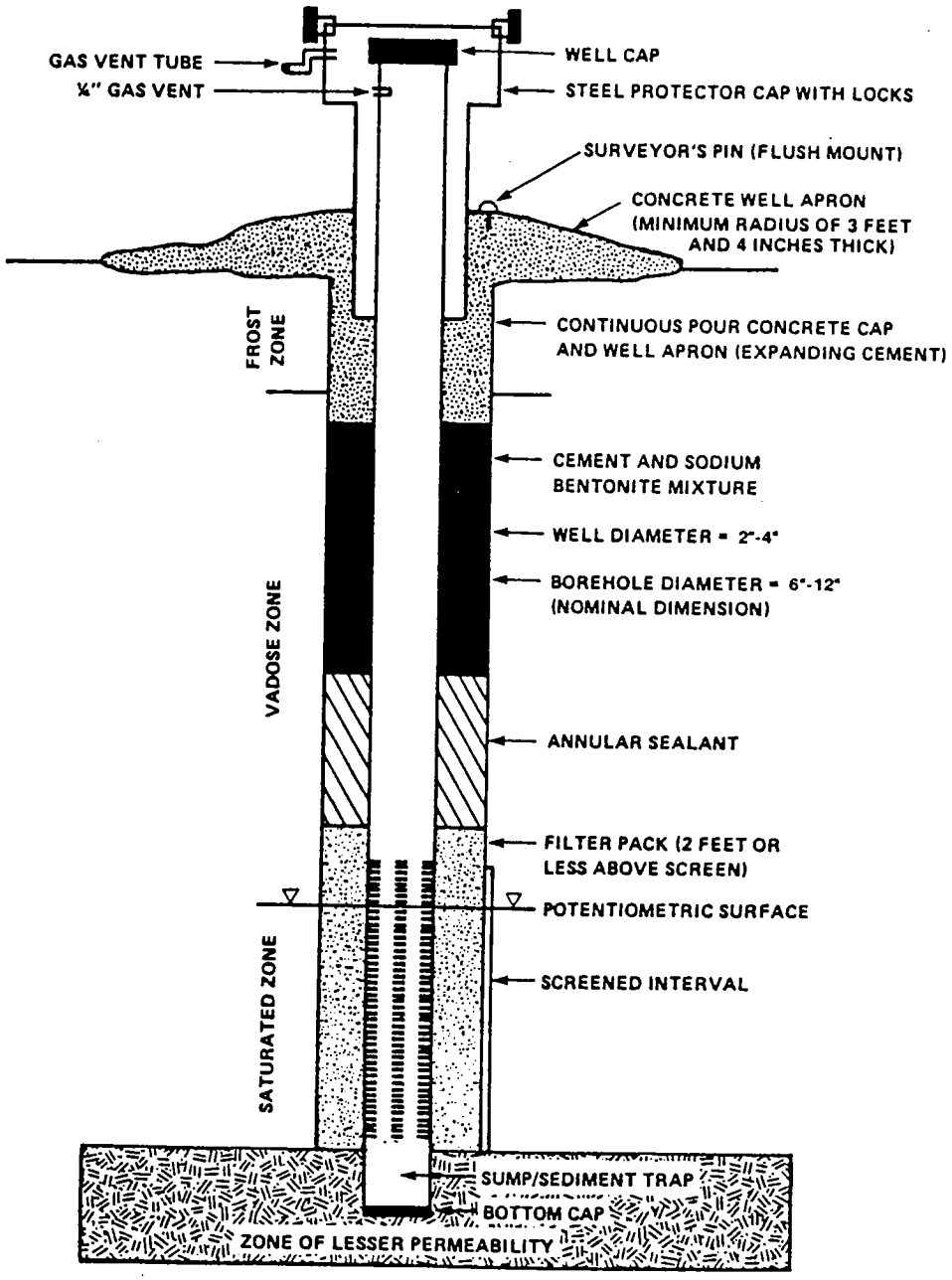
Number and Type of Samples Collected: (Please List)

Number	Type of Sample

LOCATION: _____

FIGURE 9

Driller's Log Summary



GENERAL MONITORING WELL CROSS-SECTION
(modified from USEPA, 1986)

FIGURE 10

eneral Monitoring Well Cross Section

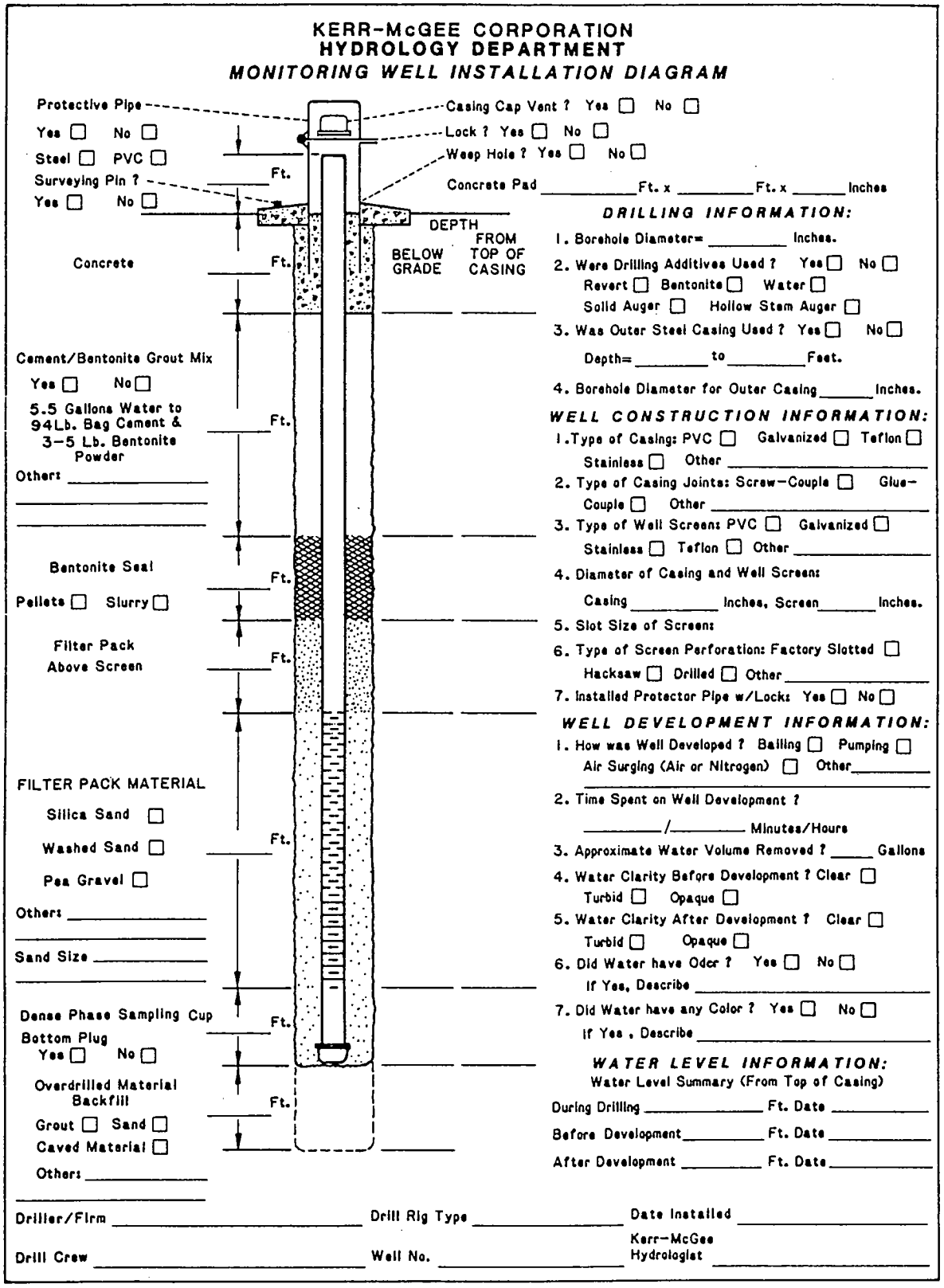


FIGURE 11

Monitoring Well Installation Diagram

REFERENCES

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American Society for Testing and Materials, 1990c, Standard method for penetration test and split-barrel sampling of soils: D1586-84, p. 298.

American Society for Testing and Materials, 1990d, Standard practice for thin-walled tube sampling of soils: D1587-83, p. 304.

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American Society for Testing and Materials, 1990f, Standard practice for description and identification of soils (visual-manual procedure): D2488-84, p. 411.

American Society for Testing and Materials, 1990g, Standard test method for liquid limit, plastic limit and plasticity index of soils: D4318-84, p. 769.

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US Environmental Protection Agency, 1982, Test methods for evaluating solid waste - physical/chemical methods, SW-846, 2nd edition, Washington, D.C.

US Environmental Protection Agency, 1986, RCRA groundwater monitoring technical enforcement guidance document, OSWER-9950.1.

US Environmental Protection Agency, 1990, Field measurements: dependable data when you need it, Washington, D.C., EPA/530/UST-90-003.

PHASE II

WORK PLAN

Section 4

Project Management

Plan

4. PROJECT MANAGEMENT PLAN

4.1 Personnel

KMCC personnel will coordinate the field related activities associated with this Work Plan. A project consultant, ENSR Consulting and Engineering will execute field sampling, related to this Work Plan, with the oversight of a Nevada Certified Environmental Manager. The work group is headed by Susan Crowley, Staff Environmental Specialist at KMCC's Henderson facility. Table 7 summarizes the key personnel for the project.

TABLE 7
Key Personnel

TITLE	COMPANY - LOCATION	NAME, PHONE
KMCC Project Manager	KMCC - Henderson, NV	Susan Crowley, 702/651-2234 (KM Net 531-2234)
ENSR Project Manager	ENSR - Camarilo, CA	Rick Simon, 805/388-3775
ENSR Asst Project Manager	ENSR - Camarillo, CA	Harold van Deinse, 805/388-3775
Site Health & Safety Officer	KMCC - Henderson, NV	Greg B. Cowley, 702/651-2228 (KM Net 531-2228)
Nevada Certified Environmental Manager	ENSR, Camarillo, CA	David Gerry, CEM # 1524 805/388-3775
Managing Hydrologist	KM - Oklahoma City, OK Corporate Office	Tom W. Reed, 405/270-2654 (KM Net 220-2654)
ENSR Hydrologist	ENSR - Camarillo, CA	D.J. Poehls, 805/388-3775
Technical Consultant	KM - OKC Tech Center	Steve R. Nelson, 405/775-5796 (KM Net 220-5796)
Regulatory Consultant	KM - OKC Headquarters	Russ Jones, 405/270-2665 (KM Net 220-2665)
Legal Counsel	KM - OKC Headquarters	Pat R. Demps, 405/270-2840 (KM Net 220-2840)
Laboratory Contact	LOCKHEED - Las Vegas, NV	Mary Ford, 702/361-1626

4.2 Reporting Requirements

After NDEP approval of this Work Plan, but not sooner than September 15, 1996, implementation of the work described in Section 2 will begin. Work will be completed as scheduled in Table 7a.

TABLE 7a

Work Schedule

Task Description	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
Task 1																	
Work Plan Approval by NDEP[1]																	
Mobilization																	
Field Activities																	
Laboratory Analysis																	
Task 2																	
Data Validation																	
Draft Report Preparation																	
KMCC Review																	
Final Report Preparation and Submittal to NDEP																	

[1] : Field activities will not begin prior to September 15, 1996, even if approval is received before this date.

PHASE II

WORK PLAN

Section 5

Health and Safety

Plan

5. HEALTH & SAFETY PLAN

5.1 Introduction

The purpose of the Health and Safety Plan (HSP) is to assign responsibilities, establish protection standards and mandatory operating procedures, and provide for contingencies that may arise while operations are being conducted at the Kerr-McGee Chemical Corporation's (KMCC) Henderson, Nevada, facility (Facility).

KMCC commissioned a Las Vegas environmental contractor, Kleinfelder, to conduct a study of the Facility environmental impacts (see resulting report titled "Final Phase I Environmental Conditions Assessment" dated April 1993). In reviewing the subsequent report, KMCC and the Nevada Department of Environmental Protection (NDEP) identified a number of locations requiring further study (Locations). KMCC developed a Phase II Work Plan describing the work to be performed (Work Plan).

This (HSP) addresses the health and safety aspects of implementing the field sampling outlined in the ECA Work Plan. The HSP contains a site-specific assessment of the hazards present and specific procedures to prevent workers from harm. The plan is divided into seven sections: Section 1 provides an overview of the plan; Section 2 provides site information; Section 3 provides a hazard assessment; Section 4 delineates work areas and procedures to move from one work "zone" to another; Section 5 delineates the personal protective equipment and procedures to be used; Section 6 consists of emergency procedures; Section 7 lists special considerations for especially dangerous operations (e.g., confined space entry).

5.1.1 Visitors

All visitors entering the exclusion and decontamination zones (see § 5.5) at the Facility will be required to read and verify compliance with HSP. In addition, visitors will be expected to comply with relevant OSHA requirements (e.g., training § 5.4 and personal protective equipment (PPE) § 5.3). As a general rule, visitors must provide their own PPE.

If a visitor does not adhere to this HSP, the visitor will be requested to leave the exclusion or decontamination zone. All nonconformance incidents will be recorded in the site log.

5.1.2 Contractors

Contractors to perform any work at the Facility, not covered under the ECA Work Plan, must submit their health and safety plans to KMCC prior to beginning work.

5.2 Site Information

The Facility is part of an industrial area known as the Basic Management Incorporated (BMI) complex (Complex). The Complex, including KMCC's property, is located in an unincorporated portion of Clark County, Nevada, and is completely surrounded by the City of Henderson. Originally sited and operated by the U. S. Government as a magnesium production facility, the Complex operated from August 1942 to November 1944 to support the war effort. KMCC's portion of the Complex will be referred to in this report as the "Facility."

Following cessation of magnesium operations, a portion of the Complex was leased from the Government by Western Electrochemical Co. (WECCO) in 1945. By 1952, WECCO had purchased various portions of the complex. In 1995, WECCO merged with American Potash and Chemical Company (AP & CC). In 1962, AP & CC purchased the current ammonium perchlorate plant, sodium perchlorate plant, and half of the sodium chlorate plant from the Government. KMCC acquired AP & CC by merger in 1967. Later, KMCC acquired the remainder of the sodium chlorate plant. In addition to the production of chemical oxidizers, the Facility also began production of manganese dioxide and several boron-based products.

5.3 Hazard Assessment

5.3.1 Potentially Present Toxic Contaminants

The toxicity of hazardous substances at the facility has a direct impact on health and safety requirements. The facility is an actively operating chemical production site and uses/stores hazardous substances for production purposes. Although many of the substances listed below are not present at the actual sampling locations, the hazard assessment portion of this document addresses the identified substances present at the facility and their toxicity characteristics with which the drilling and sampling crew may come into contact. A listing of the hazardous substances present on the site and a summary of properties related to their toxicity and hazard are presented below.

5.3.1.1 Chromium

Sodium dichromate ($Na_2Cr_2O_7$) is used in the sodium chlorate electrolytic cells to improve cell efficiency. Chromium in sodium dichromate exists in the hexavalent oxidation state — Cr(VI).

The toxicity of chromium alloys and compounds varies significantly. Chromium metals do not exhibit toxicity. Divalent and trivalent compounds of chromium have a low order of toxicity. Exposure to the dusts of ferrochrome alloys may cause lung diseases. These compounds are either insoluble or only marginally soluble in water. KMCC does not expect to encounter substantial quantities of chromium metal, di- or trivalent chromium or ferrochrome at the sampling locations.

Among all chromium compounds, only the hexavalent salts are a prime health hazard. Occupational exposure to these compounds can produce skin ulceration dermatitis, perforation of the nasal septa, and kidney damage. Hexavalent chromium from sodium dichromate has been used at the site for many years. There is a possibility of encounterering low concentrations of hexavalent chromium in the area of old P-2 and P-3 ponds and also in the area of the "Trade Effluent" Ponds which was used as the hazardous waste landfill.

Threshold limit value — time weighted average (TLV-TWA) exposure limits for di- and trivalent chromium compounds is 0.5 mg/cubic meter of air (mg/m^3) according to the American Conference of Governmental Industrial Hygienists (ACGIH). Sax's Dangerous Properties of Materials, 8th ed., Richard J. Lewis, Sr. (1992). Hexavalent chrome compounds have a TLV-TWA of 0.025 mg/m^3 according to the National Institute of Occupational Safety and Health (NIOSH). The Occupational Safety and Health Administration's (OSHA) standard for chrome VI is 0.1 mg/m^3 for short-term exposure limit (STEL).[2] Note that the TLV for hexavalent is an order of magnitude lower than for di- or trivalent chrome. Chrome VI is also considered a class A1 carcinogen (i.e., a "known" human carcinogen).

5.3.1.2 Oxidizers

Oxidizers are non-combustible substances. However, contact with combustible materials may produce flame (sometimes, with explosion). If clothing becomes soaked with chlorate or perchlorate solution which subsequently dries, flames may erupt if the clothing is exposed to friction, such as by sliding across a car seat. Note that in addition to the two compounds discussed below, dichromates are also oxidizers, however, they are not quite as strong oxidizers as perchlorate and chlorates.

[2]"Short-term" is defined as 15 minutes.

5.3.1.2.1 Perchlorates

Perchlorates are the salts (at the Facility, principally ammonium salts, NH_4ClO_4) of perchloric acid, $HClO_4$. Perchlorates are moderately soluble in water and decompose around 400°C. Health hazard is low. These compounds irritate the skin and eyes, and the dusts cause irritation of the respiratory passage.

Perchlorates are powerful oxidizing substances. These compounds may explode when mixed with combustible, organic, or other easily oxidizable compounds and subjected to heat or friction. Perchlorates may explode violently at ambient temperatures when mixed with mineral acids, finely divided metals, phosphorus, trimethyl phosphite, ammonia, or ethylenediamine. Heating to about 200°C with charcoal or hydrocarbons can cause violent explosions. A Comprehensive Guide to the Hazardous Properties of Chemical Substances, Pradyot Patnaik (1992).

5.3.1.2.2 Chlorates

Similar to perchlorates, chlorates are powerful oxidizers. Sodium chlorate is moderately soluble in water. Health hazard is low. These compounds irritate the skin and eyes, and the dusts cause irritation of the respiratory passage.

Sodium and potassium chlorates are low to moderately toxic in test animals. Oral administration produced irritation of the gastrointestinal tract, anemia, and methemoglobinemia. The oral lethal dose for 50% of test subjects (LD_{50}) for sodium, potassium, calcium and magnesium salts in rats is in the range of 1200, 1800, 2500 and 6300 mg/kg of body weight, respectively. A Comprehensive Guide to the Hazardous Properties of Chemical Substances, Pradyot Patnaik (1992).

5.3.1.3 Corrosive Materials (Acids & Bases)

By classic definition, acids are substances that dissociate yielding a proton aqueous solution. The acidity of an aqueous solution is expressed as: pH = -log[H+], where [H+] is the concentration of hydrogen ion in the solution. The lower the pH, the greater the acid strength of the solution (i.e., the greater the dissociation of the acid). Typically, acids are sour in taste.

By contrast, when bases dissociate in water, they yield a hydroxide (OH^-) ion. Since the product of [H+] and [OH^-] is a constant for water, the presence of hydroxide drives the [H^+] down, which increases the pH.

The effects of acids and bases are well known. Both acids and bases may pose acute dangers to workers. Skin exposed to acids or bases may blister and burn. If inhaled, the materials may so seriously damage the lining of the lung as to cause death.

5.3.2 Possible Hazardous Substances at Each Work Location

As described in the ECA Work Plan, KMCC plans access groundwater wells and to drill soil borings. Surface and subsurface soil and groundwater samples will be collected. Table 8 lists the hazardous substances most likely to be found at each Location.

TABLE 8
Work Locations: Possible Hazardous Substances

LOCATION	HAZARD OF CONCERN
Trade Effluent Ponds, Items No. 1 & 2.	Corrosive Materials (Acids & Caustics),
AP Pond Monitor Wells Items No. 16 & 17	Oxidizers, Chromium
Old P-2 and P-3, Items No. 7 & 8	Oxidizers, Chromium
Truck Unloading Area, Item No. 35	Corrosive Materials
AP Change House/Lab Septic Tank, Item No. 54	Corrosive Materials, Oxidizers, Organics

5.4 Work Areas

Only one of the locations is currently used by KMCC for ongoing production operations (the AP Ponds, Items No. 16 and 17). The ECA Work Plan includes a description of each location and the work to be performed. Further information is available in the "Final Phase I Environmental Conditions Assessment" prepared by Kleinfelder (April 15, 1993).

In general, none of the sites have hazardous conditions that would necessitate the assignment of specific work zones for site investigations. However, the potential for exposure to substances in the air or other direct contact is such that Level D protection, with a potential situation-specific upgrade to Level C, will be required.

Greg Cowley, the project Health and Safety Officer (HSO), will monitor all health and safety procedures.

5.5 Personnel Protection

All procedures described in this section are intended to supplement existing facility safety procedures (see Work Plan, Attachment C, the Table of Contents for the Plant Safety Manual). If more detail or clarification of an issue is needed, consult the Facility Safety Manual (Manual) or the HSO. The Manual is located in the offices of the Facility safety personnel listed in § 5.7. Note that any perceived conflict between this Plan and the Manual should be resolved in favor of the more conservative (safer) provision.

5.5.1 Medical Surveillance

Contract personnel will participate in a medical surveillance program including (but not necessarily limited to) the following:[3]

- preliminary physical examination to determine the individual's fitness for work and to establish baseline data,
- annual physical examinations to verify the effectiveness of protective measures and later determine if exposures have adversely affected the work,
- an exit physical examination if the individual has not had an examination within the last six (6) months, and
- periodic examination if an employee develops signs or symptoms indicating possible overexposure to hazardous substances or health hazards or when an employee is injured or exposed above a permissible exposure limit.

The medical exams include a medical and work history (or updated history) with special emphasis on symptoms related to the handling of hazardous substances present at the Locations, and on fitness for duty including the ability to wear any required personal protective equipment (PPE) under expected conditions (e.g., temperature extremes). All medical examination procedures will be coordinated through an individual's employer medical director.

[3] As required by 29 CFR 1910.120.

5.5.2 Air Monitoring

Air monitoring may be conducted when activities involve known or suspected atmospheric contamination, or when vapors, gases or particulates may be present. If there is a potential for toxic metal atmospheric contamination, appropriate personal exposure monitoring under the direction of a Certified Industrial Hygienist may be required.

5.5.3 Personnel Protective Equipment: Uses and Limitation

Generally, Level D PPE will be required for work activities. An upgrade to Level C PPE will be required based on the HSO's determination. Level D protection includes the following:

1) chemical resistant boots with steel toe and shank,
2) safety glasses (with side shields) or chemical splash goggles, and
3) hard hat.

Level C protection includes Items 1-3 above plus:

4) air-purifying respirator, half-face, equipment with proper filter cartridges,
5) appropriate disposable coveralls and gloves,
6) two-way communications radio (required if workers must separate beyond the point where voice communication is possible).

Half-face respirators will protect respiratory system from contaminants which are specified for the filter cartridges used. Contaminants not specified for the filter cartridges will not be properly removed and may cause acute or chronic respiratory problems. Therefore, it is imperative that the proper type of filter cartridge be installed in the respirator. Always check with the plant safety personnel to ensure that the proper cartridges are installed for the expected contaminants. Considering the range of contaminants at these sites, the following cartridges are recommended for all negative pressure respirator requirements: HEPA filter, acid-gas, and organic vapor. Facility policies may also require an "escape respirator."[4]

Further, some types of filter cartridges under go a chemical reaction with the materials to be removed from the air. These cartridges may not only become difficult to breath through if used too long, but may also lose all their effectiveness against the specified contaminant. Change filter

[4]Details will be given in the contractor orientation.

cartridges regularly as specified by the directions provided with the cartridge. Once again, if unsure check with the HSO.

No facial hair which interferes with a satisfactory seal of the respirator to the face will be allowed on personnel required to wear respiratory protective equipment.

Note that several sections of the Facility Safety Manual are relevant to the PPE to be used: § S-4 Respiratory Protection Program, § S-5 Safety Glasses, § S-10 Protective Work Clothing, and § WP-15 Glove Policy. These sections should be consulted for further guidance on proper PPE for work at the Facility.

5.5.3.1 Heat Stress

The Facility is in Henderson, Nevada, where daytime temperatures can approach 120°F in the summer. Care must be taken in order to avoid workers being overcome by heat stress due to weather conditions and the added stress of PPE. All tasks will be altered to account for conditions which pose a threat of heat-related stress. Frequency of breaks will be based upon individual worker conditions and needs, and may be mandated by the HSO. The various heat-related conditions and their symptoms are summarized below.

- Heatstroke. Linked to a failure of the thermoregulatory center of the brain, heatstroke is a serious disorder. Typically symptoms are unconsciousness or convulsions and a hot, dry appearance to the skin. Treatment includes rapid and immediate cooling (for example, by immersion in chilled water or application of wet towels). Transport the victim to a hospital while the body temperature is being reduced. Heatstroke is a medical emergency.
- Heat Exhaustion. Results from dehydration or depletion of circulating blood flow. Symptoms include fatigue, nausea, headache, giddiness, and clammy, moist skin. Victim may faint on standing and may have a weak pulse. Remove victim to a cooler environment and have victim rest and drink cool water. Electrolyte solutions may also be used. Any loss of conscience is a medical emergency.
- Heat Cramps. Consist of painful spasms of muscles. Victim should rest and drink cool liquid designed to replace electrolytes (for example, Gatorade). This condition is not a medical emergency.

Contractors shall present their heat stress program for review.

5.5.4 Training

All site personnel will be HAZWOPER trained.[5] Additionally, all personnel shall be instructed in basic hazard awareness by the HSO (or designee) prior to work on-site. Training will be augmented by crew briefings and site-specific training. Training will include:

1) history of the site,
2) acute and chronic effects of the contaminants on site,
3) requirements for PPE, its effectiveness and its limitations,
4) emergency procedures,
5) decontamination procedures, and
6) general health and safety practices.

Information concerning the health and safety hazards of the contaminants at the Locations shall be maintained at the Facility by the HSO and shall be available to personnel for examination.

The HSO shall review the contractor's respiratory protection program and coordinate training activities through the Training Supervisor at the Facility. In addition, the Facility Safety Manual contains several sections on training, in particular: § T-1, New Employee Safety Indoctrination, and § T-2 Contractor/Manpower Training Procedure Safety Indoctrination. These sections should be consulted as appropriate.

5.5.5 Exclusion Zones

An exclusion zone shall be set up when known or suspected atmospheric contaminants may be generated by site activities. The exclusion zone will immediately surround the area of contamination and will be marked with a "caution" banner. All personnel must be wearing the level of PPE specified in this plan before entering the exclusion zone.

A decontamination zone shall be set up at an area adjacent to the exclusion zone and shall be marked as such. As with the exclusion zone, personnel must be wearing HSP-specified PPE before entering the decontamination zone.

All exits from the exclusion zone must take place through the decontamination zone. All articles potentially contaminated during activities will be decontaminated or discarded prior to

[5] "HAZWOPER trained" is a person who has been trained for hazardous waste operations as per 29 CFR 1910.120(e)(3) for the purposes of this Plan.

leaving the decontamination zone. It is particularly important to remove clothing which may have been soaked with any chlorate or perchlorate solution. Remember, the dried chlorate or perchlorate may cause combustible materials (ie, cotton or synthetic clothes) to burst into flame with only the friction from sliding into a car seat.

5.5.6 Plant Security

In addition to the general security features of fencing, gates, and guards, several other features contribute to the safety of the facility: ample lighting is provided throughout the Facility; various plant personnel are equipped with two-way radios to report problems immediately; and an internal phone system (with phones in many plant areas) is provided.

Visitors and contractors entering the plant must sign a log sheet and obtain visitors passes. Visitors have narrow authorization to go only to the designated area for their work or delivery. Unlimited access once past the gate is not permitted.

5.5.7 For More Information...

Any questions about this section, the Facility Safety Manual, or PPE in general should be directed first to the KMCC contract coordinator or secondarily to KMCC corporate safety personnel. Plant safety personnel include Greg Cowley (project HSO) at 702/651-2228 and Michael Francis 702/651-2319. The KMCC Division Safety manager is Al Dooley at 405/270-2646.

5.6 EMERGENCY PROCEDURES

For the purposes of this Plan, an emergency is defined as any situation which could result in a threat to human health or the environment. Emergency situation notifications will be addressed during site orientation.

Emergency phone numbers are included in §5.6.1 of this plan. An emergency medical evacuation route is included in §5.6.2 of this plan. The Facility also has a stand-alone contingency plan. All personnel should be familiar with both the Facility plan and the information in this HSP.

5.6.1 Emergency Telephone Numbers

When calling from one location in the Facility to another, dial only the last four digits of a phone number. However, to get an outside line (ie, to call an ambulance) a nine (9) must be dialed before the number to be called. ***The in-plant emergency phone number is 3333.***

TABLE 9

Emergency Phone Numbers

Plant Emergency Number	**In case of fire, injury or any emergency...**	
	702/651-3333	**Inside facility dial 3333**
NAME	TITLE	PHONE NUMBERS(S)
Pat Corbett	Emergency Coordinator (Plant Manager)	Work 702/651-2283 Home 702/651-3229
Alternate (may be one of several people)	Alternate Emergency Coordinator	Cellular 702/596-9401
Front Desk/Receptionist	If unable to contact one of the above	702/651-2200
Susan Crowley	Environmental Specialist	Work 702/651-2234 Home 702/435-7479
Greg Cowley	Health & Safety Officer	Work 702/651-2228 Home 702/361-5414
Municipal Emergency Services	Police, Fire, Ambulance	911

5.6.2 Emergency Procedures

The emergency evacuation procedures will be covered in the contractors orientation. Figure 10 shows the in-plant evacuation meeting points (as triangles) and the off-site evacuation meeting points (as squares). Table 10 explains the emergency alarms codes.

TABLE 10

Emergency Alarm Codes

Alarm Code	Description	Meaning
* *, * *, * *, * *	1-1	Test and all clear.
* * *, * * *, * * *	3-3	No evacuation at this time.
* * * * * * * * * *	Rapid Pulse	Conduct emergency shutdown, evacuate to in-plant meeting point.
_____	Steady Tone	Evacuate to out-of-plant meeting point.

5.6.3 Special Safety Precautions; "Hot Work," Confined Space Entry, etc.

Any particularly dangerous or unusual activity must be coordinated with the HSO before work begins. In addition, a "hot work permit" (see Facility Safety Manual §WP-1) must be obtained before beginning welding, cutting, grinding, or other high temperature work. A special procedure must be followed (including obtaining a written permit) before confined space work may be performed (see Facility Safety Manual §WP-3). A confined space may be no more than entry into a ditch more than 4-feet deep, so when in doubt, contact the HSO. Other permit situations will be addressed during initial contractor orientation.

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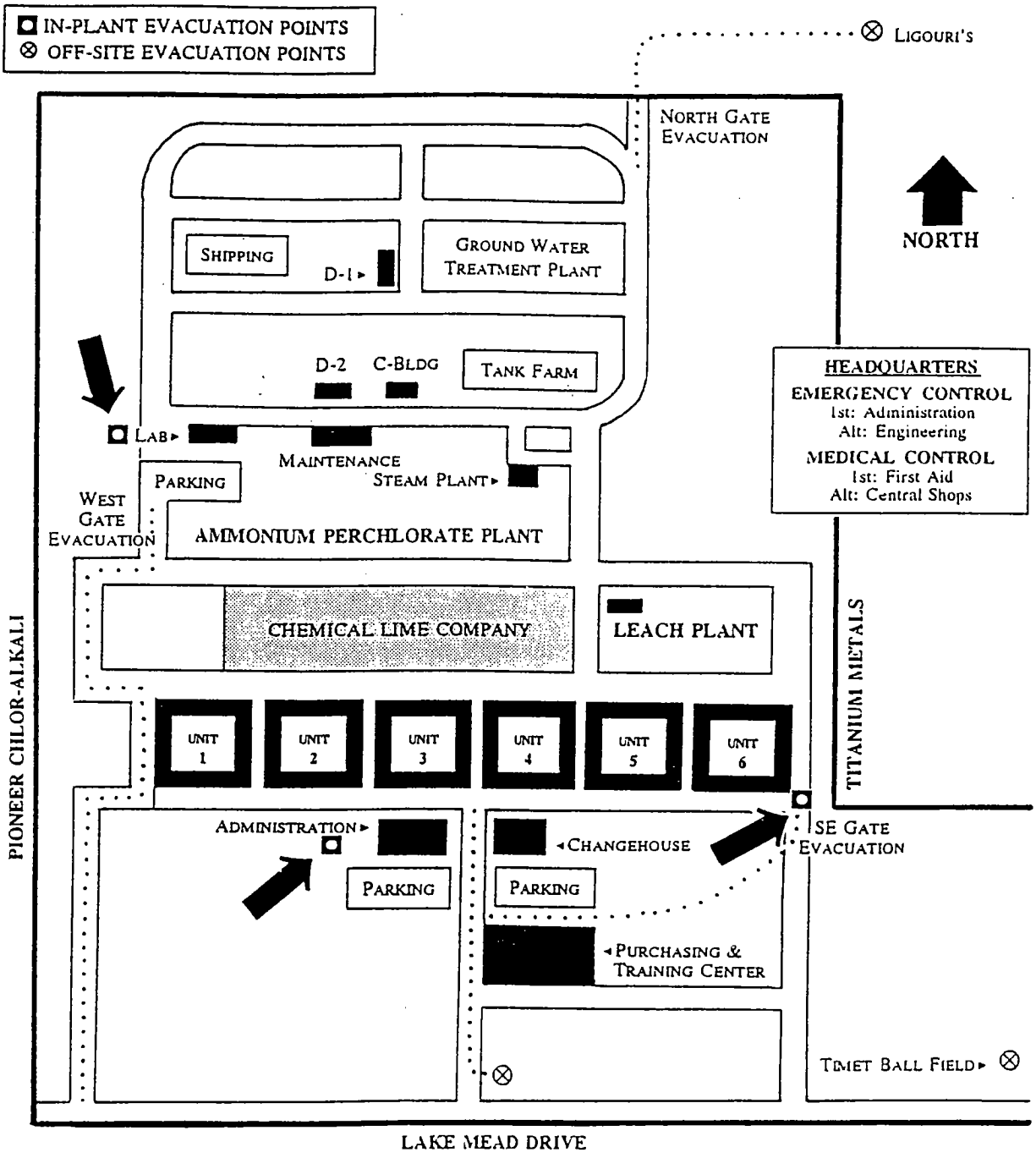
Pradyot Patnaik, A Comprehensive Guide to the Hazardous Properties of Chemical Substances, Van Nostrand Reinhold, New York City (1992).



IN-PLANT EVACUATION

For in-plant evacuation, go to the nearest meeting point. Should the nearest meeting point be unsafe, evacuate to an alternate in-plant evacuation point.

FIGURE 12
Evacuation Meeting Points

TABLE 11

List of Abbreviations and Acronyms

AP	Ammonium Perchlorate.
AP & CC	American Potash and Chemical Company (merged with WECCO and subsequently with KMCC).
BMI	Basic Management, Inc.
CFR	Code of Federal Regulations.
Complex	BMI Industrial Complex (which includes the KMCC Facility and several other facilities).
EPA	Environmental Protection Agency.
ET	Environmental Technologies Laboratories.
Facility	KMCC's Henderson, Nevada chemical complex.
HISSC	Henderson Industrial Site Steering Committee (the group has jurisdiction over, inter alia, common areas of the Complex).
KMCC	Kerr-McGee Chemical Corporation.
LOU	Letter of Understanding.
NDEP	Nevada Division of Environmental Protection.
NEL	Nevada Environmental Laboratory.
QAPP	Quality Assurance Project Plan.
RCRA	Resource Conservation and Recovery Act.
Report	Environmental Conditions Assessment report by Kleinfelder.
SAP	Sampling and Analysis Plan.
SC	Sodium Chlorate.
TCLP	Toxicity Characteristic Leaching Procedure.
WECCO	Western Electrochemical Company (merged with AP & CC).

ATTACHMENTS

ATTACHMENT A

Letter of Understanding from NDEP

L. H. DODGION
Administrator

Administration:
(702) 687-4670
Fax 687-5856

Air Quality
Mining Regulation and Reclamation
Water Quality Planning
Water Pollution Control

STATE OF NEVADA
BOB MILLER
Governor

PETER G. MORROS
Director

Fax (702) 885-0868
TDD 687-4678

Waste Management
Corrective Actions
Federal Facilities

DEPARTMENT OF CONSERVATION AND NATURAL RESOURCES

DIVISION OF ENVIRONMENTAL PROTECTION

Capitol Complex

333 W. Nye Lane

Carson City, Nevada 89710

August 15, 1994

Susan Crowley
Kerr-McGee Chemical Corporation
P.O. Box 55
Henderson, Nevada 89009-7000

Subject: Phase II Letter of Understanding Between NDEP and Kerr-McGee Chemical Corporation (KMCC)

Dear Ms. Crowley:

It is the understanding of the Nevada Division of Environmental Protection that, based upon our meetings, discussions, and correspondence with yourself and other representatives of Kerr-McGee Chemical Corporation, Kerr-McGee agrees to perform the following environmental assessment and information gathering activities at or pertaining to the KMCC's Henderson, Nevada facilities. The numbering of the particular items to be addressed follows the system used in NDEP's recommendations (dated December 16, 1992) based upon the Phase I ECA report.

1) On-Site Portions of "Trade Effluent" Settling Ponds and Associated Vitrified Clay Piping, SWMU KMCC-014:

   Provide the results of soil sampling performed by Datachem (KMCC Final Phase I Report Reference K353 "Analytical reports of soil samples taken in the vicinity of proposed SIs WC-1 and WC-2").

   Provide a work plan for characterization of potential contamination in the western portion of the KMCC "Trade Effluent" pond area (that area which lies west of Ponds WC-1 and WC-2 and east of the earthen berm which defines the eastern margin of the On-site Hazardous Waste Landfill. Historical usage and waste disposal practices are to be used to establish the list of analytes to be evaluated.

2) Open Area Due South of "Trade Effluent Disposal Ponds:

KMCC will attempt to further delineate this poorly defined historic disposal area and to establish the nature of materials deposited therein. KMCC will incorporate characterization of this area in the work plan for #1 above ("Trade Effluent" Settling Ponds).

3) Air Pollutant Emissions Associated with Industrial Processes:

Provide specific references to those passages in KMCC's Final Phase I report (and any other sources of information) which describe the nature (vapor, particulate, etc.) of historical and current air emissions at the KMCC facility. For those emissions which are determined to have been or which are presently depositional in nature, KMCC will provide information regarding patterns of dispersion and probable deposition.

4) Hardesty Chemical Company Site:

Provide analytical data obtained from sampling of the ground water monitoring wells installed on the J.B. Kelley lease site. Although these wells were installed for the evaluation of potential hydrocarbon contamination from the underground storage tanks formerly located at the J.B. Kelley site, they are in the area where Hardesty is believed to have carried out its operations. NDEP may request additional sampling of these wells with an expanded list of analytes.

KMCC will provide NDEP with any additional information regarding the past operation of Hardesty Chemical Company at the KMCC facility which may be reasonably available, including facility locations, products, waste streams, and waste disposal. KMCC and NDEP will then determine what additional investigatory work is necessary based upon the identified information concerning the activities of Hardesty at the KMCC site.

5) On-Site Portion of Beta Ditch, Including "Small Diversion Ditch" Northwest of Pond C-1:

Identify segments or tributaries of these conveyances (if any) which received waste streams from KMCC or its predecessors/tenants exclusively. Those portions of the conveyances which historically received waste streams

from two or more of the BMI companies, will be addressed as BMI Common Areas Issues. For those segments or tributaries identified as having been utilized by KMCC or its tenants exclusively, KMCC will prepare a work plan to characterize residual contamination by contaminants of concern which may exist therein.

6) Unnamed Drainage Ditch Segment:

Based upon KMCC's assertion that this ditch is in fact the Northwest Drainage Ditch which received waste streams from more than one BMI company, this area will be addressed as a BMI Common Areas issue.

7) Old P-2 Pond and Associated Conveyance Facilities:

Provide a work plan for sampling of subsurface soils in the area of the former pond to confirm that residual material concentrations are below State and Federal action levels.

8) P-3 Pond and Associated Conveyance Facilities:

KMCC will provide a work plan for sampling of subsurface soils in the area of the former pond to confirm that residual material concentrations are below State and Federal action levels. As a necessary component of this work plan, KMCC will provide additional information on the location, regulatory/closure status, and release history of this impoundment. KMCC will also provide information on the disposition of contaminated material removed from this pond.

9) New P-2 Pond and Associated Piping:

Provide engineering specifications of the impoundment including leak detection systems (e.g. double lined with leachate collection) and the location and configuration of monitor wells intended for this purpose. Provide information regarding the operational and regulatory status of this impoundment and release history (if applicable).

Issues exclusively concerning Total Dissolved Solids impacts to ground or surface water will continue to be addressed by NDEP's Bureau of Water Pollution Control.

10) On-Site Hazardous Waste Landfill, SWMU KMCC-013:

Provide the Division with copies of correspondence relating to the closure and post closure status of the landfill. This information should include the post-closure plan.

11) SWMU KMCC-005:

Provide specific information (i.e. volume of material, depth of excavation, criteria used to determine extent of contamination, etc.) relating to the removal of the "old drying pad" and underlying fill material and native soils. Provide an evaluation of the feasibility of collecting confirmatory samples of soil from beneath the area of the old pad.

12) Hazardous Waste Storage Area, SWMU KMCC-006:

No further action is required at this time.

13) Pond S-1:

No further action is required at this time. A review of the RCRA permit status of this SI may be required pending the outcome of Phase II investigations.

14) Pond P-1, and Associated Conveyance Piping:

KMCC will provide Closure documentation for this impoundment. A review of the RCRA permit status of this SI may be required pending the outcome of Phase II investigations. No further action is anticipated at this time.

15) Platinum Drying Unit, SWMU KMCC-007:

KMCC will provide either analytical data or a technically based argument supporting their contention that minor staining of the soil surrounding this unit is not a threat to either human health or the environment and is not a violation of State or Federal regulations. Included in this information shall be a discussion of how KMCC has revised housekeeping practices so as to eliminate or minimize further releases of material from this unit.

16 & 17) Ponds AP-1 and AP-2, and Associated Transfer Lines and Ponds AP-3 and Associated Transfer Lines:

Provide a technical evaluation of the appropriateness of the placement and design criteria for wells used to monitor potential contaminant migration from these impoundments. Include a list of the analytes which are currently monitored for and the latest data. Reference to the facility wide hydrologic evaluation conducted in July of 1993 may be used to provide some or all of the requested information.

Because ammonium perchlorate is highly soluble in water, and due to the fact that the ammonium ion (NH^{4+}) may be rapidly transformed to nitrate by the action of indigenous microbes in the soil through the process of nitrification, the AP pond area should be evaluated for potential ground water impacts by nitrates.

Provide an evaluation of the potential reactivity of ammonium perchlorate in the ponds and in site soils.

Provide chromium concentration data for pond contents.

Provide a summary diagram/facility map which more accurately identifies the location of the AP impoundments and the other waste management units/areas of concern at the KMCC facility. Modification of Plate 3-2 of the KMCC final Phase I report would be acceptable for this purpose.

Issues exclusively concerning Total Dissolved Solids impacts to ground or surface water will continue to be addressed by NDEP's Bureau of Water Pollution Control.

18) Pond AP-4:

Reference items 16 & 17 above. The issue of potential chromium contamination is not applicable to this impoundment.

19) Pond AP-5:

Reference items 16 & 17 above. The issue of potential chromium contamination is not applicable to this impoundment.

20) Pond C-1 and Associated Piping, SWMU KMCC-011:

This impoundment has the potential to impact ground water with elevated levels of total dissolved solids. With the

exception of manganese which has a secondary MCL of 50 ug/L, no other compounds of concern appear to have been disposed here. The potential presence of manganese in site ground water should be evaluated (reference to the KMCC hydrologic evaluation of the site performed in July of 1993 is acceptable).

Issues exclusively concerning Total Dissolved Solids impacts to ground or surface water will continue to be addressed by NDEP's Bureau of Water Pollution control. The planned closure of this impoundment should be coordinated with the BWPC as well.

21) Pond Mn-1 and Associated Piping:

Reference item 20 above. It is understood that closure of this impoundment is not anticipated by KMCC at this time.

22) Pond WC-1 and Associated Piping, SWMU KMCC-015:

No further action is required at this time.

23) Pond WC-2 and Associated Piping:

Provide information regarding the clean up of apparently contaminated soil referred to in the KMCC Final Phase I Report.

24) Leach Beds, Associated Conveyance Facilities, and Mn Tailings Area, SWMU KMCC-009:

Provide a technically based argument (which may include existing TCLP and EP Toxicity data) to demonstrate that pre-1975 disposal of slurried and solid waste to these areas will not have the potential to impact ground water with manganese.

Provide a technical evaluation of the appropriateness of the placement and design criteria for wells used to monitor potential contaminant migration from these waste management units. Include a list of the analytes which are currently monitored for and the latest monitoring data. Reference to the facility wide hydrologic evaluation conducted in July of 1993 may be used to provide some or all of the requested information.

25) Process Hardware Storage Area, SWMU KMCC-001:

No further action is required at this time.

26) Trash Storage Area:

No further action is required at this time.

27) PCB Storage Area, SWMU KMCC-003:

No further action is required at this time.

28) Hazardous Waste Storage Area, SWMU KMCC-004

Provide documentation of the remediation of hydrocarbon contaminated soil observed during Kleinfelder's site reconnaissance. This documentation should include confirmatory sampling and analysis using EPA Method 8015 modified for petroleum hydrocarbons.

29) Solid Waste Dumpsters, SWMU KMCC-008

No further action is required at this time.

30) Ammonium Perchlorate Area - Pad 35, SWMU KMCC-0017:

No further action is required at this time.

31) Drum Crushing and Recycling Area, SWMU KMCC-018:

Provide documentation of the remediation of minor soil staining in this area.

Provide information regarding improvements in area operating procedures for the removal of residual materials from drums prior to storage and crushing so as to minimize or eliminate spillage of waste materials to the ground.

32) Ground Water Remediation Unit, SWMU KMCC-019:

Provide information regarding improvements in area operating procedures for the purpose of minimizing or eliminating spillage of waste materials to the ground. Document any modifications made to the remediation unit for this purpose.

33) Sodium Perchlorate Platinum By-Product filter, SWMU KMCC-021

KMCC will provide a written statement describing the repair of floor cracks in this unit. ***Beyond this,*** no further action is required at this time.

34) Former Manganese Tailings Area, SWMU KMCC-022:

Reference item 24 above.

35) Truck Emptying/Dump Site, SWMU KMCC-025:

Provide a sampling plan for assessment/characterization of "unknown" waste materials disposed in this area.

36-38) Former Satellite Accumulation Points:

No further action is required at this time.

39) Satellite Accumulation Point - AP Maintenance Shop, SWMU KMCC-29:

Provide documentation of remediation of minor spill noted in the Phase I Report. This should include information regarding the association between the spill and the 1,1, 1-trichloroethane stored in this area.

Provide information regarding improvements in area operating procedures for the purpose of minimizing or eliminating spillage of waste materials.

40) PCB Transformer Spill:

No further action is required at this time.

41) Unit 1 Tenant Stains:

Provide documentation of remediation of hydrocarbon impacted soil in this area.

42) Unit 2 Salt Redler:

No further action is required at this time

43) Unit 4 and 5 Basements:

Provide a discussion concerning the feasibility of characterization and removal and/or stabilization of residual chromium contamination in the unsaturated zone beneath these units.

Provide, as a stand alone document, a full re-evaluation of the effectiveness of the chromium recovery system. Included should be such items as aquifer properties and characteristics, ground water flow patterns, capture and

reinjection zones, influent concentration trends, etc. A discussion of the transport and fate of chromium within the shallow aquifer and within the vadose zone beneath units 4 & 5 should also be included in this document.

44) Unit 6 Basement:

Provide a technically based discussion of the potential impacts to ground water from manganese bearing solutions and from residual high/low pH contamination in the vadose zone which may have resulted from leakage of the basement of this unit. A discussion is required of the engineering features, leak detection system(s), and periodic maintenance of the basement liner and any other appropriate method of addressing the issue of potential on-going releases. Ground water monitoring data should be used to document impacts (or lack thereof) from residual contamination beneath the unit.

45) Diesel Storage Tank:

Within 180 days of receipt of this letter of understanding, KMCC will provide the Division with a work plan designed to address visible and potential hydrocarbon contamination of soil and/or ground water in this area. If KMCC decides to renovate the tank, integrity testing (including some form of non-destructive testing of the tank bottom) will be performed. If KMCC decides to discontinue tank use, the tank will be removed and the area assessed for contamination.

46) Former Old Main Cooling Tower and Recirculation Lines:

No further action is required at this time.

47) Leach Plant Area Manganese Ore Piles:

Provide data/documentation from industrial hygiene studies to on-site workers and off-site residents from exposure to manganese ore and or manganese compounds.

48) Leach Plant Anolyte Tanks:

Provide a technical evaluation of the appropriateness of the placement and design criteria for wells used to monitor potential manganese and pH contaminant migration from this area. Include a list of the analytes which are currently monitored for and the latest data. Reference to the facility wide hydrologic evaluation conducted in July of 1993 may be used to provide some or all of the requested information.

49) Leach Plant Area Sulfuric Acid Storage Tank:

Reference item 48 above.

50) Leach Plant Area Leach Tanks:

Reference item 48 above.

51) Leach Plant Area Transfer Lines:

Reference item 48 above.

52) AP plant Area Screening Building, Dryer Building and Associated Sump:

Provide documentation of remediation of "minor white staining" from ammonium perchlorate wash downs and modifications to area procedures to mitigate or eliminate further releases of waste materials.

53) AP Plant Area Tank Farm:

Provide documentation of remediation of small visible staining and repair or replacement of the concrete pad.

Provide a discussion of procedural changes intended to mitigate or eliminate further releases of waste materials.

54) AP Plant Area Change House/Laboratory Septic Tank:

Provide a work plan for assessment/characterization of potential contamination related to waste chemical disposal via the laboratory septic system.

55) Area Affected by July 1990 Fire:

Provide documentation of the remediation of the impacted area including specific data (e.g. waste volume, etc.) regarding material disposal at U.S. Ecology.

56) AP Plant Area Old Building D-1 -- Washdown:

Provide a technically based discussion concerning the environmental fate of ammonium perchlorate in site soils (see also the requirements of item # 52 above).

57 & 58) AP Plant Area New Building D-1 -- Washdown and AP Plant Transfer Lines to Sodium Chlorate Process:

No further action is required at this time.

59) Storm Sewer System:

Provide documentation of system flow/integrity investigations as part of a technical evaluation concerning the potential for soil and/or ground water contamination resulting from waste disposal and storm water discharges through the storm sewer system.

Provide a technical evaluation of the appropriateness of the placement and design criteria for wells used to monitor potential contaminant migration from the storm sewer system. Include a list of the analytes which are currently monitored for and the latest data. Reference to the facility wide hydrologic evaluation conducted in July of 1993 may be used to provide some or all of the requested information.

60) Acid Drain System:

Provide a technically based evaluation of the potential for soil and/or ground water contamination resulting from historic waste disposal through the acid drain system.

Provide a technical evaluation of the appropriateness of the placement and design criteria for wells used to monitor potential contaminant migration from the acid system. Include a list of the analytes which are currently monitored for and the latest data. Reference to the facility wide hydrologic evaluation conducted in July of 1993 may be used to provide some or all of the requested information.

61) Old Sodium Chlorate Plant Decommissioning:

No further action is required at this time.

62) State Industries, Inc. Site, Including Impoundments and Catch Basin:

Provide a work plan for the complete assessment/ characterization of the State Industries surface impoundments. Analytes should be selected based upon known or suspected waste streams disposed to these ponds and should include TCLP metals, volatile organic compounds (if applicable), TPH (if applicable), and pH.

63) J.B. Kelley, Inc. Trucking Site:

Provide closure and/or remediation documentation for the underground storage tanks formerly located at this site. Include data from the ground water monitor wells installed by KMCC to evaluate potential hydrocarbon contamination.

Provide an assessment plan to characterize areas potentially impacted by truck washing rinsate and liquids and sludges present in the concrete vaults at this site.

64) Koch Materials Company Site:

Provide documentation of KMCC's efforts, in conjunction with those of Koch Materials Co., to remediate hydrocarbon contamination and to develop operating procedures and/or containment structures to prevent further releases of petroleum hydrocarbons and other wastes.

65) Nevada Precast Concrete Products, Green Ventures International, Buckles Construction Company, and Ebony Construction Sites:

Determine whether soil staining identified in this area is coincident with the staining referred to in item 41 above. If the staining is not coincident with this item, provide documentation of KMCC's efforts to work with these tenants for the purpose of remediating hydrocarbon contamination and developing operating procedures and/or containment structures to prevent further releases of hydrocarbon compounds and other waste materials.

66) Above-Ground Diesel Storage Tank Leased by Flintkote Co.

No further action is required at this time.

67) Delbert Madsen and Estate of Delbert Madsen Site:

Provide documentation of KMCC's efforts to work with the tenant to further assess and characterize contamination which may be present at this location.

68) Southern Nevada Auto Parts Site:

Provide documentation of KMCC's efforts to work with the tenant to further assess and characterize contamination which may be present at this location.

69) Dillon Potter Site:

No further action is required at this time.

The tasks outlined above will be incorporated (as an attachment) into the forthcoming Phase II Consent Agreement to be negotiated with KMCC. That document will provide the specific framework wherein these tasks shall be accomplished.

Should you have any questions or comments regarding any of the items, please contact either Allen Biaggi or myself at (702) 687-4670, extensions 3021 and 3017, respectively.

Sincerely,

Edward L. Basham
Environmental Management Specialist
Remediation Branch
Bureau of Corrective Actions

ELB:kmf

cc: Russell Jones, Staff Environmental Engineer, Kerr-McGee Chemical Corporation, Kerr-McGee Center, P.O. Box 25861, Oklahoma City, Oklahoma 73125

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ATTACHMENT B

LOCKHEED

Quality Assurance/

Quality Control Manual

UNCONTROLLED COPY

Lockheed
Environmental Systems & Technologies Co.

QUALITY ASSURANCE MANAGEMENT PLAN

Revision 2
June 1994

Quality Assurance Manager — Date: 06-07-94

Laboratory Director — Date: 6-7-94

TABLE OF CONTENTS

Continued...

TABLE OF CONTENTS *(Continued)*

Page No.

Continued...

TABLE OF CONTENTS *(Continued)*

Page No.

Continued...

TABLE OF CONTENTS *(Continued)*

Page No.

Continued...

TABLE OF CONTENTS *(Continued)*

APPENDICES

TABLE OF FIGURES

TABLES

ACRONYMS AND ABBREVIATIONS

AAS	atomic absorption spectroscopy
ACOE	Army Corps of Engineers
APHA	American Public Health Association
ANSI	American National Standards Institute
ASME	Americas Society of Mechanical Engineers
ASQC	American Society for Quality Control
ASTM	American Society for Testing and Materials
BFB	p-bromofluorobenzene
CCV	continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLP	Contract Laboratory Program
CPM	counts per minute
CWA	Clean Water Act
DER	duplicate error ratio
DFTPP	decafluorotriphenylphosphine
DOE	Department of Energy
DQO	data quality objective
EICP	extracted ion current profile
EML	Environmental Measurement Laboratory
EPA	U.S. Environmental Protection Agency
FIFRA	Federal Insecticide, Fungicide, and Rodenticide Act
FOM	figure of merit
FS	feasibility study
FT-IR	Fourier transform infrared spectrophotometry
FWHM	full width half maximum
GC	gas chromatograph
GC-MS	gas chromatograph-mass spectroscopy
GFAA	graphite furnace atomic absorption
GLP	good laboratory practice
GMP	good measurement practice
GPC	gas proportional counter
HEPA	high efficiency particular air
HPLC	high performance liquid chromatography
IC	ion chromatograph
ICP-AES	inductively coupled plasma/atomic emission spectroscopy
ICV	initial calibration verification
IDL	instrument detection limit
LAS	Lockheed Analytical Services
LCS	laboratory control sample
LDMS	laboratory data management system
LDR	linear dynamic range
LESAT	Lockheed Environmental Systems & Technologies Company
LLD	lower limit of detection
MDA	minimum detectable activity

(Continued...)

ACRONYMS AND ABBREVIATIONS *(Continued)*

MDL	method detection limit
MS	matrix spike
MSA	method of standard additions
MSD	matrix spike duplicate
NCAR	Nonconformance & Corrective Action Record
NEIC	National Enforcement Investigations Center
NIST	National Institute for Standards and Technology
NRC	Nuclear Regulatory Commission
OSHA	Occupational Safety and Health Administration
PE	performance evaluation
QA	quality assessment
QAMP	Quality Assurance Management Plan
QAMS	Quality Assurance Management Staff
QC	quality control
QCCS	quality control check standard
RCRA	Resource Conservation and Recovery Act
RDL	reporting detection limit
RER	replicate error ratio
RI	remedial investigation
RPD	relative percent difference
RRF	relative response factor
RRT	relative retention time
RSD	relative standard deviation
SARA	Superfund Amendments and Reauthorization Act
SDG	sample delivery group
SDWA	Safe Drinking Water Act
SOP	standard operating procedure
SOW	statement of work
SRM	standard reference material
TQM	Total Quality Management
TSCA	Toxic Substances Control Act
UPS	uninterruptable power supply
VOA	volatile organic analysis
VOC	volatile organic constituent
VTSR	validated time of sample receipt
WP	water pollution
WS	water supply

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CHAPTER 1
INTRODUCTION

This manual is an overview of the Quality Assurance Program that governs all Lockheed Analytical Services (LAS) operations. It outlines the purpose, policies, organization, responsibilities, and operations related to ensuring high-quality performance in all LAS activities. The manual is intended to provide guidance to management, project leads, laboratory analysts, and support personnel in the uniform implementation of general quality assurance requirements specific to LAS by providing a minimum set of quality management elements required to provide analytical products and services at LAS. The project-specific requirements delineated in project plans may supersede the general quality requirements described in this manual. It is essential that all LAS personnel be familiar with the policies, objectives, and procedures outlined in this management plan so that they fully understand their roles and responsibilities in the overall LAS Quality Assurance Program. Furthermore, all subcontractors employed by LAS must adhere to the set of QA requirements delineated in this manual.

1.1 POLICY

The preparation of this management plan and implementation of the quality assurance philosophy and procedures specified herein are in accordance with Lockheed Corporate quality assurance policy. Corporate regulations and guidelines require the implementation of quality assurance activities and the maintenance of sufficient documentation to demonstrate the generation of legally defensible environmental data. Lockheed corporate policies on quality are based on the following concepts:

- To achieve the mission, goals, and long-term objectives of the Corporation, we must provide our clients with products and services that satisfy their definitions and expectations of quality.

- To achieve the required quality in these products and services at a competitive price, a strategy to obtain that level of quality is necessary.

- The pursuit of quality, and its improvement, is a continuous process with measurable objectives.

- Each employee is a customer and a supplier; each is personally responsible and accountable for the quality of his or her own work.

It is the policy of Lockheed Environmental Systems & Technologies Company (LESAT) to provide products and services to its internal and external customers that satisfy or exceed their quality requirements and expectations. LAS quality policy is to produce analytical data reports that meet client and regulatory requirements, are useable for their intended purpose, are technically correct, and are produced within contract specifications. In addition, it is our corporate, company, and division policy to continually improve our procedures, products, and services. Lockheed management fully supports continuous quality improvement efforts to reduce cost without compromising quality.

Lockheed stresses the importance of quality at every level in the Corporation from the Chief

Executive Officer to the individual employee. LAS management has made a sustained commitment of personnel and resources to develop, implement, assess, and continually improve our technical and management operations. Only with management's full participation is it possible to instill this commitment to excellence in all LAS employees.

1.2 GOALS

The primary goal of the LAS Quality Assurance Program is to ensure that all measurement data generated are scientifically and legally defensible, of known and appropriate quality, and thoroughly documented so that they provide sound support for environmental decisions. A supporting goal is to comply with all environmental regulations established by local, state, and federal regulatory authorities.

The specific goals are:

- To provide a uniform framework for generating physical and chemical data.
- To operate under a comprehensive, effective, ongoing quality assurance program that focuses on preventive maintenance, which will help ensure the timely and effective completion of each measurement effort.
- To instill a commitment to quality assurance and individual excellence at all levels of the organization.
- To assist in the early detection of anomalies and nonconformances that might adversely affect data quality.
- To establish the quality assurance objectives for the measurement systems and to assess and monitor analytical data quality in terms of precision, accuracy, representativeness, comparability, completeness, and detectability through the use of proven methods.
- To establish procedures to demonstrate, through the use of control charts and other means, that analytical systems are in a state of statistical control.
- To enable personnel responsible for the production of the data to identify and implement corrective actions necessary to ensure data integrity.
- To ensure that the appropriate type and degree of quality control are applied during an analytical run.
- To ensure adequate document control.
- To eliminate data anomalies through the implementation of an automated, efficient data-handling and data-validation system.
- To develop and follow good laboratory practices (GLPs), good measurement practices (GMPs), and standard operating procedures (SOPs).
- To provide sufficient flexibility for implementing customized quality assurance procedures to meet customers' specific requirements for data quality.
- To establish guidelines for adequate control of procurement of instruments, chemicals, and services.
- To ensure proper tracking of samples and analytical data by implementing an automated laboratory data management system (LDMS).

- To ensure that computer hardware and software used in producing analytical data are independently validated and documented according to the intended use of the software.

1.3 KEY PROGRAM ELEMENTS

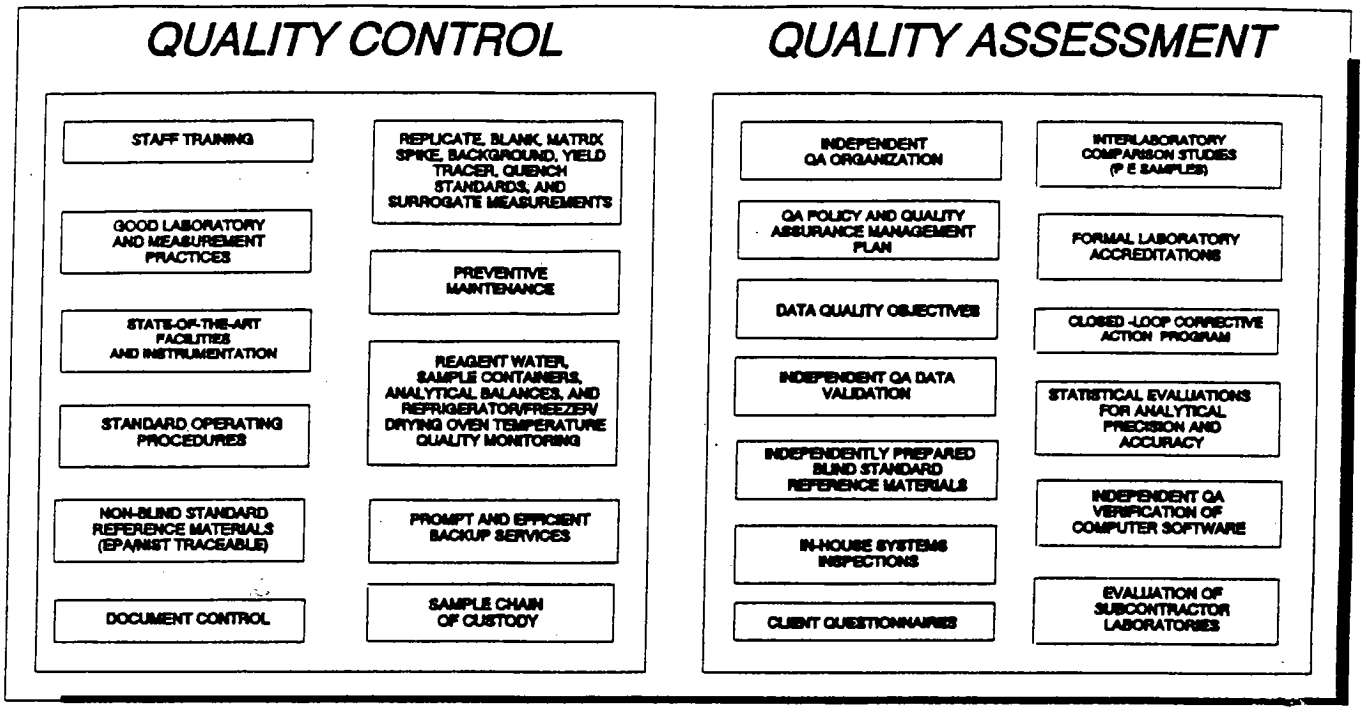
Formalizing and implementing sound quality assurance procedures are the initial steps in ensuring that data quality is within specified control limits and is well documented. The LAS Quality Assurance Program provides a quantitative approach to ensuring the integrity of all data generated by the laboratory staff. Key elements of the program, shown in Figure 1, help ensure that all analytical data meet customer, LAS, and regulatory requirements. Through application of these elements, the program monitors LAS performance in relation to quality assurance objectives and quality control requirements identified for each analytical method. This approach incorporates the proper control, assessment, and documentation of analytical data quality.

1.4 SUPPORTING DOCUMENTS

The LAS Quality Assurance Program is responsive to, and in compliance with, the guidelines and specifications described in the following documents:

- U.S. Environmental Protection Agency (EPA) Quality Assurance Management Staff (EPA QAMS-005/80).
- American National Standard Institute/American Society for Quality Control (ANSI/ASQC-E4-19xx).
- American National Standard Institute/American Society for Quality Control (ANSI/ASQC Q94-1987; ISO 9004).
- International Standard ISO/IEC Guide 25.
- U.S. Department of Energy (DOE) Order 5700.6C "Quality Assurance."
- American Society of Mechanical Engineers (ASME) NQA-1 (1989 Edition).
- Test Methods for Evaluating Solid Waste (EPA SW-846 Third Edition, 1986 and current updates).
- Methods for Chemical Analysis of Water and Wastes (EPA-600/4-79-020).
- Current revisions of the EPA Contract Laboratory Program (CLP) Statements of Work (SOW) for inorganic and organic analyses.
- Handbook for Analytical Quality Control in Water and Wastewater Laboratories (EPA-600/ 4-79-019).
- Good Laboratory Practice Standards (40 CFR Part 792).
- Standard Methods for the Examination of Water and Wastewater (18th Edition, 1992).
- Annual Standards Books of the American Society for Testing and Materials (ASTM).
- EPA Prescribed Procedures for Measurement of Radioactivity in Drinking Water (EPA-600/ 4-80/032).
- EPA Radiochemical Analytical Procedures for Analysis of Environmental Samples (EPA-LV-0539-17).

Figure 1. Key Elements of the LAS Quality Assurance Program



- EPA Eastern Environmental Radiation Facility Radiochemistry Procedures Manual (EPA/520/5-84/006).

- EPA Procedures for Radiochemical Analysis of Nuclear Reactor Aqueous Solutions (R4-73-0144).

- HASL Environmental Measurements Laboratory Procedures Manual (HASL-300).

Furthermore, the LAS Quality Assurance Program follows standards and requirements mandated by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA); the Resource Conservation and Recovery Act (RCRA); the Superfund Amendments and Reauthorization Act (SARA); the Toxic Substances Control Act (TSCA); the Nuclear Regulatory Commission (NRC); and the Occupational Safety and Health Administration (OSHA).

1.5 DEFINITIONS

The terms quality assurance, quality assessment, and quality control appear frequently in this plan and are central to the themes presented. These terms are defined as follows:

Quality assurance is the total, integrated program for ensuring the integrity of measurement data to support environmental decisions and potential litigation challenges. It consists of two separate but related activities: quality assessment and quality control.

Quality assessment (QA) is the overall system of activities enacted to ensure that the quality control activities are effective. It involves continuously evaluating the performance of the data, the production system, and the quality of the analytical data generated.

Quality control (QC) is the routine application of procedures to control the quality of

measurement data and to ensure that data meet the needs of the customer. QC procedures are used to maintain a measurement process in a state of statistical control; that is, a state in which the analytical system is stable and data are reproducible within defined limits. The aim of QC is to provide data of a quality that is satisfactory, adequate, dependable, and economical.

1.6 CONFORMANCE TO NATIONALLY RECOGNIZED STANDARDS

This plan addresses the quality assurance requirements outlined in U.S. EPA QAMS-005/80, ANSI/ASQC-E4-19xx, DOE Order 5700.6C (1991 edition), ANSI/ASQC Q94 (ISO 9004), and NQA-1. Table 1 identifies the sections of this plan and the SOPs in which requirements are addressed. The Quality Assurance Management Plan (QAMP) is supported by SOPs, which are revised routinely to reflect LAS current operations. Procedures described in the SOPs that are revised following this revision of the QAMP supersede the requirements specified in this document. References to SOPs that detail the specific elements of the LAS Quality Assurance Program are also provided in each section of the manual. A complete index of the SOPs is presented in Appendix C and is current as of the publication date of this manual.

Table 1. Sections in the LAS Quality Assurance Management Plan and Standard Operating Procedures that address EPA, NQA-1, ANSI/ASQC, DOE, and ISO Requirements (Page 1 of 3)

ASME NQA-1 Requirements	QAMP Section (SOP#)	EPA QAMS-005 Requirements	QAMP Section (SOP#)
Organization	2	Title Page	Title Page
QA Program	1	Table of Contents	Table of Contents
Design Control	3	Project Description	1
Procurement Document Control	16 (232,284)	Project Organization and Responsibility	2
Instructions, Procedures, and Drawings	5 (method SOPs)	QA Objectives	1,3 (method SOPs)
Document Control	10 (1,127)	Sampling Procedures	4(2,9,85,105,173)
Control of Purchased Items and Services	16 (232,284)	Sample Custody	4,13 (2,9)
Identification and Control of Items	15 (227,283)	Calibration Procedures and Frequency	5,6,7,8 (method SOPs)
Control of Processes	4,6,7,8,11,12 (method SOPs)	Analytical Procedures	5,6,7,8 (method SOPs)
Inspection	9 (10)	Data Analysis, Reduction, Validation	10,11,12
		and Reporting	(8,12,13,88,278)
Test Control	6,7,8 (method SOPs)	Internal QC Checks and Frequency	6,7,8 (method SOPs)
Control of Measuring and Test Equipment	14 (15,188)	Performance and System Audits	9 (10)
Handling, Storage, and Shipping	4,10,13 (2,9,85,105,173)	Preventative Maintenance	14 (15,188)
Inspection, Test, and Operating Status	9,11 (8,10)	Assessment of Precision, Accuracy and Completeness	12 (8,12,13,88)
Control of Nonconforming Items	15,16 (227,232,283,284)	Corrective Actions	15 (190)
Corrective Action	15 (190)	QA Reports to Management	17
Quality Assurance Records	10,11,13,17 (1,6,127)		
Audits	9 (10)		

Table 1. Sections in the LAS Quality Assurance Management Plan and Standard Operating Procedures that address EPA, NQA-1, ANSI/ASQC, DOE, and ISO Requirements (Page 2 of 3)

ANSI/ASQC E4-19xx Requirements	QAMP Section (SOP#)
PART A: MANAGEMENT SYSTEMS	
Management and Organization	1,2
Quality System and Description	1,2,3
Personnel Qualification and Training	2 (110)
Procurement of Items and Services	16 (232,284)
Documents and Records	10,17 (1,6,127)
Computer Hardware & Software	10 (123,124)
Planning	3,14,15
Implementation of Work Processes	4,5,6,7,8 (method SOPs)
Assessment and Response	9,11,12,17 (8,10,12,13,88)
Quality Improvement	1,3,6,7,8 (8,10,12,13,88,188,190)
PART B: COLLECTION AND EVALUATION OF ENVIRONMENTAL DATA	
Planning and Scoping	1,3,15
Design of Data Collection Operation	3
Implementation of Planned Operations	4,5,6,7,8 (method SOP)
Assessment and Response	9,11,12,17 (8,10,12,13,88)
Assessment and Verification of Data Useability	N/A
PART C: DESIGN, CONSTRUCTION, AND OPERATION OF ENGINEERED ENVIRONMENTAL SYSTEMS	N/A

DOE ORDER 5700.6C	QAMP Section (SOP#)
PART 1: MANAGEMENT	
Program	1
Personnel Qualification and Training	2 (110)
Quality Improvement	1,3 (8,10,12,13,88,188,190)
Documents and Records	10,17 (1,6,127)
PART 2: PERFORMANCE	
Work Processes	4,5,6,7,8,10, 11,12,13,14,15,16 (method SOPs & 2,9,227,283)
Design	N/A
Procurement	16 (232,284)
Inspection and Acceptance Testing	4,6,7,8,9,11,14,17 (10,284)
PART 3: ASSESSMENT	
Self/Management Assessment	9,11,12,15,17 (8,10,12,13,88)
Independent Assessment	9,11,12 (8,10,12,13,88)

Table 1. ***Sections in the LAS Quality Assurance Management Plan and Standard Operating Procedures that address EPA, NQA-1, ANSI/ASQC, DOE, and ISO Requirements (Page 3 of 3)***

ANSI/ASQC Q94 (ISO-9004) Requirements	QAMP Section (SOP#)
4. Management Responsibility	1
5. Quality System Principles	1,3
5.2 Structure of the Quality System	2
5.3 Documentation of the Quality System	1,2,3
5.4 Auditing the Quality System	9 (10)
6. Economics-Quality Related Cost Considerations	N/A
7. Quality In Marketing	N/A
8. Quality in Specification & Design	N/A
9. Quality in Procurement	16 (227,232,284)
10. Quality in Production	4,5,6,7,8,10 (method SOPs)
11. Control of Production	4,6,7,8,16 (method SOPs & 5,284)
11.2 Material Control & Traceability	4,16 (5,284)
12. Product Verification	6,7,8,9,11,12 (method SOPs)
13. Control of Measuring & Test Equipment	14 (15,188)
14. Non-Conformity	15 (190)
15. Corrective Action	15 (190)
16. Handling & Post Production Functions	4,10,13
17. Quality Documentation & Records	10 (1,6,127)
18. Personnel (Training)	2 (110)
19. Product Safety and Liability	1 (Safety Manual)
20. Use of Statistical Methods	6,7,8,12

CHAPTER 2
LABORATORY ORGANIZATION AND RESPONSIBILITIES

2.1 ORGANIZATIONAL STRUCTURE

The LAS organizational structure, shown in Figure 2, is designed to ensure that analytical operations are effective and cost-efficient. All levels of the laboratory staff are involved in implementing the formal LAS Quality Assurance Program; responsibilities of each staff level are described below.

It is Lockheed's policy to staff technical and quality assurance positions with personnel who have the education, training, and experience sufficient to competently accomplish their assigned duties. The LAS Director is responsible for ensuring that all personnel receive auxiliary training, as needed, to increase the understanding and skills that they apply to their positions.

2.2 AUTHORITY AND RESPONSIBILITY

The LAS management recognizes that the responsibility for a high-quality product starts with each employee; however, the ultimate responsibility for data and service quality and reliability resides with the Director. The LAS Director has appointed a Quality Assurance Manager to implement the LAS Quality Assurance Program and to provide continuous, independent oversight of laboratory operations. The Quality Assurance Manager is independent of daily laboratory activities and reports directly to the LAS Director to ensure unbiased evaluation of operations. The Quality Assurance Manager is the focal point for information on the LAS Quality Assurance Program and is responsible for providing advice and assistance to laboratory staff and management. The Quality Assurance Manager also has the authority to cease analytical activities that are out of control.

2.2.1 LAS Director

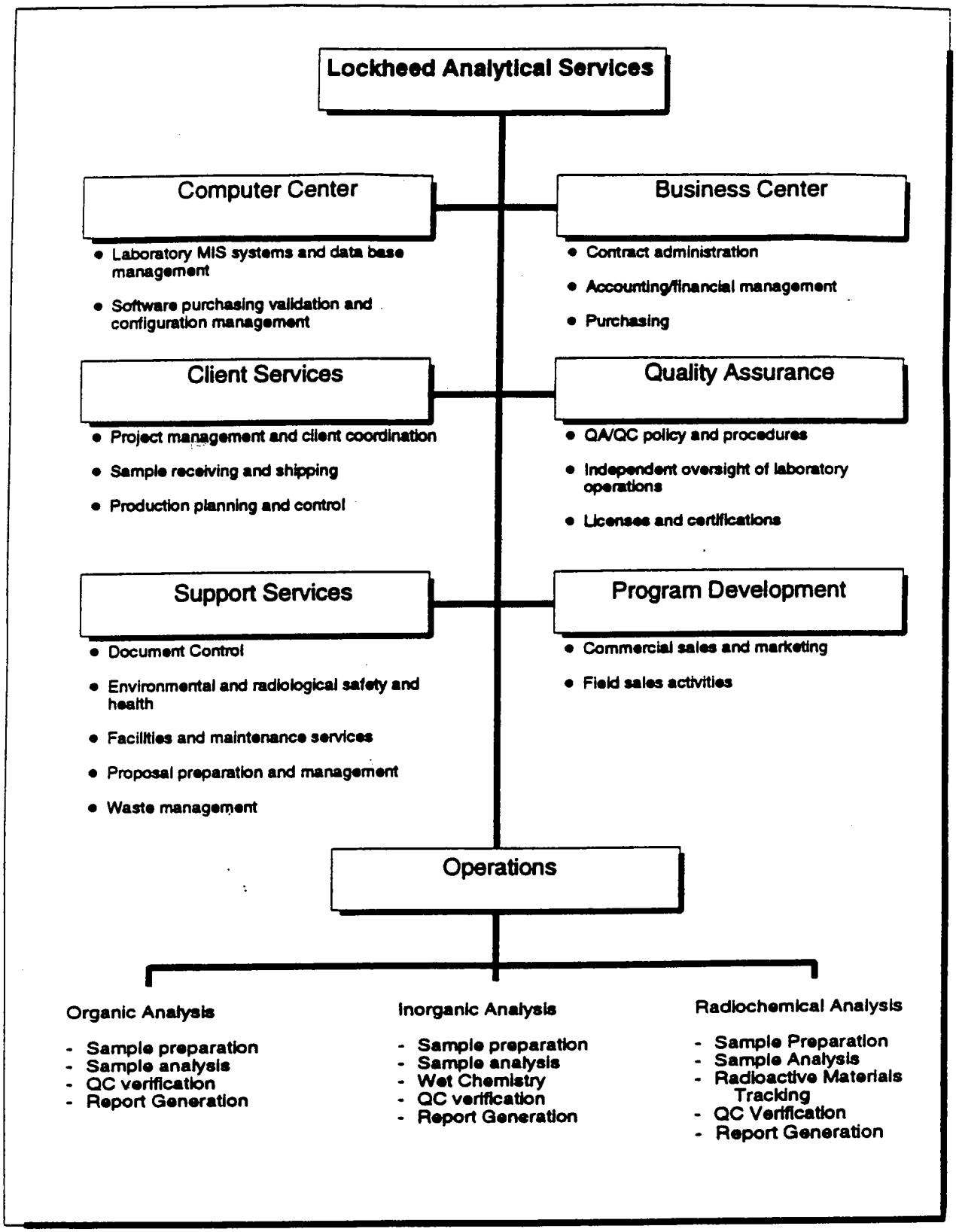
The LAS Director has the ultimate responsibility for ensuring that data and service quality meet or surpass the client's requirements. Additional responsibilities include:

- Supporting quality assurance as an essential requirement in all functional, management, and administrative areas.
- Providing the resources necessary to support an effective, ongoing, and comprehensive quality assurance program.
- Communicating management's commitment to quality assurance throughout the organization.
- Motivating all personnel to achieve increasing levels of technical competence and responsibility.
- Holding formal LAS Quality Assurance Program reviews to provide a forum for determining ways to improve LAS operations.

2.2.2 Quality Assurance Manager

The Quality Assurance Manager reports directly to the LAS Director and is responsible for designing, implementing and maintaining the LAS Quality Assurance Program in a timely, accurate, and consistent basis, and for taking or recommending corrective actions, as required. Additional responsibilities include:

Figure 2. The LAS Organizational Structure



- Establishing and maintaining a comprehensive, effective quality assurance program.

- Developing, evaluating, and documenting quality assurance policy and procedures appropriate to all laboratory functional areas with the LAS management.

- Ensuring that all laboratory operations are conducted in accordance with the LAS Quality Assurance Program and with the QA and QC requirements specific to each analytical method.

- Ensuring that all laboratory activities comply with local, state, and federal environmental regulations.

- Reviewing project-specific quality assurance plans.

- Ensuring that QC limits are established and followed for critical points in all measurement processes, and that they are based on sound statistical methods.

- Initiating internal performance audits using certified, high-purity standard reference materials (SRMs) purchased commercially.

- Performing independent QA review of a predetermined quantity of data reports.

- Informing management of system breakdowns or deficiencies, recommending corrective actions to improve the data-generation system, and defining the validity of data generated during all out-of-control situations.

- Preparing and revising quality assurance-related documents (e.g., SOPs) and periodic quality assurance reports to management.

- Advising and training laboratory staff in quality assurance practices central to their work.

- Conducting periodic technical systems evaluations of the laboratory facilities, instrumentation, and operations.

- Coordinating interlaboratory comparison studies.

- Overseeing the evaluation of locally developed computer software.

- Overseeing the LAS Training Program activities.

- Evaluating subcontractors and vendors that provide analytical and calibration services.

- Administering the laboratory accreditation and licensing activities.

2.2.3 Operations Manager

The Operations Manager reports directly to the LAS Director and is ultimately responsible for timely and accurate analysis of samples and generation of data. The Operations Manager responds to analytical requests for quotation and recommends proper analytical methods. Furthermore, this individual has the authority to reject samples that are inappropriate for analysis and to request repreparation and reanalysis of samples that are of questionable technical quality. The Operations Manager is responsible for overseeing the following laboratory functions:

- Status and progress of analytical workload.

- Coordination of sample preparation and analytical work.

- Analytical data production and review.
- Report production.

2.2.4 Client Services Manager

The Client Services Manager reports directly to the LAS Director and serves as the interface between the laboratory and the client. The Client Services Manager oversees the following functions:

- Working with clients to define requirements for analytical methodologies, quality assurance, report and deliverable timelines, and pricing.
- Working with LAS management to determine acceptable scheduling.
- Informing managers of the pending arrival of samples for analysis.
- Identifying and providing oversight of analytical services subcontractors and receipt of data reports.
- Tracking revenue/sample projections and reviewing invoices for completed work.
- Predicting potential workload.
- Sample receipt, log in, identification, storage, and tracking.
- Managing inventories of reagents, solvents, and materials.

2.2.5 Program Development Manager

The Program Development Manager reports directly to the LAS Director and oversees the following functions:

- Developing and implementing effective marketing and sales strategies.
- Identifying market trends, including new areas of service and revenue projections.

2.2.6 Support Services Manager

The Support Services Manager is responsible for providing administrative, maintenance, security, safety, and health support services for the timely implementation of laboratory operations. Specific responsibilities include overseeing the following activities:

- Maintenance and security of laboratory support equipment and facilities.
- Maintenance and archiving of laboratory records and documents.
- Developing programs to ensure compliance with laboratory environmental and radiation safety and health requirements.
- Providing waste management services.

2.2.7 Business Manager

The Business Manager oversees the functions of purchasing, accounts payable, payroll, property control, financial forecasting, invoicing, accounts receivable, and administers laboratory contracts.

2.2.8 Information Systems Manager

The Information Systems Manager reports directly to the LAS Director and oversees the following functions:

- Directing the laboratory data system (Oracle based) operations.

- Overseeing operation of network hardware and software, including distribution of personal computers (PCs).

- Developing, testing, and modifying new or existing data base applications.

- Controlling access to the laboratory minicomputer thorough management of user accounts.

- Providing technical support to LAS staff for problem solving and strategic planning.

2.2.9 Supervisors

Supervisors provide the critical link between upper management, laboratory technical personnel, and support staff. Section supervisors support the Operations Manager in formulating laboratory policy and in planning and scheduling laboratory operations. Supervisors oversee and direct the work flow in their respective groups on a daily basis. Areas of supervisory responsibility include:

- Scheduling sample preparation and analysis with regard to holding time, QA and QC, and data turnaround requirements.

- Offering guidance in the selection of equipment and methods.

- Providing guidance in resolving analytical and other technical problems encountered during sample and data preparation, analysis, and documentation.

- Assigning work priorities within the group.

- Keeping abreast of (1) preventive maintenance and repairs necessary for all equipment in the group and (2) inventory of consumables in stock.

- Ensuring that technical review of 100% of the data generated by the section is performed.

- Checking logbooks, data reporting forms, etc., on a scheduled basis.

- Reviewing completed project work.

- Ensuring that all new employees in the group receive proper training and are qualified and proficient in performing assigned tasks.

- Participating in staffing selection for the group.

Any or all of these functions may need to be delegated to technical specialists or other senior staff.

2.2.10 Laboratory Technical Personnel

The nonsupervisory technical staff of the laboratory must understand the importance of the LAS Quality Assurance Program and their individual responsibilities in ensuring the success of the program. The individual shall maintain continuous awareness of good laboratory and safety practices, recognize potential sources of error in assigned tasks, report observed substandard conditions or practices, and generally use good judgment in daily activities. In particular, each technical employee is responsible for:

- Implementing the policies contained in this document.

- Using QC procedures and SOPs properly during sample preparation, sample analysis, data generation, or any routinely performed activity.

- Properly maintaining complete documentation of laboratory activities.

- Correcting and thoroughly documenting problems and deficiencies in any portion of the measurement system.

- Evaluating 100% of data for acceptability, on the basis of method-specified QC limits and professional judgment.

- Ensuring that all laboratory operations are performed according to the appropriate governmental and laboratory health and safety program.

2.3 TRAINING PROGRAM

Training ensures (1) that the LAS staff consistently produces high-quality work; (2) the individual, regardless of his or her technical, managerial, or support role, is capable of performing the assigned task, (3) has the proper level of education and background for the specific job classification, (4) has received the appropriate orientation and indoctrination into LAS operations; and (5) proper mechanisms are in place to enhance employee skill levels. The quality assurance aspects of job performance are central to the training that LAS technical personnel receive. The LAL-91-SOP-0110 provides a detail description of the training program requirements. The following subsections provide an overview of the training program at LAS.

2.3.1 Orientation and Indoctrination

Orientation introduces the individual to existing systems and operating conditions, and indoctrination trains the individual in the principles and operations of those systems and tasks. LAS Quality Assurance Department representatives and laboratory supervisors train staff and instruct them in task-specific QC data recording and related activities. In general, in addition to instruction in environmental safety and health aspects of their positions, LAS technical staff are trained in the following areas as they apply to their positions:

- Purpose and significance of the LAS Quality Assurance Program.

- Standard operating procedures.

- LAS Quality Assurance Policy and ethics related to analytical data production.

- Overview of federal, state, and local regulations.

- LAS Notebook Policy and Guidelines for Maintaining Laboratory Logbooks and Records.

- QA and QC responsibilities.

- Nonconformance and corrective action identification and documentation.

- Client-specific data reporting requirements.

- Proper use and frequency of blanks, replicates, spikes, surrogates, and other QC samples and implementation of corrective actions.

- Technical review of analytical data.

- Standard reference material traceability.

- Proper receipt and handling of environmental samples (chain of custody).

- Data archival and retrieval practices.

Furthermore, the Operations Manager, the section supervisors and the technical staff are responsible for providing continuous, on-the-job

training by monitoring activities of subordinates and by preparing and reviewing SOPs. In some instances, individual training may be appropriate so that the new employee will learn correctly and rapidly. This approach provides knowledge and the technical skills that are necessary to perform the required tasks and it emphasizes the importance of high-quality performance. Specifically, each LAS technical employee shall be trained in the following basic laboratory operations as they relate to his/her task assignments:

- Sample receipt and logging.
- Sample handling and measuring.
- Instrument tuning and calibration.
- Data recording, handling, and reporting.
- Support equipment maintenance.
- Sample tracking through LDMS.
- Waste handling.

Dry runs and simulations of sample processing activities may be incorporated into the training program to ensure that protocols are clearly understood and that problems are identified and solved before a project begins.

2.3.2 Proficiency Monitoring and Retraining

Personnel who must perform tasks that require special skills or abilities must first demonstrate their proficiency in the assigned task under an instructor's supervision. Proficiency of each analyst is monitored by the section supervisor or designee by reviewing the analytical data generated from repetitive preparation and measurements of QC samples such as laboratory control standards, matrix spikes and matrix spike duplicates, replicates, surrogate spikes, method blanks, and blind PE samples. Independent blind PE materials obtained from approved vendors are routinely submitted by the QA Department staff to the operations staff to assess and monitor instrument, method, and analysts' performance in analyzing inorganic and organic constituents in various sample matrices. Results are evaluated against the vendor's certified acceptance limits. Any analysis that is associated with unacceptable results is further monitored by sending another set of PE materials. An overall passing score of 80 percent based on the number of acceptable analyte determinations in the PE sample is used to assess performance level. On the basis of unacceptable results, the QA staff may recommend that the sample preparation staff or the analyst need additional training. The proficiency of analysts is also monitored through successful analysis of external PE studies. The result provides a practical demonstration of the ability of the analyst to maintain performance within acceptance limits. This approach also provides a way to identify deficiencies in performance, to learn more about a new method, to understand a new project, and to learn how to use an instrument and solve problems. Qualification is considered valid for approximately two years (unless revoked for a reason) at which time the person's qualifications will need to be reevaluated and requalified.

Furthermore, to provide safe working conditions and to protect the public health, all new LAS employees who may be exposed to hazardous materials receive training from the LAS Safety Officer before they begin work. The Safety Officer schedules and presents hazardous materials awareness, hazardous materials Phase I, hazardous materials Phase II, and radiation safety courses. Training on laboratory safety is given when an employee is assigned new duties.

2.3.3 Training Records

This vital activity is thoroughly documented in the form of technical, SOP, and proficiency training records; need assessment for procedure specific training; training attendance sheets; SOP review records; personnel training records; hazard communication standard documentation; and memoranda describing the specific training activities.

Qualifications of all professional, technical, and support personnel are documented via resumes, which include academic credentials, employment history, experience, and professional registration or certification, as appropriate for the position.

A data base provides essential information on the current status of the procedure-specific training that each technical staff member has received.

Copies of all training records are maintained in a secure file cabinet. QA staff members coordinate the entry and filing of the appropriate documents to the training data base and training files.

CHAPTER 3
DATA QUALITY OBJECTIVES

Data Quality Objectives (DQOs) are the foundation for collecting environmental data that can provide a reliable basis for decisions concerning environmental remediation. The quantitative measurements that estimate the true value or true concentration of a physical or chemical property always involve some level of uncertainty. The uncertainty associated with a sample generally results from (1) natural variability of the sample, (2) sample handling operations and conditions, (3) spatial and temporal patterns, and (4) analytical variability. For an environmental data collection project, the uncertainties must be estimated and compared to standard, quantitative indicators of data quality (i.e., DQOs).

Typically, DQOs are identified during project scoping and development of sampling and analysis plans. In this manual, however, we discuss only the analytical DQOs because LAS generally does not have any jurisdiction over sample collection, shipment, or other field-related activities that may affect the data quality of the environmental sample before the sample is received in the laboratory.

DQOs are established to meet method- or client-specific requirements and to ensure that the data collected are sufficient and of adequate quality for their intended uses (EPA, 1987a). EPA has established six primary analytical DQOs for environmental studies: precision, accuracy, representativeness, completeness, comparability, and detectability. Section 3.1 addresses precision and accuracy which is augmented by Appendix A which lists analytical DQOs for precision, accuracy, and detectability for inorganic, organic, and radionuclide constituents. Sections 3.2 through 3.5 address the qualitative requirements for completeness, representativeness, and comparability, and detectability, respectively.

The components of analytical variability (uncertainty) can be estimated when QA and QC samples of the right types and quantities are incorporated into measurement procedures at the analytical laboratory. At the LAS, numerous QA and QC samples are analyzed to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QA and QC samples and their applications, described in tables 2 and 3, are selected on the basis of method- or client-specific requirements. Field blanks, field duplicates, and performance evaluation (PE) samples, which are described in Table 2, are received from the client as unknown samples. Analytical laboratory QC samples for inorganic, organic, and radionuclide analyses include calibration or instrument blanks, method blanks, background, duplicates, replicates, laboratory control samples (LCSs), calibration standards, matrix spikes (MSs), matrix spike duplicates (MSDs), surrogate spikes, and yield tracers.

3.1 PRECISION AND ACCURACY

Precision is an estimate of variability. In other words, it is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions (EPA, 1980). The precision of a measurement system is affected by random errors. Precision is expressed either as relative standard deviation (RSD) for replicate measurements greater than two or as relative percent difference (RPD) for duplicate measurements. RSD for replicate measurements ($n>2$) is calculated as a percentage:

Table 2. Descriptions and Applications of QA Samples

Sample Type	Type of Analysis[a]	Description	Application
Performance evaluation (audit) samples	I, O, R	Homogeneous, stable, and certified synthetic audit sample	Estimate intra- and inter-laboratory bias and estimate system precision
Field blank	I, O, R	ASTM Type II water that is carried through the same system as the field samples	Identify contamination resulting from sampling operations and estimate system detection limit
Field duplicate	I, O, R	Second sample collected at the sampling site	Estimate system precision

[a] = I - Inorganic analysis, O - Organic analysis, R - Radionuclide analysis.

$$\%RSD = \frac{s}{\bar{X}} \times 100$$

where s is the standard deviation of the series of individual measurements and $\bar{X}$ is the mean of the series of individual measurements.

The mean of the set of individual measurements is determined by summing the values for the individual measurements ($X_1 \ldots X_n$), then dividing by the number of measurements (n):

$$\bar{X} = \frac{X_1 + X_2 + X_3 + \ldots + X_n}{n}$$

The standard deviation for this set of measurements then is determined as follows:

$$s = \sqrt{\frac{\sum (X_i - \bar{X})^2}{n - 1}}$$

where X_i represents each of the individual measurements.

The RPD is calculated in percent for duplicate measurements as follows:

$$\%RPD = \frac{X_1 - X_2}{(X_1 + X_2) / 2} \times 100$$

where X_1 is the first sample result and X_2 is the duplicate sample result.

For radionuclide determinations, replicate measurements must agree within the 95 percent confidence level based on the summed error of the analysis, or within 3σ of the weighted average, as required by the client-specific protocol and is measured by the replicate error ratio (RER).

The RER or duplicate error ration (DER) for radionuclide determination is calculated as follows:

$$RER = DER = \frac{R_1 - R_2}{(\sigma_1 + \sigma_2)}$$

Where:

R = Result
σ = 95% confidence level

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value

Table 3. Descriptions and Applications of QC Samples

Sample Type	Type of Analysis[a]	Description	Application
QC Check Standard (ICV, CCV, QCCS)	I,O	Independent standard; prepared from source other than calibration standard	Indicates accuracy and consistency of calibration
Laboratory control sample	I,O,R	Independent standard; processed through the entire analytical procedure	Indicates accuracy of the analytical procedure
Detection limit QC check sample	I,O	Standard at a constituent concentration 2 to 3 times the detection limit	Indicates accuracy at lower end of calibration range
Check Source	R	Pure standard containing radionuclides at specified levels	Provides a check on counting instruments
Calibration blank	I,O	ASTM Type II water or better (zero constituent concentration)	Indicates instrument signal drift and sample contamination
Method blank	I,O,R	All reagents used during sample preparation steps (i.e., digestion, extraction, distillation)	Indicates sample contamination introduced during sample preparation steps
Background	R	Instrument background (e.g., deep well water for tritium)	Indicates instrument background level
Internal standard	O	Constituents added to every sample at a known concentration; not expected to be detected in environmental media	Measures the relative responses of other method constituents and surrogates
Surrogate spike	O	Constituents not expected to be detected in environmental media; added to every sample at a known concentration	Evaluates sample preparation and analytical efficiency
Quench source	R	Standard that contains quenching materials for liquid scintillation counting	Indicates liquid scintillation counting efficiency
Analytical laboratory duplicate/replicate	I,R	Sample aliquot; split at the analytical laboratory	Indicates analytical precision
Matrix spike	I,O,R	Sample plus known quantity of constituent	Indicates sample matrix effect on analysis and on accuracy of measurement system
Matrix spike duplicate	O,R	A second matrix spike sample	Indicates analytical precision influenced by sample matrix
Yield tracer	R	A radioactive or nonradioactive isotope at a known, specified quantity	Traces yields during sample preparation

[a] = I - Inorganic analysis, O - Organic analysis, R - Radionuclide analysis

(EPA, 1980). Systematic errors affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100):

$$\%R = \frac{X}{T} \times 100$$

where X is the measured standard value and T is the true or expected (reference) value.

The precision and accuracy DQOs that are to be used in evaluating inorganic, organic, and radionuclide constituents at LAS are provided in method-specific SOPs and in the documentation for the analytical method of interest.

Precision and accuracy are determined, in part, by analyzing data from matrix spike and matrix spike duplicates, unspiked duplicates, LCSs, and single blind audit samples (see tables 2 and 3). For radiochemical determinations, counting statistics can also provide an estimate of uncertainty.

3.2 COMPLETENESS

Completeness is a measure of the percentage of measurements that are judged to be valid measurements (EPA, 1980). At a minimum, the objective for completeness of data is 90% for each constituent analyzed.

3.3 REPRESENTATIVENESS

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition (EPA, 1980). Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result (concentration) is representative of the constituent concentration in the sample matrix. The LAS analysts will make every effort to analyze an aliquot that is representative of the original sample, and the homogeneity of the sample will be ensured before subsampling.

3.4 COMPARABILITY

Comparability is a measure of the confidence with which one data set can be compared to another (EPA, 1980). The comparability standards for LAS require that all laboratory analysts use uniform procedures and a uniform set of units and calculations for analyzing and reporting environmental data. To ensure that employees clearly understand the requirements and standard protocols, all personnel will participate in the uniform, ongoing LAS training program.

3.5 DETECTABILITY

Detectability refers to the minimum concentration of a constituent that can be measured by a measurement system with a stated level of confidence (Taylor, 1987). It is determined by assessing the variability of replicate measurements at zero or near zero constituent concentration, and it is reported in concentration units.

It is LAS policy to determine, for each inorganic and organic constituent, the instrument detection limit (IDL) or method detection limit (MDL) and the reporting detection limit (RDL) before any samples are analyzed. For radionuclide constituents, minimum detectable activity (MDA) is determined.

3.5.1 Instrument and Method Detection Limits

In general, a detection limit is the point or concentration at which a measured value becomes believable. In other words, it is the point at which the value is larger than the uncertainty (e.g., noise level) associated with it. The detection limit is defined as the smallest observed signal with the reliability of 1 minus α (where α is the probability of Type I error) that can be considered a signal caused by the constituent of interest.

For inorganic and organic constituents, detection limits are estimated by determining the standard deviation (s) from the results of the 7 to 10 measurements of the low-level standards or a blank analyzed on the same day. The IDL and MDL differ, not in how they are calculated, but in the way in which the low-concentration standards are handled before analysis. Because MDLs are sensitive to instrument and matrix effects, the MDL is determined by allowing the standard to undergo the appropriate sample preparation technique (i.e., extraction, digestion, distillation), as required by the analytical method before analysis. On the other hand, the IDL is calculated from standards that are not subjected to these handling steps. Thus, aside from sample preparation, all procedures for determining the IDL and MDL are the same. The distinction is that the IDL estimates the detection limit of the instrument under ideal conditions, whereas the MDL estimates the detection limit in more practical terms.

For each method and each analyte required by the method, the instrument detection limits (IDLs) are determined on a quarterly basis for metals constituents and on an annual basis for other inorganic constituents and method detection limits (MDLs) are determined annually for organic analyses.

The IDL or MDL is computed as three times the standard deviation (i.e., $3s$) of replicate (7 to 10) runs of a standard in which the concentration of the analyte of interest is at or near the detection limit specified for that technique. The replicate measurements of the low-level standards are performed on three nonconsecutive days for inorganic analyses and on the same day for organic analyses. The concentration of the standards needs to be at approximately three to five times the expected or method-specified detection limit.

The MDL studies are intended to estimate the lowest concentration of an analyte that can be routinely determined by a method with less than a 1% probability of getting a false positive measurement. The procedure involves performing multiple measurements of the analytes at a concentration that is near the expected MDL. A Student's t test is performed on the multiple measurements, and the MDL is set at the value where the measurements are distinguishable from zero at the 99% confidence level.

The variability in an analytical measurement is assumed to consist of two distinct portions. One portion is proportional to the concentration of the analyte in the sample. Small errors in volumetric measurements and transfers will create errors that are proportional to the initial analyte concentration. Since the MDL studies are run at a very low concentration, this component of the error is assumed to be low. The other portion of the variability is due to the measurement noise and chemical interferences. The objective of the MDL study is to estimate this second type of error while minimizing the first type.

3.5.2 Reporting Detection Limits

The RDL is the lowest level at which measurements become quantitatively meaningful

The RDL is defined as approximately 3 times the MDL. Because sample-handling activities, in addition to sample analysis, have an influence on detecting the presence or absence of a constituent, obtaining extremely low IDLs in the laboratory is meaningless in relation to the environmental sample result. Therefore, for reporting data, the LAS has adopted the approach of determining RDLs, which include the variability that may result from sample preparation, sample handling, and analysis techniques. For each constituent and each instrument used to quantitate that constituent, the experimentally determined IDLs and MDLs must be less than the RDLs shown in Appendix A.

The RDL is established to account for a number of factors. First, greater relative error is expected for analyte concentration at or near the IDL or MDL which decreases the level of confidence in the accuracy of the analyte quantification. Second, there is some day-to-day variability in both the IDL or MDL which means that on a given day it may not be possible to detect an analyte at the statistically determined MDL. Third, environmental samples are rarely as clean or as analytically straightforward as the matrices used to determine detection limits. Consequently, the RDL is set above the MDL and IDL (1) to reduce relative error, (2) to account for expected variability, (3) to ensure that analytes can be reported at the RDL with reasonable level confidence, and (4) to reduce the chance of reporting false negatives in environmental samples.

Two final factors in establishing RDL values include (1) the ease of data interpretation for our clients and (2) standardization of data reporting for the laboratory. If the RDL values were to change each time a new IDL or MDL study was performed, this would cause confusion to our clients and would be difficult to maintain consistency in reporting for the laboratory staff, which may result in reporting of erroneous data. Therefore, the RDL is established at a level high enough so that changes to these values are infrequent.

This is the set of statistical assumptions behind the detection limit studies. The assumptions, however, are not always correct. The results from the detection limit studies should not be taken at face value; the results should be scrutinized closely and adjusted based on the experience of the scientists who are involved in performing the study. For example, some MDL studies may show very little variability for constituents that have historically been difficult to detect. In these cases, the MDL is probably unrealistically low, and it should be adjusted upward based on the experience of the analyst and the QA specialist. In other cases, variability in the sample preparation or other factors may result in obtaining a high MDL, while observation of the raw instrument output indicates that there is adequate instrumental sensitivity to detect the analyte at concentrations far below the measured MDL. In these cases, the MDL should be lowered to reflect the true state of the instrument performance.

3.5.3 Minimum Detectable Activity

The minimum detectable activity (MDA) is a measure of the quantity of radioactive materials that could be present and detected by the analysis. The MDA is reported in activity units per unit volume or weight, such as pCi per liter or pCi per gram. Many factors affect the MDA, including calibration geometry, backgrounds (system and source-induced), detector resolution, counting systems, sample sizes, and the particular isotope measured. With the exception of the MDA formula chosen, the MDA is not affected by the analysis software. It is the LAS policy to calculate MDAs as described in ANSI 13.30 and NRC

Reg guide 86-16 also known as the "Currie Method" unless otherwise specified by the client.

By using state-of-the-art instrumentation, (e.g., a high-resolution gamma spectrometer with a 96% efficiency detector, an alpha/beta counter with less than 0.1 counts per minute [cpm] background alpha and less than 1 cpm beta, or a liquid scintillation counter with tritium channel having less than 0.8 cpm background) lower MDAs can be achieved with less counting time than required by using conventional instrumentation.

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CHAPTER 4
SAMPLE MANAGEMENT

4.1 INTRODUCTION

Sample management is the overall process by which samples are controlled, transferred, handled, and stored from the time of collection through analysis and final disposition. Sample management and chain of custody are closely related. Sample management refers to those activities aimed at ensuring sample integrity. Chain of custody involves establishing accountability for the sample; documenting how the sample is received, tracked, and stored; and defining who has access to and handles the sample. Internal chain-of-custody procedure is addressed in Chapter 13 of this plan and in LAL-90-SOP-0009, and sample receiving and log in are described in LAL-90-SOP-0002.

Sample management begins in the field when the sample is collected. The management of the collection process, with the exception of providing sample containers, is not within the jurisdiction of the LAS and is not discussed in this plan. All other sample management activities, specifically those related to sample shipment, sample containers, sample preservation, sample holding times, and sample preparation and analysis, are discussed here.

4.2 SAMPLE AND LABORATORY CONTAINERS

Container specifications depend on the client requirements, in addition to analytical method, constituent, and sample matrix of interest. LAS purchases commercially precleaned bottles for which the supplier has certified in writing that the cleaning procedure used meets or exceeds all CLP container guidelines (Chapter 16). This specification applies to all shipping and sample containers and all reagents, whether shipped off site or used only at LAS.

In general, use the following containers:

- 40-mL Teflon®-septa glass vials (clear or amber) for aqueous samples to be analyzed for volatile organic constituents (designated as "Level I").
- 1,000-mL amber glass bottles with Teflon®-lined caps for aqueous samples to be analyzed for semivolatiles, pesticides, and PCBs (designated as "Level I").
- Polyethylene bottles for aqueous samples to be analyzed for inorganic constituents (designated as "Level I").
- Wide-mouth, amber glass bottles for all soil and sediment samples.

All containers must be kept in a contaminant-free, secure area. Detailed descriptions of container specifications are given in Appendix B. Sample containers shall not be cleaned and reused for any other analyses. LAL-90-SOP-0045 provides guidelines for sample container types and uses.

In addition, use the following guidelines when selecting the material composition of sample containers and laboratory vessels:

- Use borosilicate or polyethylene bottles for storage of reagents and standard solutions, unless otherwise specified; however, do not use plastic containers for reagents and standard solutions used in organic analyses.

- Borosilicate glassware is not completely inert, especially to alkali materials. Store standard solutions of silica, boron, and the alkali metals in polyethylene bottles.

- Dilute metal standard solutions have a tendency to "plate out" on container walls over time; therefore, prepare these solutions immediately before analysis.

- Disposable glassware is acceptable for some analyses. For example, disposable vials and cuvettes are appropriate for use in automatic samplers. However, every effort must be made to ensure that these containers are free of contamination (i.e., target constituents or interferants) that can affect the analytical results.

4.3 SAMPLE PRESERVATION

Sample preservation prevents or retards the degradation and/or reaction of chemicals or biological activity in samples during transit and storage. Efforts to preserve the integrity of the samples are generally initiated at the time of sampling and should continue until analyses are performed. Preservatives are typically added to the sample container at the time of sample collection. (**Note:** Preservatives are added to aqueous samples only.) However, if requested by the client, LAS will send to the field pre-measured volumes of the preservatives in sealed ampules or in sample containers. Since pre-measured volumes of preservatives added at LAS may not always be sufficient to preserve samples, samplers are ultimately responsible for ensuring adequate preservation. Preservation and storage guidelines and requirements, by constituent, are provided in Appendix B.

4.4 SAMPLE HOLDING TIMES

The maximum time that a preserved sample may be held between sample collection and analysis depends on the stability of the constituents of interest. Holding-time limitations are intended to minimize chemical changes in a sample before it is analyzed. Maximum allowable holding times provided in Appendix B apply to aqueous, soil, and sediment samples when proper preservation procedure is followed. Holding times are calculated from sample collection to analysis, unless otherwise specified by the method.

It is LAS policy to adhere to the method or client-specified maximum holding time requirements. To expedite analysis and to minimize the possibility of exceeding holding times, it is imperative that samples be sent to the analytical laboratory by a fast, reliable method as soon as they are collected. Following sample receipt and log in, the appropriate section supervisor schedules the analysis of samples to ensure that all samples are analyzed within holding times. Holding times for sample preparation (i.e., extraction, digestion, or filtration) and analysis are entered into the sample receipt data base to ensure that samples are prepared and analyzed within the prescribed holding-time for each constituent of interest.

For some constituents, it may be necessary to hold samples in cold storage even if holding times have been exceeded. The extended storage is necessary to ensure that the samples are suitable for possible reanalysis. If the original analysis of the sample or any subsequent reanalyses exceed the holding times

for the particular constituents, a statement must be included in the case narrative of the analytical data report, and affected sample results must be qualified appropriately to indicate that the holding time requirement has been exceeded.

4.5 SAMPLE PREPARATION

The matrix of the sample and the constituents to be analyzed affect the selection of sample preparation steps. Sample preparation procedures for organic, inorganic, and radionuclide analyses are provided in various method-specific SOPs (see Appendix C).

Water used in the course of inorganic, organic, and radionuclide analyses (dilutions, preparations of standard and blank samples, etc.) must meet or exceed the standards for purity of ASTM Type II grade reagent water. All extracts, digestates, and filtered samples are collected in specified sample containers and are labeled and tracked on chain-of-custody forms and in the respective laboratory logbooks. Samples and final sample preparations (e.g., organic extracts) are stored at 4°C (± 2°C), except for metals digestates, which are stored at room temperature in acidic solutions resulting from the digestion process. Samples and standards shall be stored separately. Samples designated for VOC analyses are maintained in designated refrigerators separate from samples for other organic analyses to prevent cross contamination. The Quality Assurance staff monitors refrigerators designated for VOAs for potential cross contamination by using holding (refrigerator) blanks.

For most inorganic analyses, chemical reagents, solvents, and gases of the analytical reagent grade are used. For other analyses, such as trace organic and radiochemical, special ultrapure reagents, pesticide-quality and analytical-grade solvents, and gases are used as required for the method of interest. For methods in which the purity of reagents is not specified, LAS uses analytical grade reagents, solvents, and gases. A detailed discussion on the reagent requirements is also provided in Chapter 16.

4.5.1 Subsampling for Sample Preparation

Many environmental samples are heterogeneous upon receipt and may require some physical manipulation in order to yield representative (i.e., homogenous) sub-samples. LAS-91-SOP-0105 provides procedures for subsampling liquid, semisolid, and solid samples, and samples containing inorganic and organic constituents.

4.5.2 Filtration

The need to filter aqueous samples depends on whether total or dissolved constituents are of interest. Samples to be analyzed for dissolved inorganic constituents must be filtered in the field if preservation is needed. Samples for dissolved metals analyses must be filtered in the field before chemical preservatives are added to preclude the release of contaminants from the particulate matter. However, if the client requires that the samples be filtered in the laboratory under a controlled environment, the filter material used must be compatible with the constituents of interest.

4.5.3 Extraction and Digestion

Many organic (e.g., semivolatile, pesticides) analyses of environmental multimedia samples require the extraction of sample constituents of interest into organic solvents before analysis. Many metals analyses of aqueous samples require digestion of the sample. SOPs delineating these preparation steps are listed in Appendix C.

Soils and sediments are complex mixtures of widely varying compositions, even within a single site. Recovery of constituents depends on many factors, including organic content, mineral content, particulate size, and moisture content of the soil. It is LAS policy to report soil and sediment sample analyses for organic and inorganic methods in the as-received condition (i.e., wet weight) unless the client specifies that dry weight is to be reported. For radiochemical determinations, data are routinely reported based on dry weight of the sample.

For radionuclide determinations, grinding of samples may be necessary to ensure the collection of homogeneous representative sub-samples.

4.6 SAMPLE ANALYSIS

All samples shall be processed through the entire analytical method, as specified in Chapter 5. All analyses shall be performed within the appropriate calibration range (Chapters 6, 7, and 8) of the instrument. Each sample for which the constituent concentration exceeds the calibration range shall be diluted and analyzed within the appropriate analytical range. Records of all dilutions shall be maintained in analysis or injection logbooks, and dilution factors shall be reported on the appropriate data reporting form. The method of constituent identification and quantitation is specified in the analytical methods.

4.7 SAMPLE TRANSFER BETWEEN PREPARATION AND ANALYSIS PERSONNEL

LAL-90-SOP-0002 and LAL-90-SOP-0009 describe the procedures for sample handling and internal tracking pertaining to the original sample and the sub-samples. All analysts are trained in the proper tracking and storage of samples.

In summary, the sample is initially transferred from the sample custodian to the sample (organic, inorganic, or radiochemistry) preparation personnel who take custody of samples. For cases in which there is no sample preparation (e.g., volatiles, anions), custody is transferred directly to the analyst. The extracted, digested, or filtered portion of the sample used for analysis remains in the preparation laboratory under specified storage conditions until analysis is scheduled and the unused, raw portion of the sample is returned to the sample custodian for proper storage in the prescribed area. If reanalysis is required on the raw sample, the sample is obtained through the sample custodian, and standard chain-of-custody and sample-tracking procedures are followed.

Within the inorganic suite, there are two discernable areas: one that is refrigerated and one that is maintained at ambient temperature. Samples that are not returned to the sample log-in area after use are stored in these areas to further ensure the integrity of the environmental samples. The tracking sheets used in the laboratory and the LDMS indicate where samples are stored while they are being processed in the inorganic suite.

4.8 SAMPLE DISPOSAL

There are several possible ways to dispose of the sample after use:

- The sample will be disposed of as hazardous waste; or
- The sample may be returned to the client, if requested.

It is LAS policy to dispose of samples 60 days after the submittal of the report to the client unless otherwise specified in writing by the client or as required by regulations and licenses governing laboratory operations. However,

samples will not be disposed of until contract-required terms are expired or until the client is notified in writing of the intent to dispose. Complete records of the disposal date and method will be maintained along with all the other sample-tracking information maintained in the Document Control files. Waste disposal follows RCRA and other applicable protocols. LAL-90-SOP-0003 provides a detailed discussion of general LAS waste-handling considerations. LAL-91-SOP-0083 addresses disposal of radioactive materials.

4.9 PREVENTION OF SAMPLE CONTAMINATION IN THE LABORATORY

To ensure that sample integrity is maintained throughout the laboratory, LAS staff must follow GLPs and GMPs in the handling, preparation, and analysis of environmental samples. Chemical, physical, and radionuclide determinations must be performed in a work environment free of sample contaminants and free of constituent and measurement interferences. LAL-90-SOP-0004 addresses the specific methods to prevent potential sample contamination.

Special precautions must be taken during sample handling and analysis to minimize or eliminate cross contamination because environmental samples sent to LAS for analysis often contain trace concentrations of constituents. In general, all work areas must be kept clean and free of dust and dirt accumulation. All counter tops and chemical fume hoods where sample preparations and wet chemical analyses are performed must be cleaned regularly.

4.9.1 LAS Facility Features

Special structural design features at LAS minimize or prevent sample contamination that may occur during sample handling. The significant design features include:

- Segregated volatile and semivolatile organic analysis laboratories to minimize potential sample contamination associated with the use of common organic solvents such as methylene chloride, hexane, and acetone.

- Separate glassware washing facility supplied with high purity reagent water.

- Air balancing systems that (1) change the air 10 times per hour to help reduce the presence of airborne contamination primarily for commonly used organic solvents, (2) maintain positive pressure in the volatile organic analysis laboratory relative to the hallway to ensure that airborne contaminants do not enter the laboratory, and (3) maintain negative pressure in the other laboratories relative to the hallways to prevent airborne contaminants from escaping into the rest of the building.

- 36 chemical fume hoods, 18 snorkels, and 2 perchloric acid hoods to carry off fumes and to reduce the risk of aerosol and airborne contaminants and of personal safety hazards in the laboratory.

- Exhaust stacks from the hoods in the radioactive section that pass the exhaust from the hoods through scrubbers or HEPA filters to prevent the release of radioactive contaminants into the environment.

4.9.2 Reagents and Reagent Water

The quality of the chemical reagents, solvents, and gases used for organic and inorganic analyses must meet minimum requirements for analytical reagent grade or requirements

specified by the particular method (see Chapter 16 for details). Standards, reagents, and solvents must be stored according to manufacturer's guidelines. Photosensitive reagents and standards are stored in appropriate dark storage areas.

Laboratory reagent water used for dilutions, preparation of standards and blanks, and glassware cleaning is generated using the Barnstead/Thermolyne NANOpure Ultrapure Water System (Model D4741) in combination with dechlorination R.O. and mixed-bed ion exchange. This system is designed to supply water that meets requirements for ASTM Type II reagent water or better.

4.9.3 Sample Storage

When required, samples are stored at 4 $\pm$ 2°C to preserve their integrity, as required by the analytical method of interest. Storage includes procedures that maintain the constituent levels (through the use of chemical preservatives) and physical maintenance of the sample (through temperature or light control). When storing samples in refrigerators or light-protected enclosures, there is a risk of cross contamination. Measures are employed to prevent cross contamination, such as segregating samples to be analyzed for VOCs from those to be analyzed for semivolatile compounds, segregating environmental samples from standards (particularly for VOCs), and preparing and analyzing holding blanks to monitor cross contamination.

4.9.4 Glassware Cleaning

All glassware used in sample preparation must be cleaned according to the procedures specified for the particular analysis method. LAL-90-SOP-0017 provides protocols for glassware cleaning for organic constituents, and LAL-90-SOP-0018 provides protocols for glassware cleaning for inorganic constituents. LAL-91-SOP-0172 provides protocols for glassware cleaning for radionuclides. In general, water-soluble substances can be washed out with hot or cold water and the vessel can be rinsed with deionized water. Other substances more difficult to remove may require the use of a detergent, organic solvent, persulfate cleaning solution, nitric acid, or aqua regia. Regardless of the method of cleaning, it is good practice to rinse "dirty" glassware with the last solvent used as soon as possible after use because material allowed to dry onto glassware is much more difficult to remove.

4.9.5 Assessment of Sample Contamination Levels

Potential for background contamination that may result from the sample containers, reagent water, reagents, and solvents used in extractions and digestions; cross contamination during sample storage; or chemical and physical interference or constituent carryover during analytical operations are evaluated on the basis of QA and QC data derived from instrument, method, and holding blank samples. If blank sample data exceed method-specific or LAS-established acceptance limits, the problem is investigated and resolved. As necessary, samples associated with out-of-control situations are reprepared and/or reanalyzed.

CHAPTER 5
ANALYTICAL METHODS

5.1 ANALYTICAL METHODS SELECTION

Methods used in sample preparation or analysis are selected to meet the specific needs and requirements of the client. LAS employs standard, officially approved (e.g., EPA, ASTM, APHA) analytical methods to quantify inorganic, organic, and radionuclide constituents in environmental media (e.g., water, soil, sediment, sludge). The examples of approved methods are given in Appendix A for the inorganic, organic, and radionuclide constituents. A detailed description of these procedures is provided in EPA SW-846 (September 1986 and revisions), EPA/600/4-79/020 (EPA, 1983), and ASTM Standard Methods (17th and 18th Editions). EPA CLP methods are provided in the most recent Statements of Work or earlier versions based on clients' requirements.

5.2 ANALYTICAL QUALITY CONTROL

Rigorous QA and QC procedures are incorporated into sample preparation and analysis activities. Internal QC checks on the analytical procedures are discussed in detail for inorganic constituents in Chapter 6, organic constituents in Chapter 7, and radionuclides in Chapter 8.

In general, before an analytical run, the analyst checks schedules and records for maintenance and calibration to ensure that all necessary tasks are current. An initial or continuing calibration check verification is then completed, and the difference between analyzed values and the known standards is calculated. If any calibration check sample exceeds the control limit of the method (see method-specific SOPs as listed in Appendix C), adjustments are made and the instrument is recalibrated, as appropriate. During the course of the analytical run, the analyst incorporates all applicable QC samples in accordance with method-specific SOPs. Following each QC sample analysis, the analyst performs the necessary calculations either manually or by using appropriate software. If any QC sample falls outside method-specific control limits, the problem is investigated and resolved, and corrective action is performed in accordance with the method, the SOP, or the project-specific QA Plan. All information related to the analytical run is documented in the injection and analysis logbooks. All calculations related to QC sample analysis and the types and frequency of QA and QC samples (e.g., audits, blanks, spikes) are described in detail in the method-specific SOPs for inorganic, organic, and radioanalytical analyses.

5.3 LAS STANDARD OPERATING PROCEDURES

To ensure and document that each operational system and analytical procedure is performed in a uniform, standard way, LAS has documented a series of SOPs. A complete list of LAS SOPs, many of which are referenced in this plan, is provided in Appendix C. In general, analytical SOPs follow EPA or other approved methods. All personnel must fully understand the specific SOPs pertaining to their duties. It is the section supervisor's responsibility to ensure that each employee has read and understood all appropriate SOPs related to task responsibilities.

Each SOP is approved and signed by the section supervisor/designee, Quality Assurance staff member, Health and Safety Officer, and the LAS Director. At times, changes in SOPs are required as a result of new instrumentation, methods, client-tailored needs, or improved procedures. Any change in an SOP requires the approval of the section supervisor and the Quality Assurance Manager or designee which is documented using the SOP Change Form. On an annual basis, as required by the formal review process, SOPs are reviewed to determine if SOP reflects current operations and are approved by the individuals mentioned above.

Work instructions also provide a mechanism to document simple instructions to the laboratory staff responsible for processing and analyzing samples and in generating reports. These instructions are equivalent to internal memorandums that are also used to disseminate project-specific information. They serve as quick references to items such as specific spiking constituents, spiking levels, calculations, sample preparation techniques, project-specific information, etc. Work instructions are approved by the section supervisor, Quality Assurance Manager, project manager, or by an appropriate designee.

5.4 CONTROL CHARTS

An essential element of the QA process is the ability to detect changes in analytical performance quickly. The control chart is an effective tool for this assessment because it records in real time the accuracy (bias) and precision of the appropriate parts of the measurement process. In other words, the control chart demonstrates statistical control.

At LAS, Shewhart charts using a single measurement (X chart) of the laboratory control samples (LCSs) are generated using EXCEL to monitor analytical data quality in terms of accuracy and precision. The Shewhart chart has a center line that is defined by the process or calculated by using the data to prepare the chart. The interval between the center line and the control limit is based on the assumption that data are normally distributed on the sample size of the subgroup and on the estimated standard deviation. Using these charts, the percent recovery of the control analyte in the LCS is plotted against the control limits sequentially as a function of time. Two types of limits are established to judge whether a data set indicates lack of control: the upper and lower warning limits (95% or 2-sigma) and the upper and lower control limits (99% or 3-sigma). Initially, control limits are developed by determining the experimentally estimated standard deviation of approximately 20 independent measurements. Until the experimentally determined control limits are established, the EPA method-specified criteria (if available) are used to assess data quality. The experimentally estimated control limits must be less than or equal to the EPA-specified limits if EPA limits are available. The warning and control limits shall be updated every 20 data points or when a significant change in the measurement system is required to evaluate whether the initial limits are realistic.

The Operations Department staff members prepare and monitor control charts. The QA staff provides oversight in the development of control charts to ensure that they conform to the requirements specified in this policy.

5.4.1 Criteria for Out-of-Control Conditions

The causes for a shift or trend in control charts could result from (1) incorrect preparation of a standard or a reagent, (2) sample contamination, (3) improper storage or preservation, (4) incorrect instrument calibration, (5) poor analytical technique, and (6) deviation from the

analytical method. Out-of-control situations may result when one of the following conditions occur:

- A single point outside the control limits.
- A series of seven successive points on the same side of the central line.
- A series of five successive points trending in the same direction
- Any three consecutive points outside of the warning limits.
- A cyclical pattern of control values.

These conditions may indicate that the measurement system is out of statistical control. When this situation occurs, the data will be evaluated thoroughly to identify the most appropriate corrective action to be implemented. The problem and its solution may be documented through an Nonconformance & Corrective Action Record (NCAR) (see Chapter 15) as appropriate. Specifically, in these instances, the data in question must be further examined to identify and correct the root cause of the problem. Exceeding warning limits will only require a close observation of the measurement system. In reviewing control charts, any significant changes in key analysts, instrumentation, or processes must be kept in mind to explain potential out-of-control situations.

5.4.2 Control Analytes

An LCS is prepared by spiking matrix spiking constituents in reagent water matrix (Type II) to represent aqueous samples and in standard solid media (e.g., sodium sulfate for organic analyses) to represent solid matrix samples. For inorganic analyses, a certified PE reference material purchased from an external vendor is used for analysis of solid matrix samples. Results are compared against certified true values (central line) and control limits. An LCS is analyzed for each batch of 20 samples of the similar matrix.

The following constituents will be used to monitor analytical performance via LCS control charts for the methods specified below:

Volatile and Semivolatile Organic Analyses

Three surrogates for volatile organics and six surrogates for semivolatile organics.

Pesticides/PCBs

Two surrogates for pesticides and one Aroclor.

Metals by ICP-AES

At least three control analytes.

Metals by GFAAS

Each analyte (As, Se, Tl, Pb by GFAAS; Hg by CVAAS).

Wet Chemical Methods

Cyanide, Chloride, Nitrate, Sulfate.

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CHAPTER 6
ANALYTICAL INTERNAL QC FOR ANALYSIS OF INORGANIC CONSTITUENTS

6.1 INTRODUCTION

Internal QC is integral to ensuring that analytical results are reliable and that data integrity is maintained throughout the measurement system. Specific guidelines for instrumental calibration are given in the instrument manufacturers' instructions. Guidelines related to sample handling, sample preservation, and sample holding times for inorganic constituents are addressed in SW-846 (EPA, 1986b), in EPA 600/4-79-020 (EPA, 1983), and in Appendix B. This section summarizes the QC activities related to inorganic constituent analyses. These requirements may be superseded by a project-specific QA Plan or a method-specific SOP.

At LAS, it is prohibited to alter data solely to meet method- or contract-specified QC requirements.

6.2 HOLDING TIME COMPLIANCE

The method- or client-specified holding time requirements must be met for the sample results to be considered valid. If holding time requirements are exceeded for sample analyses, the client must be informed of the out-of-control situation, sample data must be qualified appropriately, and an explanation of the nonconformity must be provided in the case narrative of the analytical data report.

6.3 DAILY INTERNAL QC FOR EACH ANALYTICAL METHOD

The following internal QC procedures are required for atomic absorption spectroscopy (AAS), inductively coupled plasma-atomic emission spectroscopy (ICP-AES), ion chromatography (IC), and other methods applied to the analysis of inorganic constituents. These requirements may be superseded by a project QA plan or a specific SOP.

6.3.1 Initial Calibration and Instrument Tuning

Method calibrations and/or instrument calibrations must be independently verified daily. Typically, the inorganic methods require the instrument to be calibrated using at least one blank and at least three calibration standards following method SOPs and manufacturer's recommendations for specific procedures. At a minimum, the coefficient of determination (r^2) must be at 0.990, or the correlation coefficient (r) for any given calibration curve must be at 0.995. If the r or r^2 is outside the acceptance criteria, the instrument must be recalibrated. For ICP-AES systems, the instrument must be calibrated according to instrument manufacturer's recommended procedures. Similarly, each ICP system must be calibrated using two standards, one of which must be a blank. All standards used for instrument calibration must be prepared from sources that are traceable to EPA or to the National Institute for Standards and Technology (NIST).

An initial calibration is performed as required for each analytical method (e.g., AAS, ICP-AES). The concentrations of the calibration standards must bracket the expected sample concentrations. The calibration standards must be prepared by using the same type of acid or combination of acids that will be present in the

samples after preparation. All calibration standards for each analytical procedure must be prepared in compliance with the method-specific SOPs. If, during the analysis, the concentration of a sample is above the calibration range or LDR, the sample is to be diluted and reanalyzed. In this case, the diluent must have a matrix similar to the sample matrix with respect to all preservatives (acid type and concentration) used. Samples are first analyzed at the lower concentration range. If a sample concentration exceeds the upper end of the calibration range or LDR, the sample is reanalyzed to fall within the concentration range. Results are reported based on the diluted sample analyses, and the RDLs are adjusted accordingly to reflect the dilutions performed. **Note:** For mercury analysis, the calibration standards must be carried through the acid digestion process.

For inductively coupled plasma/mass spectroscopy (ICP/MS) analysis, each day, the instrument must be tuned by verifying that the system meets the mass calibration and resolution check requirements in the mass regions of interest. If the mass calibration exceeds a difference of more than 0.1 amu from the actual value, then the mass calibration must be adjusted to the correct values. The resolution must be less than 1.0 amu full width at 10 percent peak height. The tuning solution, analyzed at the beginning and end of each analytical run, must have a percent RSD of less than 10 percent. These are required criteria which must be met prior to any sample being analyzed.

6.3.2 Initial Calibration Verification

Immediately after each measurement system (e.g., ICP-AES, AAS, IC) has been calibrated and standardized, the accuracy of the calibration standards and the initial calibration must be verified and documented for each constituent by the analysis of LAS-prepared or certified independent standard solution(s). An independent standard is defined as a standard composed of the same constituents as, but from a different source than, those used in the standards for the initial calibration. If measurements exceed the control limits given in Table 4, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

For ICP-AES, the initial calibration verification (ICV) standard(s) must be run at each wavelength used for analysis. For cyanide, the ICV standard, along with the other samples, must be distilled before analysis. For mercury, the ICV standards must be carried through the acid digestion process.

Furthermore, for ICP-AES analysis using methods 6010 or 200.7, the highest calibration standard must be run as a sample before the analysis to verify the standard concentration. The measured concentration should be within $\pm 5\%$ of the true concentration.

6.3.3 Continuing Calibration Verification

To ensure calibration accuracy during each analytical run, a standard in the mid-range of the calibration curve is analyzed as verification of continued calibration. For most inorganic methods, the continuing calibration verification (CCV) standard must be analyzed at the beginning of the run, at a frequency of 10%, and after the analysis of the last sample. For ICP-AES, the standard must be analyzed for every wavelength used to analyze each constituent. The constituent concentrations in the CCV standard must be a solution at or near the mid-range concentration of the calibration curve and an LAS-prepared standard solution that is independent of the ICV standards.

Table 4. Acceptance Limits for Initial and Continuing Calibration Verification Analyses of Inorganic Constituents

		Percent of True Value	
Analytical Technique	**Constituent**	**Low Limit**	**High Limit**
ICP-AES	6010 and CLP Metals	90	110
	200.7 Metals	95	105
ICP/MS	6020 and 200.8 Metals	90	110
AAS	Metals	90	110
IC	Anions	90 (ICV)	110 (ICV)
		85 (CCV)	115 (CCV)
Cold Vapor AAS	Mercury	80	120
Other	Cyanide	85	115
	Ammonia-Nitrogen	90	110
	Dissolved Silica	90	110
	Fluoride	90	110
	Alkalinity	90	110
	Chromium (hexavalent)	90	110
	pH	-0.1	+0.1

Note: The same continuing calibration standard must be used throughout the analytical runs for each group of samples analyzed.

If the deviation of the CCV exceeds the control limits specified in Table 4, the problem must be identified and corrected, the instrument recalibrated, and the preceding 10 analytical samples since the last acceptable calibration verification reanalyzed for the constituents affected.

6.3.4 Laboratory Control Sample

To further ensure that the sample preparation and measurement processes are functioning within control, an independent liquid LCS is prepared or solid LCS purchased from an approved external source. The LCS contains the constituents of interest and is carried through the sample preparation procedure (e.g., digestion, distillation), then analyzed by using the required method. An LCS must be analyzed for each batch of 20 samples of the same matrix. The measured concentrations must fall within 20% of the true concentration or the acceptance limits specified by the vendor. If the percent recovery is outside of the acceptance criteria, all affected samples and the LCS must be redigested and reanalyzed. For solid LCS, data are compared against the advisory control windows specified by the vendor for each analyte. Data obtained for liquid and solid LCSs are further monitored using control

charts. Section 5.4 provides a detailed description of control charts.

6.3.5 Detection Limit QC Standards for AAS and ICP-AES

For ICP-AES, to verify linearity near the IDL, a detection limit QC solution prepared at approximately 2 x RDL is analyzed at the beginning and end of each sample analysis run, or twice per 8-hour work shift, whichever is more frequent. The standard is analyzed for every wavelength used for analysis, except those for ICP-AES analysis of aluminum, calcium, magnesium, sodium, and potassium. For AA, the low-level standard is run only once at the beginning of the analytical run. The measured value is recommended to be within 20 percent of the theoretical concentration; however, specific acceptance criteria has not been established by the EPA. Therefore, data are used for internal data review purposes only.

6.3.6 Initial and Continuing Calibration Blank Analyses

Immediately after every initial and continuing calibration verification, a calibration blank is analyzed (at each wavelength for ICP-AES) and at a frequency of 10% (for most inorganic analytical methods) during an analytical run to check for baseline drift and low-level calibration curve bias.

The calibration blank (theoretically a 0-μg/L standard) contains only the matrix of the calibration standards. The concentration of the analyte in the calibration blank must be less than or equal to the RDL given in Appendix A. If the absolute value of the analyte in the blank exceeds the RDL, the analysis must be terminated, the problem corrected, the calibration checked, and the preceding samples since the last acceptable calibration blank reanalyzed.

6.3.7 Method (Reagent) Blank Analysis

A method blank is a sample that has undergone the same preparation (e.g., extraction, digestion, distillation) procedures as a real sample for analysis. One method blank is processed and analyzed for each group of 20 samples or less of similar matrix for each method that requires sample preparation. Therefore, the method blank results are an indicator of possible contamination. The concentration of the analyte in the method blank must be less than or equal to the RDL (see Appendix A). If the analyte concentration exceeds this limit, the source of contamination must be investigated and if possible, eliminated. All affected samples (having constituent concentration less than 10 times the RDL) in which the high blank value exceeded the RDL of the constituent in question should be reprepared and reanalyzed. If the problem persists, the affected sample datum is qualified appropriately.

It is the policy of LAS <u>not</u> to correct the analytical data for elevated analyte levels in the blank unless otherwise specified by the client or the method.

6.3.8 Matrix (Predigestion) Spike Sample Analysis

The spike sample analysis is designed to provide information about method accuracy and the effect of the sample matrix on the digestion and on measurement methodology. For analytical methods that require sample preparation, the spike is added before sample digestion and/or distillation steps. At least one spiked sample analysis is performed on each group of 20 samples of similar matrix (i.e., aqueous, soil, sludge, or sediment).

The accuracy in terms of percent spike recovery is calculated by using the results of the sample

designated as the "original" sample. Samples identified as blanks or laboratory spikes should not be used for spiked sample analysis because these samples provide minimal information regarding matrix interference.

The required spike level for each constituent analyzed is provided in the analytical method SOP.

If the spike recovery for the matrix spike is not within the limits specified in Appendix A, the LCS data are evaluated to determine if this condition is due to a matrix interference. If the LCS data are acceptable, the data for all samples associated with that spiked sample are qualified as matrix interference in the case narrative. If the LCS results are not acceptable, all samples associated with the batch are reprepared and reanalyzed.

The percent spike recovery (%R) is calculated as follows:

$$\%R = \frac{\text{spiked sample results} - \text{sample result}}{\text{spike added}} \times 100$$

When sample concentration is less than the RDL, a sample concentration of zero can be used to calculate %R.

6.3.9 Duplicate Sample Analysis

One duplicate sample is analyzed from each group of 20 samples of similar matrix.

The within-run precision is calculated as relative percent difference (RPD) between original sample and duplicate sample values as described in Section 3.1.

The acceptance limits for the RPD specified in Appendix A should be used for original and duplicate sample values approximately greater than or equal to 5 times the RDL. For concentrations less than 5x the RDL, the absolute difference between the sample and the duplicate must be less than the RDL.

For Ph determination, precision is expressed as absolute difference between the sample and its duplicate.

If the duplicate sample results are outside of the acceptance limits for a specific matrix, an investigation is done to determine the root-cause of the observed imprecision. Generally, the data are qualified indicating that the sample heterogeneity is suspected.

6.3.10 Graphite Furnace Atomic Absorption QC Analysis

Special procedures are required for quantitation when using the graphite furnace atomic absorption (GFAA) measurement technique. These requirements apply to drinking water analyses and spike analyses.

Drinking Water Analyses - For drinking water analyses, each sample must be spiked with a known concentration of each constituent of interest. The sample is quantitated based on the recovery of the analytical spike.

Analytical (Post-digestion) Spike Analysis - Analytical spikes are added automatically by the instrument during analysis. Analytical spike concentrations are based on specific method requirements.

6.3.11 ICP-AES Interference Check Sample Analysis

To verify interelement and background correction factors, an ICP-AES interference check sample is analyzed at the beginning and

end of each analysis run or at a minimum of twice per 8-hour work shift, whichever is more frequent.

An interference check sample comprises two solutions. Solution A consists of the interferants, and solution AB consists of analytes mixed with the interferants. An interference check sample analysis consists of analyzing the two solutions consecutively (starting with solution A) for all wavelengths used and for each constituent reported by ICP-AES. Table 5 provides the constituent and interferant concentrations used for the ICP-AES interference check samples.

Results of the ICP analyses of solution AB during the analytical runs must fall within the control limit of ±20% of the true value for the constituents included in the interference check samples.

If not, the analysis must be terminated, the problem corrected, the instrument recalibrated, and all analytical samples analyzed since the last acceptable check sample reading reanalyzed.

6.3.12 Internal Standards (ICP/MS)

For ICP/MS, internal standards are used to monitor and correct for changes that occur between standards and samples as a result of physical interferences. A minimum of three internal standards must be selected to bracket the mass ranges 1-70, 71-125, and 126-250. The internal standard must be added to every samples and intensities of any internal standard must fall between 60 to 125 percent (EPA Method 200.8) and 30 to 120 percent (EPA Method 6020) of that internal standard in the initial calibration standard. If recoveries are unacceptable, a different suitable internal standard must be selected, samples must be diluted and reanalyzed, or a higher level spike must be prepared and analyzed.

6.3.13 ICP-AES Serial Dilution Analysis

If the client requires CLP-level analyses, for all constituents analyzed by ICP-AES, the results of the ICP-AES serial dilution analysis on each group of samples of a similar matrix type should be analyzed and reported. A serial dilution analysis involves performing a five-fold dilution on a given sample. Blank samples are not used for serial dilution analysis.

If the constituent concentration is sufficiently high (at a minimum, a factor of 50 times above the IDL in the original sample), an analysis of five-fold dilution must agree within 10% of the original determination. If the % difference exceeds the 10% criteria for an analyte, chemical or physical interference is suspected. Results for that analyte for all samples associated with the batch are qualified appropriately.

The percent difference is calculated as follows:

$$\% \text{ Difference} = \frac{\text{abs } (I - S)}{I} \times 100$$

where I is the initial sample result and S is the serial dilution result (5 times the instrument reading).

6.3.14 ICP-AES Linear Range Analysis

Linear range determination must be performed quarterly for each constituent. The standard must be analyzed during a routine analytical run. The concentration of this standard must be within 5% of the true concentration. This concentration represents the upper limit of the ICP linear range. If measured sample concentrations exceed this level, the samples in question must be diluted and reanalyzed within the linear range.

Table 5. Constituent and Interferant Concentrations Used for ICP-AES Interference Check Sample

Constituent	Concentration (mg/L)	Interferant	Concentration (mg/L)
Barium	1	Aluminum	500
Beryllium	1	Calcium	500
Cadmium	2	Iron	200
Chromium	1	Magnesium	500
Cobalt	1		
Copper	1		
Lead	2		
Manganese	1		
Nickel	2		
Silver	2		
Vanadium	1		
Zinc	2		

6.3.15 ICP-AES Interelement Correction Determination

On an annual basis, correction factors for spectral interference due to aluminum, calcium, iron, and magnesium must be determined for all ICP instruments at all wavelengths used for each constituent.

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CHAPTER 7
ANALYTICAL INTERNAL QC FOR ANALYSIS OF ORGANIC CONSTITUENTS

7.1 INTRODUCTION

Quality control is integral to ensuring that analytical results for organic constituents are reliable and that data integrity is maintained throughout the measurement system. Specific guidelines for instrumental calibration and tuning and for methods of sample handling, sample preservation, and holding times are described in LAS method-specific SOPs, instrument manufacturers' guidelines, and EPA methods. This section summarizes the QC activities related to organic constituent analyses. These requirements may be superseded by a project-specific QA Plan or a method-specific SOP.

At LAS, it is prohibited to alter data solely to meet method- or contract-specified QC requirements.

7.2 HOLDING TIME COMPLIANCE

The method- or client-specified holding time requirements must be met for the sample results to be considered valid. If holding time requirements are exceeded for sample analyses, the client must be informed of the out-of-control situation, sample data must be qualified appropriately, and an explanation of the nonconformity must be provided in the case narrative of the analytical data report.

7.3 SYSTEM TUNING OF THE GC-MS SYSTEM

It is necessary to establish that a given gas chromatograph-mass spectroscopy (GC-MS) system meets the standard mass spectral abundance criteria specified in the method before sample analysis begins. This system tuning is accomplished through the analysis of p-bromofluorobenzene (BFB) for volatile organic analyses and decafluorotriphenylphosphine (DFTPP) for semivolatile organic analyses.

Before any sample, blank, or standard is analyzed, the hardware for each GC-MS system must be tuned to meet the method-specified ion abundance criteria. The ability to meet the abundance criteria must be demonstrated for each 12-hour period, unless otherwise specified. Whenever corrective action is taken that may affect the tuning condition (e.g., ion source cleaning or repair), the tune must be verified regardless of the 12-hour tuning requirements. If background subtraction is required, it must be designed to eliminate interferences that may result from column bleed or instrument background ions, and must not be performed solely to meet QC requirements. The documentation of the tuning is provided as a bar graph spectrum and as a mass listing.

7.4 INITIAL CALIBRATION OF THE ANALYTICAL SYSTEM

7.4.1 Initial Calibration of the GC-MS System

Before samples and required blanks are analyzed and after tuning criteria have been met, calibration standards that contain all the target compounds, surrogates, and internal standards are analyzed at method-required concentrations to calibrate the GC-MS initially to determine the sensitivity of the system and the linearity of response. Once the system has been calibrated, the initial calibration must be verified every 12

hours, unless otherwise specified, for each GC-MS system.

Secondary ion quantitation should be performed only when there are sample interferences with the primary ion. All standards shall be analyzed under the same conditions as the method blank and the routine samples. The relative retention times of each constituent in each calibration run should agree within method-specified criteria.

The initial calibration is considered valid only after the minimum relative response factor (RRF) and the %RSD criteria have been met. Sample analysis can begin only after these criteria have been met. When the accuracy of initial calibration is verified, the average RRFs and the %RSD for all target compounds must be calculated and reported on the appropriate calibration summary sheet.

7.4.2 Initial Calibration of the GC, HPLC, and FT-IR Systems

For the gas chromatography (GC), high performance liquid chromatography (HPLC), and Fourier transform infrared spectrophotometry (FT-IR) systems, the following calibration procedures must be performed. Before samples and required blanks are analyzed, calibration standards that contain all the target compounds and required surrogates are analyzed at method-required concentrations to calibrate the analytical system initially to determine the linearity of response.

If the method specifies the use of response factors for compound quantitation (i.e., CLP), the %RSD for each compound must meet the method-specified criteria. If %RSD is exceeded, an appropriate corrective action is instituted and a new initial calibration is performed.

Other methods, particularly SW-846, allow the analyst to use a calibration method best suited to the analytical technique. The calibration method used primarily at LAS is a quadratic fit forced through zero, followed by linear regression forced through zero, and linear non-forced through zero. The coefficient of determination (r^2) must be greater than or equal to 0.990 or the correlation coefficient (r) must be greater than or equal to 0.995 for the calibration to be considered valid in determining constituent concentration. Point-to-point calibrations are not used at LAS.

7.5 RETENTION TIME WINDOWS

Identification of target analytes is achieved by the use of retention time windows. For methods (i.e., GC/MS methods) that use internal standards, this is accomplished by establishing the retention time of each analyte relative to the internal standard during the daily continuing calibration. Relative retention times (RRTs) of target analytes are then calculated for all subsequent analysis. The RRT criteria must be met for analyte identification to be considered accurate.

External standard methods (i.e., GC, HPLC, FT-IR methods) differ in that the retention time criteria are established by using the absolute retention time for each analyte established during the initial calibration. A retention time window is established from the absolute values. The retention time window criteria may vary depending on the method. LAS' method-specific SOPs detail the requirements for establishing retention time windows for applicable methods.

7.6 INTERNAL STANDARDS

For the methods that employ internal standards for target constituent quantitation (i.e., GC/MS

methods), the internal standard solution must be added to every standard, blank, matrix spike, matrix spike duplicate, sample (for VOAs) and sample extract (for semivolatiles analyses).

Internal standard responses and retention times in all standards must meet the method-specified criteria. If these criteria are not met, the chromatographic and/or the mass spectrometric system must be inspected for malfunctions. When corrections are made and the system is demonstrated as in control, the affected samples shall be reanalyzed.

The extracted ion current profile (EICP) and retention times of the internal standards must be monitored and evaluated for each sample, blank, matrix spike, matrix spike duplicate, and LCS. If method-specified criteria were not met, the sample in question must be reanalyzed. If reanalysis does not solve the problem, both analyses results are reported.

7.7 CONTINUING CALIBRATION VERIFICATION

7.7.1 Continuing Calibration Verification of the GC-MS System

A mid-level calibration standard containing all target compounds, required surrogates, and internal standards must be analyzed every 12 hours during analysis, unless otherwise specified, to verify the initial calibration.

The internal standard responses, retention times, minimum RRFs, and percent difference (%D) criteria specified in the method or LAS SOPs must be met for the continuing calibration to be considered valid. If these criteria are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some potential problems may result from standard mixture degradation, purge-and trap system contamination (VOAs only), injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatography system. If the source of the problem cannot be identified after corrective action has been taken, a new initial calibration is required.

7.7.2 Continuing Calibration Verification of the GC, HPLC, and FT-IR Systems

A mid-level calibration standard containing all target constituents and required surrogates must be analyzed at the frequency specified by the method or the client.

Percent difference (%D) of concentration (i.e., the difference between the concentration of the continuing calibration and the midlevel initial calibration standard) must meet method- or client-specified QC criteria for the continuing calibration to be considered valid.

If the %D criterion is exceeded for any analyte, corrective action must be taken. The experience and the professional judgement of the analyst play a key role in determining the most suitable action. If the source of the problem cannot be identified after corrective action, a new initial calibration curve must be generated for the analyte that exceeded the criterion. These criteria must be met before sample analysis is continued.

7.8 LABORATORY CONTROL SAMPLE

An LCS is a volume of reagent water (meeting the specifications for ASTM Type II water or better) for aqueous samples or a contaminant-free solid matrix for soil or sediment samples, which is spiked with known quantities of target analytes and required surrogates. An LCS is prepared independently from the calibration standards and carried through the entire analytical process. An LCS must be analyzed

for each batch of up to 20 samples of the same matrix.

In the event that LCS data exceed the QC limits, the LCS recovery data are evaluated in conjunction with other QC analyses (i.e., MS/MSD, QCCS, surrogate spikes, method blank) to determine if the analytical process is in control. If the process is judged out-of-control, all affected samples and method blanks must be reextracted and reanalyzed.

7.9 INSTRUMENT AND METHOD BLANK ANALYSIS

An instrument blank consists of deionized, distilled water spiked with surrogates and is carried only through the analytical process. For low-level volatile organic analyses, instrument blank serves as a method blank because no preparation procedure is required for this method. The instrument blank measures any contamination that may result during analysis. The instrument blanks must be analyzed at a frequency specified by the method.

A method blank consists of all reagents and required surrogates in a volume of deionized, distilled laboratory water (meeting the specifications for ASTM Type II water or better) for aqueous samples or in a contaminant-free solid matrix for soil or sediment samples. However, the method blank is carried through the entire analytical process (i.e., extraction, concentration, and analysis). Its volume or weight must be approximately equal to the sample volumes or sample weights being processed.

A method blank must be analyzed for each batch consisting of up to 20 samples. For the analysis of volatile target constituents, a method blank must be analyzed before sample analysis, within each 12-hour period, or for each 10 samples, as specified in the method. Laboratory personnel must ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample-processing hardware that lead to discrete artifacts or elevated baselines are minimized.

No contaminants shall be detected above the RDLs in the instrument and method blanks. If a blank exceeds the RDL, the source of the contamination is investigated, and appropriate corrective actions are taken and documented before sample analysis proceeds. All samples associated with the method blank that contain high target constituent(s) must be reextracted/reanalyzed or the affected sample results properly qualified. The measured concentration of common laboratory contaminants (i.e., acetone, methylene chloride, and phthalates) must not exceed five times the RDL. Otherwise the samples associated with the unacceptable blanks must be reextracted and reanalyzed (Semivolatile organics) or reanalyzed (VOAs).

It is the policy of LAS not to correct the analytical data for elevated analyte levels in the blank unless specified by the client or method.

7.10 SURROGATE SPIKE ANALYSIS

The surrogate standards, specified in each method-specific SOP, are to be added to each sample, blank, duplicate, LCS, matrix spike, and matrix spike duplicate before purging or extraction. The surrogate spike recovery data provide information regarding the efficiency of the sample preparation and the analytical process. Surrogate analysis is evaluated by determining whether the surrogate spike percent recovery (measured as concentration) falls within the acceptance criteria for each method.

7.10.1 Surrogate Spike Recovery in Method Blanks

If recovery of surrogate compound in the method blank exceeds QC limits, the problem must be investigated to identify the root cause. If the extraction and/or analytical process is judged to be out-of-control and sample data quality is adversely affected, all affected samples shall be reextracted and reanalyzed when sufficient sample aliquots are available and holding times are not expired. The problem must be corrected before sample analysis proceeds. The specific corrective action is determined by the instrument operator and his/her technical lead and supervisor.

7.10.2 Surrogate Spike Recovery in Samples

If recovery of surrogate compound in client sample exceeds QC limits, the problem must be investigated to identify the root cause. The surrogate results in question must be evaluated in conjunction with other QC data (i.e., LCS data and method blank data). The same sample extract may be reanalyzed to determined if the out-of-control condition resulted from isolated, poor instrument performance. If the extraction and/or analytical process is judged to be out-of-control and sample data quality is adversely affected, the sample in question shall be reextracted and reanalyzed when sufficient sample aliquots are available and holding times are not expired. The problem must be corrected before sample analysis proceeds. The specific corrective action is determined by the instrument operator and his/her technical lead, supervisor, and the project manager.

7.11 MATRIX SPIKE AND MATRIX SPIKE DUPLICATE ANALYSIS

The matrix spike analysis is designed to provide information about the effect of the sample matrix on the measurement methodology. The minimum QC requirements for matrix spike and matrix spike duplicate analyses are listed as follows:

- A matrix spike analysis and a matrix spike duplicate analysis must be performed for each batch of up to 20 samples of a similar matrix and processed through the same procedure. Prepare the matrix spike solutions according to the method-specific SOPs.

- The percent spike recovery for matrix spike/matrix spike duplicate and relative percent difference (RPD) between the matrix spike and matrix spike duplicate are calculated as specified in the method-specific SOPs.

- If the QC criteria are not met for matrix spike or matrix spike duplicate results, use the results in conjunction with other QC data (i.e., LCS results, surrogate data, internal standard response) and determine the need for corrective action.

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CHAPTER 8
ANALYTICAL INTERNAL QC FOR ANALYSIS OF RADIOCHEMICAL CONSTITUENTS

8.1 INTRODUCTION

Internal QC is integral to verifying that data integrity is maintained throughout the measurement process. The validity of the radionuclide data generated can only be ensured through accurate and precise instrument calibration and through implementing rigorous internal QC practices. This section summarizes the QC activities related to radiochemical constituent analyses. The types and uses for various QC samples are also described in tables 2 and 3 in Chapter 3. The requirements in this chapter may be superseded by a project QA plan or a specific SOP.

At LAS, it is prohibited to alter data solely to meet method- or contract-specified QC requirements.

8.2 HOLDING TIME COMPLIANCE

The method- or client-specified holding time requirements must be met for the sample results to be considered valid. If holding time requirements are exceeded for sample analyses, the client must be informed of the out-of-control situation, sample data must be qualified appropriately, and an explanation of the nonconformity must be provided in the narrative of the analytical data report.

8.3 CALIBRATION OF THE COUNTING INSTRUMENT

The calibration of detection instrumentation used in radiochemical determinations, including initial setup and method-specific calibrations, is time consuming. Unlike calibration of other analytical measurement systems, however, calibration of radiochemical instruments is stable for long durations. Nevertheless, each instrument shall be calibrated and checked for drift as specified in the instrument manual, EPA and other nationally recognized standards, and practical laboratory experience. If more restrictive calibration requirements are requested by the client, calibrations will be performed according to project specific requirements when counting the project samples. All QC requirements regarding calibration must be met before sample analysis can proceed.

The calibration standards must be prepared or obtained from NIST-certified standards, NIST-traceable commercial standards, standards available from EPA, or commercial standards traceable to a national laboratory equivalent to NIST. The activity of each source shall be certified by the manufacturer, including uncertainty of the measurements.

8.3.1 Gross Alpha and Beta Counting by Gas Proportional Detector

8.3.1.1 Initial Instrument Setup and Calibration. The instrument must include a low-background, anticoincidence proportional counter, a sample detector, and cosmic detector. It must also discriminate between alpha and beta pulses. The instrument is to be configured according to the manufacturer's instructions; any changes or modifications to the configurations must be documented in the appropriate SOP.

Before samples and required blanks are counted, the instrument must be initially calibrated to establish operating high voltage, adjust for proper alpha and beta separation, and determine

alpha and beta background count rate, and alpha and beta counting efficiencies. Standard sources Am-241, Tc-99 and other sources as required by the project shall be used for calibration.

The plateau voltage is determined by counting one standard over a range of voltage in increments. Plot voltage versus counting activity to determine proper operating voltages for both alpha and beta counting. After plateau voltage is set, cross talk (i.e., sensitivity) and counting efficiency shall be determined. Cross talk shall be determined by counting known alpha activity on a beta counter, and known beta activity on alpha counter. Counting efficiency shall be determined by counting a known activity for each counting geometry over the sample weight range expected to be encountered during analysis.

LAL-91-SOP-0078 and LAL-91-SOP-0079 provide instructions and guidance for the calibration, maintenance, and operation of the Tennelec Alpha/Beta counting systems employed at the LAS. All QC criteria specified in the SOPs regarding initial instrument setup and calibration must be met before sample analysis can proceed. The instrument shall be recalibrated per the appropriate SOPs and instrument manuals after repair, change of detector, or when continuing calibration verification cannot meet QC requirements.

8.3.1.2 Calibration Verification. Background counts of at least 1-hour duration must be performed daily on each detector. Duration of the background count should be the same as the expected sample count duration. If the background counts are greater than two times the long-term average, the instrument is considered out of control.

Standard alpha and beta sources must be counted on each detector on daily basis. A minimum of 10,000 net counts shall be acquired for Tc-99, and a minimum of 10,000 net counts shall be acquired for Am-241. The value must fall within the 3 standard deviations of a long term mean value.

A plateau count shall also be performed whenever there is concern regarding the integrity of the system, as indicated by background and check source counts, to verify whether the initial calibration is valid.

A new initial calibration shall be performed when calibration verification checks cannot meet SOP or project specified QC requirements.

8.3.2 Alpha Spectroscopy System

8.3.2.1 Initial Instrument Setup and Calibration. The alpha spectroscopy system shall consist of a detector system capable of measuring alpha isotopes in the range of 3 to 7 MeV. The system shall have a resolution of less than 50 KeV for the isotopes Am-241 (5.49 MeV) or Pu-239 (5.14 MeV).

Before samples and required blanks are counted, the instrument must be initially calibrated for energy and counting efficiency by using multi-point alpha standards. A multinuclide source containing two or more nuclides such as Am-241, Cm-244, and Pu-239, or equivalent, shall be used for calibration.

LAL-91-SOP-0077 provides instruction and guidance for the calibration, maintenance, and operation of the alpha spectrometry system. All of the QC criteria specified in the SOP regarding initial instrument setup and calibration must be met before sample analysis can proceed. The instrument shall be recalibrated per the appropriate procedures and instrument manuals after repair, maintenance to the detector or electronics, and when continuing calibration verification cannot meet QC requirements.

8.3.2.2 Calibration Verification. Background counts of at least 12-hour duration for all regions of interest in use must be performed on a weekly basis for each detector. Duration of the background count should be the same as the expected sample count duration. If the background counts are greater than the project specific MDA's, the instrument is considered out of control.

Instrument check standards shall be counted once a week or prior to sample analysis, whichever is less frequent, to verify energy and efficiency calibration for a minimum of two nuclides. A minimum of 2,000 counts in each peak shall be acquired to compromise between the probability of detector contamination and counting statistics. The value must fall within the 3 standard deviations of a long term mean value.

If the calibration verification check exceeds the control limits, no samples are counted until the problem is investigated and the instrument is brought back into control. A new initial calibration shall be performed when calibration verification checks cannot meet SOP or project specified QC requirements.

8.3.3 Liquid Scintillation Counting System

8.3.3.1 Initial Instrument Setup and Calibration. A low-background counter consisting of two photomultiplier tubes that recognize coincidence events is required. The counter is operated with windows that maximize the figure of merit (FOM) when the samples are counted. The calibration standards are used to set the windows.

Before samples and required blanks are counted, the instrument must be initially calibrated for counter windows, counting efficiency and quench curve. The counter shall be calibrated with windows that maximize the FOM when possible (i.e., FOM = E^2/B; where E = detector efficiency, and B = background count per minute [CPM]).

A quench curve shall be established for each radionuclide of interest over a range of varying quenching similar to that is normally encountered during analysis. At least 10,000 counts are accumulated for each unquenched and quenched standard. For quench curve determination, both internal and external quench methods are used for at LAS. The external method is used routinely because it is more efficient for large batches of samples. The internal method (i.e., matrix spikes) is used as a verification of the external quench determination and when small numbers of samples make generating the external quench curve less efficient. The quench standards for each radionuclide to be analyzed shall be prepared according to the procedure specified in the corresponding method SOP for that radionuclide. The quench standards shall be contained in the same type of scintillation vial to be used for sample analysis.

LAL-91-SOP-0080 and LAL-91-SOP-0081 provide instructions and guidance for the calibration, maintenance, and operation of the liquid scintillation counters employed at the LAS. All of the QC criteria specified in the SOPs regarding initial instrument setup and calibration must be met before sample analysis can proceed. The instrument shall be recalibrated per the appropriate procedures and instrument manuals after repair, maintenance to the detector or electronics, and when continuing calibration verification cannot meet QC requirements.

8.3.3.2 Calibration Verification. Background counts of at least 1-hour duration must be performed on a daily basis. If the background counts exceeds the 3 standard deviations of a long term mean value, the instrument is considered out of control.

Standard sources of H-3 and C-14 shall be counted daily. A minimum of 100,000 net counts shall be acquired. The value must fall within the 3 standard deviations of a long term mean value.

If the calibration verification check exceeds the control limits, no samples are counted until the problem is investigated and the instrument is brought back into control. A new initial calibration shall be performed when calibration verification checks cannot meet SOP or project specified QC requirements.

8.3.4 Gamma Spectroscopy System

8.3.4.1 Initial Instrument Setup and Calibration. A beta absorber consisting of about 6 mm of aluminum, beryllium, or plastic may be used for samples that have a significant beta activity and high beta energies. Germanium detectors with high resolution are used for gamma spectrometry. The detector output is digitized and stored by using a multichannel analyzer. The system must be set up according to manufacturer's specifications.

Before samples and required blanks are counted, the instrument must be initially calibrated for energy and counting efficiency.

Depending upon the intended use, the gamma spectroscopy system should be calibrated for different energy ranges. In general, the energy range is set from 50 KeV to 2000 KeV which is suitable for most applications. However, other energy ranges may be utilized as necessary to analyze nuclides with energies lower or higher then this range.

For energy calibration, use an NIST traceable standard source containing a gamma emission near the lower and upper end of the desired energy range. For the 50-2000 KeV range, the mixed source SRM4275, containing the 123.14 KeV and 1274.5 KeV europium-154 peaks, may be used. Each photopeak of interest in the final spectra must contain no less than 10,000 net counts. A two-point calibration on the spectra is required to generate calibration data for energy versus channel number and the peak shape (FWHM) versus energy number.

Efficiency calibrations should be performed over the range of matrices and geometries normally encountered to obtain attenuation curves for each procedure. After the energy calibration is completed for the system, collect an energy spectrum using a calibrated radioactivity standard in a desired and reproducible counting geometry. The efficiency calibration may be performed using one or more spectra to obtain the required numbers of isolated singlets over the entire energy range of interest. Each full-range gamma-ray peak of interest must contain at least 10,000 net counts.

LAL-91-SOP-0075 provides instruction and guidance for the calibration, maintenance, and operation of the Gamma Spectroscopy System. All of the QC criteria specified in LAL-91-SOP-0075 regarding initial instrument setup and calibration must be met before sample analysis can proceed. The instrument shall be re-calibrated per the appropriate procedures and instrument manuals after repair, maintenance to the detector or electronics, and when continuing calibration verification cannot meet QC requirements.

8.3.4.2 Calibration Verification. Background counts of at least one hour duration shall be performed on each detector on no less than a weekly basis. Background count duration shall be as long as the longest sample count duration. The background counts must fall within the three standard deviation of the long term average of the LLD.

A three-point energy and efficiency calibration verification shall be performed daily using a sealed check source. The source used shall

meet the SOP specifications. The peak centroid energy, FWHM, and counting efficiency must fall within the 3 standard deviation uncertainty limits for each of the three peaks.

If the calibration verification check exceeds the control limits, no samples are counted until the problem is investigated and the instrument is brought back into control. A new initial calibration shall be performed when calibration verification checks cannot meet SOP or project specified QC requirements.

8.4 METHOD BLANK ANALYSIS

A method blank is a sample composed of all the reagents (in the same quantities) in the reagent-grade water carried through the chemical separation process and are used to determine sample contamination introduced during sample preparation.

Method blanks are analyzed at a frequency of 5% per batch and are analyzed in the same manner and with the same aliquot and count time as the samples. As required to obtain a statistically significant number of counts, the reagent blank may be counted longer than the samples.

The method blank value must be less than or equal to two times the MDA (or the RDL). If the reagent blank data indicate an out-of-control condition, the cause of the contamination must be eliminated before more samples are analyzed.

The samples counted with the contaminated blanks must be qualified or reprepared and reanalyzed, depending on the customer's needs.

It is the policy of LAS not to correct the analytical data for elevated radionuclide levels in the blank unless specified by the client or method.

8.5 LABORATORY CONTROL SAMPLE

To ensure that the sample preparation and counting processes are functioning within control, an independent LCS is prepared for each batch containing up to 20 samples. LCSs must be prepared and analyzed in the same manner as the samples, and must have the same aliquot size and count time as the samples. In the absence of absorption corrections and yield, the LCS will have the same MDA as the sample.

The LCS data must meet the SOP- or project-specified QC limits. The samples results generated with the out-of-control LCS shall be qualified or the sample reanalyzed, depending on the requirements of the customer.

8.6 CHEMICAL YIELD

Method performance on individual samples subject to chemical process and separation is established by means of spiking with tracer which is a radionuclide of the same element of the radionuclides of interest or with stable carrier of the same or a chemically similar element. All samples and QC samples shall be spiked prior to sample preparation. Sample specific chemical recoveries must meet the SOP- or project-specified QC requirements for LCS and method blank samples which are free from matrix interference.

Since the effects of sample matrix are frequently outside the control of the laboratory and may present relatively unique problems associated with each sample, the evaluation of data quality will be performed based on other QC results (i.e., LCS, method blank and duplicate recoveries), analytical experience, and professional judgement. If QC limits are not met for sample analysis, an explanation shall be provided in the case narrative of the final report.

8.7 DUPLICATE SAMPLE ANALYSIS

One duplicate sample shall be performed for each 10 samples (i.e., at a frequency of 10%) in a batch. Duplicate sample must be analyzed in the same manner and with the same aliquot and count time as the sample. Samples identified as field blanks shall not be used for duplicate sample analysis because poor precision is expected near the MDA. The method precision is determined as per SOP or project specifications.

If the duplicate analysis exceeds QC limits, the matrix homogeneity shall be evaluated to determine if reanalysis is required. If the duplicate analysis is out-of-control, a second, different sample, of the similar matrix, may need to be analyzed in duplicate or data must be qualified and a narrative shall be attached to the analysis batch worksheet.

8.8 MATRIX SPIKE AND MATRIX SPIKE DUPLICATE ANALYSES

Matrix spike (MS) and matrix spike duplicate (MSD) samples are prepared and analyzed only as required by the client. These samples are used to determine the sample matrix effect on the accuracy and precision of the measurement process.

The percent spike recovery and duplicate precision calculated as per SOP or project-specific requirements must meet the QC limits specified by the SOP or project. If the QC criteria are not met for MS or MSD results, the results from other QC analyses (i.e., LCS, duplicates, and other chemical recovery) are evaluated to determine the extent of corrective action.

CHAPTER 9
SYSTEMS AND PERFORMANCE AUDITS

9.1 INTRODUCTION

Technical systems audits, management assessments, and performance evaluations are essential in every quality assurance program. These audits are used to determine on-going compliance with the quality assurance program and project plans and to assess the overall quality of data collected during the measurement process. Furthermore, audits help in evaluating sample collection, sample analysis, and data handling procedures. The objectives of these audits are (1) to confirm proper conduct of all sample handling, sample analysis, and data handling and reporting procedures and (2) to minimize the generation of invalid data by detecting potential problems at the earliest stage possible in these processes. Such practices can save time and reduce costs associated with resampling and reanalysis.

9.2 TECHNICAL SYSTEMS AUDITS

9.2.1 Internal Audits

A technical system audit is an in-depth, qualitative, on-site evaluation of a sample handling, measurement, and data handling system. These audits are accomplished through (1) observing project activities, (2) inspecting operating conditions and documentation, and (3) interviewing project participants. The primary objective of an internal system audit is to assess and document all facets of the measurement process: facilities, sample preparation, instrument operation, instrument calibration, analytical measurement, data generation, data validation, data reporting, document control, waste handling, and overall QC practices.

These evaluations enable LAS management to ensure that three important actions are being performed:

- Record keeping is implemented in accordance with LAS Notebook Policy (LAL-90-SOP-0006) and sample chain-of-custody activities are implemented in accordance with Sample Receiving and Login (LAL-90-SOP-0002) and LAS Internal Chain-of-Custody and Evidentiary Procedures (LAL-90-SOP-0009).

- Current versions of SOPs (Appendix C) are readily available, properly controlled, and are being followed by the laboratory staff.

- Analytical QC is being followed in accordance with the LAS Quality Assurance Management Plan, the method or SOP, and all other client specifications. For this purpose, analytical QC is defined as (1) the analysis of the proper types and numbers of QA and QC samples (e.g., standard reference or performance evaluation samples, blanks, spikes, duplicates), (2) the maintenance of proper standards traceability, (3) the data reporting from those analyses on laboratory reporting forms, and (4) the use of the proper corrective action measures.

In order to eliminate any question of conflict of interest, the technical system audits of analytical processes must be performed by quality assurance personnel or other independent Lockheed staff who have no other responsibilities related to the data generation operations and who report outside the Operations Department. At the

LAS, auditors are part of the Quality Assurance Department; they report directly to the Quality Assurance Manager who, in turn, reports directly to the LAS Director (see Chapter 2). LAL-90-SOP-0010 provides details of the LAS approach to internal technical systems auditing.

9.2.1.1 Systems Audit Procedures. During the audit, the auditor may use the LAS laboratory technical systems evaluation questionnaire to document observations. This process ensures that the auditor has examined all elements of the system under evaluation. The questionnaire also aids in discussing data quality issues related to the QA and QC sample analyses (e.g., blank, spike, duplicate, and single-blind samples). Documentation, such as chain-of-custody forms, analysis request forms, SOPs, logbooks, reagent bottle labeling, and instrument printouts, is inspected and randomly cross checked (e.g., dates, initials) when applicable. A detailed discussion of the technical systems evaluation is given in LAL-90-SOP-0010.

The auditor summarizes all observations in a technical systems evaluation report and brings all problems observed to the attention of the Quality Assurance Manager, Operations Manager, and responsible supervisors for corrective action. The auditor also maintains a log used to track corrective action requests and results. Chapter 15 provides details of the corrective action mechanisms in place at LAS.

9.2.1.2 Internal Auditing Schedule. Routine technical systems evaluations are performed semiannually. These evaluations may or may not be announced to the operations staff. Furthermore, unannounced follow-up evaluations are performed as required to ensure that any deficiencies identified during the routine evaluations were corrected in a timely manner. In addition, preliminary systems audits may be performed for major clients before any environmental samples are analyzed for a particular project to ensure that the laboratory is generating products of an acceptable quality.

9.2.2 External On-site Systems Audits

It is LAS policy to respond in writing to any corrective action requirements identified by external auditors. The response includes the corrective actions implemented, or the proposed resolution and the proposed schedule for its implementation.

9.3 PERFORMANCE EVALUATIONS

9.3.1 Internal Performance Evaluations

A performance evaluation (PE) audit is a quantitative evaluation of the laboratory analytical system. PE audits are performed annually or more often as new major methods are introduced into routine operations. The evaluation generally involves the measurement of a PE reference material that has a known value or composition. These samples are key factors in environmental sample analysis; thus, they must be of high quality. It is important that the reference material be certified (e.g., NIST, EPA, private supplier) or, at a minimum, verified before use, and that the certification or verification be adequately documented. Certification documents are maintained by the Quality Assurance Department.

The Quality Assurance staff ensures that the PE materials are selected in such a way that the concentration of PE samples are representative of the media and the levels of inorganic, organic, and radionuclide constituents typically processed at LAS.

Two types of PE samples are used to monitor analytical system performance, such as single-blind and QC check standards (QCCSs). Single-blind PE samples are samples that the

analyst knows are audit samples, but for which the analyst does not know the constituent levels. QCCS are samples submitted in such a manner that the analyst knows that the sample is a PE sample and is also aware of the theoretical concentration of the constituents in the sample. The main function of the QCCS is to provide immediate feedback to the analyst during the sample analysis, so that if the results of the QCCS analysis do not fall within predetermined levels of precision or accuracy, appropriate corrective actions within the analysis system can be pursued.

Data obtained from the analyses of the audit samples are used for the following purposes:

- To judge the ongoing capability of the analyst and sample preparation technician, the reliability of the instrumentation, and the proficiency of the method(s).

- To establish a statistically valid estimate of the accuracy and precision of the measurement system.

- To assess whether or not the system is operating within the established control limits on a daily basis and over extended periods.

Acceptance criteria may be established for the measurement of each constituent in the audit samples. The recommended acceptance limits provided by the PE material supplier and method-specified QC acceptance criteria are used to assess the acceptance of data. If the analytical results fall outside the criteria, the Quality Assurance auditor documents this condition on the Nonconformance & Corrective Action Record and immediately contacts the analytical laboratory personnel to request corrective action. Typically, corrective actions may require (1) recheck and recalculation of data, (2) reevaluation of other related QC results, or (3) instrumental or procedural refinements. If major deficiencies are identified, another suitable PE material may be submitted to verify that the proper actions have been executed to eliminate or minimize the potential for recurrence of the problem.

9.3.2 External Intercomparison and Performance Evaluation Studies

Semiannually, LAS participates in the analysis of water pollution (WP) and water supply (WS) PE samples issued by the EPA EMSL-Cincinnati as required by the Clean Water Act (CWA) and Safe Drinking Water Act (SDWA) Programs. The results achieved from these studies verify our capability to analyze low-level, drinking water and waste water samples for inorganic and organic constituents.

As specified by the LAS Radioactive Material License issued by the Nevada State Health Division, LAS consistently and actively participates in U.S. EPA's Environmental Radioactivity Laboratory Intercomparison Studies Program. Simulated environmental performance evaluation (PE) samples containing known amounts of one or more radionuclides of interest are analyzed to verify LAS' capability to determine low-level radionuclides in multimedia environmental samples (e.g., water, air, vegetation, milk, etc.) with a stated level of confidence. The LAS also participates in the Department of Energy's (DOE's) Environmental Measurement Laboratory (EML) Radiochemical Proficiency Program for the analysis of various radionuclides in environmental and low-level mixed waste samples that consist of water, soil, and vegetation. In addition, each year, LAS analyzes PE materials to obtain accreditations through various federal, state, and local accrediting authorities (e.g., U.S. Army Corps Of Engineers, State Departments of Health).

The analyses of these PE samples independently demonstrates LAS' capability to perform required testing and to produce analytical results of known and documented quality.

Table 6 presents the external PE Programs administered by the EPA and DOE in which LAS routinely participates.

9.4 MANAGEMENT ASSESSMENTS

Each year, the LAS Director or designated management staff evaluates the LAS Quality Assurance Program to ensure its continuing suitability and effectiveness of its implementation and to introduce any necessary changes or improvements. The purpose of this independent, qualitative assessment is to verify the effectiveness of the LAS Quality System by determining the adequacy of policies, objectives, organization, procedures, and practices to ultimately ensure data quality. This type of an assessment provides a management tool for continuous evaluation and improvement of LAS activities. This assessment typically includes a detailed review of the following matters:

- Results from external on-site audits conducted by clients.
- Results from internal evaluations, including corrective actions implemented.
- Results of PE intercomparison studies.
- Results of internal blind PE studies.
- Details of any complaints from clients.
- Staff training for new and existing staff.
- Adequacy of staff, equipment, and facility resources.

The management assessment findings and actions are documented in the form of an internal memorandum.

Table 6. External PE Intercomparison Studies in Which LAS Routinely Participates

Agency	PE Study Type
U.S. EPA Environmental Monitoring Systems Laboratory at Cincinnati, OH	Water Pollution/Water Supply for inorganic and organic constituents
U.S. EPA Environmental Monitoring Systems Laboratory at Las Vegas, NV	Uranium in water Alpha, Beta in water Mixed Gamma in water Pu-239 in water Ra in water
DOE Environmental Measurements Laboratory (EML)	Uranium in soil Uranium in water Mixed gamma in water and soil Total U in water and soil Pu in water and soil
U.S. Army Corps Of Engineers	Inorganic and organic constituents

CHAPTER 10
DATA HANDLING, MANAGEMENT, AND REPORTING

10.1 INTRODUCTION

A carefully executed Quality Assurance Program emphasizes sufficient document control and data management, minimizes the generation of data that are not scientifically or legally defensible, and results in efficient, cost-effective data management. Data needs differ depending on the requirements of a specific project or client. Rigorous, broad-based analysis, QA, and QC are especially important in cases involving site clean-up because legal actions rely heavily on the quality of analytical data generated during site characterization. For environmentally related measurements, the quality assurance program established for the CLP is considered to be the minimum necessary to provide defensible data for regulatory, enforcement, legal, or policy matters. At LAS, proper documentation is an important facet of scientifically sound, high-quality data.

The National Enforcement Investigations Center (NEIC) in Denver, Colorado, has established policies and procedures for data handling in analytical laboratories that participate in the CLP (Laidlaw, 1986). The significance of NEIC enforcement is that data and documents are evidentiary materials and, as such, must be able to withstand legal scrutiny. NEIC policies and procedures have been incorporated into the data handling operations at LAS.

10.2 DATA CONFIDENTIALITY

It is LAS policy to preserve the confidentiality of data and reports generated by the laboratory and to respectfully decline release of this information to persons other than authorized representatives of clients. Reports and supporting records maintained in the Document Control Section are not released to persons without approval from laboratory management. Furthermore, records are not released to persons or organizations outside of Lockheed unless directed to by competent authority in the client's organization. If directed by courts-of-law or other competent authorities, such as regulatory agencies, we will provide records as necessary and notify our clients and provide information as to the identification of the requestor and the records that were released.

It is also our policy to respect client and/or project requirements for confidentiality of projects. When confidentiality clauses are contained in contractual documents, we will not release any information without first obtaining written approval from the client.

10.3 SAMPLE RECEIPT DATA

Chapter 4, Sample Management, and Chapter 13, Internal Sample Chain-of-Custody and Evidentiary Procedures, describe the sample handling and transfer systems in detail; this section pertains specifically to the handling of the sample data.

Upon arrival at LAS, environmental samples are logged into the sample management data base. Sample information provided in the system must include:

- Job and client name.
- Date and time of sample collection (to track holding time).
- Date of sample arrival at the laboratory.
- Client sample ID number.

- Corresponding internal LAS sample ID.
- Types of analyses requested.
- General observations concerning the conditions under which the samples arrived.
- Number and types of samples.
- Designation of samples for quality assurance analysis, if required.
- Storage location.

10.4 SAMPLE BATCHING

Before analysis, samples shall be grouped into analytical batches and ordered according to assigned LAS batch IDs. A distinct sample ID number should be assigned to each QA and QC sample to ensure correct identification and inclusion of the correct number of quality assurance samples in each batch.

The LAS follows all method-specified requirements for sample batch size by sample matrix type and by QC sample type and frequency requirements. Because of specialized client requirements, often due to short holding time requirements or other needs, a smaller than normal ratio of environmental samples to QC samples may be necessary. Although this may not be the most efficient approach for sample throughput, the client will be provided with technically sound, high-quality data. The LAS generally conforms to the EPA CLP "batch" definition of a sample delivery group (SDG), which includes 20 consecutive samples, whether they are received in one day or are cumulatively received over a maximum 14-day period. The 14-day limit is frequently prohibitive if holding time must be considered, especially for volatile and cyanide analyses, and semivolatile and pesticide extractions.

10.5 ANALYTICAL DATA DOCUMENTATION

Accountability for analysis begins with receipt of the samples. At LAS, laboratory personnel typically use bound logbooks with prenumbered pages or sample preparation and analysis bench sheets to record data. Validation of measurement data is easily accomplished by requiring the analyst to review, date, and sign data for each analysis on the day completed. This validation can be further strengthened by providing space for the laboratory supervisor's (or designee) signature, which indicates that he or she has witnessed the data production and the completion of the analysis. Hard-copy data generated by a computer can be permanently affixed in the logbook; hard copy so affixed is acceptable as an original record of sampling and laboratory logging. All original raw data (e.g., chromatograms, logbooks) are maintained in the laboratory while in use, then forwarded to Document Control for long-term storage.

10.5.1 Standards and Reagents Data

The working standards made from certified materials must be labeled with complete information (i.e., standard preparation dates, standard ID, concentration of each constituent (if possible), solvents used, expiration dates, and preparer's name). To ensure standards traceability, this information and further details regarding the preparation of the working standards should be recorded in a bound standards logbook. The documentation should include lot #, concentrations, preparation data, date prepared, storage conditions, preparer's initials, and expiration dates.

10.5.2 Instrument Operation Data

Specific injection/analysis and maintenance logbooks are maintained for each instrument.

These logbooks contain records of all routine and emergency maintenance, tuning, calibration, and analytical activities conducted on the instrument. The project name, the date that the analysis is performed, and the names of the analyst(s) who operated the instrument should be recorded on each page. Upon completion of an operational period, each responsible analyst must validate the information by signing and dating the bottom of the page. Twice each month, each supervisor or designee verifies the accuracy of the information recorded by signing and dating the bottom of the page. Periodically (typically during an internal on-site evaluation) the Quality Assurance Department representative reviews the LAS notebook to ensure that standard procedures are being followed, then verifies the review by initialing and dating each page. The main portion of each page may contain information regarding instrument maintenance and modification, tuning and calibration activities, instrument settings, instrument operating conditions, and the sample analyzed. If automated data management systems are used, reference to the data file for each standard or sample should be recorded.

Hard-copy instrument readouts (such as chromatograms and integrator tapes) must be labeled with analysis date, time, type of analysis, sample ID, and reference to the calibration curve used for quantification; the identification of chromatographic peaks also should be noted. The analytical data package is filed in the Document Control Department.

10.6 MAINTENANCE OF LOGBOOKS

Logbooks are invaluable documentation tools, whether they are used in sample receiving or sample preparation and analysis operations. Regardless of their specific purpose, some general rules apply to the maintenance of LAS logbooks. LAL-90-SOP-0006 details the procedures for logbook maintenance; an overview is provided below.

Logbook entries should be completed in black or blue ballpoint ink. Complete information (e.g., dates, data, sample numbers, observations) should be legibly entered so that an examination by a supervisor, auditor, or another analyst can easily determine what was done, by whom, when, and the results. After the last entry is made, the analyst signs the page. If more than one entry is made on the same page, the analyst should initial and date for each entry. Corrections are made by drawing a single line through the incorrect entry; the line must not obscure the entry. The correct information is then entered and is initialed and dated. The use of correction fluid is prohibited. If the page is not completely filled out, a "Z" or a "slash" should be drawn covering the blank section of the page.

Loose sheets, such as computer printouts or certification information, may be permanently affixed to the logbook provided that the analyst initials and dates over the pasted record. Original pages are never removed from the logbook. The use of bound logbooks encourages a chronological sequence of data insertion. Numbering of pages encourages use of data in sequence, and a table of contents ordered according to date, time, sample ID, type of analysis, type of sample (i.e., routine, blank, and duplicate), and identity of analyst aids in referencing data.

Bound, numbered laboratory logbooks are issued by the Document Control staff. Each logbook is tracked by the Document Control staff, and all completed logbooks are transferred to Document Control for long-term storage if they are not used as a reference document.

10.7 DATA REPORTING FORMATS

The way in which data are reported depends on the specific needs of the client. To meet the special needs of our clients, LAS produces numerous types of "standard" deliverable

packages appropriate for reporting of inorganic, organic, and radionuclide analyses data. The standard packages are suitable for (1) clients who are interested only in determining the concentration of specific constituents of interest; (2) sampling and analysis projects that require analytical data to be presented for review by local and state regulatory agencies; and (3) environmental projects that involve remedial investigations (RIs), feasibility studies (FSs), and site cleanup operations mandated by EPA or by state regulatory authorities. The standard types of data deliverables include data packages that provide CLP-level documentation for inorganics and organics and CLP-like documentation for radiochemical analyses.

General information such as dates of sample collection (if known), sample receipt, sample extraction (if applicable), and analysis are also provided on the data reporting forms for every data package.

Data in electronic format are also provided as required by the clients.

10.8 DATA MANAGEMENT SYSTEM

Large portions of the analytical data generated in laboratories are initially recorded on bench sheets, then transferred onto data reporting forms or into computer data bases, and the danger of data transfer error increases each time the data are copied. To minimize such errors, at LAS every effort is made to enter data directly into the computer data base, or direct output of data is provided from instruments (e.g., AAS, ICP-AES, GC, GC-MS) into a data base.

At LAS, the Laboratory Data Management System (LDMS) serves as the central repository for sample data. Five subsystems are integrated into the LDMS: Sample Management, Test Scheduling, Sample Preparations Specification, Quality Control Charts, and Disposal. These subsystems are combined using the Oracle Relational Data Base Management System. The LDMS provides each sample with a unique bar-code identifier which links sample data (e.g., preparation data, analysis data, result data, turnaround times) to the customer or SDG. This system of barcodes allows the samples to be tracked throughout the laboratory, providing location, status, and time constraints. The LDMS also provides an excellent audit trail, capturing such information as the name of the analyst who made the change (user ID and password), the reason for the change (the analyst is required to input the reason), the data existing before the change, and the changed data. This audit trail is always accessible for review by management and the Quality Assurance staff only. Reports in the LDMS are generated in standard or tabular formats to meet the customer's specific needs.

Computer security is controlled by a User Name/Password System at three levels: Local Area Network, Unix Operating System, and the LDMS. Security access to the subsystems is restricted to only those personnel allowed to add, modify, and change data. Other personnel within the LAS can be defined as "view only." This status entitles these users to examine status and result data, but not to change the data. The Audit Module monitors and tracks access activity and requires electronic comments to be entered should results be modified.

Testing of software and hardware at LAS is initiated through request forms: Change Request, New Requirements, and Discrepancy Reports.

Software QA. LAS employs LDMS software purchased from an external source. Project definition, functional design, and implementation phases of the LDMS software is required to be implemented and documented adequately.

All internally developed software related to data generation activities is evaluated by Computer Center staff, the Operations staff, and the independent QA Department staff to ensure that computer-generated data are accurate and meet the end users' specific requirements. The software review is documented on the Lockheed Software Verification Form presented in Figure 3 and requires the approval of the independent QA Department staff. The LAL-91-SOP-123 and LAL-91-SOP-124 delineate LAS-specific procedures related to the independent validation, verification, and documentation of the software according to its intended use.

10.9 DOCUMENT CONTROL

LAS is equipped with a centralized document control facility managed through the Document Control Section. Document Control provides a secure location to store and account for all official LAS records. The Document Control Section is responsible for:

- Storing all applicable client data, including sample analysis data, QC data, and the final delivery report.

- Storing, maintaining, and managing records for LAS SOPs, quality assurance data files, laboratory logbooks, laboratory certifications, performance evaluation reports, health and safety records, administrative operations, electronic media data, and facility security data.

- Maintaining and monitoring off-site facilities for long-term storage and archiving of all records described above.

The location of each document control item is given in the Document Control Inventory Index Notebook. A document control number, following a standard numbering convention, is assigned to every accountable item in the Document Control system.

Quality assurance files in hardcopy and/or electronic media include: internal and external PE Studies; corrective action reports; federal and state certifications; internal and external systems audit reports; certificates of internal PE materials, standard reference materials, and support equipment; MDL study data; calibration and internal QC data; control charts; annual balance/weight certifications by external vendors; sample preparation, analysis, and maintenance logbooks; training data; and project-specific QA/QC information.

Access to the Document Control room is restricted. Only Document Control Section and designated technical leaders and management personnel have access to the document control files. Upon request, section personnel retrieve file item(s). A work table is available to laboratory personnel for reviewing requested items. When an item is removed, it is recorded on a sign-in/sign-out sheet.

All records of chemical analyses, including all raw data, calculations, quality control data, and reports, are kept for a minimum of three years unless otherwise specified by the customers.

LAL-90-SOP-0001 details the procedures outlined above and specifies document control for sample analysis data, SOPs, QA and QC data, LAS administrative files, and electronic media.

Figure 3. Lockheed Software Verification Form

LOCKHEED SOFTWARE VERIFICATION FORM

SOFTWARE TITLE			DATE	NUMBER
DEVELOPER	DEPT CODE	PHONE	TYPE OF SOFTWARE (MACRO, PROGRAM, ...)	VERSION.RELEASE
OPERATING HARDWARE	SOFTWARE DEVELOPED IN (LOTUS, EXCEL, DBASE ...)	MANUFACTURED ☐ IN-HOUSE ☐	MANUFACTURER	

DESCRIPTION OF SOFTWARE/FUNCTION

DESCRIPTION OF TEST PLAN (FUNCTIONAL TESTS, RANGE TESTS, PERFORMANCE TESTS, CALCULATION TESTS, INPUTS, OUTPUTS, ...)

TESTER(S) ASSIGNED/DATE

ASSESSMENT OF TEST RESULTS (FUNCTIONAL, RANGE, PERFORMANCE, CALCULATIONS, INPUTS, OUTPUTS, SITE ANY DEFICIENCIES, ...)

OTHER COMMENTS (BE CANDID)

TESTER(S) SIGNATURE/DATE

☐ APPROVED FOR IMPLEMENTATION.

☐ ADDITIONAL DEVELOPMENT OR ANALYSIS REQUIRED. ASSIGNED TO:_____ By:_____

(DESCRIBE UNDER COMMENTS SECTION.)

☐ DISAPPROVED. (EXPLAIN UNDER COMMENTS SECTION.)

COMMENTS SECTION

TESTER/SUPERVISOR SIGNATURE/DATE

☐ SOFTWARE VERIFICATION ACCEPTED BY QA.
REQUEST CLOSED.

QA SIGNATURE/DATE

CHAPTER 11
DATA EVALUATION AND VALIDATION

11.1 INTRODUCTION

Data validation is the process in which data are assessed for acceptability on the basis of established criteria. The primary objective of this quality assurance function is to assess and document the technical quality of the data generated from inorganic, organic, and radionuclide determinations performed by the laboratory analysts. A supporting objective is to evaluate the overall performance of the measurement processes on a continuous basis. The LAS staff do not interpret data useability for a client. We assess and document the technical quality of data in order to help the client evaluate the useability of the data and make sound environmental decisions.

The LAS Quality Assurance Department personnel use a structured mechanism for validating analytical data, thereby minimizing subjectivity. This chapter provides an overview of the procedures used in reviewing and validating analytical data generated by the laboratory staff. LAL-90-SOP-0008, LAL-90-SOP-0012, LAL-90-SOP-0013, and LAL-91-SOP-0088 describe in detail the procedures and the acceptance limits involved in reviewing and validating data. LAL-93-SOP-0274 delineates the procedures for assessing data integrity using electronic media. These SOPs are also useful in training Quality Assurance and technical staff to validate and review data correctly and consistently.

The data validation procedures described here comply with the requirements specified in:

- U.S. EPA Laboratory Data Validation Functional Guidelines for Evaluating Inorganic, Organic, and Pesticide/PCB Analyses (1988).
- U.S. EPA National Functional Guidelines for Organic Data Review (1991).
- U.S. EPA Guidelines and Specifications for Preparing Quality Assurance Project Plans (QAMS-005/80).
- U.S. EPA Contract Laboratory Program Statement of Work (current version).

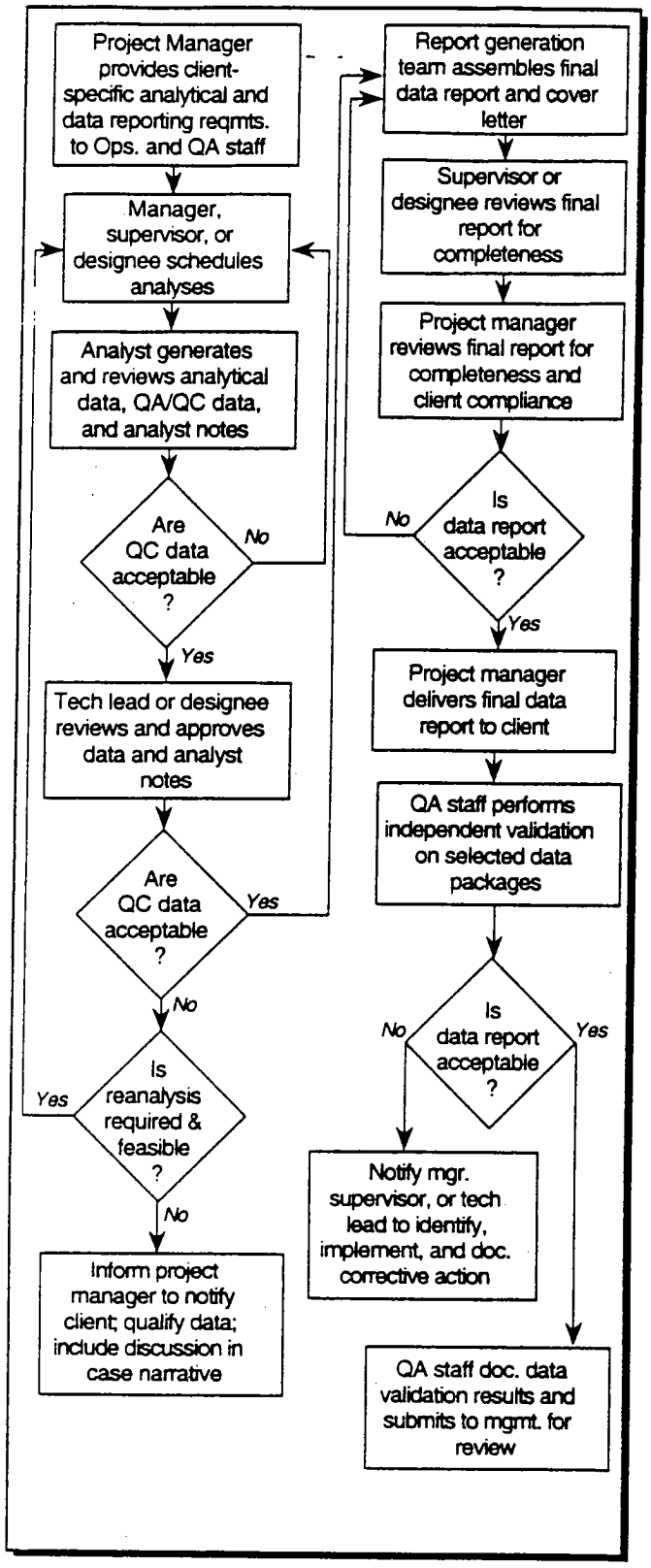
11.2 TECHNICAL DATA REVIEW

To ensure the identification and correction of potential anomalies early in the data generation process, all measurement data produced during laboratory analysis are reviewed, first by the analyst, then by the supervisor or a designated technical specialist. Quality Assurance personnel review selected data packages in depth to further ensure data integrity. Figure 4 is an overview of the LAS data review flow. LAL-90-SOP-0008 describes the technical review of data packages.

11.2.1 Data Review By Analyst

During the course of the analytical run, the analyst incorporates all applicable QC check samples as specified by the standard method of interest documented in the LAS SOPs. Following each QC sample analysis, the analyst performs necessary calculations either manually

Figure 4. Overview of the LAS Data Review Process



or using appropriate software. If a QC check exceeds acceptance criteria, an appropriate corrective action (e.g., redigestion/reextraction, dilution, recalibration, reanalysis) is identified and implemented. In the event that QC analyses are not acceptable and an appropriate corrective action cannot be performed, data are qualified using standard data qualifiers. Information related to the analytical run is thoroughly documented in the injection or instrument logbooks. Also, the analyst completes a checklist or a batch narrative to indicate that he or she has reviewed the data.

11.2.2 Data Review by Section Supervisor or Technical Specialist

Following completion of a batch of samples, the section supervisor or designated technical specialist reviews the data package (generated by the analyst in either electronic or hard-copy format) to ensure that the calculations are accurate, that internal QC samples are analyzed at the required frequency, and that QC data meet method- or LAS-established criteria. If discrepancies are identified, the supervisor discusses and resolves them with the analyst by assigning an appropriate corrective action, which may include recalibration, repreparation, and reanalysis of samples in question. The Supervisor or designee also performs a completeness review of the data report before it is forwarded to the Project Manager. The reviewer documents his or her review on the checklist, the data reporting forms, or the raw data.

11.2.3 Data Review by Project Manager

The final review is performed by the Project Manager (i.e., client services representative) who evaluates the data package for completeness, accuracy, consistency, and client compliance. A data package that meets all the requirements is then submitted to the client. The final data package is also submitted to

Document Control for long-term storage to ensure data custody.

11.2.4 Data Review by Quality Assurance Department Staff

For selected data packages, the Quality Assurance Department staff performs independent, extensive evaluation of data quality, report completeness, and client compliance to identify systematic problems associated with the data production process. LAL-90-SOP-0012, LAL-90-SOP-0013, and LAL-91-SOP-0088 describe the procedures for inorganic, organic, and radionuclide data validation, respectively. LAL-93-SOP-0274 describes the procedures for assessing data integrity using electronic media (tape audits) for analyses that employ GC and GC/MS techniques.

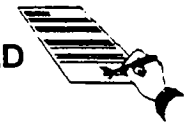
A preliminary review of the chain-of-custody record and Sample Discrepancy Report for sample-specific information (e.g., sample collection, preservation, holding time requirements, cooler/sample condition upon arrival) is performed to assess sample integrity. Holding times between sample collection and analysis are checked to assess potential degradation or loss of analytes of interest.

Analytical precision and accuracy are evaluated by using QC check standards, LCSs, surrogate spikes, matrix spikes, matrix spike duplicates, and unspiked duplicates and tracer recovery to estimate the degree of variance around the reported value and any bias effect due to matrix or laboratory sample processing procedures. In addition, data obtained from external SRMs, submitted as unknowns (blind) samples to the analyst, are evaluated to assess the accuracy of the overall laboratory system and the reliability of the data. Potential for background contamination that may result from sample containers, reagent water, reagents or solvents used during digestion or extraction, cross contamination during storage, or carryover during analysis are evaluated using instrument (calibration), method, and holding blank data. MDLs and RDLs are evaluated to ensure that minimum detectability requirements specified by the method or client are met.

Random errors resulting from incorrect calculations, transcriptions, unit conversions, and switched samples are also examined by independent recalculations against the raw data.

If systematic errors are identified during the QA data review, a Nonconformance & Corrective Action Record (NCAR, Figure 5) is initiated by the reviewer and submitted to the responsible LAS staff. The corrective action implemented is then documented on the NCAR by the responsible party and verified by the QA staff. Collectively, the evaluation comments regarding the data quality are documented in a checklist, which is specific to a client's batch of samples or to an analytical batch, to indicate whether DQOs are met and that resultant data are valid. A detail description of the corrective action program is provided in LAL-92-SOP-0190 and in Chapter 15.0.

Figure 5. LAS Nonconformance & Corrective Action Record



NONCONFORMANCE & CORRECTIVE ACTION RECORD

NCAR - Page 1 of ___

Nonconformance

___ Analytical Method	___ Sample Handling & Management	___ Communication
___ Analytical System	___ Data Review & Validation	___ External PE Studies
___ Sample Preparation	___ Data Reporting	___ Other *(describe below)*
___ Standard Prep & Traceability		_____

Detailed Description of Nonconformance

Project Name/Job Name	Method	Prep Batch ID	Analysis Batch ID
Originator's Signature	Date	Supervisor's Signature	Date

Corrective Action

Detailed Description of Corrective Actions Implemented or Planned

Responsible Person's Signature	Date
Supervisor's Signature	Date
Client Notification Required? ___ yes ___ no ___ n/a	Date Contacted

Corrective Action Verification

Detailed Description

QA Staff's Signature	**Date**

CHAPTER 12
ASSESSMENT OF ANALYTICAL DATA QUALITY

12.1 INTRODUCTION

Data corresponding to four of the six primary analytical DQOs (see Chapter 4) can be assessed quantitatively. Quantitative assessment of precision, accuracy (as bias), and completeness is discussed here. Detectability, which must be assessed during the analytical process, is discussed in Chapters 4, 7, 8, and 9.

12.2 PRECISION

Precision is an estimate of variability. It is a measure of the agreement among individual measurements of the same sample or same concentration (e.g., performance evaluation samples, matrix spikes and matrix spike duplicates, or unspiked duplicates). For analytical measurements performed at LAS, precision is expressed as intralaboratory precision (precision within a single laboratory).

Intralaboratory precision estimated from field duplicate data represents variability that results from sample collection, processing, analysis, and inhomogeneity. On the other hand, analytical laboratory duplicate data (i.e., spiked and unspiked duplicates and LCS duplicates, if applicable) represent variability that results from the measurement process (i.e., sample processing and analysis in the laboratory). Further, precision within a single laboratory can be evaluated in terms of repeatability (within-run precision) and reproducibility (between-run precision).

Interlaboratory precision can be best estimated through repeated measurements of the same sample type at the same concentration. These samples (external performance evaluation audit samples) are used to establish overall analytical laboratory performance.

Precision for a duplicate pair (a sample and its duplicate) is calculated as RPD or as RSD when more than two data points are involved. For radiochemistry, RER is also used to determine precision. (See Section 4.1 for further explanation of the RPD, RSD, and RER equations.) Large percent RPD/RSD indicates poor precision between the sample and its duplicate for a given constituent. Ideally, values for a sample and its duplicate would be equal, and the standard deviation would be zero. However, as the mean concentration of the duplicate pair approaches the detection limit for that measurement, a high RPD or RSD is expected because large relative errors may occur at low concentrations. Consequently, the precision limits given in Appendix A represent precision at approximately 5 times the RDL, a level at which the precision is expected to stabilize.

12.3 ACCURACY (BIAS)

Accuracy (bias) is determined by analyzing a reference material of known constituent concentration or by analyzing a sample to which a known concentration or amount of constituent has been added (i.e., LCS, matrix spike, surrogate spike samples, and yield tracer). The accuracy estimate may apply only to a specific portion of the measurement system rather than to the entire measurement system. Bias can be caused by the sample matrix, sample preparation procedures, analytical method, measurement system, and improper sample handling practices. Accuracy is calculated as a percent recovery or as a percent bias. (See

Section 4.1 for further explanation of the accuracy equations.)

Analytical bias can be determined through the analysis of a standard reference material (e.g., QCCS, ICV, CCV) or a certified performance evaluation audit sample which has a known concentration. Bias resulting from sample matrix is determined through the matrix spike analysis. Sample-handling accuracy is estimated through the analysis of matrix spikes and of LCSs that undergo a digestion or extraction process performed on each sample matrix associated with a group of 20 or fewer samples.

12.4 COMPLETENESS

Completeness of data collected can be compared directly to the DQO (95%). An environmental project can produce 100% data completeness; however, the results may not be representative of the constituent concentrations actually present. For example, the analytical method might be biased, or the sampling frequency or locations may not provide a representative indication of the actual distribution of the constituent in the matrix sampled.

CHAPTER 13
INTERNAL SAMPLE CHAIN-OF-CUSTODY AND EVIDENTIARY PROCEDURES

13.1 INTRODUCTION

The LAS chain-of-custody procedures are applicable to all samples received by the LAS or its subcontractors, regardless of sample origin or disposition. All environmental samples received are considered physical evidence and must be handled in accordance with certain procedural safeguards. The LAS chain-of-custody program, through careful documentation, ensures traceability of the handling and possession of each sample from time of receipt through completion of analysis and data reporting. The program is in compliance with procedures established by the NEIC Contract Evidence Audit Team for evidentiary handling of samples in the CLP.

13.2 RESPONSIBILITIES

The primary responsibility for the maintenance of the chain-of-custody records belongs to the Sample Custodian; however, all LAS personnel are responsible for maintaining the integrity and traceability of samples that are assigned to them, in accordance with LAL-90-SOP-0009. Furthermore, the Operations Manager, Quality Assurance Manager, and each supervisor must ensure that all personnel are familiar with and follow the LAS chain-of-custody policy and procedures.

13.2.1 Sample Custodian

The Sample Custodian has the primary responsibility for receiving and accurately logging samples into LDMS as detailed in LAL-90-SOP-0002. Samples and standards containing radioactive constituents are received, logged in, and transferred according to procedures detailed in LAL-91-SOP-0085 and in LAL-91-SOP-0113. In addition to sample receipt, the Sample Custodian must fully document sample custody and communicate information to the appropriate parties. Other custody duties include:

- Radiation screening of all coolers and samples.
- Checking sample containers for breakage and leakage.
- Placing samples in appropriate and secure storage areas.
- Controlling access to samples in storage and ensuring that the chain-of-custody SOPs are followed when samples are removed from and returned to storage.
- Maintaining sample identification files, including documentation for any missing or disposed samples.
- Ensuring that conditions of storage facilities are properly monitored and maintained (e.g., refrigerator temperatures) located in the sample receiving area.
- Cleaning LAS shipping containers.
- Returning shipping containers to the client's field operations team(s).
- Notifying the Client Services Department

representatives of issues related to cooler and sample condition and any discrepancies upon receipt.

Sample Custodian alternates are also available as needed. The alternates are fully trained to perform the duties of the Sample Custodian and are proficient in all aspects of sample shipping, receiving, and tracking. The alternates also assist the Sample Custodian during peak activity periods.

13.2.2 Analysts

All analysts and technicians handling samples must follow the chain-of-custody protocols described in LAL-90-SOP-0009. These procedures include:

- Maintaining sample integrity, which may involve refrigeration, prevention of light exposure, or protection from dust, moisture, or other forms of contamination.
- Maintaining accurate, up-to-date logbooks and records (LAL-90-SOP-0006).
- Updating sample status in LDMS.
- Communicating pertinent information to applicable parties.
- Returning extracts, digests, and samples to the proper location upon completion of processing or analysis.

13.3 CHAIN-OF-CUSTODY ELEMENTS

13.3.1 Sample Labels

At LAS, field samples are typically received with labels affixed at the sampling site in order to prevent misidentification of samples. The information on the label generally include:

- Sampling location
- Client sample ID
- Date and time of sampling
- Constituents of interest (if space permits)
- Name of sample collector
- Chemical preservative(s) used
- Other relevant information

Figure 6 shows the internal chain-of-custody sample seal and label. Each sample received at LAS has affixed to it an internal label that includes a unique LAS sample ID, the contract number, the sample matrix, and the required methods of analysis.

13.3.2 Sample Seal

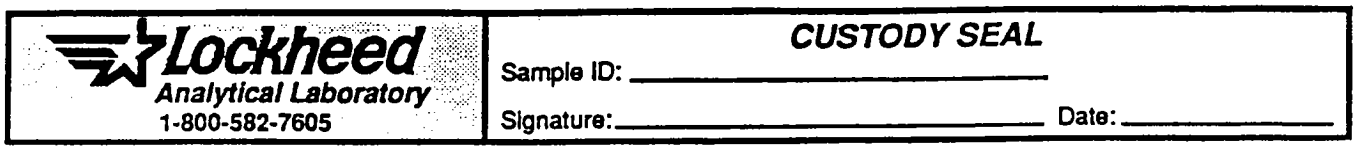
When samples are shipped to LAS by common carrier (e.g., air freight), the shipping container and individual sample bottles should be sealed to ensure the integrity of samples during transportation. The sample seal may contain the date of sample collection and the client's sample ID numbers as per project requirements.

13.3.3 Chain-of-Custody Form

In order to establish the documentation necessary to trace sample possession from the time of collection, a serially numbered chain-of-custody form should be completed and should accompany every sample. Figure 7 is the LAS chain-of-custody form to be used for environmental analyses. This form must contain the following types of information:

- Client name and address
- Sample identification

Figure 6. LAS Internal Chain-of-Custody Sample Seal and Label



- Signature of sample collector
- Date and time of sample collection
- Sample type (e.g., ground water, soil)
- Location description of sampling site
- Number of containers
- Chemical and physical constituents and methods for which analysis will be conducted
- Preservatives
- Signature(s) of person(s) involved in the chain of possession
- Inclusive dates and times of possession
- Internal temperature of shipping container (cooler chest) upon arrival at the laboratory
- Condition of samples upon arrival at the laboratory
- Cooler and sample survey for radioactivity

13.3.4 Log-in Chain-Of-Custody Report

The sample log-in data sheet generated by the Sample Custodian must accompany the sample(s) on delivery to the individual analytical laboratory and should clearly identify which sample containers have been designated for each required chemical and physical constituent. This form should include the following types of information:

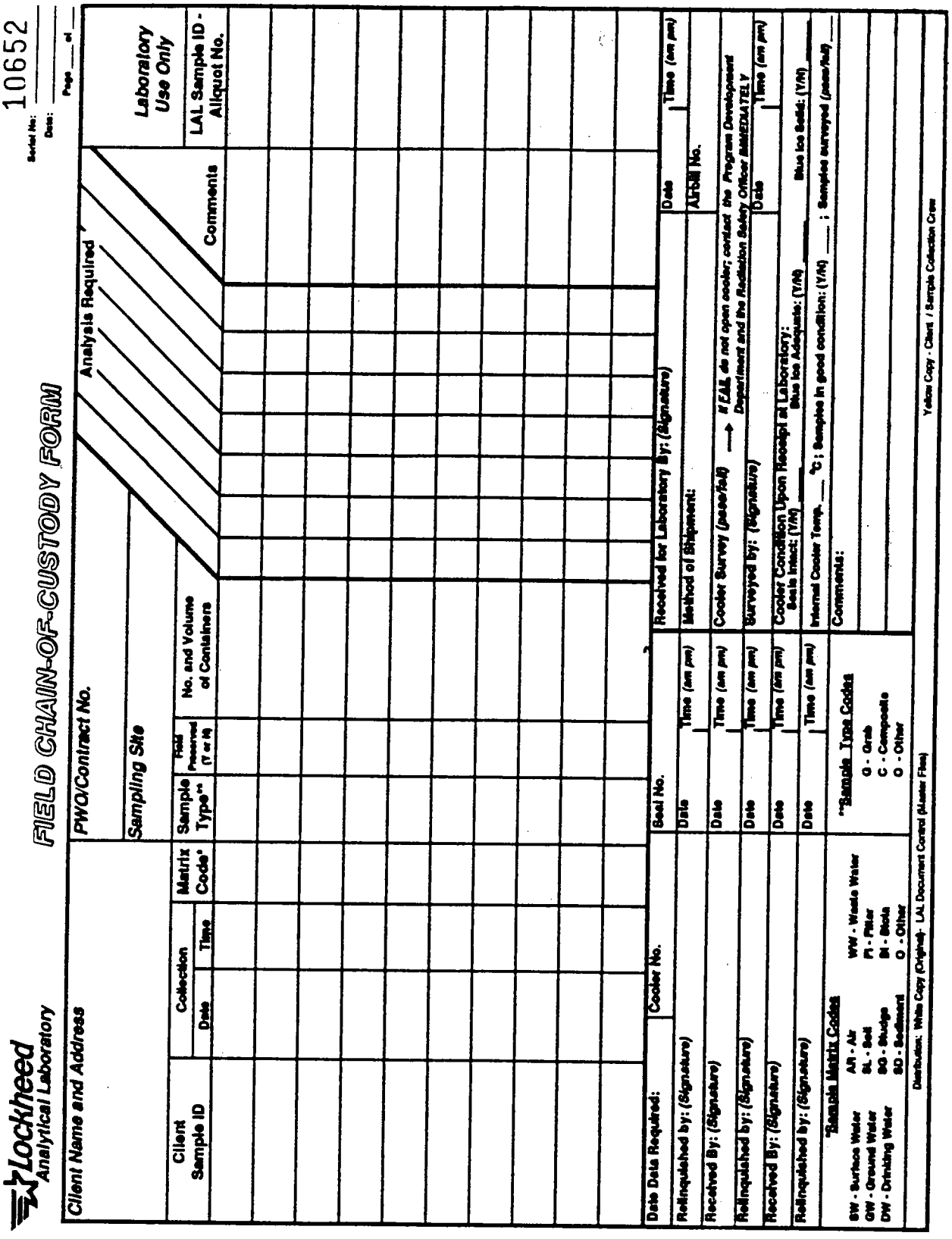
- Name of person receiving the sample (Sample Custodian)
- Client and LAS sample ID numbers
- Date of sample receipt
- Analyses to be performed
- Cooler and sample condition
- Date sample collected (to trace holding time requirements).

13.3.5 Sample Receiving Checklist

The Sample Receiving Checklist generated by the Sample Custodian must accompany the sample(s) on delivery to the individual analytical laboratory and should clearly identify cooler/sample condition upon receipt and any discrepancies identified during sample log in. This form should include the following types of information:

- Cooler condition (i.e., presence of custody seals, chain-of-custody form, and sufficient coolant material and radioactivity survey).
- Sample condition (i.e., bottle labeling, proper containers types, preservation, sample volume, headspace for VOA vials).

Figure 7. LAS Chain-of-Custody Form



- Miscellaneous items (e.g., identification of samples requiring short holding times and samples to be subcontracted).

13.3.6 Analysis Records

After the environmental sample has been received in the laboratory, the Sample Custodian or the appropriate laboratory personnel shall clearly document the processing steps that are applied to the sample. All sample preparation techniques (e.g., extraction or digestion) and analytical methods used must be documented in the bound logbooks or on bench sheets. Experimental conditions, such as the use of specific reagents (e.g., solvents or acids), temperatures, sample pH, and instrument settings, should be noted. The results of the analysis of all QC samples should be recorded if generated using manual instrumentation; these results should be identified in a manner that allows them to be easily associated with the corresponding batch of routine samples. Automated analyses generate electronic format data. The laboratory logbook also should include the date and name of the person who performed each sample processing and analytical step.

All pertinent laboratory information discussed above may be recorded on preprinted forms (e.g., bench sheets) or on computer-generated data reporting forms.

13.4 SAMPLE RECEIPT PROCEDURES

Sample receipt is to be completed only by the Sample Custodian or designated alternates in accordance with LAL-90-SOP-0002 and LAL-91-SOP-0085 for samples potentially containing radionuclides.

13.4.1 Shipping Container Check-In

When the courier has delivered shipping containers (e.g., coolers) to the loading dock, the Sample Custodian (1) checks the number of containers against the airbill or similar form, (2) signs appropriate courier forms, (3) notifies the courier, Operations Manager, Quality Assurance Manager, and the Client Services representatives of any damage to the container(s), and (4) verifies that the chain-of-custody seal on the shipping container is intact. The boxes are then monitored for radiation levels, and if the container is deemed safe, the shipment is then moved to the sample receiving room, where the Sample Custodian or designee (1) records the sender's (e.g., client's) name, date of shipment, and shipping container condition, (2) places the container under the fume hood and removes the chain-of-custody form from the shipping container, (3) inspects the interior of the shipping container, (4) checks the temperature of the shipping container interior, and (5) documents all applicable information on the chain-of-custody form and on the sample receiving checklist. If the sample containers inside the shipping containers are undamaged, the shipping container can be transferred from the fume hood to a work bench for sample check-in.

If samples are determined to be potentially radioactive, the packing is inspected to ensure that it is intact and that it is not leaking. Then sample(s) are moved to the radioactive material receiving laboratory for further handling as specified in LAL-91-SOP-0085. If the shipping container is damaged or suspected of leaking, the Radiation Safety Officer is immediately notified.

13.4.2 Sample Check-In

Once container check-in is completed and documented, the Sample Custodian checks each sample container (e.g., bottle, vial) for leakage or damage, and, if necessary, the container is wiped off and its cap tightened. Broken or damaged containers are documented and set

aside for later disposal. The sample label is then checked against the chain-of-custody form, and, if correct, is entered into the LDMS. An LAS sample ID number is then assigned to the container and entered into the data base, and a bar-coded sample label is affixed to the container. After the contents of the cooler go through this process, the samples are placed on an assigned shelf in a designated refrigerator. Client sample batches are cross-referenced with LAS batch identifiers for tracking purposes.

13.5 SAMPLE STORAGE, TRANSFER, AND DISPOSAL

All samples are stored in locked areas that meet standard storage and preservation criteria for the matrix and analyte. Several refrigerators are designated for storing samples for VOC analyses to prevent cross contamination from other environmental samples. Samples and standards are also maintained separately. Only the Sample Custodian and alternates and Operations Manager have keys to these secured areas. Only the Sample Custodian and alternates are permitted to remove the samples from the storage area; therefore, sample analysis and preparation personnel can obtain samples only with the properly prepared sample batching form and sample tracking form. Once the samples are checked out from the Sample Receiving Area, the LDMS will be sued to track and document the internal chain of custody of all client samples. The new location of each sample will be recorded in the LDMS by Sample Receiving laboratory technicians. This location will be specific and will be a refrigerator within LAS if required. The LDMS will keep a record of all location changes for each sample. This record will be available for retrieval at anytime. If sample containers are changed during processing, the sample preparation analyst prepares new LAS labels, affixes them to the top of the container, and documents this action in the laboratory logbook and on the sample preparation tracking form. Any movement of sample between analysis laboratories or to or from refrigerators will be recorded in the LDMS by the analyst involved. Analysts are instructed to refer to appropriate SOPs for method-specific sample handling and storage requirements. All samples remaining in analysis laboratories awaiting further work will be stored as required. Samples not requiring refrigeration will be stored in designated secure areas in each laboratory. When processing or analysis is complete, the analyst returns the unused portion of the sample and the respective sample tracking form to the Sample Custodian, who then changes the location of the samples in the LDMS data base.

At the discretion of the Sample Custodian, and when storage space is at a premium, samples for which all analyses and data reports have been completed may be transferred to an off-site location for storage and archival for later disposal or, if requested, they may be returned to the client. It is the policy of LAS to dispose of samples 60 days after the submittal of the report to the client unless otherwise specified in writing by the client or as required by regulations and licenses governing laboratory operations. All transfers will be documented (1) on the sample tracking form, which is stored in the Document Control files after the sample analysis and archiving and the data reporting activities have been completed and (2) through the LDMS.

- Miscellaneous items (e.g., identification of samples requiring short holding times and samples to be subcontracted).

13.3.6 Analysis Records

After the environmental sample has been received in the laboratory, the Sample Custodian or the appropriate laboratory personnel shall clearly document the processing steps that are applied to the sample. All sample preparation techniques (e.g., extraction or digestion) and analytical methods used must be documented in the bound logbooks or on bench sheets. Experimental conditions, such as the use of specific reagents (e.g., solvents or acids), temperatures, sample pH, and instrument settings, should be noted. The results of the analysis of all QC samples should be recorded if generated using manual instrumentation; these results should be identified in a manner that allows them to be easily associated with the corresponding batch of routine samples. Automated analyses generate electronic format data. The laboratory logbook also should include the date and name of the person who performed each sample processing and analytical step.

All pertinent laboratory information discussed above may be recorded on preprinted forms (e.g., bench sheets) or on computer-generated data reporting forms.

13.4 SAMPLE RECEIPT PROCEDURES

Sample receipt is to be completed only by the Sample Custodian or designated alternates in accordance with LAL-90-SOP-0002 and LAL-91-SOP-0085 for samples potentially containing radionuclides.

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CHAPTER 14
PREVENTIVE MAINTENANCE AND REPAIR

14.1 INTRODUCTION

Preventive maintenance and instrument and equipment repair responsibilities are coordinated through the LESAT Environmental Sciences and Technologies Division Instrumentation Maintenance and Repair Section. It is the responsibility of the engineers in this section to perform maintenance and repairs in coordination with the instrument operators and in accordance with the standard formats and procedures described in LAL-90-SOP-0188 and LAL-90-SOP-0015. Minor instrument and equipment maintenance is also performed by the analysts and other laboratory staff as required.

14.2 PREVENTIVE MAINTENANCE

Preventive maintenance is the scheduled routine action taken to help ensure the proper operation of instrumental systems. A proper preventative maintenance program consists of, but is not limited to (1) the periodic calibrating, tuning, and cleaning of instruments, (2) the periodic changing of oils and filters, and (3) the monitoring of known areas of wear or degradation to ensure the timely replacement of worn parts or components. The criteria used to determine the scope and frequency of preventative maintenance are (1) the instrument manufacturer's recommendations, (2) compiled maintenance data, (3) past experience of the instrument operators, and (4) the maintenance engineers.

In general, preventive maintenance is scheduled quarterly as the instrumental systems are available, even if there is no indication of a negative effect on data quality resulting from the system performance. Personal computers are serviced every 6 months. A preventative maintenance schedule for LAS instrumentation is prepared quarterly by the Instrumentation Maintenance and Repair Section and is distributed to all laboratory departments. The engineer assigned to perform the preventive maintenance on a particular instrument contacts the instrument operator at least one working day before the scheduled maintenance in order to confirm the schedule with the operator. Occasionally, project (e.g., client) priorities conflict with this schedule. It is the responsibility of the instrument operator or section supervisor to reschedule the servicing at an earlier or later date. This practice ensures minimizing instrument "down time" that could affect sample holding times and project deliverable deadlines. All preventive maintenance work is documented on a work order request form prepared by the engineer and signed by the operator or requestor. A carbon copy of the form is retained in the laboratory files and is also documented in the instrument operations logbook.

In addition to the routine maintenance performed quarterly, each instrument operator performs maintenance as needed. This work may include cleaning or replacing analytical columns, injection ports, or transfer lines.

14.3 REPAIRS

Repairs are defined as any unscheduled service or maintenance required on equipment and instrumentation. This work is performed expressly at the request of the operator or supervisor and can include repairs of nonfunctioning instrumentation or of functioning instrumentation not performing optimally.

Requests for repairs are first entered into the service logbook. The engineer assigned to each request then makes a preliminary evaluation of the work required and initiates a work order request form. The work may include such activities as diagnosis, parts procurement and installation, or inventory control. Upon completion of the repair, the work order request form is completed by the engineer and signed by the requestor. A carbon copy of the form is retained in the laboratory files; the work is also documented in the instrument operations logbook.

14.4 MAINTENANCE OF LABORATORY SUPPORT EQUIPMENT

Important aspects of maintaining sample integrity, ensuring precise and accurate sample measurements, and providing worker safety include the proper maintenance, calibration, and inspection of a wide array of support equipment used for sample storage and preparation. This support equipment includes analytical balances, micropipets, thermometers, balances, weights, refrigerators, freezers, ovens, reagent water systems, waste water discharge monitors, fume hoods, ventilation systems, and radiation survey detectors. Routine and periodic checks and maintenance of the support equipment are described in detail in LAL-90-SOP-0015, and a summary is provided below. Maintenance of major analytical equipment, such as GC-MS, AAS, and ICP-AES, is discussed in LAL-90-SOP-0188. Routine checks and maintenance of fire protection equipment, high efficiency particular air (HEPA) filters, hoists, perchloric acid scrubbers, safety equipment (i.e., emergency light, eye wash, safety shower and ground fault circuit), and waste water discharge monitors are also discussed in detail in LAL-90-SOP-0015.

14.4.1 Refrigerators and Freezers

Daily refrigerator and freezer temperatures are recorded in a temperature logbook. The temperature log includes date, time, temperature, corrective action (if required), and initials. Refrigerator temperatures should be at 4 °C ± 2 °C and freezer temperatures at -20 °C to -10 °C, measured with NIST-traceable thermometers. If the units deviate by more than the specified temperature tolerances, adjustments are made to bring them within specifications.

14.4.2 Ovens

The temperature of each oven is measured before use at the dial setting appropriate for the method of determination using NIST-traceable thermometers. The temperature readings are recorded on the Oven Temperature Log assigned to each oven.

14.4.3 Analytical Balances

The accuracy of all analytical balances is checked using "NBS Class S" or "ASTM Class 1" NIST-traceable weights. Balances used daily are checked daily before use. Balances used infrequently are checked before use. The balance accuracy checks are recorded in the logbook designated for each balance. Each analytical balance is certified annually by an independent vendor, and the certification is documented by a label affixed to the balance and on certification forms maintained by Document Control. Details of analytical balance check procedures are provided in LAL-90-SOP-0046.

14.4.4 NIST Class "S" Weight Calibration

On an annual basis, one set of Class "S" weights is sent to an external, qualified vendor for independent calibration. The other Class "S" weights or equivalent used to check the accuracy of balances at LAS are verified against the externally calibrated Class "S" weight set each year. Copies of calibration certificates and the verification data are maintained with the weights and in Document Control files.

14.4.5 Micropipets

Each micropipet is calibrated monthly at three frequently used volume settings in accordance with procedures described in LAL-91-SOP-0175 and in LAL-93-SOP-0205 for inorganic and radionuclide determinations, respectively. Each measurement must be within 1 percent for volumes $\geq$ 100 μL, and 2 percent for volumes $<$ 100 μL. If the QC limit is exceeded, the micropipet is adjusted according to the SOP specifications. The micropipets should be labeled appropriately to indicate the status of calibration. The micropipet calibration checks, calculations, and required adjustments are recorded in a bound logbook.

14.4.6 Reagent Water System

Each day, the resistivity of reagent water for each Nanopure reagent water system is measured and recorded on the form posted on the system. The resistivity for ASTM Type II reagent-grade water must be greater than 1.0 MΩ.cm at 25 °C. If the resistivity drops below 3.0 MΩ.cm, the system's filters are replaced. In general, although directly related to the frequency of use of the system and the feed water condition, prefilters are changed periodically by the LAS maintenance staff. The quality of the reagent water is further monitored continuously through the analyses of method (reagent) blanks for inorganic, organic, and radiochemical analyses.

14.4.7 Fume Hoods

Air flow velocities through hoods are checked and recorded every six months. The air handling system is adjusted, if necessary. If adjustments are made, the velocities must be rechecked. A detailed description of this activity is provided in LAL-91-SOP-0099.

14.4.8 Ventilation System

LAS Maintenance personnel check the positive and negative flow of the LAS ventilation system and adjust the flow as needed. Filters are inspected monthly and are cleaned or replaced, if necessary.

14.4.9 Radiation Survey Detectors

All radiation survey detectors are inspected daily, if used, to ensure proper operation, in accordance with the manufacturer's operation manual and LAL-91-SOP-0173. The portable radiation survey equipment shall be sent to an authorized vendor for annual calibration or when the instrument cannot meet QC limit for daily calibration check.

14.4.10 Thermometer Calibration

On an annual basis, an independent thermometer calibration check is performed using an NIST-traceable thermometer, as described in LAL-90-SOP-0015.

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CHAPTER 15
CORRECTIVE ACTIONS AND CONTINGENCY PLANNING

15.1 INTRODUCTION

If, on the basis of internal or external systems or performance audits, sample handling, routine monitoring of laboratory support equipment, or QC sample analysis results, analytical systems fail to meet the established criteria, an appropriate corrective action must be implemented. The Operations Manager, project manager, Quality Assurance Manager, supervisor, and analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected or if the corrective action will impact the project budget or schedule, the action may directly involve the LAS Director.

15.2 CORRECTIVE ACTIONS

Corrective actions are generally of two types, immediate actions and long-term actions.

An immediate action is designed to correct or repair nonconforming instruments and measurement systems. The need for such an action most frequently will be identified by the analyst as a result of calibration checks and other QC sample analyses.

A long-term action is designed to eliminate causes of nonconformance. The need for such actions is identified by quality assurance and QC systems and performance audits. The systematic nonconformances identified during the data generation process and the appropriate corrective measures taken are thoroughly documented on the NCAR. Examples of this type of action include:

- Training and qualification of staff in technical skills or in implementing the Quality Assurance Program.
- Rescheduling of analytical laboratory routine to ensure analysis within allowed holding times.
- Identifying vendors to supply standards of sufficient purity.
- Revising the quality assurance system or replacing personnel, as appropriate.
- SOP revisions.

For either type of corrective action, the sequential steps that compose a closed-loop corrective action system are as follows:

- Define the problem.
- Assign responsibility for investigating the problem.
- Investigate and determine the cause of the problem.
- Determine a corrective action to eliminate the problem.
- Assign and accept responsibility for implementing the corrective action.
- Establish effectiveness of the corrective action and implement the correction.
- Verify that the corrective action has eliminated the problem.

Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be documented properly. On-the-spot actions are used to correct minor problems, such as recalibration, retuning, or a minor repair (e.g., replacement of a minor part) of a malfunctioning instrument or the correction of poor analytical technique being used by an analyst. These occurrences are documented in the appropriate injection, run, or analysis logbooks. Routine instrument maintenance, malfunctions, and power failures are documented in the appropriate instrument maintenance logbooks. The nonconformances systematic in nature are documented and monitored through the NCAR forms. The closed-loop corrective action program is described in detail in LAL-92-SOP-0190. Corrective actions specific to methods are discussed in appropriate SOPs.

15.3 CONTINGENCY PLANNING

A comprehensive Quality Assurance Program must emphasize contingency planning and actions to prevent problems from occurring and to ensure timely, effective completion of a measurement effort. The LAS has contingency plans for the areas listed below. Contingency plans specific to the work areas are also important to the smooth functioning of the LAS and should be addressed and updated periodically by all LAS managers and supervisors.

- *Staffing* - A primary objective is to ensure that qualified staff are always available to perform the necessary analytical work, regardless of employee turnover, vacation, illness, or other absence. The resolution to this issue is to (1) anticipate critical staffing needs and recruit qualified staff to maintain work flow, (2) ensure timely hiring of candidates, and (3) continuously ensure cross-training of existing staff to provide back-up capabilities. Other Lockheed staff who have particular expertise in analyzing difficult samples can also be consulted for advice and problem resolution.

- ***In-house Service Experts*** - Our preventive maintenance program is designed to minimize malfunctions, permit simple adjustments, and ensure fewer and shorter breakdowns of critical analytical equipment. Procedures in place to maximize instrument up-time are described in Chapter 14.

- ***Redundant Instrumentation and Support Equipment*** - In most cases, duplicate instrumentation is available to ensure uninterrupted work flow. It is the responsibility of the section supervisors to ensure that analytical personnel are trained in identifying approved alternative methods of analysis, if necessary. For example, most constituents analyzed by using ICP-AES can also be determined by using furnace or flame AAS and ICP/MS, provided that the required detection limits are attainable. Redundant equipment is available for providing the laboratory reagent water and gases necessary for the analytical instruments. In addition, LAS has procedures in place for leasing major instruments, equipment, and computers within a short time frame, should the situation dictate.

- ***Instrument Service Contracts*** - LAS vendor service contracts ensure that vendors supply 24-hour emergency response. These responses include overnight parts delivery or service-engineer assistance to maintain operating capacity.

- ***Subcontractor Analytical Laboratory*** - To support the laboratory during peak periods or in the event of a critical instrument malfunction, LAS has arranged to use qualified analytical laboratories as subcontractors for short-term backup analytical support. Through an extensive process, LAS QA personnel evaluate, identify, and select qualified analytical laboratories before an analytical contract is awarded. In order to qualify, a subcontractor laboratory must pass this evaluation and, potentially, an on-site inspection.

- ***Uninterruptable Power Supply*** - The Exide Electronics Powerware System 50 Uninterruptable Power Supply (UPS) provides line conditioning and backup power to the LAS HP 9000 845 computer system/server. In case of power failure, the laboratory generator becomes the main source of power. The UPS still provides the conditioning and backup. If, during a power failure, the generator becomes ineffective, the UPS serves as the main power source, providing power for 30 to 60 minutes to the HP 9000 Computer. This contingency plan allows sufficient time for the main computer system to be shut down and data archival. All electronically generated data are stored on the main computer system and on the individual PC hard drives. In the event that the main laboratory computer system fails, the analytical data can be retrieved from the PC hard drives.

15.4 IDENTIFICATION AND CONTROL OF NONCONFORMING ITEMS AND MATERIALS

The LAS personnel must follow procedures to identify, segregate, evaluate, and document nonconforming items or materials to prevent inadvertent installation or use. The section supervisors are responsible for overseeing the identification, segregation, review, disposition, and documentation of nonconforming instruments, equipment, and materials at LAS. This activity is described in detail in LAL-93-SOP-0283.

15.4.1 Identification

Upon identification of nonconforming items (e.g., analytical instruments, support equipment, chemicals, reagents, solvents, etc.), appropriate section supervisors, tech leads, or designees are notified and a legible and easily recognizable identification is used to indicate that the item is not in use. Identification of nonconforming items are typically done by marking, tagging, or other methods that shall not adversely affect the end use of the item. The identification shall be legible and easily recognizable.

If identification of each nonconforming item is not practical, the container, package, or segregated storage area, as appropriate, shall be identified.

The out-of-control condition must be documented in the related logbook, worksheet, maintenance record, Nonconformance and Corrective Action Record (NCAR), or in an internal memo.

15.4.2 Segregation

When practical, nonconforming items shall be segregated from conforming items by placing them in a clearly identified area until proper corrective action is taken or until disposition.

For example, expired standards or chemicals shall be segregated prior to further verification or disposal. The small nonconforming analytical or support equipment, such as balances, thermometers, and pipettors, can be

physically segregated from the routine equipment to avoid possible misuse.

15.4.3 Review

Nonconforming items should be reviewed by appropriate section supervisors, technical leads, or designees to determine whether they can be used as they are or whether they shall be repaired or reclassified.

15.4.4 Disposition

The justification for disposition, such as use-as-is, rework, reject, or repair of nonconforming items must be approved by the appropriate section supervisors or technical leads and documented in the related logbook, worksheet, maintenance record, NCAR, or in an internal memorandum.

Repaired, replacement, or reworked items must be tested to verify that required operational conditions and all the QC specifications can be met before use.

15.5 HANDLING OF CLIENT INQUIRIES

Client inquiries are generally received through the project manager or a member of the Program Development group. Typically, the project manager communicates with the client to ascertain the details of the inquiries, including technical data problems, deliverable issues, turn-around-time problems, etc. Technical and deliverable issues are coordinated by the Project Manager and usually involve input from operations, QAD, and managerial personnel. A formal response to the client is coordinated by the Project Manager, but may on occasion be delivered by a member of the Program Development group.

CHAPTER 16
CONTROL OF PURCHASED ITEMS AND SERVICES

16.1 PROCUREMENT

The procurement of LAS instruments, equipment, chemicals, standards, and services is controlled to ensure compliance with specified requirements. LAS operations, facility, quality assurance and purchasing personnel ensure the adequacy and quality of all contractor-purchased articles, materials, and services. LAS personnel plan and implement procurement quality activities to ensure timely and adequate integration with all other elements of the organization having responsibility for control and performance of subcontractors and suppliers.

At LAS, the Source Selection Process is established to review all competitive procurements when the competition involves an evaluation and comparison of cost or price and other technical factors. The source selection procedures are designed to (1) maximize competition, (2) minimize the complexity of the solicitation, evaluation, and the selection decision, (3) ensure impartial and comprehensive evaluation of all offers, and (4) ensure selection of the source whose offer has the highest degree of quality and whose performance is expected to best meet the solicitation requirements. The findings that result from this process provide permanent procurement file documentation.

LAS maintains a documented receiving inspection system which ensures:

- Procured articles, materials, or services indicate evidence of inspections and tests performed by the supplier in accordance with purchase requirements and are accompanied by required certifications (if necessary).

- Chemical analyses and physical tests are conducted according to the approved analytical protocols.

When an article, material, or service procured by LAS does not conform to applicable specifications or other requirements, it is identified as nonconforming, segregated to the extent practicable, and held for review action. LAS has established a documented, systematic technique for the identification, documentation, and control of nonconformances.

16.2 SELECTION AND QUALIFICATION OF SUBCONTRACTOR ANALYTICAL LABORATORIES

Selecting the analytical laboratory that will provide the best complement of subcontract services for an environmental project is of primary importance. It requires an approach to ensure that all data generated by LAS subcontractors are of known, acceptable, and documented quality and are in compliance with the LAS QA Program- and client-specific requirements.

The LAS has established a policy to perform an in-depth evaluation of a laboratory's capabilities to provide analytical services as a subcontractor before an analytical contract is awarded. The objective is to select laboratories that are capable, technically qualified, credible, and competitive in terms of analytical cost. A detailed description of the specific requirements, procurement, planning, on-site systems

evaluations, and control of supplier nonconformances is provided in LAL-93-SOP-0232.

16.3 MATERIALS PROCUREMENT AND CONTROL

The quality of all materials used in the handling, preparation, analysis, and storage of samples at LAS facilities must be of known and acceptable quality so that the effect of the materials on analytical results can be defined. Reagents, solvents, reagent water, gases, and sample containers, as well as laboratory glassware, vessels, and implements purchased by LAS or prepared internally (e.g., compressed air) shall meet all the requirements that are stated in the particular analytical methods or that are otherwise specified by the client.

Chemical reagents, solvents, and gases are available from a variety of sources and in a variety of purity grades, ranging from technical to ultrapure grades. The constituents measured and the sensitivity and specificity levels of the analysis system are key elements in determining the required purity of these materials. In general, if the analytical method does not specify the grade required, "analytical grade" or higher purity will be used. Procedures for acceptance of laboratory chemicals is addressed in LAL-93-SOP-0284.

16.3.1 Responsibility

The section supervisor is responsible for overseeing and ensuring that (1) suitable grades of materials are specified in requisitions, (2) the materials procured meet the applicable requirements, (3) the appropriate certification or other documentation regarding the materials has been provided and is maintained in the Document Control files, (4) the materials are stored safely and properly, and (5) the materials are removed from use when the shelf-life (or other criteria) is expired or otherwise outdated.

16.3.2 General Materials Requirements

16.3.2.1 Inorganic Analyses. In general, analytical reagent grade reagents and solvents are adequate for inorganic analyses; however, trace metal analyses by atomic absorption and emission spectroscopy shall be spectro-quality. Fuel and oxidant gases may be commercial grade. Compressed air can be commercially supplied (dry grade) or supplied by LAS air compressors, provided that proper pressure and filtration of oil, water, and trace metals are maintained.

16.3.2.2 Organic Constituent Analyses. In general, pesticide grade (i.e., nanograde) is the minimum grade acceptable for materials used in organic analyses. Reference grade standards shall be used as necessary. Some gas chromatography systems require that solvents and standards (and environmental samples, as well) be free of certain compound classes. For example, photoionization detectors require that reagents and solvents be free of sulfur and phosphorus compounds because of their interfering properties.

For sample cleanup procedures, the adsorbent materials (florisil, carbon, silica gel, and alumina) are most commonly used. These materials, as well as all analytical reagents, solvents, and other chemicals must be checked to determine suitability for the analyses.

16.3.2.3 Laboratory Reagent Water. In general, deionized ASTM Type II grade water (or better) is used for dilution of samples in preparation of reagent and standard solutions, and for final rinsing of glassware. The specifications for ASTM Type II grade water is for resistivity to be greater than 1.0 M Ω.cm or conductivity less than or equal to 1 μS/cm at

25 °C. Organic-free water is required for VOC analyses; however, when determining trace organics by gas chromatography following solvent extraction, "specialty" water such as HPLC grade water must be used. The quality of the reagent water must be monitored through daily resistivity checks and method blank analyses.

16.3.2.4 Laboratory Containers, Vessels, and Implements. Material composition and volumetric tolerances of containers and other vessels used in the sample storage, preparation, and analysis processes can affect the quality of the analytical data.

Soft glass containers are not recommended for general use, especially for the storage of reagents. Chemically resistant borosilicate glass, such as Pyrex® or Kimax® is generally used unless otherwise specified in the analytical protocols. Plastic vessels, containers, and other apparatuses made of Teflon®, polystyrene, polyethylene, and polypropylene are also desirable when specified. Guidelines for selecting the material composition of laboratory containers are provided in Chapter 4. In addition, implements used in the handling of samples, such as spatulas and spoons, and supplies such as aluminum foil and Parafilm® must meet specifications for the matrices and constituents of interest.

In general, volumetric glassware will be of sufficient accuracy for the analytical reagent volumes measurement. This glassware includes volumetric flasks, volumetric pipets (and static and adjustable micropipettors), and calibrated burets. Less accurate types of glassware, such as graduated cylinders and beakers, are also used for specific applications.

16.3.2.5 Calibration Services. Certain items at LAS are calibrated by independent vendors. Inaccurate calibrations performed by independent vendors could lead to malfunctioning equipment, uncertain data quality, and safety problems. The qualifications of the vendors that provide calibration services will be verified by any or all of the following steps:

- checking references for the vendor;
- checking the vendor's certifications;
- Checking the vendor's quality assurance documentation;
- verifying the vendor's qualifications with the equipment manufacturer;
- checking the accuracy of the calibration with certified standards; and
- reviewing previous experience with the vendor.

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CHAPTER 17
QUALITY ASSURANCE REPORTS TO PROJECT MANAGEMENT

17.1 INTRODUCTION

An effective quality assurance program should include formal and frequent reports to inform management and technical staff of progress in the on-going implementation of the quality assurance plan. At a minimum, the following LAS parties should receive regular updates on project quality assurance status: (1) director, (2) operations manager, (3) program development staff, (4) client services (project) staff, (5) section supervisors, and (6) analysts and other technical staff.

17.2 INTERNAL QUALITY ASSURANCE REPORTS

LAS Quality Assurance personnel develop reports routinely for the LAS staff on the following topics:

- General LAS Quality Assurance Program status (weekly and monthly).
- Long-term control charts (monthly).
- Results of external PE and laboratory intercomparison studies (semiannually).
- Results of external and in-house technical system audits (as reports are received from clients or generated).
- Status of completed and outstanding Nonconformance and Corrective Action Records (weekly).
- Assessment of the blind and nonblind data audit (PE) sample data (as data are produced).
- Laboratory accreditation, licensing, and permitting updates (weekly).
- All significant quality-related problems identified during independent data validation and the corrective action procedures that are recommended (as performed).
- Information regarding QA training, regulatory changes, QA-related issues, and recommendations (as performed).
- Local, state, and federal regulatory information (as updates are received).
- Schedules for external on-site audits (as updated).

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APPENDIX A

ANALYTICAL DATA QUALITY OBJECTIVES FOR PRECISION ACCURACY, AND DETECTABILITY FOR INORGANIC, ORGANIC, AND RADIONUCLIDE CONSTITUENTS

Table A-1. Estimated Reporting Detection Limits and Precision and Accuracy Objectives for Inorganic Constituents (4 Pages)

Constituent	Analytical Method	Estimated Reporting Detection Limit		Precision (RPD)[b]	Accuracy (% Rec)[c]
		Aqueous (mg/L)	Solids[a] (mg/kg)		
Alkalinity - High	310.1	30	300	20	100 ± 25
Alkalinity - Low	310.1	30	300	20	100 ± 25
Aluminum	200.7/6010/CLP	0.20	40	20	100 ± 25
Aluminum	200.8/6020	0.05	10	20	100 ± 25
Ammonia-Nitrogen	350.1	0.05	0.50	20	100 ± 25
Antimony	200.7/6010/CLP	0.06	12	20	100 ± 25
Antimony	204.2/7041/CLP	0.06	0.50	20	100 ± 25
Antimony	200.8/6020	0.005	1.0	20	100 ± 25
Arsenic	200.7/6010/CLP	0.20	40	20	100 ± 25
Arsenic	200.9/7060/CLP	0.01	2.0	20	100 ± 25
Arsenic	200.8/6020	0.01	2.0	20	100 ± 25
Arsenic	TCLP	0.50	100	20	100 ± 25
Barium	200.7/6010/CLP	0.20	40	20	100 ± 25
Barium	200.8/6020	0.05	10	20	100 ± 25
Barium	TCLP	10	2000	20	100 ± 25
Beryllium	200.7/6010/CLP	0.005	1.0	20	100 ± 25
Beryllium	200.8/6020	0.005	1.0	20	100 ± 25
Boron	200.7/6010	0.20	40	20	100 ± 25
Boron	200.8/6020	0.10	20	20	100 ± 25
Bromide	300.0	0.10	1.0	15	100 ± 25
Cadmium	200.7/6010/CLP	0.005	1.0	20	100 ± 25
Cadmium	213.2/7131/CLP	0.005	1.0	20	100 ± 25
Cadmium	200.8/6020	0.005	1.0	20	100 ± 25
Cadmium	TCLP	0.10	20	20	100 ± 25
Cerium	200.8/6020	0.005	1.0	20	100 ± 25
Cesium	200.8/6020	0.005	1.0	20	100 ± 25
Cesium	3500-CS	1.0	200	20	100 ± 25
Chemical Oxygen Demand (COD)	410.4	20	N/A	20	100 ± 25
Chloride	325.2	1.0	N/A	20	100 ± 25
Chloride	300.0	0.020	0.20	15	100 ± 25
Chromium (total)	200.7/6010/CLP	0.50	100	20	100 ± 25
Chromium (total)	200.8/6020	0.010	2.0	20	100 ± 25

Table A-1. Estimated Reporting Detection Limits and Precision and Accuracy Objectives for Inorganic Constituents (4 Pages)

Constituent	Analytical Method	Estimated Reporting Detection Limit		Precision (RPD)[b]	Accuracy (% Rec)[c]
		Aqueous (mg/L)	Solids[a] (mg/kg)		
Chromium (total)	218.2/7191/CLP	0.010	2.0	20	100 ± 25
Chromium (total)	TCLP	0.50	100	20	100 ± 25
Chromium (IV)	7196	0.020	0.20	15	100 ± 15
Cobalt	200.7/6010/CLP	0.050	10	20	100 ± 25
Cobalt	200.8/6020	0.005	1.0	20	100 ± 25
Copper	200.7/6010/CLP	0.025	5.0	20	100 ± 25
Copper	200.8/6020	0.005	1.0	20	100 ± 25
Cyanide	335.2/9010/CLP	0.020	2.0	20	100 ± 25
Fluoride	340.2	0.050	0.50	20	100 ± 25
Hydrazine	LAL-92-SOP-0189	0.0010	0.0010	20	100 ± 25
Iron	200.7/6010/CLP	0.10	20	20	100 ± 25
Iron	200.8/6020	0.050	10	20	100 ± 25
Lead	200.7/6010/CLP	0.10	20	20	100 ± 25
Lead	200.8/6020	0.003	0.60	20	100 ± 25
Lead	239.2/7421/CLP	0.003	0.60	20	100 ± 25
Lead	TCLP	0.50	100	20	100 ± 25
Lithium	200.7/6010	0.10	20	20	100 ± 25
Magnesium	200.7/6010/CLP	2.0	400	20	100 ± 25
Magnesium	200.8/6020	0.50	100	20	100 ± 25
Manganese	200.7/6010/CLP	0.015	3.0	20	100 ± 25
Manganese	200.8/6020	0.015	3.0	20	100 ± 25
Mercury	245.2/7470/7471	0.0002	0.15	20	100 ± 25
Mercury	TCLP	0.020	10	20	100 ± 25
Molybdenum	200.7/6010	0.20	40	20	100 ± 25
Molybdenum	200.8/6020	0.005	1.0	20	100 ± 25
Monomethylhydrazine	LAL-92-SOP-0189	0.0010	0.0010	20	100 ± 25
Nickel	200.7/6010/CLP	0.040	8.0	20	100 ± 25
Nickel	200.8/6020	0.005	1.0	20	100 ± 25
Nitrate as Nitrogen	300.0	0.020	0.20	15	100 ± 25
Nitrate/Nitrite as Nitrogen	353.2	0.050	0.50	20	100 ± 25

Table A-1. Estimated Reporting Detection Limits and Precision and Accuracy Objectives for Inorganic Constituents (4 Pages)

Constituent	Analytical Method	Estimated Reporting Detection Limit		Precision (RPD)[b]	Accuracy (% Rec)[c]
		Aqueous (mg/L)	Solids[a] (mg/kg)		
Nitrite as Nitrogen	300.0	0.010	0.10	15	100 ± 25
Ortho-Phosphate as Phosphorus	365.2	0.030	0.30	20	100 ± 25
Osmium	200.7/6010	0.10	40	20	100 ± 25
Phosphorus	200.7/6010	0.050	10	20	100 ± 25
Potassium	200.7/6010/CLP	2.0	400	20	100 ± 25
Potassium	200.8/6020	0.50	100	20	100 ± 25
Selenium	200.7/6010/CLP	0.30	60	20	100 ± 25
Selenium	200.8/6020	0.005	1.0	20	100 ± 25
Selenium	270.2/7740/CLP	0.005	1.0	20	100 ± 25
Selenium	TCLP	0.10	20	20	100 ± 25
Silica	370.1	1.0	N/A	20	100 ± 25
Silicon	200.7/6010	0.10	20	20	100 ± 25
Silicon	200.8/6020	0.10	20	20	100 ± 25
Silver	200.7/6010/CLP	0.10	2.0	20	100 ± 25
Silver	200.8/6020	0.005	1.0	20	100 ± 25
Silver	200.9	0.010	2.0	20	100 ± 25
Silver	TCLP	0.50	100	20	100 ± 25
Sodium	200.7/6010/CLP	2.0	400	20	100 ± 25
Sodium	200.8/6020	0.50	100	20	100 ± 25
Sulfate	300.0	0.10	1.0	15	100 ± 25
Sulfate	375.4/9038	5.0	50	20	100 ± 25
Sulfide	9030	3.0	N/A	20	100 ± 25
Strontium	200.7/6010	0.10	20	20	100 ± 25
Strontium	200.8/6020	0.005	1.0	20	100 ± 25
Thallium	200.7/6010/CLP	0.50	100	20	100 ± 25
Thallium	200.8/6020	0.005	1.0	20	100 ± 25
Thallium	279.2/7841/CLP	0.010	2.0	20	100 ± 25
Tin	200.7/6010	0.20	40	20	100 ± 25
Tin	200.8/6020	0.005	1.0	20	100 ± 25
Titanium	200.7/6010	0.10	20	20	100 ± 25
Titanium	200.8/6020	0.005	1.0	20	100 ± 25

Table A-1. Estimated Reporting Detection Limits and Precision and Accuracy Objectives for Inorganic Constituents (4 Pages)

Constituent	Analytical Method	Estimated Reporting Detection Limit		Precision (RPD)[b]	Accuracy (% Rec)[c]
		Aqueous (mg/L)	Solids[a] (mg/kg)		
Total Dissolved Solids	160.1	40	N/A	10	100 ± 20[d]
Total Kjedhal Nitrogen	351.2	0.20	N/A	20	100 ± 25
Total Organic Carbon	351.2	0.20	N/A	25	N/A
Total Organic Halides	9020	0.040	0.40	20	100 ± 25
Total Phenolics	420.1	0.15	1.5	20	100 ± 25
Total Phosphorus	365.2	0.030	N/A	20	100 ± 25
Total Residual Chlorine	330.5	0.10	N/A	20	100 ± 25
Total Suspended Solids	160.2	12	N/A	10	100 ± 20[d]
Unsymetrical Dimethylhydrazine	LAL-92-SOP-0189	0.0010	0.0010	15	100 ± 25
Uranium	200.8/6020	0.001	0.20	20	100 ± 25
Vanadium	200.7/6010/CLP	0.05	10	20	100 ± 25
Vanadium	200.8/6020	0.005	1.0	20	100 ± 25
Zinc	200.7/6010/CLP	0.020	4.0	20	100 ± 25

[a] Typically based on 1:10 soil to water ratio
[b] RPD-Relative Percent Difference on the basis of sample and duplicate analyses
[c] Percent recovery of matrix spike sample
[d] Accuracy estimate on the basis of LCS recovery

Table A-2. Reporting Detection Limits for Volatile Organic Analyses by GC/MS (Capillary Column) Using Methods 624/8240A/8260 (2 Pages)

Constituent	Reporting Detection Limit	
	Aqueous (μg/L)	Solid (μg/kg)
Chloromethane	5	5
Vinyl Chloride	5	5
Bromomethane	5	5
Chloroethane	5	5
Trichlorofluoromethane	5	5
Acetone	10	10
2-Chloroethyl vinylether	20	20
1,1-Dichloroethene	5	5
Methylene Chloride	5	5
Carbon Disulfide	5	5
Vinyl Acetate	10	10
1,1-Dichloroethane	5	5
2-Butanone	10	10
trans-1,2-Dichloroethene	5	5
cis-1,2-Dichloroethene	5	5
Chloroform	5	5
1,1,1-Trichloroethane	5	5
Carbon Tetrachloride	5	5
1,2-Dichloroethane	5	5
Benzene	5	5
Trichloroethene (TCE)	5	5
1,2-Dichloropropane	5	5
Bromodichloromethane	5	5
4-Methyl-2-pentanone	10	10
2-Hexanone	10	10
cis-1,3-Dichloropropene	5	5
trans-1,3-Dichloropropene	5	5
1,1,2-Trichloroethane	5	5
Toluene	5	5
Dibromochloromethane	5	5
Tetrachloroethene (PCE)	5	5
Chlorobenzene	5	5
Ethylbenzene	5	5
m,p-Xylene	5	5
o-Xylene	5	5
Styrene	5	5

Table A-2. Reporting Detection Limits for Volatile Organic Analyses by GC/MS (Capillary Column) Using Methods 624/8240A/8260 (2 Pages)

Constituent	Reporting Detection Limit	
	Aqueous (μg/L)	Solid (μg/kg)
Bromoform	5	5
1,1,2,2-Tetrachloroethane	5	5
1,3-Dichlorobenzene	5	5
1,4-Dichlorobenzene	5	5
1,2-Dichlorobenzene	5	5

Table A-3. QC Acceptance Criteria for Matrix Spike and LCS Recoveries for Volatile Organic Analyses by GC/MS using Methods 624\8240A\8260

Matrix Spike Compound	QC Limits (%)			
	Aqueous Sample		Solid Sample	
	% Recovery[1]	RPD[2]	% Recovery[2]	RPD[2]
1,1-Dichloroethene	64-124	14	59-172	22
Benzene	67-127	11	66-142	21
Trichloroethene (TCE)	60-120	14	62-137	24
Toluene	72-132	13	59-139	21
Clorobenzene	68-128	13	60-133	21

[1] - Criteria adopted from Table 7 of SW-846 Method 8260, Revision 0, July 1992.
[2] - Criteria adopted from CLP SOW 3/90 OLM01.0-OLM01.8.

Table A-4. Reporting Detection Limits for Pesticides/PCBs Analyses by GC/ECD Using Methods 608/8080

Constituent	Reporting Detection Limit	
	Aqueous (µg/L)	Solid (µg/kg)
A-BHC	0.05	1.7
B-BHC	0.05	1.7
G-BHC (Lindane)	0.05	1.7
D-BHC	0.05	1.7
Heptachlor	0.05	1.7
Aldrin	0.05	1.7
Heptachlor epoxide	0.05	1.7
G-Chlordane	0.05	1.7
Endosulfan I	0.05	1.7
A-Chlordane	0.05	1.7
4,4'-DDE	0.1	3.3
4,4'-DDT	0.1	3.3
Dieldrin	0.1	3.3
Endrin	0.1	3.3
Endosulfan II	0.1	3.3
4,4'-DDD	0.1	3.3
Endrin Aldehyde	0.1	3.3
Endrin Ketone	0.1	3.3
Endosulfan Sulfate	0.1	3.3
Methoxychlor	0.5	17
Toxaphene	5	170
PCB-1016	1	13
PCB-1221	2	13
PCB-1232	1	13
PCB-1242	1	13
PCB-1248	1	13
PCB-1254	1	13
PCB-1260	1	13
(Technical) Chlordane	1	40

Table A-5. QC Acceptance Criteria for Matrix Spike and LCS Recoveries for Pesticides/PCBs Analyses by GC/ECD Using Methods 608/8080

Matrix Spike Compound	QC Limits (%)			
	Aqueous Sample		Solid Sample	
	% Recovery[1]	RPD[2]	% Recovery[2]	RPD[2]
G-BHC (Lindane)	32-127	15	46-127	50
Heptachlor	34-111	20	35-130	31
Aldrin	42-122	22	34-132	43
Dieldrin	36-146	18	31-134	38
Endrin	30-147	21	42-139	45
4,4'-DDT	25-160	27	23-134	50

[1] Criteria specified in Table 3 of EPA SW-846 for Method 8080, Revision 0, September 1986 which is adopted from Method 608 in 40 CFR Part 136.
[2] Criteria adopted from CLP SOW 3/90, OL01.0-OLM01.8.

Table A-6. QC Acceptance Criteria for Matrix Spike and LCS Recoveries for PCBs Analyses by GC/ECD Using Methods 608/8080

Matrix Spike Compound	QC Limits (%)			
	Aqueous Sample		Solid Sample	
	% Recovery[1]	RPD[2]	% Recovery[1]	RPD[2]
PCB-1260	8-127	30	8-127	50

[1] Criteria specified in Table 3 of EPA SW-846 for Method 8080, Revision 0, September 1986 which is adopted from Method 608 in 40 CFR Part 136.
[2] LAS' best estimates; no criteria specified by Method 608/8080 or CLP SOW.

Table A-7. Reporting Detection Limits for SemivolatileOrganic Analyses by GC/MS Using Methods 625/8270A (2 Pages)

Constituent	Reporting Detection Limit	
	Aqueous (µg/L)	Solids (µg/kg)
Phenol	10	660
bis(2-Chloroethyl)ether	10	660
2-Chlorophenol	10	660
1,3-Dichlorobenzene	10	660
1,4-Dichlorobenzene	10	660
Benzyl alcohol	20	1300
1,2-Dichlorobenzene	10	660
2-Methylphenol	10	660
bis(2-Chloroisopropyl)ether	10	660
4-Methylphenol	10	660
N-Nitroso-di-n-propylamine	10	660
Hexachloroethane	10	660
Nitrobenzene	10	660
Isophorone	10	660
2-Nitrophenol	10	660
2,4-Dimethylphenol	10	660
Benzoic acid	50	3300
bis(2-Chlorethoxy)methane	10	660
2,4-Dichlorophenol	10	660
1,2,4-Trichlorobenzene	10	660
Naphthalene	10	660
4-Chloroaniline	20	1300
Hexachlorobutadiene	10	660
4-Chloro-3-methylphenol	20	1300
2-Methylnaphthalene	10	660
Hexachlorocyclopentadiene	10	660
2,4,6-Trichlorophenol	10	660
2,4,5-Trichlorophenol	10	660
2-Chloronaphthalene	10	660
2-Nitroaniline	50	3300
Dimethylphthalate	10	660
Acenaphthylene	10	660
2,6-Dinitrotoluene	10	660
3-Nitroaniline	50	3300
Acenaphthene	10	660
2,4-Dinitrophenol	50	3300

Table A-7. Reporting Detection Limits for SemivolatileOrganic Analyses by GC/MS Using Methods 625/8270A (2 Pages)

Constituent	Reporting Detection Limit	
	Aqueous (μg/L)	Solids (μg/kg)
4-Nitrophenol	50	3300
Dibenzofuran	10	660
2,4-Dinitrotoluene	10	660
Diethylphthalate	10	660
4-Chlorophenyl-phenylether	10	660
Fluorene	10	660
4-Nitroaniline	20	3300
4,6-Dinitro-2-methylphenol	50	3300
N-Nitrosodiphenylamine	10	660
4-Bromophenyl-phenylether	10	660
Hexachlorobenzene	10	660
Pentachlorophenol	50	3300
Phenanthrene	10	660
Anthracene	10	660
Di-n-butylphthalate	10	660
Fluoranthene	10	660
Pyrene	10	660
Butylbenzylphthalate	10	660
3,3'-Dichlorobenzidine	20	1300
Benzo(a)anthracene	10	660
Chrysene	10	660
bis(2-Ethylhexyl)phthalate	10	660
Di-n-octylphthalate	10	660
Benzo(b)fluoranthene	10	660
Benzo(k)fluoranthene	10	660
Benzo(a)pyrene	10	660
Indeno(1,2,3-cd)pyrene	10	660
Dibenz(a,h)anthracene	10	660
Benzo(g,h,i)perylene	10	660

Table A-8. QC Acceptance Criteria for Matrix Spike and LCS Recoveries for Semivolatile Organic Analyses by GC/MS Using Methods 625/8270A

Matrix Spike Compound	QC Limits (%)			
	Aqueous Sample		Solid Sample	
	% Recovery[1]	RPD[2]	% Recovery[2]	RPD[2]
Phenol	5-112	42	26-90	35
2-Clorophenol	23-134	40	25-102	50
1,4-Dichlorobenzene	20-124	28	28-104	27
N-Nitroso-di-n-propylamine	D-230	38	41-126	38
1,2,4-Trichlorobenzene	44-142	28	38-107	23
4-Chloro-3-methylphenol	22-147	42	26-103	33
Acenaphthene	47-145	31	31-137	19
4-Nitrophenol	D-132	50	11-114	50
2,4-Dinitroltoluene	39-139	38	28-89	47
Pentachlorophenol	14-176	50	17-109	47
Pyrene	52-115	31	35-142	36

[1] Criteria specified in Table 6 of EPA SW-846 for Method 8270A, Revision 1, July 1992 which is adopted from Method 625 in 40 CFR Part 136.

[2] Criteria adopted from CLP SOW 3/90, OLM01.0-OLM01.8.

Table A-9. Reporting Detection Limits for Total Petroleum Hydrocarbons Extractables Analyses by GC/FID Using Method 8015-Modified

Constituent	Reporting Detection Limit	
	Aqueous (mg/L)	Solid (mg/kg)
Diesel	1.0	30
Gasoline	1.0	30

Table A-10. QC Acceptance Criteria for Matrix Spike and LCS Recoveries for Total Petroleum Hydrocarbons Analyses by GC/FID Using Method 8015-Modified

Matrix Spike Compound	QC Limits (%)			
	Aqueous Sample		Solid Sample	
	% Recovery[1]	RPD[2]	% Recovery[1]	RPD[2]
Gasoline	25-145	20	30-130	30
Diesel	25-145[3]	20[3]	30-130[3]	30[3]

[1] LAS-established criteria
[2] LAS' best estimates
[3] Criteria adopted from gasoline analysis

Table A-11. Minimum Detectable Activities for Radionuclide Determinations

Constituent	Minimum Detectable Activity	
	Aqueous (pCi/L)[1]	Solids (pCi/g)
Tritium (H-3)	300	300
Gross α/β[1]	2/4	5/10
Gamma Spec - Nuclide specific (see below)		
Strontium, Isotopic (Sr-89 & Sr-90)	1	1
Strontium, Total	1	1
Uranium, Isotopic	0.5	0.5
Uranium, Total (μg/L or μg/g)	0.1	0.1
Radium-226	0.5	0.5
Radium-228	2.0	N/A
Radium, Total Alpha	2.0	N/A
Radon-222	50	N/A
Technetium-99	1.0	1.0
Thorium, Isotopic	0.5	0.5
Plutonium, Isotopic	0.1	0.1
Americium, Isotopic	0.1	0.1
Cerium, Isotopic	0.1	0.1
Polonium-210	1.0	1
Lead-210	1.0	2
Carbon-14	10	2
Nickel-63	2	2
Iron-55	5	5
Plutonium-241	5	5

[1] For clean water samples (i.e., total solids < 30 mg/250 mL) and if the counting statistics allow.

NOTE: As required, lower MDAs/RDLs can be achieved for all radionuclides.

Table A-12. QC Acceptance Criteria for LCS Recoveries and Duplicates for Radionuclide Determinations

Constituent	LCS Recovery (%)		Relative Percent Difference[1]	
	Aqueous	Solid	Aqueous	Solid[2]
Tritium (H-3)	100 ± 20%	100 ± 20%	20	20
Gross α/β[1]	100 ± 30%	100 ± 30%	30	30
Gamma Spec	100 ± 20%	100 ± 20%	20	20
Strontium, Isotopic (Sr-89 & Sr-90)	100 ± 25%	100 ± 25%	25	25
Strontium, Total	100 ± 25%	100 ± 25%	25	25
Uranium, Isotopic	100 ± 20%	100 ± 20%	20	20
Uranium, Total	100 ± 10%	100 ± 10%	20	20
Radium-226	100 ± 20%	100 ± 20%	20	20
Radium-228	100 ± 30%	N/A	30	N/A
Radium, Total Alpha	N/A	100 ± 20%	N/A	20
Radon-222	100 ± 30%	N/A	30	N/A
Technetium-99	100 ± 25%	100 ± 25%	20	20
Thorium, Isotopic	100 ± 20%	100 ± 20%	20	20
Plutonium, Isotopic	100 ± 20%	100 ± 20%	20	20
Americium, Isotopic	100 ± 20%	100 ± 20%	20	20
Cerium, Isotopic	100 ± 20%	100 ± 20%	20	20
Polonium-210	100 ± 20%	100 ± 20%	20	20
Lead-210	100 ± 20%	100 ± 20%	20	20
Carbon-14	100 ± 25%	100 ± 30%	25	25
Nickel-63	100 ± 20%	100 ± 25%	20	20
Iron-55	100 ± 20%	100 ± 25%	20	20
Plutonium-241	100 ± 20%	100 ± 20%	20	20

N/A: Not applicable

[1] RER ≤ 1, if activity of the sample is less than 10 X MDA.

[2] Solids may have outliers due to potential sample inhomogeneity.

APPENDIX B

GUIDELINES AND REQUIREMENTS FOR SAMPLE CONTAINERS, PRESERVATION, STORAGE, VOLUME, AND HOLDING TIMES

Table B-1. Requirements Containers, Preservation Techniques, Sample Volumes, and Holding Times for Inorganic and Organic Analyses (3 Pages)

Constituent	Analytical Method	Container[1]	Storage & Preservation [2,3]	Minimum Sample Quantity	Maximum Holding Time
INORGANICS:					
Acidity	305.1	P,G	4°C	100 mL	14 days
Alkalinity	310.1	P,G	4°C	100 mL	14 days
Ammonia-N	350.1	P,G	4°C H_2SO_4 pH<2	400 mL	28 days
Bromide	300.0	P,G	None Required	50 mL	28 days
Chemical Oxygen Demand (COD)	410.4	P,G	4°C H_2SO_4 pH<2	50 mL	28 days
Chloride	300.0/325.3/9251	P,G	None Required	50 mL	28 days
Cyanide, total and amenable	335.1/335.4/9010	P,G,T	4°C NaOH pH>12[2] $C_6H_8O_6$	500 mL or 4 ounces	14 days (water & soil)
Filterable Residue (TDS)	160.1	P,G	4°C	100 mL	7 days
Fluoride	340.2	P,G	None Required	300 mL	28 days
Non-Filterable Residue (TSS)	160.2	P,G	4°C	100 mL	7 days
pH	150.1	P,G	None Required	25 mL	Immediate
Total Kjeldahl Nitrogen	351.2	P,G	4°C H_2SO_4 pH<2	500 mL	28 days
Nitrate, as N	300.0/353.2/9200	P,G	None Required	100 mL	48 Hours
Nitrate-Nitrite	300.0/353.3	P,G	4°C H_2SO_4 pH<2	100 mL	28 days
Nitrite, as N	300.0/351.2	P,G	None Required	100 mL	48 hours
Orthophosphate, as P	365.2	P,G	Filter; 4°C	50 mL	48 hours
Total Phosphorous, as P	365.2	P,G	4°C H_2SO_4 pH<2	50 mL	28 days
Specific Conductance	120.1/9050	P,G	None Required	100 mL	28 days
Temperature	170.1	P,G	None Required	1000 mL	Immediate
Total Hardnes ($CaCO_3$)	200.7	P,G	4°C HNO_3 pH<2	100 mL	180 days
Total Organic Carbon	415.1/9060	P,G,T	4 °C HCl/H_2SO_4 pH<2[2]	500 mL or 4 ounces	28 days (water and soil)
Turbidity	180.1	P,G	4°C	100 mL	48 hours
METALS:					
Chromium +6	7196	P,G,T	4°C	500 mL or 8 ounces	24 hours[4] (water and soil)
Mercury	245.2/7470/7471	P,G,T	4°C HNO_3 pH<2[2]	500 mL or 8 ounces	38 days in glass 13 days in plastic 28 days for CLP/TCLP

Table B-1. Requirements Containers, Preservation Techniques, Sample Volumes, and Holding Times for Inorganic and Organic Analyses (3 Pages)

Constituent	Analytical Method	Container[1]	Storage & Preservation [2,3]	Minimum Sample Quantity	Maximum Holding Time
INORGANICS:					
All metals (except Cr^{+6} and Hg)	200.7/6010; 200/7000 Series	P,G,T	4°C HNO_3, to pH < 2[2],	500 mL or 8 ounces	180 days (water and soil)
ORGANICS:					
Total Recoverable Petroleum Hydrocarbons (TRPH)	418.1	G,T	4°C H_2SO_4 pH < 2	1000 mL or 8 ounces	28 days (water and soil)
Oil and Grease	413.1/413.2/ 9070/9071	G	4°C H_2SO_4 pH < 2	1000 mL or 8 ounces	28 days (water and soil)
Total Petroleum Hydrocarbons-*Gasoline*	8015 (Modified)	G	4°C $Na_2S_2O_3$; HCl pH < 2	1000 mL or 8 ounces	14 days (water and soil)
Total Petroleum Hydrocarbons-*Diesel*	8015 (Modified)	G	None required	1000 mL or 8 ounces	7 days to extraction; 40 days to analysis
Purgeable Halocarbons	8010	G, Teflon-lined septum	4°C $Na_2S_2O_3$	3x40 mL	14 days
Aromatic Volatile Organics	8020	G, Teflon-lined septum	4°C HCl pH < 2[2]; $Na_2S_2O_3$	3x40 mL or 4 ounces	14 days (water and soil)
Chlorinated Herbicides	8150	G, Teflon-lined septum	4°C pH 5-9	1000 mL or 8 ounces	Water - 7 days to extraction; 40 days to analysis Soil - 14 days to extraction; 40 days to analysis
Pesticides and Polychlorinated Biphenyls (PCBs)	8080/8140	G, Teflon-lined cap	4°C pH 5-9	1000 mL or 8 ounces	Water - 7 days to extraction; 40 days to analysis Soil - 14 days to extraction; 40 days to analysis
Phenols	8040	G, Teflon - Lined, cap	4°C $Na_2S_2O_3$	1000 mL or 8 ounces	Water - 7 days to extraction; 40 days to analysis Soil - 14 days to extraction; 40 days to analysis
Semivolatile Organics	625/8270	G, Teflon-lined cap	4°C $Na_2S_2O_3$	1000 mL or 8 ounces	Water - 7 days until extraction; 40 days to analysis Soil - 14 days until extraction; 40 days to analysis
Volatile Organics	8240/8260	G, Teflon - lined septum	4°C $Na_2S_2O_3$ HCl pH < 2[2]	3 x 40 mL or 4 ounces	14 days (water and soil); 7 days (water-unpreserved)

Table B-1. Requirements Containers, Preservation Techniques, Sample Volumes, and Holding Times for Inorganic and Organic Analyses (3 Pages)

Constituent	Analytical Method	Container[1]	Storage & Preservation [2,3]	Minimum Sample Quantity	Maximum Holding Time
INORGANICS:					
Polycyclic Aromatic Hydrocarbons (PAHs)	8310	G, Teflon - lined cap	4°C $Na_2S_2O_3$	1000 mL or 8 ounces	Water - 7 days until extraction; 40 days after extraction Soil - 14 days until extraction; 40 days after extraction
Toxicity Characteristic Leaching Procedure (TCLP)	1311	G, teflon - lined cap	Cool, 4°C	1000 mL or 8 ounces	VOAs - 14 days to TCLP extraction SemiVOAs - 14 days to TCLP extraction and 40 days to analysis Mercury - 28 days to TCLP extraction; 28 days to analysis Metals - 180 days to TCLP extraction; 180 days to analysis
Explosives	8330	P, G, T	Cool, 4°C	1000 mL or 8 ounces	Water - 7 days to extraction Soils - 14 days to extraction Analyze w/in 40 days

1 Polythylene (P); Glass (G); Brass sleeves.
2 No pH adjustment is required for soils.
3 Preservation with 0.008% $Na_2S_2O_3$ is only required when residual chlorine is present.
4 Holding time requirement for Chromium
6 in soils has not been established. The recommended hold. time for extracting into water is 48 hours. The sample must be analyzed w/in 24 hrs of extraction.

Sources: Table 2-21 in EPA SW-846 (Revision 1, July 1992); EPA-600/4-79-020 (Revised March 1983); CLP SOWs.

APPENDIX C

STANDARD OPERATING PROCEDURES INDEX

SOP INDEX

Number	Document Title
LAL-90-SOP-0001	Document Control
LAL-90-SOP-0002	Sample Receipt and Login
LAL-90-SOP-0003	Waste Handling and Disposal
LAL-90-SOP-0004	Preventing Sample Contamination
LAL-90-SOP-0005	Standards Traceability in the Inorganic and Organic Laboratories
LAL-90-SOP-0006	Maintenance of Laboratory Logbooks and Records
LAL-90-SOP-0007	(SOP RETIRED - Information in # 6) Laboratory Notebook Policy
LAL-90-SOP-0008	Technical and Managerial Review of Analytical Data
LAL-90-SOP-0009	Internal Sample Chain-of-Custody and Evidentiary Procedures
LAL-90-SOP-0010	Internal System Evaluation
LAL-90-SOP-0011	(SOP RETIRED 7/93) Statistical Review of QA Data
LAL-90-SOP-0012	Independent QA Validation of Inorganic Analysis Data
LAL-90-SOP-0013	Independent QA Validation of Organic Analysis Data
LAL-90-SOP-0014	Security
LAL-90-SOP-0015	Routine and Periodic Maintenance of Laboratory Support Equipment
LAL-90-SOP-0016	Analysis of VOCs by GC-MS (EPA 624/8240/8260/CLP)
LAL-90-SOP-0017	Glassware Cleaning in the Organic Chemistry Laboratory
LAL-90-SOP-0018	Glassware Cleaning for Inorganic Analyses
LAL-90-SOP-0019	Procedure for the Determination of Anions by Ion Chromatography
LAL-90-SOP-0020	Determining Ammonia in Surface Water and Waste Water by Flow Injection Analysis
LAL-90-SOP-0021	Determination of Specific Conductance
LAL-90-SOP-0022	Determination of the pH
LAL-90-SOP-0023	Analysis of Volatile Organic Compounds in Drinking Water by Capillary Column GC/MS
LAL-90-SOP-0024	Analysis of Semivolatile Organics Compounds by Capillary Column GC/MS
LAL-90-SOP-0025	Analysis of Purgeable Aromatics and Halocarbons in Wastewater and Drinking Water by Gas Chromatograph Using Methods 8010/8020, 601/602, and 502.2
LAL-90-SOP-0026	Continuous Liquid-Liquid Extraction on Organic Compounds
LAL-90-SOP-0027	Separatory Funnel Extraction of Organic Compounds
LAL-90-SOP-0028	Extractable Organics in Soil/Solid Matrices by Sonication Extraction
LAL-90-SOP-0029	Extractable Organics in Soil/Solid Matrices Soxhlet Extraction
LAL-90-SOP-0030	Analysis of Volatile Organic Compound in Drinking Water by Gas Chromatography (Hall/PID)
LAL-90-SOP-0031	Analysis of Trihalomethanes in Drinking Water by GC/MS Selected Ion Monitoring

SOP INDEX

Number	Document Title
LAL-90-SOP-0032	Block Digestor Sample Preparation for the Determination of Metals by Inductively Coupled Plasma Emission Spectroscopy
LAL-90-SOP-0033	Sample Preparation for the Determination of Metals by Graphite Furnace AAS (GFAAS)
LAL-90-SOP-0034	Procedure for Sample Preparation for the Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy (CVAAS)
LAL-90-SOP-0035	Procedure for Determination of Percent Solid in Soils, Sediments, and Sludges
LAL-91-SOP-0036	Determination of Total Hardness of Water by EDTA Titration
LAL-90-SOP-0037	Determination of Metals by Inductively Coupled Plasma-Atomic Emission Spectrometer (ICP-AES) Using Leeman Model PS-3000 ICP-AES
LAL-90-SOP-0038	Determination of Metals by GFAAS
LAL-91-SOP-0039	Determination of Sulfide in Water
LAL-90-SOP-0040	Determination of Total Filterable Residue
LAL-91-SOP-0041	Data Completeness and Client Compliance Review
LAL-90-SOP-0042	Potentiometric Determination of Fluoride in Aqueous Samples
LAL-90-SOP-0043	(SOP RETIRED) Containment Facility and Standards Laboratory Safety and Operations
LAL-91-SOP-0044	Determination of Mercury by CVAAS
LAL-90-SOP-0045	Guidelines for Sample Containers, Preservation, Packaging, and Shipment
LAL-90-SOP-0046	Proper Use and Care of Analytical Balances
LAL-90-SOP-0047	(SOP RETIRED) Colormetric Determination of Hydrazine in Water Samples
LAL-90-SOP-0048	TCLP Non-Volatile Extraction
LAL-91-SOP-0049	Titrimetric Determination of the Acidity of Aqueous Samples
LAL-93-SOP-0050	Titrimetric Determination of the Alkalinity of Aqueous Samples Using the Orion 940/960 pH/ISE/Millivolt Meter
LAL-91-SOP-0051	(SOP RETIRED 9/93) Determination of Total Kjeldahl Nitrogen in Aqueous Samples Using the Spectronic 21D Spectrophotometer
LAL-90-SOP-0052	Determination of Total Organic Carbon (TOC) in Aqueous Samples
LAL-91-SOP-0053	Determination of Total Organic Halides (TOX) in Aqueous, Oil, and Solid Samples by Combustive Oxidation Using the Dohrman DX020 A/B TOX Analyzer
LAL-90-SOP-0054	Cleanup Procedures by Gel Permeation Chromatography (GPC)
LAL-91-SOP-0055	Analysis of Chlorinated Herbicides by GC/ECD
LAL-91-SOP-0056	Determination of Total Petroleum Hydrocarbons (EPA Method 8015 Modified by GC-FID)
LAL-91-SOP-0057	Determination of Metals by Flame AAS Perkin Elmer 5100
LAL-90-SOP-0058	(SOP RETIRED) Sample Receipt and Tracking at Richland Receiving Facility in Washington
LAL-91-SOP-0059	Colorimetric Determination of Hexavalent Chromium

SOP INDEX

Number	Document Title
LAL-91-SOP-0060	Sample Preparation and Analysis Gross Alpha and Beta Activity in Aqueous Samples
LAL-91-SOP-0061	Gross Alpha and Beta Activity in Solids Sample
LAL-91-SOP-0062	Sample Preparation and Analysis for Radioactive Cesium in Aqueous Samples
LAL-91-SOP-0063	Samples Preparation and Analysis for Gamma Activity in Aqueous Samples
LAL-91-SOP-0064	Sample Preparation and Analysis for Gamma Activity in Solids
LAL-91-SOP-0065	Sample Preparation and Analysis for Radioactive Strontium in Aqueous Samples
LAL-91-SOP-0066	Sample Preparation and Analysis for Tritium in Aqueous Samples
LAL-91-SOP-0067	Sample Preparation and Analysis of Tritiated Water Extracted from Solid Samples
LAL-91-SOP-0068	Radiochemical Determination of Uranium in Aqueous Samples, Ion Exchange Method
LAL-91-SOP-0069	Determination of Actinide Elements in Water Sample Preparation and Analysis
LAL-91-SOP-0070	Sample Preparation and Analysis of Carbon-14 in Aqueous Samples
LAL-91-SOP-0071	Sample Preparation and Analysis of Alpha-Emitting Radium Isotopes in Water
LAL-91-SOP-0072	Sample Preparation and Analysis for Radioactive Iodine
LAL-91-SOP-0073	Radium-226 in Water Samples Radon Emanation Technique
LAL-91-SOP-0074	Sample Preparation and Analysis Radium-228 Activity in Aqueous Samples
LAL-91-SOP-0075	Calibration, Maintenance, and Operation of a High-Resolution Gamma Spectroscopy System
LAL-91-SOP-0076	(SOP RETIRED) Calibration, Maintenance, and Operation of a Sodium Iodide Gamma Spectroscopy System
LAL-91-SOP-0077	Calibration, Maintenance, and Operation of an Alpha Spectroscopy System
LAL-91-SOP-0078	Calibration, Maintenance, and Operation of a Tennelec LB4000 Alpha/Beta Counting System
LAL-91-SOP-0079	Calibration, Maintenance, and Operation of a Tennelec LB5500 Alpha/Beta Counting System
LAL-91-SOP-0080	Calibration, Maintenance, and Operation of a Quantalus Liquid Scintillation Counter
LAL-91-SOP-0081	Calibration, Maintenance, and Operation of a Rackbeta Liquid Scintillation Counter
LAL-91-SOP-0082	PROJECT SPECIFIC
LAL-91-SOP-0083	Disposal and Shipping of Radioactive Material
LAL-91-SOP-0084	Radiation and Contamination Surveys
LAL-91-SOP-0085	Receipt of Radioactive Samples
LAL-91-SOP-0086	Multiple-tube Fermentation Technique for Members of the Coliform Group
LAL-91-SOP-0087	Membrane Filter Technique for Members of the Coliform Group
LAL-91-SOP-0088	Radiochemical Data Validation

SOP INDEX

Number	Document Title
LAL-93-SOP-0213	The Analysis of Oganochlorine Pesticides and PCB'S Using Method CLP 3/90 and SOW (Revisions OLM01.0-1.8) by CLP 3/90 SOW - Revisions OLMO 1.0 - 1.8
LAL-93-SOP-0214	PROJECT SPECIFIC
LAL-93-SOP-0215	PROJECT SPECIFIC
LAL-93-SOP-0216	Grinding, Milling and Mixing of Solid Samples for Radiochemical Analysis
LAL-93-SOP-0217	Analysis of Semivolatile Organic Compounds by Capillary Column GC/MS by CLP 3/90 SOW (Revisions OLM0 1.0-1.8)
LAL-93-SOP-0218	Analysis of VOCs by GC-MS By CLP 3/90 SOW (Revisions OLMO1.0-1.8)
LAL-93-SOP-0219	Semivolatile Extraction Following CLP 3/90 SOW (Revisions OLM01.0-1.8)
LAL-93-SOP-0220	Determination of Metals by Inductively Coupled Plasma-Atomic Emission Spectrophotometer (ICP-AES) Using Thermo Jarrell Ash Model 61-E
LAL-93-SOP-0221	Block Digestor Sample Preparation for the Determination of Metals by Inductively Coupled Plasma Emission Spectroscopy for Burbank B-6 Soil Samples
LAL-93-SOP-0222	Sample Preparation for the Determination of Metals by Graphite Furnace AAS Atomic Absorption Spectroscopy for Burbank B-6 Soil Samples
LAL-93-SOP-0223	Analytical Project Initiation
LAL-93-SOP-0224	Determinations of Nickel-59, 63 in Water Samples and Soil Samples
LAL-93-SOP-0225	Determination of Nitrite-Nitrogen, Nitrite + Nitrate - Nitrogen, and Nitrate-Nitrogen in Water by flow Injection Analysis
LAL-93-SOP-0226	Operation of the One Half Ton Hoist
LAL-93-SOP-0227	Control of Hazardous Energy (Lockout/Tagout) Program for Lockheed Analytical Services
LAL-93-SOP-0228	Determination of Chloride in Water by flow Injection Analysis
LAL-93-SOP-0229	Extraction of Nitro Aromatics
LAL-93-SOP-0230	Radioactive Sample Extract Screening
LAL-93-SOP-0231	Determination of Corrosivity of Liquids by EPA Method 1110
LAL-93-SOP-0232	Selection and Qualification of Subcontractor Analytical Laboratories
LAL-93-SOP-0233	Determination of Reactivity
LAL-93-SOP-0234	Maintaining Security of RCRA Confidential Business Information (CBI)
LAL-93-SOP-0235	(SOP CANCELLED)
LAL-93-SOP-0236	Determination of Heat of Combustion of Solids and Liquids by EPA Method 5050
LAL-93-SOP-0237	Determination of Viscosity of Liquids
LAL-93-SOP-0238	Determination of Density of Liquid Samples
LAL-93-SOP-0239	Determination of Total Halides in Oil by EPA Method 9076
LAL-93-SOP-0240	Analysis of Volatile Organic Compounds by Isotope Dilution GC/MS Methods 1624
LAL-93-SOP-0241	Determination of 2, 3, 7, 8-TCDD by GC/MS Using Method 613

SOP INDEX

Number	Document Title
LAL-93-SOP-0242	Determinations of Polynuclear Aromatic Hydrocarbons by GC/FID Using Method 8100
LAL-93-SOP-0243	Method 1625 Analysis of Semivolatile Organic Compounds by Isotope Dilution GC/MS
LAL-93-SOP-0244	Determinations of Phthalate Esters by GC/FID/ECD
LAL-93-SOP-0245	Preparation of Trip Blanks
LAL-93-SOP-0246	Determination of Oil and Grease in Sludge Samples Using Method 9071
LAL-93-SOP-0247	PROJECT SPECIFIC
LAL-93-SOP-0248	Determination of Metals by Hydride Generation Atomic Absorption Spectroscopy
LAL-93-SOP-0249	RETIRED SEE SOP 086 Determination of Total Coliforms by the Multiple Tube Fermentation Technique
LAL-93-SOP-0250	RETIRED SEE SOP 087 Determination of Total Coliforms by the Membrane Filtration Technique
LAL-93-SOP-0251	Determination of Total Residue
LAL-93-SOP-0252	Determination of Chromium (VI) by Chelation-Extraction and Direct Aspiration Atomic Absorption Spectroscopy
LAL-93-SOP-0253	Determination of Sulfite
LAL-93-SOP-0254	Determination of Total Residual Chloride
LAL-93-SOP-0255	Determination of Dioxins Using CLP Multi-Media-Concentration Protocol
LAL-93-SOP-0256	Determination of Total Kjeldahl Nitrogen
LAL-93-SOP-0257	Extraction for Pesticides/PCBs Following Methods 8080/608
LAL-93-SOP-0258	PROJECT SPECIFIC
LAL-93-SOP-0259	PROJECT SPECIFIC
LAL-93-SOP-0260	Extraction of Polynuclear Aromatic Hydrocarbons by Method 8310
LAL-93-SOP-0261	Radiochemical Acid Waste Handling
LAL-93-SOP-0262	Radiochemical Operation of Blue-M Ashing Oven
LAL-93-SOP-0263	Radiochemical Fume Hood Cleaning
LAL-93-SOP-0264	Determination of Diesel Range Organics (DRO) (State of Tennessee Method)
LAL-93-SOP-0265	605 Extraction
LAL-93-SOP-0266	Determination of Alkalinity by Colorimetric Alternator Methyl Orange
LAL-93-SOP-0267	Temperature Measurements (Thermo Metro)
LAL-93-SOP-0268	Determination of Gasoline Range Organics
LAL-93-SOP-0269	Determination of Amines in Aqueous Samples by Ion Chromatography
LAL-93-SOP-0270	TCLP Volatile Extraction
LAL-93-SOP-0271	Determination of Alcohol
LAL-93-SOP-0272	Determination of Ash from Petroleum Products

SOP INDEX

Number	Document Title
LAL-93-SOP-0273	Determination of Heating Value
LAL-93-SOP-0274	Internal Evaluation of Data Integrity of Electronic Media
LAL-93-SOP-0275	Technical Review of Data for Army Environmental Center
LAL-93-SOP-0276	Spiking and Spike Verification in the Organic Laboratory
LAL-93-SOP-0277	Guideline for Developing Case Narratives
LAL-93-SOP-0278	Generation of Report Packages
LAL-93-SOP-0279	Determination of Formaldehyde in Aqueous Samples by Spectrophotometry
LAL-93-SOP-0280	(SOP CANCELLED)
LAL-93-SOP-0281	(SOP CANCELLED)
LAL-93-SOP-0282	Determination of Total Nitrogen, Phosphorus, Sulfur, and Halogens by Combustion Bomb Oxidation
LAL-93-SOP-0283	Identification and Control of Nonconforming Items
LAL-93-SOP-0284	Procedures for Acceptance of Laboratory Chemicals
LAL-93-SOP-0285	Gross Radium Alpha Screening
LAL-93-SOP-0286	Determination of Benzadines by Method 605
LAL-93-SOP-0287	Analysis of Organochlorine Pesticides and PCBs by Method 608
LAL-93-SOP-0288	Titrimetic Determination of Bromide
LAL-93-SOP-0289	Colorametric Determination of Hardness
LAL-93-SOP-0290	Titrimetric Determination of Hardness
LAL-93-SOP-0291	Screening of Gross Alpha in Water Samples Using Liquid Scintillation
LAL-93-SOP-0292	Determination of Trace Levels of Volatile Amines by Ion Chromatography
LAL-94-SOP-0293	(SOP CANCELLED)
LAL-94-SOP-0294	Iron 55 & 59
LAL-94-SOP-0295	Sample Management in the Organic Section
LAL-94-SOP-0296	Independent QA Review of Analytical Data for WHC
LAL-94-SOP-0298	Long Term Archival of Electronic Data
LAL-94-SOP-0299	Determination of Chlorinated Acid Herbicides by Method 515.1
LAL-94-SOP-0300	The Analysis of Alcohols
LAL-94-SOP-0301	Determination of Organic Acids in Aqueous Samples by Ion Chromatography
LAL-94-SOP-0302	Determination of TOC Using the Modified Mebius Method
LAL-94-SOP-0303	Semi-Volatiles Extraction by Method 8270
LAL-94-SOP-0304	Tritium Enrichment

ATTACHMENT C

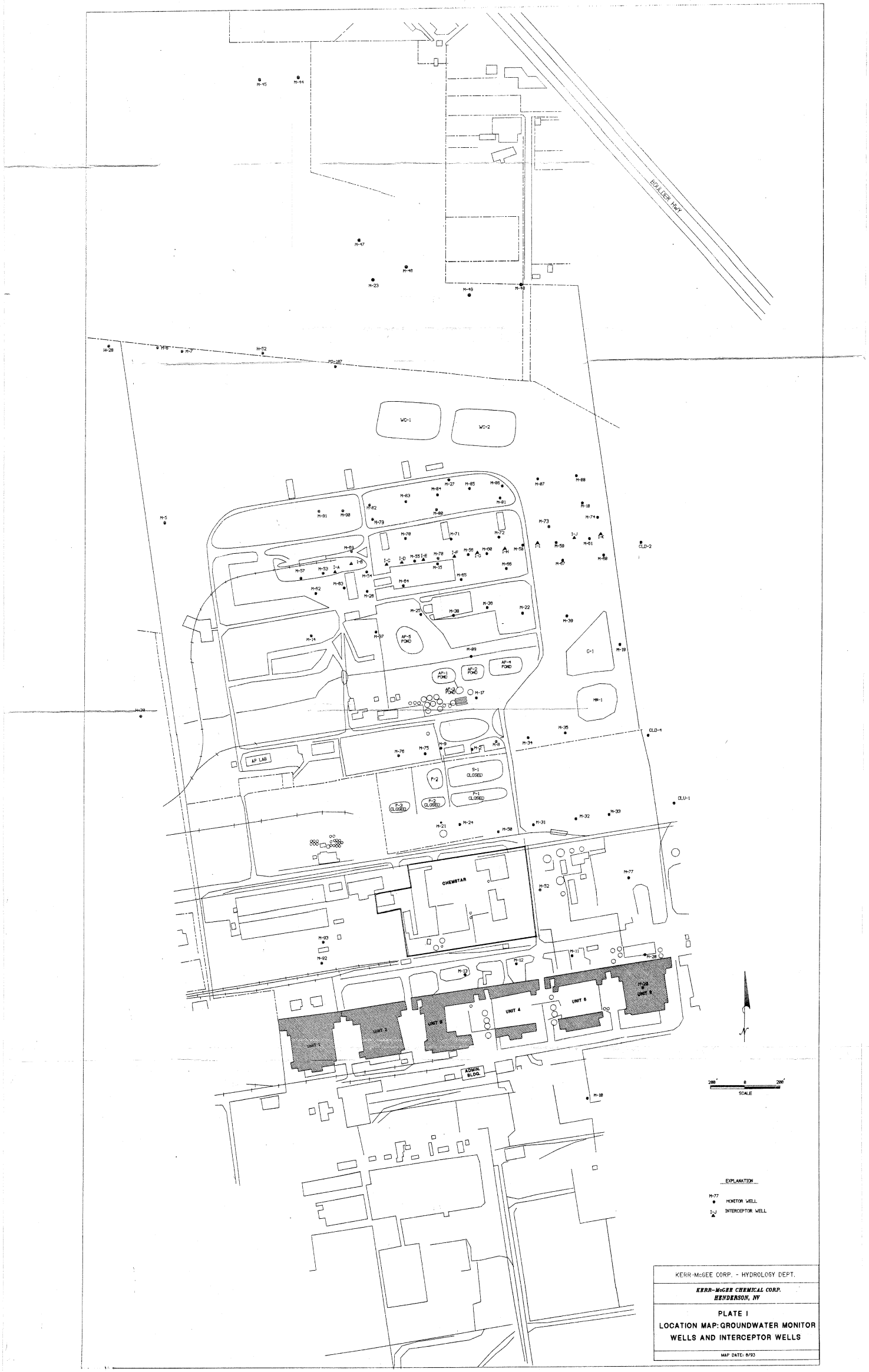
Henderson Facility Safety Procedure Manual

Table of Contents

UNCONTROLLED
This document will not be updated.

Last Issue Date

X-A. Safety and Health


BOULDER HWY
M-45
M-44
M-47
M-46
M-23
M-49
M-48
M-28
M-6
M-7
M-52
PZ-107
WC-1
WC-2
M-87
M-88
M-27
M-85
M-86
M-84
M-83
M-81
M-82
M-18
M-91
M-98
M-79
M-74
M-73
M-5
M-78
M-71
M-72
I-J
M-61
CLD-2
M-59
M-63
I-C
I-D
M-55
I-E
M-78
I-F
M-58
M-68
I-H
M-58
M-57
M-53
I-A
I-B
M-54
M-15
M-66
M-67
M-60
M-65
M-64
M-62
M-63
M-26
M-25
M-38
M-36
M-22
M-39
M-14
M-37
AP-5 POND
M-89
C-1
M-19
AP-1 POND
AP-2 POND
AP-4 POND
AP-3 POND
M-17
MW-1
M-38
M-35
CLD-4
M-34
AP LAG
M-76
M-75
M-9
M-7
M-8
S-1 CLOSED
P-2
P-1 CLOSED
P-3 CLOSED
P-4 CLOSED
CLU-1
M-21
M-24
M-30
M-31
M-32
M-33
CHEMSTAR
M-77
M-52
M-93
M-92
M-17
M-29
M-12
M-13
UNIT 1
UNIT 2
UNIT 3
UNIT 4
UNIT 5
UNIT 6
ADMIN BLDG.
M-10
N
200' 0 200'
SCALE
EXPLANATION
M-77 MONITOR WELL
I-J INTERCEPTOR WELL
KERR-McGEE CORP. - HYDROLOGY DEPT.
KERR-McGEE CHEMICAL CORP.
HENDERSON, NV
PLATE I
LOCATION MAP: GROUNDWATER MONITOR WELLS AND INTERCEPTOR WELLS
MAP DATE: 8/93