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June 15, 2001

Mr. Cornell Long
Technical Program Manager
AFIERA/RSRE
2513 Kennedy Circle
Brooks AFB, Texas 78235-5123

Subject: Transmittal of
Interim Final Scientific and Technical Report for
Perchlorate Biotransport Investigation
Contract F41624-95-D-9018, Delivery Order 0055, CDRL A003 and
Delivery Order 0060, CDRL A003

Dear Mr. Long:

Enclosed are four copies of the Interim Final Scientific and Technical Report for Perchlorate Biotransport Investigation for Delivery Order 55 and Delivery Order 60 for your review.

This deliverable is being issued as interim final with the understanding that additional work is required. This additional investigation will be included in the final report.

Parsons ES appreciates the opportunity to serve the Air Force under this important requirement. If you have any questions or require additional information, please contact me at 512/719-6067.

Sincerely,

Stephen J. Rembish, Ph.D.
Delivery Order Manager

cc: Lt. Col. Dan Rogers, 314 AW/JA
Randy Burns, USEPA (2 copies)
Annie Jarabek, USEPA
Mark Sprenger, USEPA
Clarance Callahan, USEPA

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Air Force
Institute for
Environment, Safety, and Occupational Health
Risk
Analysis

Interim Final
Scientific and Technical Report for Perchlorate Biotransport Investigation

A Study of Perchlorate Occurrence in Selected Ecosystems

Prepared for:

Air Force Institute for Environment, Safety and Occupational Health Risk Analysis

Contract No. F41624-95-D-9018
Delivery Orders 0055 and 0060

Prepared by:

PARSONS ENGINEERING SCIENCE, INC.
Austin, Texas

June 2001

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ACRONYMS AND ABBREVIATIONS

µg/L	Micrograms per liter
ABL	Allegany Ballistics Laboratory
AFB	Air Force Base
AFIERA	Air Force Institute for Environment, Safety and Occupational Health Risk Analysis
APPL	Agriculture and Priority Pollutants Laboratories, Inc.
BLM	Bureau of Land Management
CCL	Contaminant Candidate List
Clayton	Clayton Group Services, Inc.
ClO_4^-	Perchlorate
COC	Chain of custody
DN	Downstream
EnChem	EnChem, Inc.
Holloman	Holloman Air Force Base, New Mexico
ICV	Initial calibration verification
ID	Identification
Indian Head	Indian Head Naval Surface Warfare Center, Maryland
IOC	Army Industrial Operations Command
IPC	Instrument performance check
IPSC	Interagency Perchlorate Steering Committee
Lake Mead	Lake Mead NRA and Las Vegas Wash, Nevada
LCS	Laboratory control sample
LHAAP	Longhorn Army Ammunition Plant, Texas
LVOA	Low vulnerability ordnance ammunition
LVVWD	Las Vegas Valley Water District
MB	Method blank
MDL	Method detection limit
MDS	Method development study
MGD	Million gallons per day
mg/kg	Milligrams per kilogram
MS/MSD	Matrix spike/matrix spike duplicate
NMDGF	New Mexico Department of Game and Fish
NPDWR	National Primary Drinking Water Regulations
NRA	National Recreation Area
OGWDW	Office of Ground Water and Drinking Water
POC	Points of contact
ppb	Parts per billion
ppm	Parts per million
PVC	Polyvinyl chloride

ACRONYMS AND ABBREVIATIONS
(continued)

QC	Quality control
RSD	Relative standard deviation
SDG	Sample delivery group
SDWA	Safe Drinking Water Act
SNWA	Southern Nevada Water Authority
SOP	Standard operating procedure
SWMU	Solid Waste Management Unit
TNRCC	Texas Natural Resource Conservation Commission
TNT	2,4,6-trinitrotoluene
TSH	Thyroid-stimulating hormone
UP	Upstream
USEPA	United States Environmental Protection Agency
Yuma	Yuma, Arizona

EXECUTIVE SUMMARY

Introduction

Perchlorate (ClO4-) is an oxidizing compound that is very mobile in aqueous systems and can persist for decades under typical conditions. Ammonium perchlorate is used as an oxidizer component and primary ingredient in solid rocket propellant for rockets, missiles, and fireworks. Large-scale production of perchlorate began in the mid-1940s. Since the 1950s, large volumes of perchlorate have been disposed of in several US states. Environmental impacts of perchlorate contamination are being studied through a unique partnership between the Department of Defense and the Environmental Protection Agency (EPA), together with the Interagency Perchlorate Steering Committee (IPSC). The Ecological Impacts subcommittee of the IPSC met in Little Rock on April 20-21, 1999 and identified data gaps. The present study was designed to address some of these gaps.

The primary goal of this project is to answer one of the most frequently asked questions during previous IPSC meetings: "Can perchlorate be found in biological receptors?" Therefore, the primary objectives for the project include characterization of complete contaminant exposure pathways for ecological receptors of concern, evaluation of the potential for biotransport and identification of any specific food web issues. To complete these tasks, physical and biological media were sampled and analyzed to determine perchlorate concentrations. A total of 965 samples, of various media, were collected and analyzed for perchlorate on this project. This report does not attempt to determine bioaccumulation factors (BAFs) for perchlorate.

To support the primary goals, an additional objective was to conduct a Method Development Study (MDS). The objectives of the MDS were to determine and document accurate and reliable method(s) to quantify perchlorate concentrations in various matrices including soil, sediment, vegetation, aquatic life, mammals, reptiles, birds, and insects.

There are presently many data gaps concerning perchlorate contamination. Therefore, this study was limited in scope to address only some of the data gaps. Table ES-1 describes the limitations of this study:

2. Study sites

Six sites from throughout the United States were selected for this study. The sites were chosen to include diverse soil types and climatic conditions. The sites also included a range of aquatic system types, facility types, and discharge and transport pathways. Additional site selection criteria included accessibility, known areas of contamination, political considerations, availability of background information, local stakeholder involvement, physical control of the site, and availability of a range of environmental media concentrations.

Table ES-2 provides a brief description of the six sites studied.

3. Methodology

In order to determine the concentration of perchlorate in soil, sediment, plant tissue, and animal tissue, the development of analytical methods to measure perchlorate in these media was required. Part of this investigation included the development of these methods. All analytical methods were based on USEPA's Method 314.0 for perchlorate in water, and required the development of different extraction procedures for various biological media. All methods were successfully developed and verified for use in this study.

Since this project was to document the occurrence of perchlorate at the six sites, and not to develop bioaccumulation factors, the project goal for species selection was to collect representative samples from as many different matrices and biological groups as possible. The number and types of samples collected were dependent upon species availability at each of the sample locations, state and federal scientific collection permit limitations, and field personnel safety. All sample collection methods were performed in ways to minimize impact to local animal and plant populations. This study did not attempt to isolate perchlorate to particular body parts or plant tissue. All animals collected were analyzed as discrete samples from aliquots extracted from the whole body composite of each discrete animal unless a field composite of small animals (i.e., minnows) was necessary to provide the required volume for the analytical method to be successful.

4. Results and Discussion

Analytical laboratory methods were developed and documented for the matrices required, soil, sediment, plant tissue, and animal tissue. The findings of the method development study indicate that detection of perchlorate was possible in all of the matrices. Clayton Environmental Services was able to consistently attain a reporting limit of 4 µg/L for water, 80 µg/kg for soil and sediment, and 400 µg/kg for tissue samples. These reporting limits were sufficient to detect the presence of perchlorate in numerous tissue samples, soil samples, and water samples.

In order to answer the question of whether perchlorate is present in biological receptors, approximately 1000 samples were collected and analyzed for perchlorate. These samples cover a wide range of potential sources, exposure pathways, and receptors. Perchlorate was detected in abiotic media (e.g., soil and water) at all six sites investigated. The Lake Mead area has the highest mean concentration of perchlorate across all media types, and samples collected at Yuma, Arizona had the lowest concentrations of perchlorate on a weight-weight basis. The range of concentrations of perchlorate in surface water across the sites varied up to six orders of magnitude from Yuma, Arizona (near the reporting limit of 4 µg/L) to Location 1 of the Lake Mead area (130,000 µg/L).

The sites and locations with the highest perchlorate concentrations in the surface water also have the highest concentrations of perchlorate in the biological media. The highest concentrations of perchlorate were found predominately in vegetation. In aquatic

media, vegetation had the highest concentrations of perchlorate at three of the six sites. In terrestrial media, vegetation had the highest concentrations at five of the six sites.

5. Uncertainties

The goal of this investigation was to report the occurrence of perchlorate in physical media (soil, sediment, and water) and biological media (flora and fauna). This investigation detected the presence of perchlorate in all physical media as well as some biological media. Due to the breadth and scope of this investigation, there are a number of uncertainties that are introduced into the results. These uncertainties should be considered before attempting to interpret the data. Table ES-3 describes the primary uncertainties with this investigation:

6. Conclusions

The goal of this investigation was to document the occurrence of perchlorate in physical and biological media at six perchlorate contaminated sites across the United States representing different perchlorate levels and different climatic conditions. This goal was accomplished in that perchlorate was identified in numerous media at the sites. The present investigation provides new information on the presence of perchlorate in the environment.

In addition, we can conclude:

- Uptake of perchlorate occurs into various biological media,
- Both terrestrial and aquatic vegetation have the highest perchlorate concentrations and frequency of detection of all the media sampled at the sites investigated,
- There is an apparent correlation between surface water perchlorate concentrations and concentrations in other media, and
- Preliminary investigation of salt cedar leaf, seed, and leaf litter show that different parts of the plant accumulate different levels of perchlorate.

While it is apparent that perchlorate is available for uptake by plants and animals, this study does not address the mechanism for the uptake of perchlorate from the environment. Overall, it appears that this study successfully documented the occurrence of perchlorate in the environment.

7. Recommendations for future investigations

The following are recommendations for additional investigations that may supplement the information in this report and provide a better understanding of perchlorate in the environment.

- Perform additional field sampling of similar media at the sites and locations with high perchlorate concentrations and good species diversity. This could include specific co-location of samples collected, performance of field stomach content analysis of collected animals with follow-up sampling of the food sources identified, and organ specific analysis to determine specific tissue location of

stored perchlorate. In addition, this study could be designed to address potential seasonal effects.

- Conduct inter-laboratory investigations on sample preparation procedures for biological media to improve reproducibility and documentation of steps to get from field sample to analytical extract.
- Perform an investigation on agricultural crops irrigated with water containing perchlorate to determine the possible occurrence of perchlorate in the crops.
- Determination of a water quality standard for perchlorate using microcosm studies on biological species in conjunction with existing environmental effects data.

Table ES-1

This investigation *does*:	**This investigation *does not*:**
• Assess six sites across the United States with known sources of perchlorate that represents only a snapshot in time; • Analyze various physical media, plants and animals representing multiple trophic levels for the presence of perchlorate; • Document the analytical method development for perchlorate detection in the parts per billion (ppb) range for soil, sediment, flora, and fauna samples.	Investigate possible perchlorate contamination at any other sites or locations; Attempt to delineate the extent of contamination at the six sites, nor in-depth source investigations, nor perchlorate sampling to satisfy any regulatory requirements at the site; Include ecological or human health risk assessments; Analyze specific animal tissue or plant tissue (only whole body analysis was performed), Include in-depth analysis of contaminant pathways and food webs, and conclusions regarding the relationship of perchlorate found in physical and biological media; Compare perchlorate occurrence in flora and fauna among sites or locations; Calculate bioaccumulation factors; Investigate remediation technologies; nor does it Include valid sample sizes for statistical computations.

Table ES-2

Yuma, Arizona	• Located on the Colorado River, which is known to be contaminated with low levels of perchlorate (approximately 5 ppb) • Area surrounding the river includes irrigated farmlands, desert, and desert mountains
Lake Mead National Recreation Area and the Las Vegas Wash, Nevada	• Industrial discharges of perchlorate from groundwater seeps near the Las Vegas Wash have concentrations in the 100,000 ppb range • The Las Vegas Wash is the primary storm water and treated wastewater effluent drainage feature for the city of Las Vegas • Lake Mead is the receptor of all outflow from the Las Vegas Wash • The drinking water treatment plant intake for the Las Vegas area is in Lake Mead downstream of the outflow of the Las Vegas Wash The surroundings of the Wash include the convergence of four North American deserts
Allegany Ballistics Laboratory, West Virginia	• The site is bordered by the North Branch of the Potomac River • Surroundings include mountain vegetation and deciduous forests
Holloman AFB, New Mexico	• The White Sands pupfish is a threatened species • The area is adjacent to White Sands National Monument and White Sands Missile Range
Indian Head Naval Surface Warfare Facility, Maryland	• The site is located on the Potomac River • Abundant freshwater aquatic species present
Longhorn Army Ammunition Plant, Texas	• Located in East Texas pine forests • Site perchlorate contamination has been well studied by Texas Tech University

Table ES-3

Site-related Uncertainties • The samples obtained represent a snapshot in time and do not document the occurrence of perchlorate under any climatic conditions other than those present when sampling occurred, • Meteorological differences occurred among the sites and no attempt was made to standardize the results for meteorological conditions (i.e., rainfall rates varied among the sites), • Some of the sample locations are not naturally occurring water bodies (treated water storage ponds, manmade drainage structures), • Sample locations were not necessarily those expected to have the highest perchlorate concentrations, but the sample locations were chosen for ease of sampling, • Numbers and types of biological samples at each location vary greatly, and • No attempt was made to identify all potential sources of perchlorate contamination (e.g., fertilizer use in irrigated areas).
Biological/ecological uncertainties • Specific receptors could not always be targeted – an opportunistic sampling strategy was employed, • There were low sample counts of each medium at each location, • The routes of exposure (direct contact, incidental intake, ingestion) to each receptor were not evaluated, • No gut analysis was performed to evaluate food chain exposure, • Performing a trend analysis across the sites would require combining classes of receptors (e.g. small mammals, fish, etc.,), and • Migration patterns and daily spatial range of mobile biological samples was not studied.
Analytical uncertainties • Detection limits for tissues were comparatively higher than physical media samples, • Laboratory sample clean up methods for extraction aliquot and dilutions for analysis may greatly affect detection limits.

SECTION 1
INTRODUCTION

Concerns about potential human or ecological exposure to perchlorate in the environment have been largely unexplored until recently for several reasons. These include the lack of an analytical method capable of detecting perchlorate in the parts per billion (ppb) range, limited knowledge about the potential toxicological effects of perchlorate, and the lack of information regarding the fate and transport of perchlorate in the environment. In April 1997, methodology was developed to detect perchlorate at low concentrations in water (approximately 4 ppb). Within several months, perchlorate had been discovered at various manufacturing sites and in well water and drinking water supplies in California, Nevada, and Utah.

The potential effects of perchlorate in the environment are just beginning to be assessed. Areas of uncertainty and informational needs include: analytical methodologies for media other than water, toxicological information on human health exposure to low levels in drinking water, the prevalence of perchlorate in surface water and groundwater, treatment technologies, transport and transformation in environmental media, and ecological effects. This study addresses two of those information needs: the method development and validation for perchlorate analysis in soil, sediment, and biological media, and characterization of the potential for perchlorate to move through potential exposure pathways.

1.1 PROJECT OBJECTIVES

The primary goal of this project is to answer one of the most frequently asked questions raised during previous Interagency Perchlorate Steering Committee (IPSC) meetings: "Is perchlorate appearing in biological receptors throughout the food chain?" Therefore, the primary objectives for this project include characterization of complete contaminant exposure pathways for ecological receptors of concern and evaluation of the potential for biotransport. To complete these tasks, physical and biological media were sampled and analyzed to determine perchlorate concentrations.

To support the primary goal, an additional objective was to conduct a Method Development Study (MDS). The objectives of the MDS were to determine accurate and reliable method(s) to quantify perchlorate concentrations in various matrices (beyond the existing methods for water) including, soil, sediment, terrestrial and aquatic vegetation, fish, invertebrates, mammals, reptiles, birds, and insects.

1.2 DOCUMENT OBJECTIVES

This document reports results of the study conducted according to the "Workplan for Perchlorate Biotransport Analysis" (Parsons ES, October 2000) prepared for the Air Force Institute for Environment, Safety and Occupational Health Risk Analysis under

Contract F41624-95-D-9018, Delivery Order 0055. Specific items included in this report are:

- Background information on perchlorate issues;
- Description of the six study sites including perchlorate sources and environmental conditions;
- Description of the study methods, including field, laboratory, and data management methods and method development for non-aqueous samples;
- Study results, discussing perchlorate concentrations by site, by medium;
- Identification of ecological receptors or locations for which perchlorate has the potential to generate ecological risk;
- Discussion of issues contributing to uncertainty in this investigation;
- Discussion of the overall investigation success and how results relate to other studies; and
- Recommendations for additional investigations based on the results of this study.

Because there are presently many data gaps concerning perchlorate contamination, this study was limited in scope to address only some of the gaps. Therefore, this report does not include information or discussion of the following:

- Possible perchlorate contamination at any other sites or locations,
- An attempt to delineate the extent of contamination at the six sites, nor in-depth source investigations,
- Ecological risk or human risk assessments, nor water quality criteria development,
- Specific animal body part of plant part analysis (only whole body analysis was performed),
- In-depth analysis of contaminant pathways and food webs,
- Comparison of flora and fauna between sites or locations,
- Remediation techniques, and
- Valid sample sizes for statistical computations.

1.3 BACKGROUND OF PERCHLORATE

Perchlorate (ClO_4^-) is an oxidizer component used in explosives, chemical fertilizers, and as solid propellant for rockets, missiles, emergency ejection seats, fireworks, and automobile air bag inflators. Perchlorate is a highly soluble, non-volatile, kinetically stable ion that does not adsorb readily to most minerals and can persist for many decades under typical groundwater and surface water conditions (USEPA, May 2000). Environmental perchlorate contamination has resulted from discharges from manufacturing and production operations, waste handling and washing operations, research and testing, and use of perchlorate in industrial, military, and agricultural practices.

Minimal information exists on ecological effects of perchlorate or any of its salts. Existing data suggest that at concentrations ranging from 10 to 1,000 parts per million (ppm), effects on biota include changes in thyroid-hormone production, alterations in metamorphosis, changes in development and population growth, and inhibition of seed germination and growth in agricultural crops. Treatment methods for removal of perchlorate removal from water are limited at present; however, ion exchange separators have been shown to be effective, and bioremediation tests suggest uptake by certain plants may also be possible (USEPA 1999; USEPA, May 2000).

Perchlorate has been found to modify thyroid function by competitive inhibition of iodide uptake, resulting in reduced thyroid hormone production. Thyroid hormone deficiencies can affect normal metabolism, growth, and development. The limited database on the toxicology of perchlorate confirms its potential to disrupt thyroid hormone production in mammalian test species, but limited data exist to evaluate the dose-response relationship for this thyroid effect or to evaluate other potential target tissues. There are no data to evaluate the effects of perchlorate in developing fetuses or its effects on species in natural ecological systems (USEPA, 1998).

A recent epidemiological study evaluated the potential effects of perchlorate on newborns in Yuma, Arizona. Yuma obtains its drinking water from the Colorado River, which contains low levels of perchlorate contamination (5-10 micrograms per liter [μg/L]). This study concluded that thyroid stimulation hormone (TSH) levels were reduced in newborns exposed to perchlorate-contaminated drinking water (Brechner, *et al.*, 2000).

The Safe Drinking Water Act (SDWA), enacted by Congress in 1974 and amended in 1986 and 1996, provides the basis for safeguarding public drinking water systems from contaminants that pose a threat to public health. The United States Environmental Protection Agency (USEPA) Office of Ground Water and Drinking Water (OGWDW) develops National Primary Drinking Water Regulations (NPDWR) to protect human health by limiting the levels of contaminants that may occur in public drinking water systems. The 1996 amendments to the SDWA require the USEPA to publish a list of contaminants not currently subject to NPDWR and known to be or anticipated to occur in public water systems. This list, the Contaminant Candidate List (CCL), is used to establish the primary sources of priority contaminants for research, guidance development, and regulatory determinations or monitoring by the individual states (USEPA, May 2000).

The CCL consists of 50 chemical and 10 microbial contaminants and is divided into two categories: (1) contaminants for which sufficient information exists to begin making regulatory determinations by 2001, and (2) contaminants for which additional research and occurrence information is necessary before regulatory determinations can be made. Perchlorate was added to the final CCL on March 2, 1998, and is identified in the second category as a contaminant needing additional research in the areas of health effects, treatment technologies, analytical methods, and more complete occurrence data (USEPA, May 2000).

Some states are beginning to set human health criteria for perchlorate in drinking water. The California Department of Health Services adopted 18 µg/L as its provisional action level for perchlorate in 1997. The Nevada Division of Environmental Protection determined that the health-based action level of 18 µg/L, as established in California, would be the recommended action level for cleanup pending a more current risk assessment (USEPA, May 2000). Texas utilizes a human health criterion for perchlorate at 22 µg/L (Texas Natural Resource Conservation Commission [TNRCC], 1999) for remedial actions. The Arizona Department of Health Service has determined that 14 µg/L is protective of human health with a margin of safety (ADHS, 2000).

1.4 SITES INVESTIGATED

Six sites, known to be contaminated with perchlorate, from throughout the United States were selected for this study. The six sampling sites are shown in relation to the contiguous United States on Figure 1.1. Figure 1.2 provides a more detailed area map for each of the six sites. The sites selected by the IPSC and the Air Force were chosen to include diverse soil types and climate conditions. They also included a range of aquatic systems, types of facilities, and discharge and transport pathways. A summary of site characteristics is provided in Table 1.1, and a detailed description of each site can be found in Section 2. Additional site selection criteria included accessibility, previously studied areas of contamination, political considerations, availability of known background information, local stakeholder interest, physical control of the site, and availability of a range of environmental media concentrations.

1.5 PROJECT RESPONSIBILITIES

Parsons Engineering Science, Inc. (Parsons) is the environmental services division of Parsons Corporation, one of the world's largest full service engineering and construction firms. The Austin, Texas office of Parsons ES is managing this investigation by performing study development, contracting, implementation of the investigation, management of laboratory activities, and production of the investigation report.

United States Air Force Institute for Environment, Safety and Occupational Health Risk Analysis (AFIERA), Brooks Air Force Base, Texas is representing both the Department of Defense and IPSC for this project. AFIERA is responsible for initiating the project, developing the scope of work, and managing the project. AFIERA also assisted in site visits and field tasks, development of plans, and development of the methods used to present findings.

Clayton Laboratory is a division of Clayton Services Group, Inc. (Clayton) with offices throughout the United States. Clayton's Novi, Michigan laboratory provided analytical laboratory services, including method development, analysis, quality control, and reporting for the various samples collected in this investigation. EnChem, Inc. (Madison, Wisconsin) and Covance Laboratories, Inc. (Madison, Wisconsin) both provided sample preparation services.

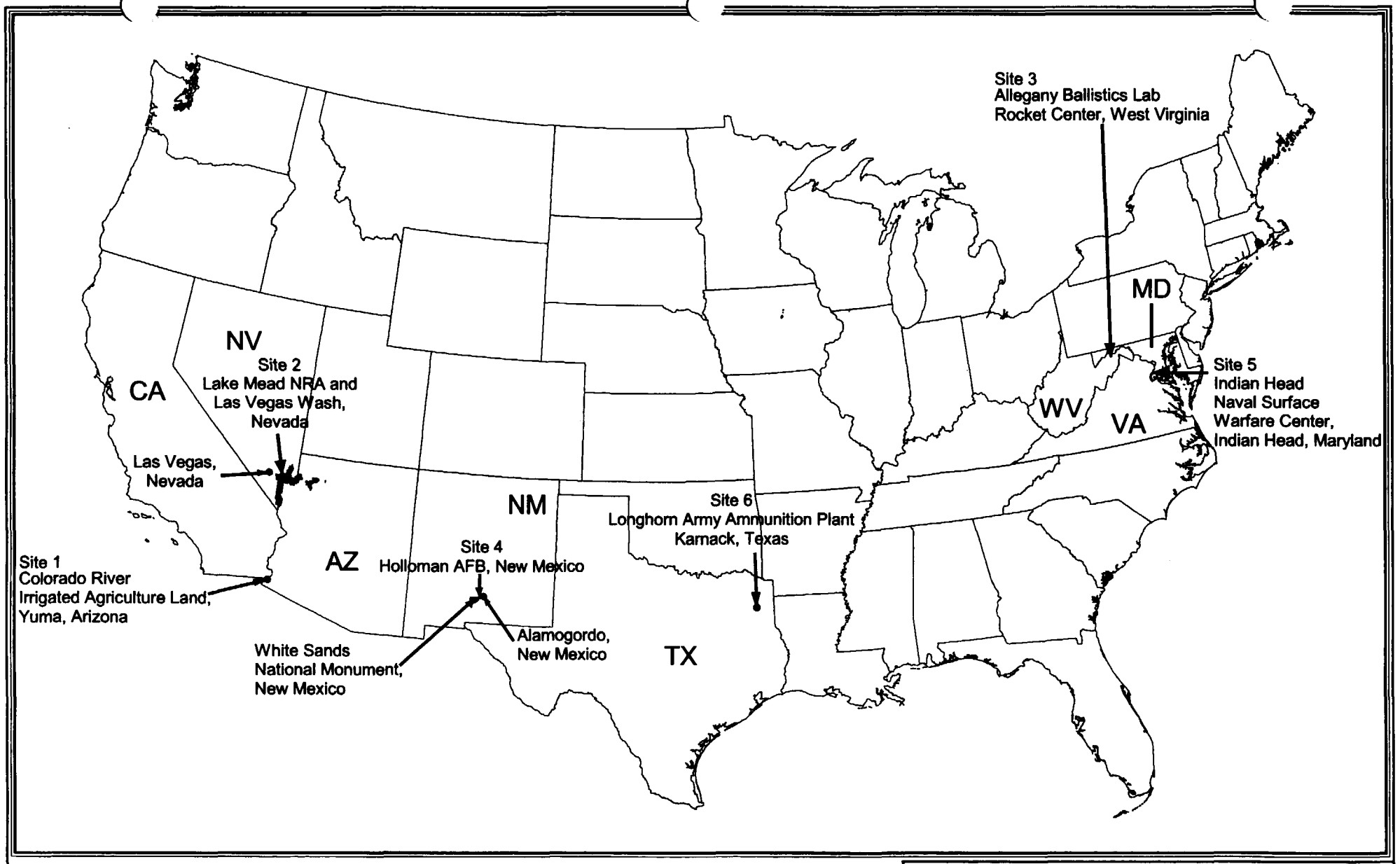

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Figure 1.1

Perchlorate Biotransport Investigation
National Site Locations

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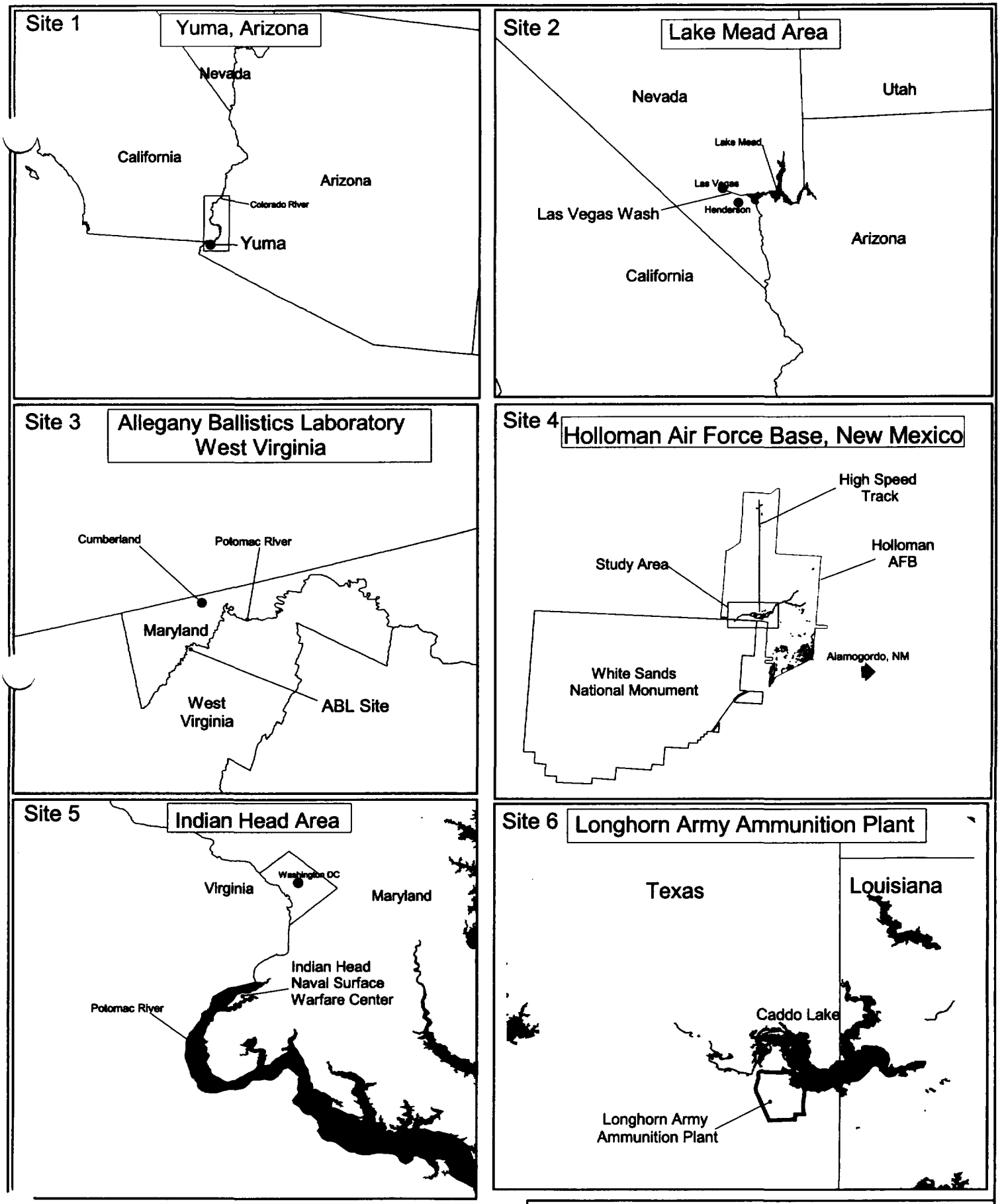

Figure 1.2

Perchlorate Biotransport Investigation
Site Locations

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Table 1.1
Study Sites Representing a Variety of
Source, Discharge and Transport Pathways for Perchlorate

Site	Location	Aquatic System(s)	Sources	Transport Route	Facility Type
1	Yuma, AZ	Irrigation systems Colorado River	Application of fertilizer contaminated with perchlorate and Colorado River contamination from Lake Mead/Las Vegas Site	River water and irrigation wastewater	Agricultural area
2	Lake Mead Area	Las Vegas Wash Lake Mead	Historical seepage from manufacturing facility lagoons	Contaminated groundwater seeps releasing to Las Vegas Wash	Perchlorate manufacturing facility
3	Allegany Ballistics Laboratory	Wastewater discharge Potomac River	Sumps, burn pits, machining of propellants	Wastewater discharge Stormwater runoff Groundwater transport	Research, testing, and production of solid propellant motors
4	Holloman AFB	Perennial pools along the Lost River	Rocket test track, Wash racks	Stormwater runoff Wash water	High speed testing of rocket motors
5	Indian Head Area	Town Gut Marsh Potomac River	Spills, disposal	Wash water, Stormwater runoff	Storage, milling, testing, and burning of perchlorate
6	Longhorn Army Ammunition Plant	Harrison Bayou Goose Prairie Creek	Burn areas, Treated wastewater	Wastewater discharge, Stormwater runoff	Production, testing, and manufacturing of rocket motors

Points of contact at each site provided site history, source information, and direction for sampling location selection during kick-off meetings and preliminary site visits. Site personnel reviewed and commented on the workplan and provided support (*e.g.*, boat and boat operators) for sampling activities at some locations.

Regulators and stakeholders at some of the sites also provided site history, source information, and direction for sampling location selection during kick-off meetings and preliminary site visits. Some regulators and stakeholders also reviewed and commented on the workplan.

1.6 DOCUMENT ORGANIZATION

This document uses the term "site" to describe the six geographic or institutional facilities where perchlorate was studied. The term "location" is used to describe the specific sampling points or areas within each site.

Section 1 of this document outlines project scope and objectives, and provides an introduction to perchlorate and sample locations. Section 2 details study methodologies, including information collection, field methods, laboratory methods (including results of the method development study), data management, and Quality Control (QC) processes. Section 3 presents results of perchlorate analyses in general, and then for different locations and media. Results from the Lake Mead Area are presented in detail to highlight differences in species. Additionally, results from this study are compared to data available from other organizations/agencies collected at the same sites within the same timeframe. Section 4 presents conclusions drawn from data interpretation and makes recommendations for further studies. Patterns of accumulation and transport are described. Several appendices to this document provide raw data.

We wish to thank all the site managers from the Air Force, Army, Navy, Bureau of Land Management, and National Park Service for participation in this study. Their support was crucial in obtaining background information and facilitating the sample collection process.

SECTION 2
INVESTIGATION METHODS

2.1 INITIAL STUDY METHODS

This project was divided into two phases. Phase I consisted of site reconnaissance, workplan development, and the analytical laboratory method development and validation discussed in the workplan (Parsons, October 2000). Phase II consisted of implementing the workplan; including sampling, analysis, data validation, data processing, and production of the final report.

A brief scientific and technical review of existing information on perchlorate sources and dynamics in ecological systems was conducted. This review provided background information and was used to develop the sampling strategy.

AFIERA personnel provided information on sites with known perchlorate contamination and helped shape details of the sampling procedures. AFIERA also provided coordination with site points of contact (POC) to obtain their commitment to participate in this research project and obtain access to their sites.

Telephone conference calls were conducted with POCs from each site to gather knowledge of the sources, estimated extent and levels of perchlorate at each site, and the potential receptors and exposure pathways. Preliminary site visits were conducted to assimilate information from all six sites to produce a comprehensive sampling program that would maximize the field and analytical efforts. Table 2.1 lists the site POCs and other personnel involved at each site in the investigation.

Scientific collection permits were required to collect many of the fauna species for the field effort. These permits were obtained from the U.S. Fish and Wildlife Service and each individual state natural resource agency responsible for permitting. Permits were obtained prior to collection activities. At some sites, minor amendments to the species list were made via telephone conference calls and facsimile correspondence with permit authorities to optimize species and abundance observed during field efforts. Table 2.2 summarizes the scientific collection permit information for this project.

A comprehensive workplan (*Workplan for Perchlorate Biotransport Analysis*, Parsons ES, October 2000) was developed to describe the sampling, shipping, laboratory procedures, quality control, and report preparation phases of the investigation. The workplan contains detailed descriptions of the project goals and tasks performed to fulfill those goals. The workplan was reviewed by AFIERA, site POCs, stakeholders, state regulators, and USEPA personnel. Field work commenced upon receipt of review comments and approval with a list of modifications agreed to by all parties.

Table 2.1
Site POC Information

Site #: Site Name:	1 Yuma, AZ	2 Lake Mead Area, NV	3 Allegany Ballistics Laboratory, WV	4 Holloman AFB, NM	5 Indian Head Area, MD	6 Longhorn Army Ammunition Plant, TX
Information						
Name	Dave Daniels	Bill Burke	Mr. Dominic O'Conner	Fred Fisher	Mr. Carey Yates	Cyril Onewokae
Organization	Bureau of Land Mgmt	National Park Service	NAVFAC	Holloman AFB	NSWC, Indian Head	OSC/MAC
Group/Division	Yuma Office	Lake Mead NRA	Envr Eng	49 CES	Envr Eng	--
Phone	520 317 3228	702 293 8935	757 322 4795	505 572 5062	301 744 2271	309 782 1350
Email	dave_daniels@blm.gov	william_j._burke@nps.gov	oconnerdt@efdlant.navfac.navy.mil	Fred.Fisher@Holloman.af.mil	yatesca@ih.navy.mil	OnewokaeC@OSC.Army.mil
Name		Brenda Pohlmann	David McBride		Jeff Bosser	David Tolbert
Organization		NDEP	ABL		NSWC, Indian Head	LHAAP
Group/Division		--	--		Facility Biologist	Envr Protection Specialist
Phone		702 486 2857	304 726 5354		301 744 4705	318 459 5109
Email		--	--		--	D_Tolbert@msn.com
Name		Kim Zikmond				
Organization		SNWA				
Group/Division		--				
Phone		702 862 3880				
Email		--				

ABL = Allegany Ballistics Laboratory
LHAAP = Longhorn Army Ammunition Plant
POC = Point of contact
NRA = National Recreation Area
SNWA = Southern Nevada Water Authority
NDEP = Nevada Department of Environmental Protection

Table 2.2
Collection Permit Information

Site #: Site Name:	1 Yuma, AZ	2 Lake Mead Area, NV	3 Allegany Ballistics Laboratory, WV	4 Holloman AFB, NM	5 Indian Head Area, MD	5 Indian Head Area, MD	6 Longhorn Army Ammunition Plant, TX
State							
POC		Bill Burke	Barbara Sargent	Gregg Schmitt	Mary Jo Scanlan	Tamara O'Connell	Rosie Roegner
Organization	Arizona Game and Fish Department	National Park Service	WV Dept. of Natural Resources and Natural Heritage Program	NM Dept. of Game and Fish	MD Dept. of Natural Resources	MD Dept. of Natural Resources	Texas Parks and Wildlife Department
Group/Division		Nevada Division of Wildlife	---	---	Wildlife Division	Fisheries Service	---
Phone		(702) 293-8951	(304) 637-0245	(505) 827-9925	(410) 260-8545	(410) 260-8323	(512) 389-4800
Fax			(304) 637-0250	---	---	(410) 260-8279	---
Permit #	SP563482	WEST-09-00 S20069	180-2000	3057	SCO-30848	SCP-200074	SPR-0494-681
Permittee	Ed West	Ed West	Michael Sipos	Michael Sipos	Michael Sipos	Michael Sipos	Randy Palachek
Expiration	12/31/2000	9/30/2000 8/31/2000	8/31/2000	12/31/2000	12/31/2000	12/31/2000	--
Amendments	--	--	Dated: 8/22/2000	--	Dated: 7/18/2000	Dated: 8/28/2000	Dated 7/12/2000
Annual Rpt Submitted	Yes	Yes	Yes (submitted on 12/6/2000)	Yes (submitted on 12/6/2000)	Yes (submitted on 12/6/2000)	Yes (submitted on 12/6/2000)	No (submitted 5/30/01)
Federal							
POC	Tamela Tate-Hall	Tamela Tate-Hall	Kamile McKeever	Kamile McKeever	Kamile McKeever	Kamile McKeever	Kamile McKeever
Organization	USFWS	USFWS	USFWS	USFWS	USFWS	USFWS	USFWS
Group/Division	Migratory Bird Permits Office	Migratory Bird Permits Office	Migratory Bird Permits Office	Migratory Bird Permits Office	Migratory Bird Permits Office	Migratory Bird Permits Office	Migratory Bird Permits Office
Phone	--	--	(505) 248-7882	(505) 248-7882	(505) 248-7882	(505) 248-7882	(505) 248-7882
Fax	--	--	(505) 248-7885	(505) 248-7885	(505) 248-7885	(505) 248-7885	(505) 248-7885
Permit #	MB029781-0	MB029781-0	MB030375-0	MB030375-0	MB030375-0	MB030375-0	MB030375-0
Permittee	Ed West	Ed West	Michael Sipos	Michael Sipos	Michael Sipos	Michael Sipos	Michael Sipos
Expiration	12/31/2002	12/31/2002	12/31/2002	12/31/2002	12/31/2002	12/31/2002	12/31/2002
Amendments	MB029781-1	MB029781-1	MB030375-1 Dated: 8/22/2000	MB030375-1 Dated: 8/22/2000	MB030375-1 Dated: 8/22/2000	MB030375-1 Dated: 8/22/2000	MB030375-1 Dated: 8/22/2000
Annual Rpt Submitted	Yes	Yes	Yes (submitted on 12/6/2000)	Yes (submitted on 12/6/2000)	Yes (submitted on 12/6/2000)	Yes (submitted on 12/6/2000)	Yes (submitted on 12/6/2000)

POC = Point of contact
USFWS = U. S. Fish and Wildlife Service

Table 2.3 illustrates the schedule for field sampling events at each site during the summer and fall of 2000. Meteorological conditions were typical for each region preceding and during the sampling efforts.

2.2 FIELD STUDY METHODS

This section describes the methods utilized in the field to gather the samples and information required for the investigation. Details on sampling schedule, site and location information, collection methods, a summary of all samples collected, photo documentation of field activities, and documentation of anomalies observed are provided in the following sections.

2.2.1 Sampling Sites and Locations

The six sampling sites are shown in relation to the contiguous United States on Figure 1.1. Figure 1.2 provides a more detailed area map for each of the six sites. Each site has an 11-inch by 17-inch drawing showing the sample locations and major features at the site. Table 2.4 lists the sample sites and locations discussed below.

2.2.1.1 Site 1 - Yuma, Arizona

Yuma, Arizona is located in the southwestern corner of Arizona near the borders of California and Mexico. Figures 1.1 and 1.2 provide general illustrations of the area surrounding the sampling site. Yuma is the third largest metropolitan area in Arizona with a 1999 year-round metropolitan statistical area population of 105,000, with approximately 89,000 visitors per year. The Yuma Field Office of the U.S. Department of Interior's Bureau of Land Management (BLM) encompasses 1.6 million acres of public land in Arizona and California.

The Colorado River flows through the City of Yuma before flowing into Mexico. The Colorado River basin is widely used in the area for irrigated farming through a series of water distribution canals. Otherwise, the area is a mix of desert and desert mountains with less than 3 inches of average rainfall per year.

The potential perchlorate sources at this site are irrigation water from the Colorado River which has exhibited low concentrations of perchlorate from upstream sources (see Site: 2: Lake Mead National Recreation Area and Las Vegas Wash) and potential runoff contamination from agricultural use of fertilizers which could contain ammonium perchlorate. There is no evidence to suggest that fertilizer used in these areas contains (or ever contained) perchlorate, however there is historical evidence that some fertilizers may be contaminated with perchlorate. The extent of contamination in fertilizer has not been studied.

Table 2.3
Schedule of Sampling Events

Site Name	Site Number	Sampling Dates
Yuma, Arizona	1	Monday 7/31/00 to Friday 8/4/00
Lake Mead NRA and the Las Vegas Wash, Nevada	2	Saturday 8/12/00 to Wednesday 8/16/00
Allegany Ballistics Laboratory, West Virginia	3	Monday 8/21/00 to Thursday 8/24/00
Holloman AFB, New Mexico	4	Monday 9/18/00 to Wednesday 9/20/00
Naval Surface Warfare Center, Indian Head, Maryland	5	Friday 8/25/00 to Wednesday 8/30/00
Longhorn Army Ammunition Plant, Karnack, Texas	6	Monday 9/11/00 to Friday 9/15/00

Table 2.4
Site and Location Information

Site Name	Site No	Location No	Location Name
Yuma, AZ	1	1	Palo Verde Lands
	1	2	Palo Verde Drain
	1	3	Imperial Dam
	1	4	Agriculture Lease 1
	1	5	Agriculture Lease 2
Lake Mead Area, NV	2	1	Spring
	2	3	ECS3-Upstream of Lake Las Vegas
	2	5	Lake Mead, Las Vegas Delta Area
Allegany Ballistics Laboratory, WV	3	1	Burn Area
	3	2	SWMU 37L
	3	3	SWMU 37E
	3	4	SSL2
	3	5	Upstream of Weir (WWTP)
	3	6	Downstream of Weir (WWTP)
	3	7	Storm Drain
	3	8	CERCLA Site 1 Outfall
Holloman AFB, NM	4	0	Monitoring Wells
	4	1	South Bldg 1176
	4	2	East Bldg 1176
	4	3	South of Test Track
	4	4	First Lost River
	4	5	Second Lost River
	4	6	Third Lost River
	4	7	Fourth Lost River
Indian Head Area, MD	5	1	LVOA West (West of Bldg 1018)
	5	2	LVOA East (East of Bldg 1018)
	5	3	West Creek of Town Gut Marsh
	5	4	East Creek of Town Gut Marsh (at IW85)
	5	5	Town Gut Marsh 1 @ Atkin's Road
	5	6	Town Gut Marsh 2 @ Atkin's Spur Road
	5	7	Town Gut Marsh 3 @ Noble Rd
	5	8	Creek East of Town Gut Marsh 2
Longhorn Army Ammunition Plant, TX	6	1	Building 25-C
	6	2	INF Pond
	6	3	Discharge Point on Harrison Bayou
	6	4	Goose Prairie Creek (between 51st St and
	6	5	Harrison Bayou downstream (Sampling Site 9)

The five sampling locations were selected at points of irrigation water withdrawal from the Colorado River, at a return drain from irrigated and fertilized farmlands, and one site near a public use area at Imperial Lake. Figure 2.1 shows the sampling area and sample locations for the Yuma site along the Colorado River from Yuma, Arizona to Blythe, California. Location 1 is along the Colorado River at an agriculture lease east of Palo Verde, California. Location 2 is at the irrigation return flow canal near Palo Verde, California which collects water and run-off from over-irrigated fields. Location 3 is along the shore of Imperial Reservoir near the BLM concession on the east shoreline, approximately 800 feet upstream from Imperial dam. Location 4 is at the BLM Agriculture Lease 1, immediately upstream of Laguna Dam. Location 5 is at the BLM Agriculture Lease 2, downstream from Laguna Dam.

The natural environment surrounding Yuma is a mix of desert and desert mountains. Vegetation communities along the river include cattail (*Typha* spp.), willow (*Salix* spp.), and cottonwood (*Populus fremontii*). In drier areas, arrowweed (*Pluchea sericea*), and screwbean mesquite (*Prosopis pubescens*) are mixed with the cottonwood and willow. Farther from the river, honey mesquite (*Prosopis glandulosa*), saltbush (*Atriplex* spp.), and quailbush (*Atriplex lentiformis*) are typical.

2.2.1.2 Site 2 – Lake Mead National Recreation Area and the Las Vegas Wash

Lake Mead National Recreational Area (NRA) contains over 1.5 million acres within its legislated boundaries, approximately 181,000 acres of which are surface waters within Lake Mead and Lake Mojave. The recreation area recorded over 10 million recreational visits in 1997. Lake Mead NRA is adjacent, on its western border, to the City of Las Vegas, Nevada, and at its southern border to the Laughlin, Nevada-Bullhead City, Arizona urban areas. Development in these areas is occurring rapidly. Lake Mead NRA's eastern border is the Grand Canyon (Lake Mead, December 1999).

The NRA lies within two geologically diverse areas. The area most utilized by visitors lies west of the Grand Wash Cliffs within the Basin and Range Province. It is a north-south trending series of isolated mountain ranges and basins featuring alluvial fans, vast bajadas and playa deposits of silts, clays, and gravels. Extensive igneous intrusions with interspersed granitic- or volcanic-capped mountain blocks are present (Lake Mead, December 1999). Elevations vary from 517 feet to 5,639 feet above sea level.

Three of the four North American deserts converge in this area, with vegetation and faunal representatives of the Mojave, Sonoran, and Great Basin Deserts present within the Lake Mead NRA. The area centers on the two reservoirs, Lake Mead and Lake Mojave, which together represent over 142 miles of the former river channel of the Colorado River (Lake Mead, December 1999).

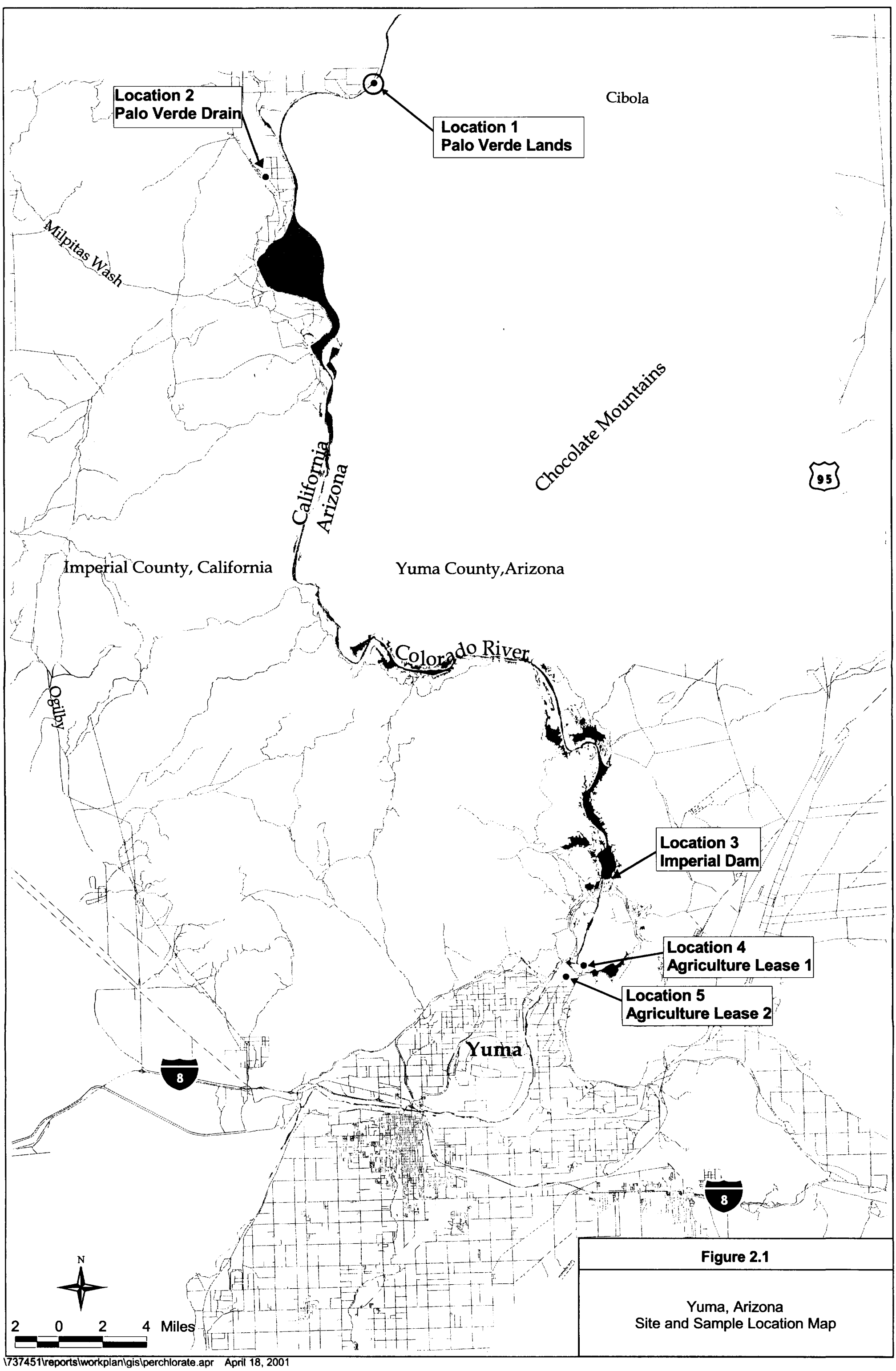

Figure 2.1

Yuma, Arizona
Site and Sample Location Map

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Plant communities associated with Las Vegas Wash include creosote bush (*Larrea tridentata*), saltbush, and salt cedar (*Tamarix* spp.), as well as typical marsh and riparian communities. The creosote bush community occurs primarily in the undisturbed and lightly disturbed desert environment on the uplands surrounding the wash. Saltbush occurs in areas of wet saline soils and supports a community of shadscale (*Atriplex confertifolia*), wingscale (*Atriplex canescens*), cattle spinach (*Atriplex polycarpa*), and quailbrush (*Atriplex lentiformis*). Salt tolerant species such as inkweed (*Sueda torreyana*) and pickleweed (*Allenrolfea occidentalis*) dominate the more saline portions of the community. Saltgrass (*Distichlis spicata*) is occasionally found as an understory species. Stands of cattail, bullrush (*Scirpus* spp.) and phragmites (*Phragmites australis*) characterize an open marsh in the Wash. Extensive stands of salt cedar are also found along the Wash. Cheesbush (*Hymenochlea salsola*), mesquite (*Prosopis* spp.), catclaw (*Acacia greggii*), desert willow (*Chilopsis linearis*) and salt cedar characterize the riparian areas of the Wash.

The Las Vegas Wash plays an important role in the environmental and water resource issues facing southern Nevada. The Las Vegas Wash is the primary outfall for all stormwater flows, landscape and surface street runoff, treated wastewater flows, and shallow groundwater seepage in the Las Vegas Valley (Las Vegas Wash, May 2000). The Wash presently flows at approximately 160 million gallons per day (MGD) and is projected to increase to 300 MGD with increased wastewater discharges over the next 30 years. Figure 1.2 provides a general illustration of the Lake Mead NRA, the City of Las Vegas, and the Las Vegas Wash. Figure 2.2 shows the sampling area and sample locations for this project.

The predominant source of perchlorate at this site is contaminated groundwater seepage which has been traced to historical discharges from a perchlorate manufacturing facility south of the Wash. Perchlorate has been detected in the groundwater seeps at concentrations up to approximately 100,000 μg/L (Kerr-McGee, 1998).

Beginning in November 1999, Kerr McGee Corporation began operating an ion exchange water treatment system at the location where contaminated shallow groundwater surfaces in the Wash. The treated water and any water exceeding the flow capacity of the present treatment system travels via surface drainage into the Wash where it mixes with other seepage, and treated wastewater flows. Water in the Wash then flows just above Lake Las Vegas where it is diverted into a bypass tunnel (underneath Lake Las Vegas) and discharges immediately below the Lake Las Vegas Dam. Below the dam, the flow follows the original Wash pathway and drains into the Las Vegas Bay arm of Lake Mead located at a large delta created by sediment from the Wash.

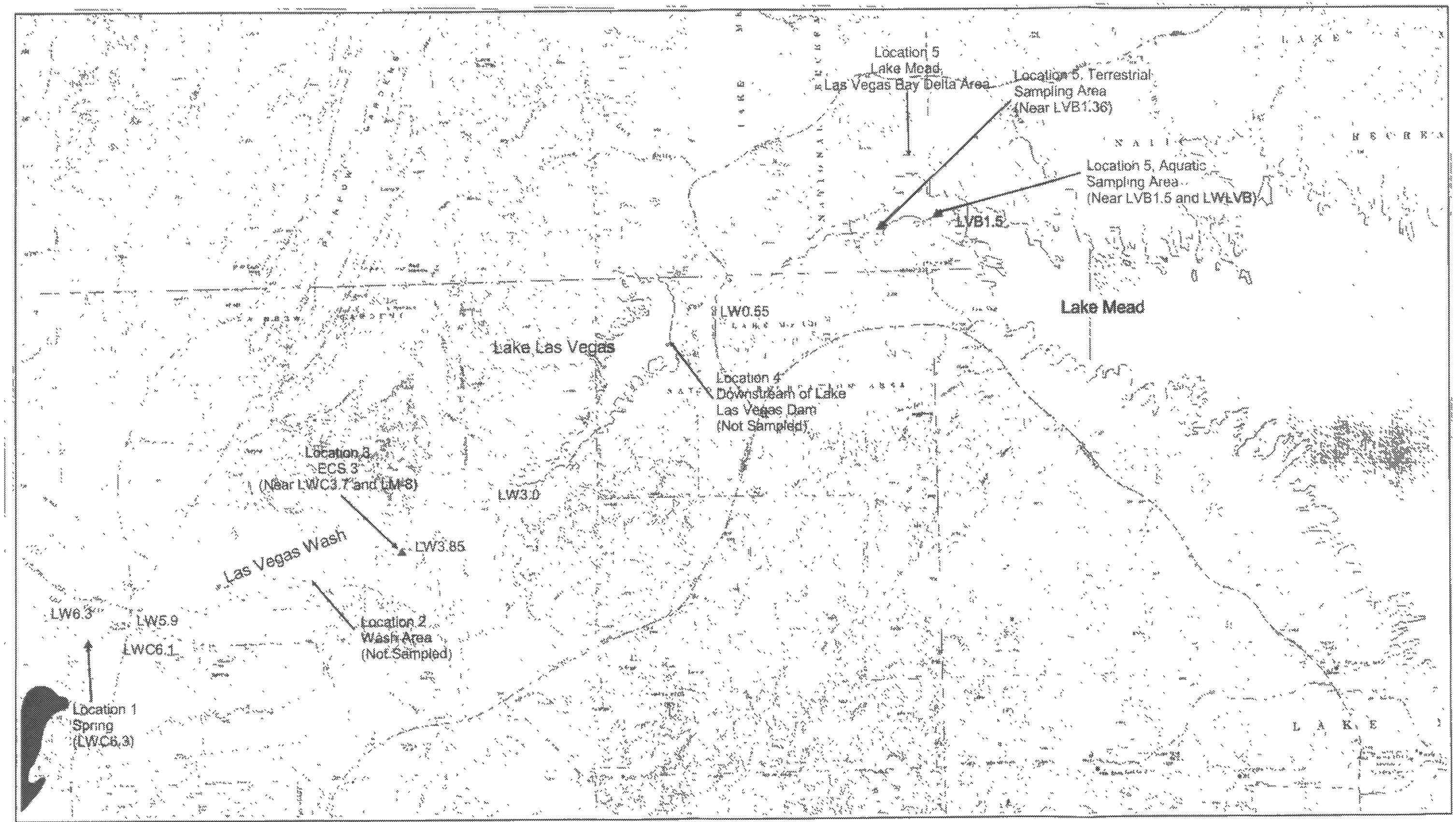

Figure 2.2

Lake Mead National Recreation Area
and the Las Vegas Wash, Nevada
Site and Sample Location Map

The Southern Nevada Water Authority (SNWA) performs routine perchlorate sampling along the Wash and in Lake Mead. Historical sampling shows perchlorate concentrations up to 64 µg/L in the Wash upstream of the contaminated groundwater seeps associated with the manufacturing facilities. Historical sampling at the SNWA raw drinking water intake (located at depth) at Saddle Island in Lake Mead exhibited perchlorate concentrations up to 45 µg/L.

The sample locations for this study were selected to reflect a gradient of perchlorate concentrations. Samples collected near the source of perchlorate represented the highest concentration, while decreasing concentrations were found as samples were collected further from the source. Samples were collected along the Wash and into Lake Mead to assess concentrations in different media and biotransport potential along the path of flow.

Samples collected at Location 1 of this site were actually collected at three sub-locations. Location 1 UP (upstream) is the surface discharge pond of one of the major groundwater seeps from the Kerr McGee contaminant plume. This is considered the primary point source for perchlorate into the Wash. This is also the location of the intake for the ion exchange treatment plant. The actual sample location is the upstream (west) side of the concrete dam/intake structure for the ion exchange wastewater treatment plant. Also, included with the data from Location 1 UP are two fish samples and two water samples which were collected at an impoundment created by a dirt road located approximately 150 feet upstream of the ion exchange treatment plant intake. Location 1 DN (downstream) is the downstream side (east) of the concrete dam/intake structure for the ion exchange wastewater treatment plant.

The figures and tables of this report use Location 1 UP data unless otherwise noted. These upstream data are being used because they are most representative of the concentrations that plants and animals in the area are subjected to. In this sample area, the marshes created by the water of Location 1 UP cover approximately 0.5 acre, while the ponded area of Location 1 DN covers only approximately 500 square feet. In addition, most of the samples collected at this location were obtained closer to Location 1 UP water. The perchlorate concentration in Location 1 DN would be more indicative of the concentration for the spring discharge into the Wash (after treatment); however, it is likely there are other groundwater pathways directly into the Wash.

Locations 2 and 4 were omitted from the final workplan to allow increased sample collection at the remaining locations. Location 3 is at the erosion control structure ECS 3, on the Wash, upstream of Lake Las Vegas.

Location 5 samples were collected on and around the sediment delta created by the Wash in the Las Vegas Bay area of Lake Mead. Most samples collected at Location 5 were from the terrestrial sample location which is the south shoreline and escarpment in the vicinity of the existing SNWA sample location LVB1.36M. Many of the aquatic samples were collected from Las Vegas Bay in the vicinity of existing SNWA sample location LVB1.5M and LWLVB.

2.2.1.3 Site 3 – Allegany Ballistics Laboratory, Rocket Center, West Virginia

Allegany Ballistics Laboratory (ABL) is located in Rocket Center, West Virginia, approximately 9 miles south of Cumberland, Maryland (Figures 1.1 and 1.2). The facility is a research, development, testing, and production center for solid propellant motors for ammunition, rockets, and armaments. ABL has been operational since the 1940s. The surrounding natural habitat is characterized by mountain vegetation communities including vast deciduous forests composed of a variety of oaks, maples, beech, poplar, and other hardwoods. Figure 2.3 shows the sampling area and sample locations for the ABL site.

Potential perchlorate sources at this site are a series of outdoor burn areas at the north end of the facility, Solid Waste Management Unit (SWMU) 37L, SWMU 37E (a former burn pit area), and the sanitary wastewater treatment building area. Location 1 includes the areas immediately surrounding three burn pans (Pans D, E, and F) in the burn area. This area also includes the location of three historic disposal pits where perchlorate waste was released onto the ground to percolate into the soil. Location 2 is the former sump area where machining of propellants containing perchlorate occurred. This area is identified as SWMU 37L. Location 3 is the former sump area adjacent to an area where machining of propellants containing perchlorate occurred. This area is now identified as SWMU 37E. Location 4 is the existing ABL sampling location SSL-2, which is the outfall for most drainage and discharge from ABL to the Potomac River. This location includes areas where surface water or groundwater from listed sites may have migrated. Location 5 is the weir overflow from the sanitary wastewater treatment plant. Location 6 is downstream of the 10-inch diameter polyvinyl chloride (PVC) pipe discharge for treated sanitary wastewater. Location 7 is the storm drain which collects runoff from the SWMU 37L area. Location 8 is located at the outfall of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Installation Restoration Site 1 pump and treat facility. This facility collects groundwater throughout Site 1 and discharges the treated water into the North Branch Potomac River. The Site 1 pump and treat facility was designed and installed under CERCLA guidelines to remove other known contaminants from Site 1. Therefore, the pump and treat facility at Site 1 does not currently remove perchlorate from the groundwater prior to discharge.

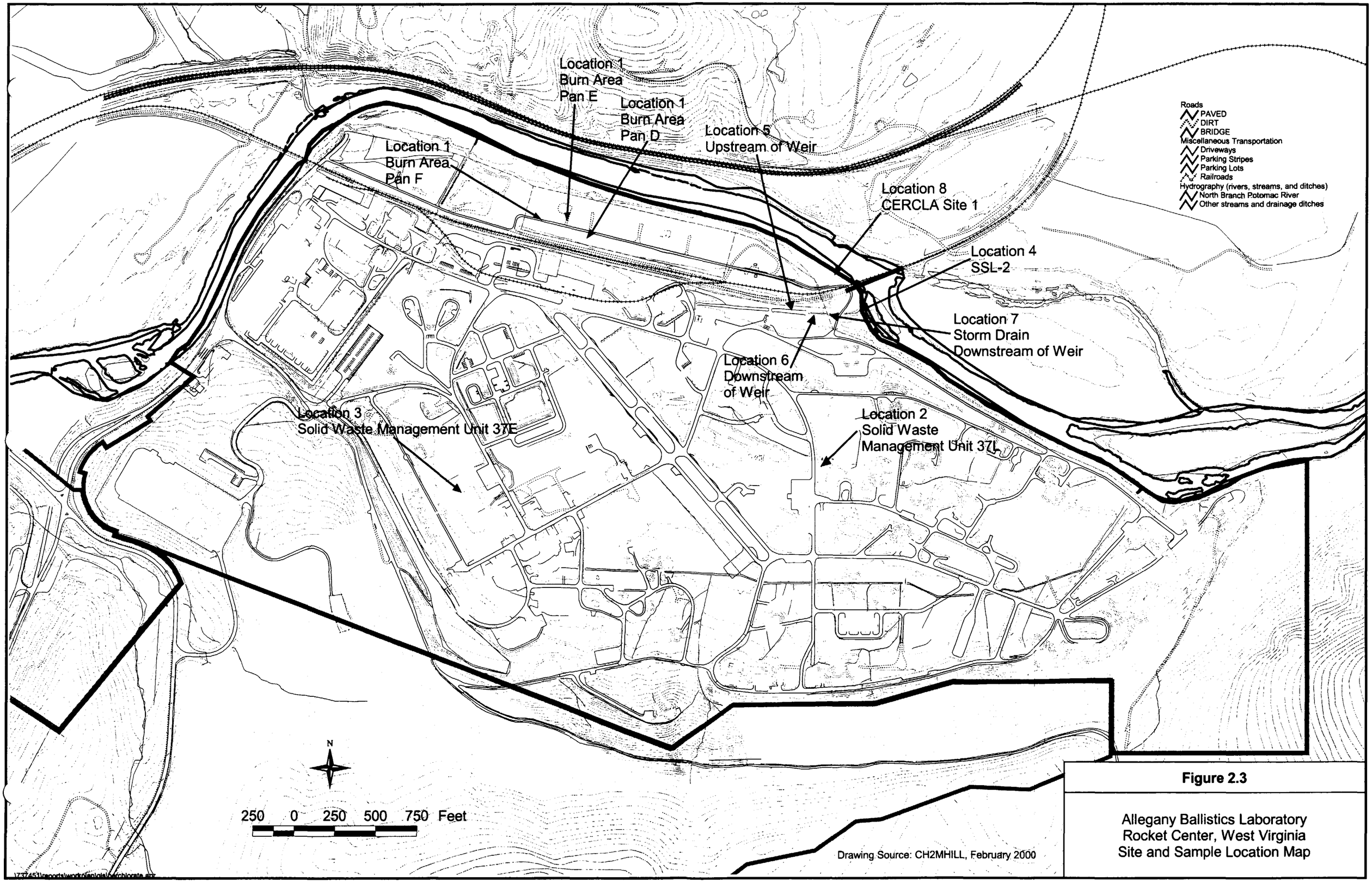

Figure 2.3

Allegany Ballistics Laboratory
Rocket Center, West Virginia
Site and Sample Location Map

2.2.1.4 Site 4 – Holloman AFB

Holloman Air Force Base (Holloman) is located in Otero County, in southeastern New Mexico, approximately 6 miles west of Alamogordo, NM (see Figures 1.1 and 1.2). Since 1942, Holloman Air Force Base (AFB) has played a decisive role in U. S. national security. Clear skies, a moderate climate, and low population densities provide a highly favorable environment for nearly year-round training and military readiness operations.

Holloman is currently home to the 49th Fighter Wing and provides important research and testing integral to maintaining military excellence. The primary mission of the 49th Fighter Wing is to support national security objectives with the F-117A Nighthawk stealth fighter. Holloman is home to a number of tenant units, including the German Air Force Flying Training Center, the 46th Test Group, the 4th Space Surveillance Squadron, the Phillips Laboratory Balloon Division, and the Primate Research Institute. Many of the tenant units have research and development missions that date from the early days of the Air Force, including missile development and testing, high speed rocket sled tests, high altitude balloon projects, and Aeromedical Field Laboratory experiments. The Primate Research Facility trained the first chimpanzee (HAM) to make a suborbital flight, and the first chimpanzee (Enos) to orbit the earth (Holloman, January 2000).

White Sands National Monument, administered by the National Park Service, is located to the southwest of, and contiguous with, portions of the western border of the base. Over 500,000 visitors each year utilize the park's recreational facilities.

Holloman is located in a semi-arid region within the northern portion of the Chihuahuan Desert (Schmidt, 1986). It is uniquely situated at the center of the Tularosa Basin within easy reach of several ecologically distinct areas. Managed areas located nearby include volcanic peaks and mesas covered in high mountain grasslands and conifer forests, dropping to desert shrublands, with some cactus and yucca. Vegetation within the base is dominated by four-wing saltbush (*Atriplex canescens*) and alkali sacaton grass (*Sporobolus airoides*). Other common plants are honey mesquite (*Prosopis glandulosa)*, creosote bush (*Larrea tridentata)*, salt cedar (*Tamarix ramosissima*), pickleweed (*Allenrolfea occidentalis)*, and Indian saltgrass (*Distichlis spicata*).

Surrounding mountain fronts vary in vegetation cover from xerophytic shrublands to juniper-pinyon savannas, on high mountain meadows. Dominating the area are diverse, broad expanses of lowland desert environments, including gypsum dunelands punctuated by playas and ephemeral drainages.

Seasonal fluctuation in precipitation rates is a result of prevailing wind directions, which can bring in frontal storms from the north, or Pacific or Caribbean cyclonic systems. Holloman averages 8.58 inches of rainfall annually. Nearly half this amount falls within the months of July through September, known as the summer monsoons. Monsoon thunderstorms are generally short in duration and high in intensity.

There are no natural perennial streams within Holloman AFB or in the nearby surrounding landscape; however, the Lost River is an intermittent stream with perennial

pools. The pools are located in the final one-third of Lost River, near the confluence of Ritas and Malone Draws.

The White Sands pupfish, endemic to the Tularosa Basin, inhabits clear, shallow, strongly alkaline pools and streams with fine mud-silt and sand bottoms. The pupfish are limited to water bodies at Holloman and White Sands Missile Range.

The White Sands pupfish is a species of concern at Holloman (formerly a Federal Category 2 species). Its protection is the responsibility of the New Mexico Department of Game and Fish (NMDGF). It has no legal protection under the Endangered Species Act, but it could be listed under the Act if the species fails to be managed properly under a 1994 cooperative agreement. The species is classified as State Endangered, Group 2, and was first listed on 24 January, 1975 (NMDGF Reg. 563). This designation means that prospects of survival or recruitment of the species in the state are likely to be in jeopardy within the near future (NMDGF, 2000). In addition, this species is considered a species of concern by the American Fisheries Society (Williams *et al.*, 1989). It is listed in The Nature Conservancy's global ranking protocol as G1, meaning it is critically imperiled globally because of extreme rarity or because of some factor(s) making it especially vulnerable to extinction (Biological Conservation Database, 1998). The New Mexico Natural Heritage Program ranks the White Sands pupfish as S1, meaning it is critically imperiled in New Mexico because of extreme rarity (Biological Conservation Database 2000).

Figure 2.4 shows the middle western area on base (south of the test track), and shows the sample locations for this project. Perchlorate sources at the Holloman site are near the test track area. This area is used for high speed tests of rocket motors. Contamination sources include runoff from storage of spent rocket motors and drainage pathways conveying washwaters and runoff from the test track area.

Sampling was concentrated in areas downgradient of the test track and storage areas, with continued sampling along the Lost River and into the sand dunes towards the boundary with White Sands National Monument. Location 1 is downgradient of the storage area for spent rocket motors, south/southwest of Building 1176. Location 2 is along the drainage feature heading from the test track to the playa, east of Building 1176. Location 3 was not sampled. Location 4 is the farthest upstream sampling location of the Lost River as it forms at the end of the playa and heads west towards the White Sands National Monument. Location 5 is on the Lost River approximately 1,800 feet downstream from Location 4. This location corresponds to one of the White Sands pupfish trapping locations being studied by Holloman personnel (called pupfish 11). Location 6 is also on the Lost River approximately 2,500 feet downstream from Location 5 and corresponds to one of the White Sands pupfish trapping locations (called pupfish 8). Location 7 is also on the Lost River approximately 1,800 feet downstream from Location 6 and corresponds to one of the White Sands pupfish trapping locations (called pupfish 18).

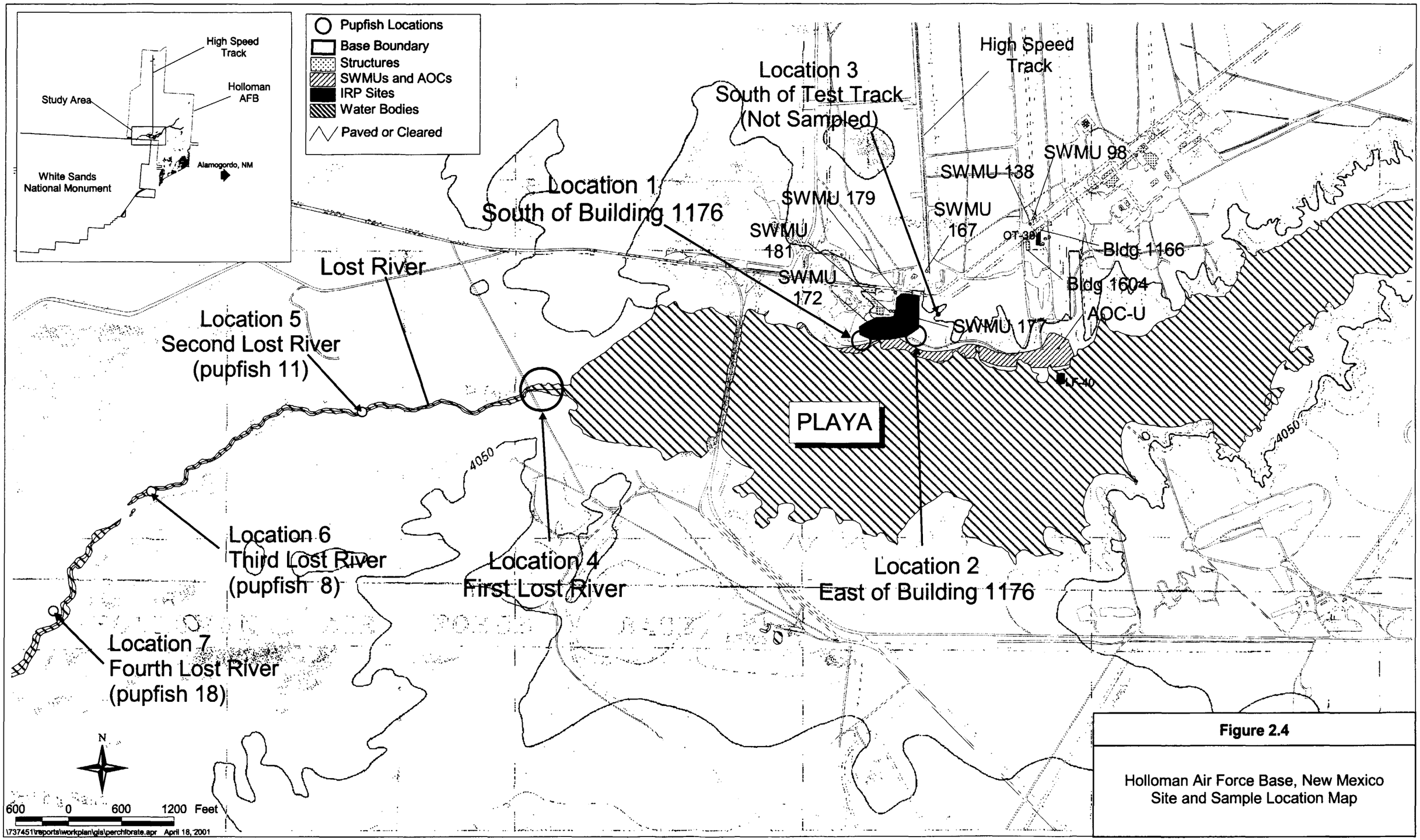

Figure 2.4

Holloman Air Force Base, New Mexico
Site and Sample Location Map

2.2.1.5 Site 5 – Naval Surface Warfare Center, Indian Head, MD

Indian Head Naval Surface Warfare Center (Indian Head) is located approximately 30 miles south of Washington D.C., just east of the Potomac River, and is bounded by Mattawoman Creek on the east (Figures 1.1 and 1.2). The Indian Head facility began as a proving ground for naval guns in 1890. Indian Head now focuses on research, design, testing, engineering, manufacturing, safety training, and support for energetics for the Naval Surface Warfare Center.

Milling, testing, and burning of perchlorate provide the source of contamination at Indian Head. This investigation focused on the Low Vulnerability Ordnance Ammunition (LVOA) area and the Town Gut Marsh system. Figure 2.5 shows the sampling area and sample locations for the Indian Head site. The LVOA area is used for mixing perchlorate, and the Town Gut Marsh series of ponds are immediately downstream of the LVOA area. This area was chosen because the LVOA area is known to be contaminated with perchlorate, and the three ponds and surrounding land making up Town Gut Marsh provide good habitat for biological species.

Sampling locations at this site were selected to provide characterization of the perchlorate source area and a series of small water impoundments. No samples were collected at either Location 1 or 2 because no water was flowing from either location at the time of sampling. Location 3 is the west creek of Town Gut Marsh, just before entering Town Gut Marsh. Location 4 is the existing IW85 sampling location on the east creek of Town Gut Marsh. Location 5 is the lower area of the first impoundment of Town Gut Marsh. This is immediately upstream of the Atkins Road crossing of Town Gut Marsh. Location 6 is the lower area of the second impoundment of Town Gut Marsh and is immediately upstream of the Atkin's Spur Road crossing of Town Gut Marsh. Location 7 is the lower area of the third impoundment of Town Gut Marsh and is immediately upstream of the Noble Road crossing of Town Gut Marsh. Location 8 is in the creek entering the Town Gut Marsh pond system south of Atkins Road from the northeast and is located approximately 60 feet northwest of the sign identifying IW82.

2.2.1.6 Site 6 – Longhorn Army Ammunition Plant, Karnack, TX

Longhorn Army Ammunition Plant (LHAAP) is a former government-owned, contractor-operated and maintained industrial facility under the jurisdiction of the United States Army Industrial Operations Command (IOC). There is currently no manufacturing/operating contractor at the LHAAP facility. The facility is inactive and scheduled to be excessed (Sverdrup, April 2000).

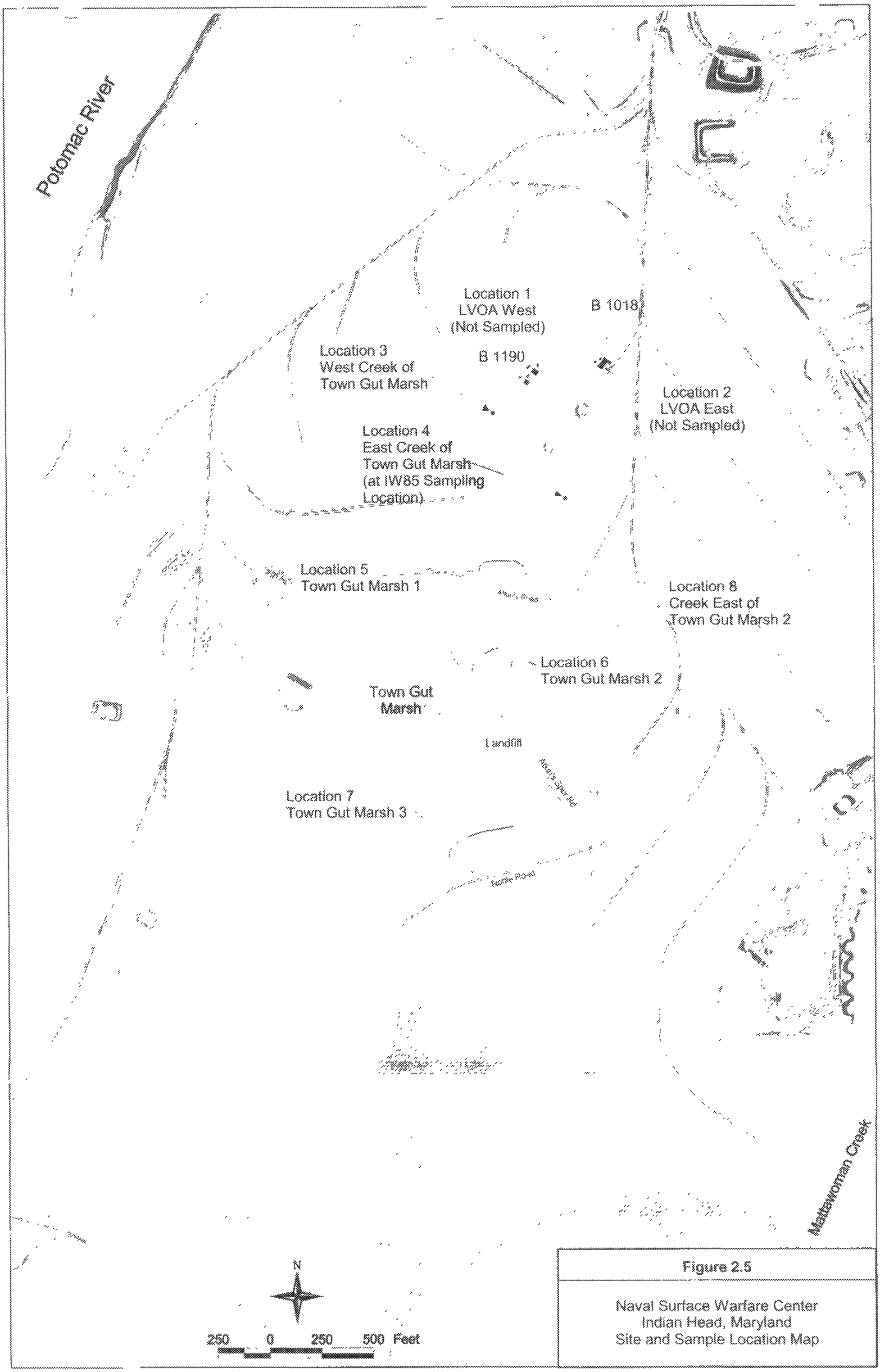

Figure 2.5

Naval Surface Warfare Center
Indian Head, Maryland
Site and Sample Location Map

location. The number of samples collected at other sampling locations was increased to compensate.

Location 2 is the INF Pond where water is held after treatment at the new pump and treat system operating near Burning Ground No. 3. Location 3 is the discharge location for treated wastewater from the INF Pond (Location 2). The discharge enters Harrison Bayou just south of Avenue Q. Location 4 is along Goose Prairie Creek at Karnack Avenue. This creek collects drainage from the Plant 3 manufacturing area (including Location 1). Location 5 is on the downstream end of Harrison Bayou near its entrance to Caddo Lake. This location is downstream of the INF Pond (Location 2), the discharge point (Location 3), and Burning Ground No. 3. This location also corresponds to a site being studied by LHAAP personnel (called sampling site #9).

2.2.2 Summary of Sample Collection Methods

A wide assortment of collection methods were necessary to obtain the abiotic and biotic media samples for this project. Over 960 samples were collected, and this section provides a brief description of the methods which were employed during the sampling efforts. Table 2.5 summarizes the procedures for sample collection, handling, and shipping. The following subsections provide more specific information on sampling methods for each sample medium. The workplan (Parsons, 2000) provides further detail on sample collection methods and health and safety issues for the investigation. In general, it was not possible to precisely co-locate samples (e.g. soil and animal tissue samples). However, samples were generally co-located within a sampling location based on the ease of sample collected and the presence of biological media.

2.2.2.1 Soil

Soil samples were collected directly into a sample container or with a trowel from approximately the top 3 inches of the soil column.

2.2.2.2 Surface Water

Discrete surface water samples were collected from approximately 1 foot below water surface using a dipper (when water depth permitted) and placed directly into the sample container. Samples were collected in a manner that ensured undisturbed conditions at the point of sampling (sample collected upstream of path walked to sample location and collected facing upstream). Surface water measurements (pH, dissolved oxygen, conductivity, and temperature) were collected in the same manner, after collection of the laboratory sample.

2.2.2.3 Sediment

Discrete sediment grab samples were collected from the top ½-inch to 1-inch of sediment in the water body and placed directly into the sample container. Typically three discrete samples were collected from the water body so a representative sample of the entire water body was obtained.

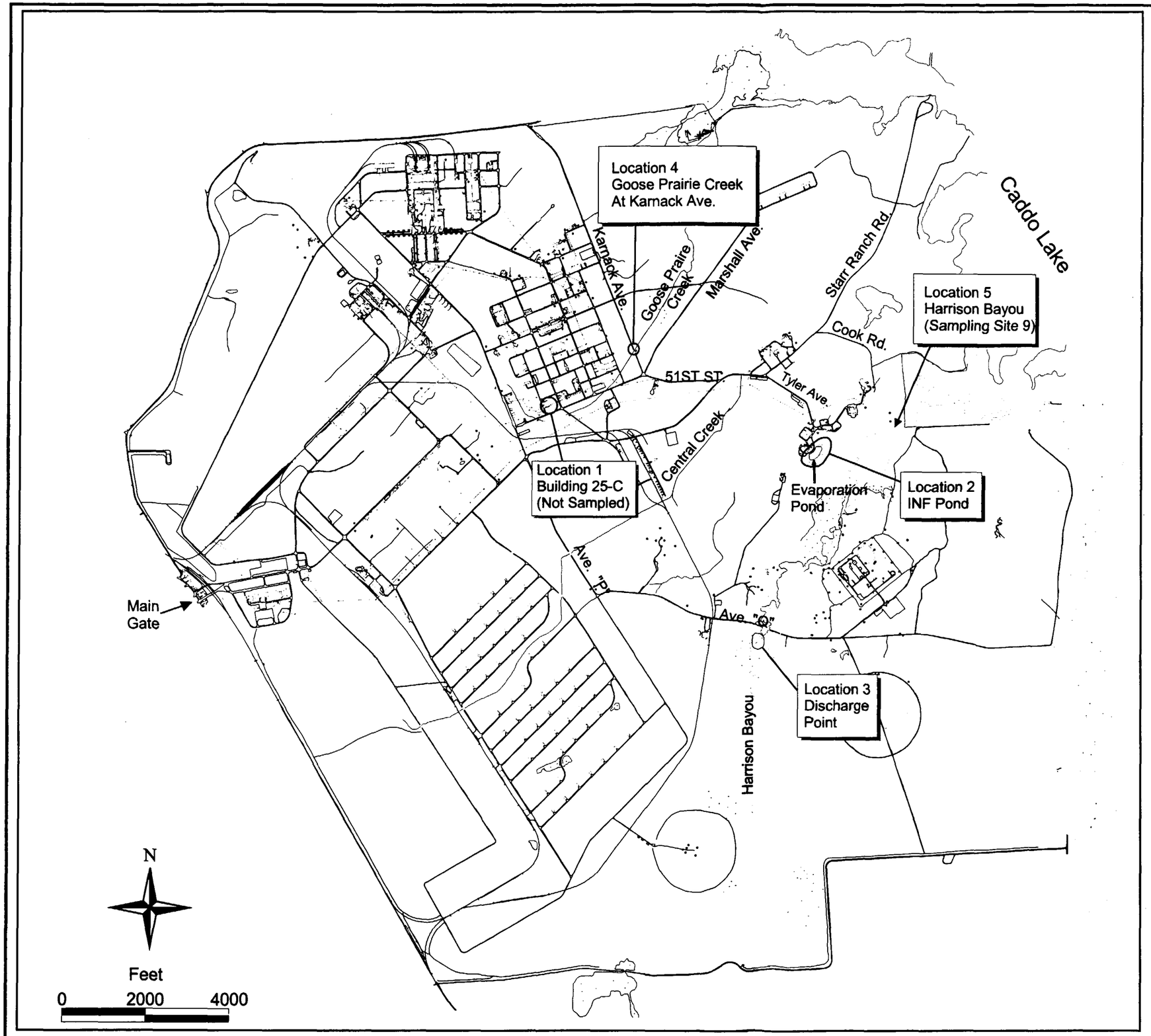

Figure 2.6

Longhorn Army Ammunition Plant
Karnack, Texas
Site and Sample Location Map

\737451\reports\workplan\gis\perchlorate.apr April 18, 2001

Table 2.5
Summary of Sample Collection Methods

Media Name	Media Code	Collection Method	Field Prep	Sample Container	Initial Preservation	Shipping Preservation
Aquatic						
Surface Water	01	Dipper or directly into sample bottle	NA	Plastic bottle	on ice	on ice
Groundwater	02	Only by others (not in SOW)	NA	Plastic bottle	on ice	on ice
Sediment	03	Trowel or directly into sample bottle	NA	Plastic bottle	on ice	on ice
Vegetation	04	Trowel or hand	NA	Ziplock	on ice	on ice
Fish	05					
Small fish		Electroshock, seine, trap	WB	Ziplock	on ice	on ice
Large fish		Electroshock, seine	Comp. tissue	Ziplock	on ice	on ice
Pupfish		Trap	WB	Ziplock	on ice	on ice
Invertebrates	06					
Crayfish		Kicknet, trap, hand	WB	Ziplock	on ice	on ice
Aquatic Insects		Kicknet	WB	Ziplock	on ice	on ice
Amphibians	07	Pit trap, Kicknet, shock	WB	Ziplock	on ice	on ice
Waterfowl	08	Shoot	Comp. tissue	Ziplock	on ice	on ice
Terrestrial						
Surface Soil	09	Trowel or directly into sample bottle	NA	Plastic bottle	on ice	on ice
Vegetation	10	Trowel or hand	NA	Ziplock	on ice	on ice
Mammals	11					
Small		Trap	WB	Ziplock	on ice	on ice
Large		Trap, shoot	WB	Ziplock	on ice	on ice
Bats		Shoot or mist net	WB	Ziplock	on ice	on ice
Birds	12	Shoot	WB	Ziplock	on ice	on ice
Invertebrates						
Insects	13	hand				
Earthworm	13	Trowel or hand	NA	Ziplock	on ice	on ice
Reptiles	14	Shoot, net, hand	NA	Ziplock	on ice	on ice

Legend:
- C = Celcius
- NA = Not Applicable
- comp = Composite of individual specimens to obtain necessary mass for analysis.
- WB = Whole body
- SOW = Scope of work

2.2.2.4 Sediment Pore Water

In addition to analyzing the sediment, analysis was performed on the interstitial water (pore water) which the laboratory decanted from the sediment preparation on approximately 10 percent of the sediment samples. The goal of this additional analysis was to determine if perchlorate concentrations differ in surface water, pore water, and in the sediment layer.

2.2.2.5 Aquatic Vegetation

Discrete aquatic vegetation samples were collected by hand or with a dip net.

2.2.2.6 Aquatic Invertebrates

Dip nets and hand picking were used to collect aquatic invertebrates from the water body, man-made structures, and snags. Attempts were made to collect an appropriate mass of the same species for each sample.

2.2.2.7 Fish

Fish collection was performed by hand net, rod and reel, traps, and electroshocking. Due to personnel safety concerns, attempts were made to minimize electroshocking use. All fish not retained as samples were released at the point of capture.

2.2.2.8 Amphibians

Amphibians were collected by hand and dip net.

2.2.2.9 Waterfowl

Waterfowl were shot with a 20 gauge shotgun with steel or bismuth shot.

2.2.2.10 Terrestrial Vegetation

Terrestrial vegetation was collected by hand. Sample collection was directed at the vegetation parts which were most likely consumed by the biota in the area (typically leaves, seeds and flowering stalks, when available).

2.2.2.11 Terrestrial Insects

Terrestrial insects were collected using sweep nets during the day and light traps (with sheet) and blacklights at night.

2.2.2.12 Reptiles

Reptile collection was performed by hand or through the use of nets or traps.

2.2.2.13 Birds

Birds were shot with a 12 or 20 gauge shotgun with steel or bismuth shot.

2.2.2.14 Mammals

Sherman live-capture traps were used for small mammal collection (*e.g.* mice). Bait typically consisted of oatmeal rolled with peanut butter. Removal of mammals from traps was performed by shaking the trap into a zip lock bag. After identification, target specimens were euthanized. Non target and surplus animals were released at the point of capture.

Larger mammals (*e.g.* cottontails) were shot with a 12 or 20 gauge shotgun with steel or bismuth shot, or with larger live traps. Bats were also collected with a 12 or 20 gauge shotgun. Attempts were made to capture bats using mist nets; however, none were captured by this method.

Special health and safety precautions were taken for field sampling team members, sample shipping, and laboratory personnel dealing with mammals which had the potential for transmitting diseases such as rabies or hantavirus. These procedures are discussed in detail in the workplan (Parsons, 2000).

2.2.3 Photo Log of Field Work

Photographs documenting habitat characteristics and sample collection procedures at each sampling site are included in Appendix A.

2.2.4 Sample Handling and QA/QC

This section provides an overview of the sample handling and quality assurance/quality control procedures that were utilized in the field and laboratory to ensure sample integrity. For much greater detail on the sample handling procedures and goals, see the document, *Workplan for Perchlorate Biotransport Analysis*, Parsons ES, October 2000.

2.2.4.1 Field Measurements and Documentation

Personnel responsible for sample collection maintained field records to document all sampling and measurement activities. Information gathered during field activities was recorded with indelible black or blue ink in logbooks used specifically for this study or field data sheets for specific activities. Corrections were crossed out with a single line, dated, and initialed.

The logbooks and field logsheets provide a daily handwritten record of all field activities. Entries were made immediately before, during, and at the end of each work session. Other pertinent information that was noted in the logbooks and logsheets includes:

- Notation of weather conditions and any preceding meteorological or anomalous conditions which might affect sample results or sampling success;
- Sampling conditions, collection methods and success, decontamination, and sample handling, identification, and preparation;
- Sketches of sample locations when necessary;
- Specific comments on problems that occurred during daily activities, their final resolution, and anticipated impact; and
- Record of telephone calls pertaining directly to the decision-making process of the field investigation.

2.2.4.2 Sample Handling, Identification, Shipping, and Storage

Because perchlorate is very stable and nonvolatile, wet ice was used to preserve the samples. Sample bottles and ziplock bags were labeled with sample identification (ID), date, time, analysis, and sampler's initials.

The sample IDs consist of two digit numbers for a series of codes which define the samples. An example of the ID for sample 01030502 is presented in Table 2.6. This sample was collected at site #1 (01=Yuma, Arizona, see Table 2.4), sampling location #3 (03=Imperial Dam, see Table 2.4), aquatic group, fish sample media (05, see Table 2.5), and sample number 2 (02) for this medium at this location. Common name and/or scientific names were recorded in the field logbook with the sample ID to assist verification during data processing to ensure the correct species identification for each sample.

Table 2.6
Example identification for sample 01030502

Site #	Sample Location #	Sample Media ID	Number of this Sample at this Location
01	03	05	02

All samples were stored in ice chests cooled with wet ice. The samples were bottled or contained in ziplock bags, and ice was ziplock bagged to prevent samples contacting water from melted ice. Samples were logged on Chain of Custody forms and delivered to the laboratory under sealed custody via overnight delivery service.

Upon arrival and check-in at the preparation laboratory or analytical laboratory, samples were stored at <0 degrees Celsius to minimize decomposition until preparation and analysis. Detailed sample handling procedures followed by the analytical laboratory are provided in Appendix B of this report and detailed sample handling procedures followed by the preparation laboratory are provided in Appendix C.

2.2.4.3 Summary of Samples Collected

Table 2.7 provides a summary of the number of samples collected at each site listed by location and media. The biological sample numbers were dependent upon collection permit requirements, abundance of species, and collection effectiveness.

Table 2.7 does not include QC samples (field duplicates, laboratory duplicates, and laboratory split samples).

2.2.5 Anomalies Associated with Field Work

At the first two sites sampled, Yuma, Arizona and Lake Mead, Nevada, some biological sample identifications (ID) included alphabetical codes for species indication. This labeling scheme was changed during sampling to the use of solely numerical sample ID with documentation of common or species name in the field logbook. The samples with alphabetical codes were modified in the Access® database to be similar in structure to sample IDs of the remainder of the project. Hardcopies of laboratory reports may have the original sample ID. In addition, on rare occasions two separate field teams at a single location collected similar biological samples and labeled them with the exact same numerical ID. Table 2.8 lists the modifications to sample IDs made in the database.

Field duplicate samples were not identified as such on the sample ID to keep the analytical laboratory "blind." These samples were documented in the field logbook and were differentiated in the Access database with the sample type notation "FD" (instead of "N" for normal).

Other notes of interest from the field sampling activities were:

- The heads were severed from all poisonous snakes and discarded prior to shipping for laboratory personnel safety.
- Mammals captured in traps likely ate all bait used in the trap which could affect perchlorate concentrations.
- At locations where small fish were collected, and total biomass was low, fish samples were composite samples of from two to 40 fish, as necessary.
- Many insect samples were composites of several species, with the most common (by weight) species name given to the sample, and composite being recorded in the species data category.

Table 2.7
Summary of Samples Collected

		Site Name	Yuma, AZ					Lake Mead Area			Allegany Ballistics Laboratory								Holloman AFB							Indian Head Area						LHAAP				Total
		Location Number	1	2	3	4	5	1	3	6	1	2	3	4	5	6	7	8	0	1	2	4	5	6	7	3	4	5	6	7	8	2	3	4	5	
	Sample Type																																			
Animal tissue	Aquatic	Bird				1				8																										9
Animal tissue	Aquatic	Reptile																										6	1				3			10
Animal tissue	Aquatic	Insect																									1					3				4
Animal tissue	Aquatic	Invertebrate							1	1																	2			3					2	9
Animal tissue	Aquatic	Fish	1			1	5	2	7	10				10	2								3	3	3		1	13	11	7			8	10	10	107
Animal tissue	Aquatic	Amphibian					2		5	1					2											2	2	1		1		4		1	1	22
Plant tissue	Aquatic	Vegetation	3	2		3	3	3	4	2					9	3									3		3			3		3	8			50
Pore water			1			1		2		1						3				1						1				1					1	12
Sediment			5	3		4	5	12	5	5				3	3	3	3	3		3		3	3	3	3	3	3	3	3	3	3	3	3	3	3	99
Groundwater																			19																	19
Surface water			5	4	5	4	6	14	6	9				3	6	6		6					6	6	6	6	6	6	6	6	6	6	6	6	7	153
Soil			4	3		2	5	5	5	5	9	6	3		3	3		3		13	3		3	3	3	4	3	3	3	3	3	4	3	3	3	113
Plant tissue	Terrestrial	Vegetation	10			9	8	9	15	9	9	6	6							15	15		9	9	9	3	6	3		6	3	6	6		6	177
Animal tissue	Terrestrial	Insect	2			4	3	1	2	1	2	1	3	2	2		1	2									1	2	1	3		1	1			35
Animal tissue	Terrestrial	Invertebrate										1					2	1											3							7
Animal tissue	Terrestrial	Reptile					1														1													2	3	7
Animal tissue	Terrestrial	Bird	6				1	5		2	5										4		1		1			3				3	9			42
Animal tissue	Terrestrial	Mammal	5			8	5	8	4	6	2				3					7	12		5		6	4	1	3	1	2	1	3	3	1		90
			44	12	5	37	44	61	54	60	27	14	12	18	30	18	6	15	19	39	35	3	30	24	34	23	29	43	29	38	16	36	48	26	36	965

Number of samples collected by site, location, and media group.

Table 2.8
Summary of Sample ID Modifications

Revised Sample ID (in Access Database)	Revised Sample ID (in Laboratory Report)	Original Sample ID (in field)	Sample Date	Sample Time
Duplicated Sample IDs				
06031101A		06031101	9/11/2000	1800
05071301A		05071301	8/27/2000	1200
03081352		03081302	8/22/2000	1450
06051451		06051401	9/11/2000	1200
05060502		05060592	8/27/2000	1600
01041151		01041101	8/1/2000	730
01041152	01041152	01041102	8/1/2000	730
01041153	01041153	01041103	8/1/2000	730
06041451		06041401	9/12/2000	1520
01011291		010112MD01	8/3/2000	1920
Sample IDs originally with alphabetic code				
01010501		010105MW01		
01011001		010110AF01		
01011002		010110AW01		
01011003		010110AW02		
01011004		010110AW03		
01011005		010110HM01		
01011006		010110HM02		
01011007		010110TA01		
01011008		010110TA02		
01011009		010110TA03		
01011010		010110TA04		
01011011		010110QB01		
01011202		010112MD02		
01011203		010112SW01		
01011204		010112SW02		
01011205		010112SW03		
01011206		010112SW04		
01011207		010112SW05		
01011291		010112MD01		
01040401		010404JS01		
01040402		010404JS02		
01040403		010404JS03		
01040501		010405LM01		
01040801		010408CT01		
01041001		010410HM01		
01041002		010410HM02		
01041003		010410HM03		
01041004		010410AW01		
01041005		010410AW02		
01041006		010410AW03		
01041007		010410AF01		
01041008		010410AF02		
01041009		010410AF03		
01041301		010413DF01		
01050401		010504CT01		
01050402		010504CT02		
01050403		010504CT03		
01050501		010505SF01		
01050502		010505P001		
01050503		010505MT01		
01050504		010505MT02		
01050505		010505MT03		
01050601		010506DM01		
01051001		010510QB01		
01051002		010510QB02		
01051003		010510AW01		
01051004		010510AW02		
01051005		010510AW03		
01051006		010510SM01		
01051007		010510SM02		
01051008		010510SM03		

2.3 LABORATORY METHOD DEVELOPMENT AND DISCUSSION

Anal. method

In order to meet the primary objective of this project, to determine the presence of perchlorate in biological media, an analytical method for determining perchlorate concentration in tissue was required. Therefore, Clayton was tasked with developing and verifying analytical methods for perchlorate determination in soil, sediment, plant tissue, and animal tissue. Clayton utilized USEPA's Method 314.0, "*Analysis of Perchlorate in Drinking Water using Ion Chromatography,*" Revision 1.0, November 1999 (USEPA Cincinnati), and innovative extraction techniques to successfully develop methodology for determining perchlorate concentrations in media other than water. Collaboration was made with Dr. Jackson Ellington (Ellington, 2000) for sample preparation and analysis.

The ion chromatography method for perchlorate determination in water matrices is detailed in USEPA's Method 314.0. Clayton prepared their Laboratory Standard Operating Procedure (SOP) entitled *"WATER: M-CC-68/N, Rev. 2, June 2000"* according to this method and their instrument capabilities. Clayton also developed the following SOPs for the respective matrices: *"M-CC-69/N, Rev. 2, Oct. 2000"* for soil; *"M-CC-70/N, Rev. 1, Nov. 2000"* for vegetation; and *"M-CC-71/N, Rev. 0, Oct. 2000"* for animal tissue. Copies of these four SOPs are included in Appendix B.

Sample preparation for soil matrices involved extracting an aliquot of soil with deionized water, using Alumina (basic wash) to remove inorganic ions from the sample extract, diluting the sample extract by ten with deionized water, and following with C_{18} cartridge clean-up. The effluent was then filtered through a 0.45-micron Acrodisc filter prior to injection on the ion chromatography instrument.

Vegetation samples were manually reduced in size by cutting the specimen into strips approximately 1 inch wide. Each sample was then homogenized by passing it through a commercial style meat grinder three times and then blending the sample in a commercial stainless steel blender containing liquid nitrogen. A description of the sample preparation used by EnChem, Inc. (EnChem) is included in Appendix C. The processed samples were then sent to Clayton where the extraction and clean-up steps established for soil were used to finalize sample preparation.

Whole animal samples were ground and blended by EnChem following the same procedure used for the vegetation samples. One set of fish tissue samples was prepared by Covance Laboratories, Inc. following the preparation guidelines established by EnChem. The processed samples were then sent to Clayton where the appropriate extraction and clean-up steps established for soil were used.

Overall, the method development performed by Clayton was shown to produce reliably accurate and precise measurement on the amount of perchlorate in these difficult matrices. Perchlorate was detected in both plant and animal tissue using the modified methodology and sample preparation procedures developed by Clayton and EnChem. Detailed method development processes are described in the Parsons Perchlorate Method Development and Validation Report, included in Appendix D. Analytical Method Validation Processes

The instrument detection limit, linear range and ruggedness for each Dionex DX-120 ion chromatography instrument were determined.

Clayton established the method detection limit (MDL) and assessed the precision and accuracy for water matrices following their SOP. A MDL of 1.03 µg/L was statistically calculated for water (see Appendix B). However, the chromatographic resolution and response of perchlorate is limited to a minimum concentration of 4 µg/L. Thus, Clayton used 4 µg/L as the reporting limit (RL) for the method. Results of the precision study were 11.6 percent relative standard deviation (RSD) at the 95 percent confidence level, an accuracy of 105.5 percent.

A similar analytical method validation was conducted for soils (using a composite field soil sample), vegetation (using a field arrowweed sample), and animal tissue (using a field cottontail sample). A composite soil sample was used for method validation because of the limited sample size obtained in the field.

Reporting limits

To calculate the RL for solid and tissue samples, the lowest point of the instrument calibration curve (in µg/L) is multiplied by the final extraction volume of the sample (in mL), divided by the percent solid of the sample and multiplied by the dilution factor (if applicable). For example, assuming the lowest point of the calibration curve is 4 µg/L, the final extraction volume is 20 mL, the sample weight is 2 grams, the percent solid is 75%, and the dilution factor is 5. An example calculation is as follows:

4 µg/L * 20 mL / 2 g * 10 / 0.75 * 5 = 2,666 µg/Kg-dry

For the solids matrix, the method precision was 26.9 percent RSD at the 95 percent confidence level, and the method accuracy was 88.0 percent. The specific vegetation and animal tissue matrices used for method validation were selected because they served as the "worst-case" option for their particular group of samples. For vegetation, the precision was 22.5 percent RSD at the 95 percent confidence level, and the accuracy was 95.5 percent. For the animal tissue, the precision was 8.74 percent RSD at the 95 percent confidence level, and the accuracy was 86.4 percent.

The precision and accuracy observed for each matrix demonstrate that Clayton's methods development was successful. While the water matrix tests exhibited the best results, all matrices had accuracy recoveries above 85 percent and RSDs of less than 30 percent. These results demonstrate the success of the developed methodology in producing high quality (accurate and precise) data.

2.3.1 Laboratory Quality Control Procedures

Prior to sample analysis, a valid initial calibration for the ion chromatograph system is required. For this study, an initial calibration was performed using perchlorate levels of 4, 10, 20, 50 and 100 ppb. The resulting linear regression calibration curve was required to have a coefficient of determination (r^2) of at least 0.995 to meet acceptable performance standards. An instrument performance check (IPC) sample, which contained perchlorate and a mixture of common anions, was analyzed immediately after

the initial calibration curve. An initial calibration verification (ICV) using a secondary source standard was analyzed immediately after the IPC to verify the initial calibration. The calibration curve was verified with a low-level continuing calibration verification standard (LCCV, 4 ppb calibration standard) each working day. Alternating mid-level continuing calibration verifications (MCCV, 20 ppb), and high level continuing calibration verifications (HCCV, 100 ppb) were analyzed after every 10 field samples.

QC samples were prepared and analyzed with each analytical batch (a maximum of 20 field samples) to access the accuracy and precision of the instrumentation. Each analytical batch, consisting of field samples of similar matrix, was required to include a method blank (MB), a laboratory control sample (LCS), and a matrix spike/matrix spike duplicate pair (MS/MSD). Additional QC samples, such as laboratory duplicates or field duplicate samples, were included in some analytical batches, but these were not required.

An MB was prepared and analyzed prior to sample analysis for each analytical batch. The MB is used to demonstrate whether interference or contamination from the analytical system, glassware, and reagents, is present at levels that would affect the sample results. If perchlorate was detected above the RL in the MB, corrective action was initiated to reduce the contamination and bring the analytical system back into control.

An LCS was prepared and analyzed with each analytical batch to assess the accuracy and precision of the analytical process. The LCS was analyzed at a concentration of 20 µg/L. A batch of samples was considered acceptable only if the LCS met tolerance.

An MS/MSD pair was analyzed for each batch to assess the accuracy and precision of the process within the native sample matrix. All samples were selected at random by the analyst, but samples containing elevated concentrations of perchlorate were avoided. The sample chosen for the MS/MSD was spiked at a concentration of 200 µg/L. A summary of specific QC acceptance criteria are included in Table 2.9.

2.3.2 Laboratory Report

All aqueous samples results for perchlorate were reported in µg/L. All solid sample results for perchlorate were reported on dry weight basis in µg/kg-dry. The percent moisture results were provided for all soil, sediment, tissue, and vegetation samples. Sample results were converted from dry weight to wet weight concentrations using the following equation:

Wet Weight Concentration = Dry weight concentration x [1-(percent moisture of the sample/100)]

Table 2.9
Specific QC Acceptance Criteria

Matrix	SOP	ICAL[1]		ICV[2] 2nd Source	LCCV[3] 4 ppb	MCCV[4] (20 ppb) and HCCV (100 ppb)	IPC[5]	Sample Duplicates	LCS[6]	MS/MSD[7]
		4 ppb	10 - 100 ppb							
Solid	M-CC-69/N Rev.2 Oct. 2000	±25%	±15%	None Given	±25%	±15%	±20%	±15%	±25%	Statistical Limits
Plant Tissue	M-CC-70/N Rev.1 Nov. 2000	Statistical Limits	±15%	None Given	Statistical Limits	±15%	±20%	Statistical Limits	±25%	Statistical Limits
Water	M-CC-68/N Rev.2 June 2000	Statistical Limits	±15%	15%	Statistical Limits	±15%	±20%	Statistical Limits	±25%	Statistical Limits
Animal Tissue	M-CC-71/N Rev.0 Oct. 2000	±25%	±15%	None Given	±25%	±15%	±20%	±15%	±25%	Statistical Limits

[1] ICAL = Initial Calibration

[2] ICV = Initial Continuing Verification

[3] LCCV = Low Continuing Check Verification

[4] MCCV = Middle Continuing Check Verification

[5] IPC = Instrument Performance Check

[6] LCS = Laboratory Control Sample

[7] MS/MSD = Matrix Spike/Matrix Spike Duplicate

2.3.3 Data Verification and Validation Procedures

Data validation by Parsons ES included an extensive review of the Chain of Custody (COC), case narrative, analytical sample results reports, QC reports, preparation summaries, calibration curves, instrument sequence files, raw data, and manual integrations. Data Validation Summary Reports are included in Appendix E.

Data verification included a general overview of the Sample Delivery Group (SDG), including review of the COC, case narrative, analytical sample results reports, QC reports, and calibration curves. A Verification Checklist (VC) was completed and stored with each SDG data package. Data Verification Reports are included in Appendix E.

SECTION 3
RESULTS AND DISCUSSION

This section presents and discusses the analytical results for samples collected from each of the six perchlorate study sites; Yuma, Arizona (Yuma), Lake Mead NRA (Lake Mead Area), Allegany Ballistics Laboratory (ABL), Holloman Air Force Base (Holloman), Indian Head Area (Indian Head), and Longhorn Army Ammunition Plant (LHAAP). The analytical data included in this section has been summarized and presented in tabular and graphical format to aid in comparison and discussion of potential perchlorate biotransport at each of the six sites.

All perchlorate results and reporting limits (RLs) for analytical samples discussed in this section have been adjusted to "wet weight" values. The wet weight of a sample is obtained by multiplying the dry weight perchlorate concentration of the sample by [1 – (% moisture of the sample/100)]. A comprehensive listing of all analytical data received, as well as analytical results and laboratory RLs adjusted to wet weight values, is included in Appendix F.

To aid in the management and interpretation of the analytical data, adjusted analytical results for all samples collected at each of the six sites mentioned above are grouped by site, location and media (i.e. surface water, sediment, vegetation, fish, birds, invertebrates, etc.). Two different mean concentrations were calculated within each site specific media group to obtain representative perchlorate concentrations. The first mean concentration was the "mean for all samples." To calculate this mean, the perchlorate concentrations from all samples were used. For samples where perchlorate concentrations were not detected above the laboratory RL, the laboratory RL, rather than zero, was used in the calculation of the mean value. The other mean concentration was the "mean for detected samples." This procedure calculates a mean using only the samples that had perchlorate concentrations detected, excluding the nondetected samples. The use of both of these procedures allows for different ways of evaluating the data.

In addition, all tables and figures within this section incorporate only "normal" or "parent" samples, with one exception. When a field duplicate contained a higher perchlorate concentration than the normal/parent sample, the field duplicate, with the higher perchlorate concentration, was used to calculate the media specific mean perchlorate concentration. The mean perchlorate concentrations are used in graphical representations within this section to simplify the large quantity of species-specific analytical data collected as part of this investigation and aid in the interpretation of potential perchlorate biotransport pathways.

3.1 DATA PRESENTATION

This study represents a major perchlorate sampling effort conducted at six independent sites across the country. These sites represent both low and high rainfall

areas with significantly different biota. In addition, the perchlorate sources and ecological pathways are substantially different at the sites, as discussed in Section 2. Therefore, the goal was to identify trends and biotransport pathways across those varying sites.

Table 3.1 summarizes the quantity of samples collected at each site and presents the number of normal samples that were collected in this study. In addition to the samples included in Table 3.1, approximately 20% more samples were analyzed in the laboratory as field duplicates and laboratory split samples.

Mean perchlorate values, calculated using all samples (the RL was used for samples without detected perchlorate) for each of the six sites, are compared by medium type and medium group in order to assess the distribution of perchlorate on a large scale. Table 3.2 summarizes analytical results and mean perchlorate concentrations for all samples collected. The table presents mean perchlorate concentrations calculated using only analytical results detected above laboratory RLs, as well as mean concentrations for all results, calculated using the laboratory RL for values below detection limits.

Figures 3.1a and 3.1b provide a graphical representation of perchlorate concentrations detected in various media types and groups at each site. Medium type include soil, sediment, surface water, and aquatic and terrestrial biota. Aquatic biota consists of birds, reptiles, invertebrates (including insects), fish, amphibians, and vegetation. Terrestrial biota consists of vegetation, invertebrates (including insects), reptiles, birds, and mammals. Figures 3.1a and 3.1b present the same data, however, Figure 3.1b presents the data with a lower scale in order to make the results of the media with the lower perchlorate concentrations visible.

A total of 18 medium type/group combinations were evaluated as part of this study. Due to varying environmental conditions at the time of sample collection, a representative sample of every type of target biotic media could not be collected at all sites. Therefore, several "gaps" in the data for various media exist at several sites. These gaps are represented in the figure as blank squares. However, with the quantity of data collected at each of the perchlorate study areas, site and media comparisons can still be made.

As seen in Figures 3.1a and 3.1b, perchlorate was detected in biological media at all six sites investigated with concentrations ranging over several orders of magnitude. In aquatic media, perchlorate was not detected above laboratory RLs in birds, reptiles, or invertebrates at any of the sites in which they were sampled. However, all other aquatic media were detected above laboratory RLs at least once.

Table 3.1
Number of Samples Collected at Each Site

Sample Type		Site Name	Yuma, AZ	Lake Mead Area	Allegany Ballistics Laboratory	Holloman AFB	Indian Head Area	LHAAP	Total
Animal tissue	Aquatic	Bird	1	8	0	0	0	0	9
Animal tissue	Aquatic	Reptile	0	0	0	0	7	3	10
Animal tissue	Aquatic	Insect	0	0	0	0	1	3	4
Animal tissue	Aquatic	Invertebrate	0	2	0	0	5	2	9
Animal tissue	Aquatic	Fish	7	19	12	9	32	28	107
Animal tissue	Aquatic	Amphibian	2	6	2	0	6	6	22
Plant tissue	Aquatic	Vegetation	11	9	12	3	6	9	50
Pore water			2	3	3	1	2	1	12
Sediment			17	22	15	15	18	12	99
Groundwater			0	0	0	19	0	0	19
Surface water			24	29	21	18	36	25	153
Soil			14	15	27	25	19	13	113
Plant tissue	Terrestrial	Vegetation	27	33	21	57	21	18	177
Animal tissue	Terrestrial	Insect	9	4	13	0	7	2	35
Animal tissue	Terrestrial	Invertebrate	0	0	4	0	3	0	7
Animal tissue	Terrestrial	Reptile	1	0	0	1	0	5	7
Animal tissue	Terrestrial	Bird	9	7	5	6	3	12	42
Animal tissue	Terrestrial	Mammal	18	18	5	30	12	7	90
Total			**142**	**175**	**140**	**184**	**178**	**146**	**965**

Number of samples collected by site, location, and media group.
The table only includes "normal" samples.

Table 3.2 Site and Media Specific Perchlorate Concentrations

Site	*Medium/ Matrix*	*No. of Samples*	*No. of Detects*	*Maximum Adj Result*	*Minimum Adj Result*	*Mean of All Results*	*Mean of Detected Results*	*Maximum Adj RL*	*Minimum Adj RL*
1 Yuma, AZ									
	Aquatic Bird	1	0	ND	ND	400		400	400
	Fish	7	0	ND	ND	396		400	392
	Amphibian	2	0	ND	ND	421		432	410
	Aquatic Vegetation	11	1	260	ND	377	260	408	369
	Pore Water	2	1	2.00	ND	3.00	2.00	4.00	4.00
	Sediment	17	0	ND	ND	81.0		83.6	79.2
	Surface Water	24	15	5.60	ND	4.18	4.29	4.00	4.00
	Soil	14	1	90.0	ND	80.6	90.0	80.4	79.0
	Terrestrial Vegetation	27	15	1790	ND	553	686	418	370
	Terrestrial Insect	7	0	ND	ND	462		822	392
	Terrestrial Reptile	1	1	531	531	531	531	412	412
	Terrestrial Bird	9	0	ND	ND	398		417	388
	Terrestrial Mammal	18	0	ND	ND	403		421	386

RL = reporting limit.
ND = not detected about the reporting limit.
In calculation of the mean of all results, the RL is used for nondetected values.
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Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
Reported results less than RLs are estimated values.
13-Jun-01

Table 3.2 Site and Media Specific Perchlorate Concentrations

Site	*Medium/ Matrix*	*No. of Samples*	*No. of Detects*	*Maximum Adj Result*	*Minimum Adj Result*	*Mean of All Results*	*Mean of Detected Results*	*Maximum Adj RL*	*Minimum Adj RL*
2 Lake Mead Area									
	Aquatic Bird	8	0	ND	ND	397		402	390
	Aquatic Invertebrate	2	0	ND	ND	389		398	381
	Fish	19	2	44300	ND	2910	24300	5740	381
	Amphibian	6	0	ND	ND	402		421	396
	Aquatic Vegetation	9	4	176000	ND	47900	107000	20900	377
	Pore Water	2	2	98000	140	49100	49100	4.00	4.00
	Sediment	16	11	56000	ND	16800	24400	83.6	78.0
	Surface Water	23	21	130000	ND	37100	40600	20000	4.00
	Soil	15	13	1470000	ND	163000	188000	80.5	77.0
	Terrestrial Vegetation	33	33	428000	516	69400	69400	40000	372
	Terrestrial Insect	4	3	4730	ND	3120	4030	407	385
	Terrestrial Bird	7	4	4190	ND	1200	1800	400	381
	Terrestrial Mammal	18	15	53000	ND	12000	14300	6220	385
	Pore Water - downstream	1	1	1000	1000	1000	1000	4.00	4.00
	Sediment - downstream	6	2	21700	ND	4220	12500	82.6	78.0
	Surface Water - downstream	6	6	19000	12000	13300	13300	4.00	4.00

RL = reporting limit.
ND = not detected about the reporting limit.
In calculation of the mean of all results, the RL is used for nondetected values.
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Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
Reported results less than RLs are estimated values.
13-Jun-01

Table 3.2 Site and Media Specific Perchlorate Concentrations

Site	Medium/ Matrix	No. of Samples	No. of Detects	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
3 ABL									
	Fish	12	1	451	ND	406	451	410	388
	Amphibian	2	0	ND	ND	410		417	404
	Aquatic Vegetation	12	0	ND	ND	397		408	390
	Pore Water	2	2	23.0	14.0	18.5	18.5	4.00	4.00
	Sediment	15	2	119	ND	84.2	112	81.9	77.0
	Surface Water	21	15	280	ND	174	212	80.0	4.00
	Soil	27	13	22100	ND	1260	2520	83.6	76.8
	Terrestrial Vegetation	21	14	300000	ND	36000	53800	39600	384
	Terrestrial Insect	13	1	12600	ND	1340	12600	2040	388
	Terrestrial Invertebrate	4	0	ND	ND	503		800	392
	Terrestrial Bird	5	1	423	ND	403	423	419	394
	Terrestrial Mammal	5	0	ND	ND	404		417	390

RL = reporting limit.
ND = not detected about the reporting limit.
In calculation of the mean of all results, the RL is used for nondetected values.
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Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
Reported results less than RLs are estimated values.
13-Jun-01

Table 3.2 Site and Media Specific Perchlorate Concentrations

Site	*Medium/ Matrix*	*No. of Samples*	*No. of Detects*	*Maximum Adj Result*	*Minimum Adj Result*	*Mean of All Results*	*Mean of Detected Results*	*Maximum Adj RL*	*Minimum Adj RL*
4 Holloman AFB									
	Fish	9	0	ND	ND	399		419	385
	Aquatic Vegetation	3	0	ND	ND	407		416	396
	Pore Water	1	0	ND	ND	400		400	400
	Sediment	15	1	105	ND	131	105	819	78.0
	Surface Water	18	0	ND	ND	400		400	400
	Groundwater	19	15	38.0	ND	11.7	13.7	4.00	4.00
	Soil	25	9	2800	ND	241	447	803	78.0
	Terrestrial Vegetation	57	9	7600	ND	666	2110	420	370
	Terrestrial Reptile	1	0	ND	ND	408		408	408
	Terrestrial Bird	6	0	ND	ND	403		414	385
	Terrestrial Mammal	28	0	ND	ND	404		421	389

RL = reporting limit.
ND = not detected about the reporting limit.
In calculation of the mean of all results, the RL is used for nondetected values.
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Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
Reported results less than RLs are estimated values.
13-Jun-01

Table 3.2 Site and Media Specific Perchlorate Concentrations

Site	Medium/ Matrix	No. of Samples	No. of Detects	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
5 Indian Head Area									
	Aquatic Reptile	7	0	ND	ND	396		412	388
	Aquatic Insect	1	0	ND	ND	398		398	398
	Aquatic Invertebrate	5	0	ND	ND	407		419	402
	Fish	32	0	ND	ND	401		417	366
	Amphibian	6	1	465	ND	415	465	410	394
	Aquatic Vegetation	6	4	420	ND	325	290	408	384
	Pore Water	2	0	ND	ND	4.00		4.00	4.00
	Sediment	18	5	318	ND	133	120	810	78.0
	Surface water	36	27	25.0	ND	14.4	17.9	4.00	4.00
	Soil	19	7	546	ND	139	158	164	76.8
	Terrestrial Vegetation	21	7	931	ND	423	477	418	384
	Terrestrial Insect	7	0	ND	ND	402		417	394
	Terrestrial Invertebrate	3	0	ND	ND	401		412	394
	Terrestrial Bird	3	0	ND	ND	399		402	394
	Terrestrial Mammal	12	0	ND	ND	400		419	388

RL = reporting limit.
ND = not detected about the reporting limit.
In calculation of the mean of all results, the RL is used for nondetected values.
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Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
Reported results less than RLs are estimated values.
13-Jun-01

Table 3.2 Site and Media Specific Perchlorate Concentrations

Site	Medium/ Matrix	No. of Samples	No. of Detects	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
6 LHAAP									
	Aquatic Reptile	3	0	ND	ND	397		404	388
	Aquatic Insect	3	3	996	603	741	741	412	396
	Aquatic Invertebrate	2	0	ND	ND	401		413	390
	Fish	28	0	ND	ND	397		417	385
	Amphibian	6	4	1660	ND	934	1200	412	388
	Aquatic Vegetation	9	3	15700	ND	4250	12000	2020	381
	Pore Water	1	0	ND	ND	4.00		4.00	4.00
	Sediment	12	2	286	ND	103	215	82.8	77.0
	Surface Water	25	6	3800	ND	887	3680	2000	4.00
	Soil	13	0	ND	ND	79.6		82.5	77.0
	Terrestrial Vegetation	18	11	2340	ND	905	1230	408	388
	Terrestrial Insect	2	0	ND	ND	388		392	385
	Terrestrial Reptile	5	0	ND	ND	400		404	394
	Terrestrial Bird	12	0	ND	ND	401		412	384
	Terrestrial Mammal	7	1	571	ND	422	571	410	386

RL = reporting limit.
ND = not detected about the reporting limit.
In calculation of the mean of all results, the RL is used for nondetected values.
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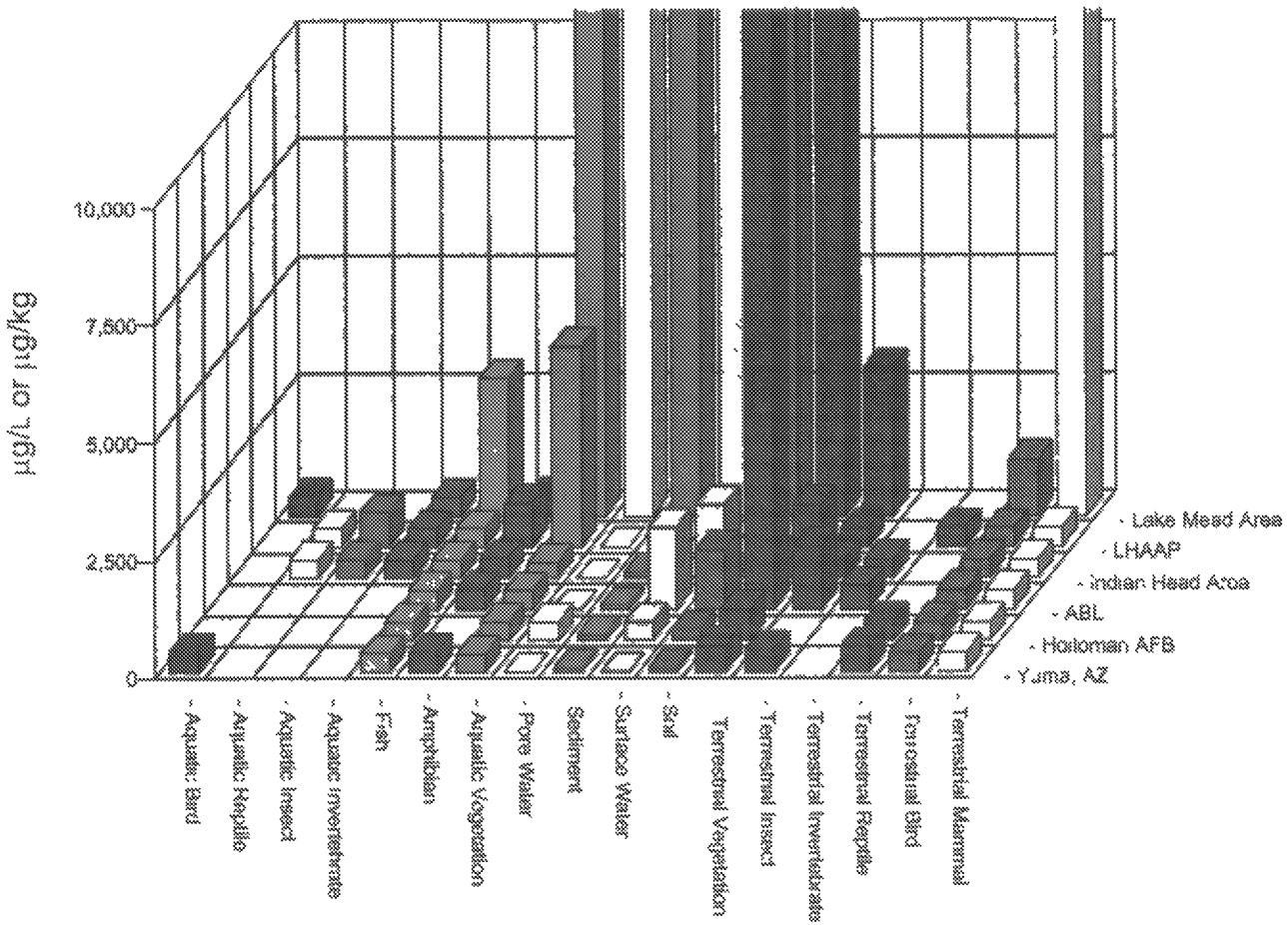
Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
Reported results less than RLs are estimated values.
13-Jun-01

Figure 3.1b
Media and Site-Specific Mean Perchlorate Concentrations



In calculation of the mean, the reporting limit is used for nondetected values
Sediment, soil, and tissue sample results are reported in µg/kg
Surface water and pore water results are reported in µg/L

Aquatic insect samples were collected at both LHAAP and Indian Head. However, only insects collected at LHAAP contained perchlorate at a mean concentration exceeding laboratory RLs (741 µg/kg). Fish samples were collected at all sites. However, only the Lake Mead Area and ABL contained fish samples with mean perchlorate concentrations exceeding laboratory RLs (24,300 µg/kg and 451 µg/kg, respectively). Amphibian samples were collected at all sites except Holloman. However, only LHAAP and Indian Head contained amphibian samples with mean perchlorate concentrations exceeding laboratory RLs (1,200 µg/kg and 465 µg/kg, respectively). Aquatic vegetation samples were collected at all six sites, but perchlorate was not detected above laboratory RLs at ABL or Holloman. Mean concentrations of perchlorate detected in aquatic vegetation were relatively low at Yuma (260 µg/kg) and Indian Head (290 µg/kg), higher at LHAAP (12,000 µg/kg), and higher again in the Lake Mead Area (107,000 µg/kg).

Aquatic abiotic media were collected at all sites. However, sediment collected at Yuma, AZ, sediment pore water and surface water at Holloman, and sediment pore water at LHAAP and Indian Head did not contain perchlorate at concentrations exceeding laboratory RLs. Mean concentrations of perchlorate detected in sediment are relatively consistent at Holloman (105 µg/kg), ABL (112 µg/kg), Indian Head (120 µg/kg) and LHAAP (215 µg/kg), and much higher in the Lake Mead Area (24,400 µg/kg in the upstream location and 12,500 µg/kg in the downstream location). Mean concentrations of perchlorate in sediment pore water was relatively low at Yuma (2 µg/L) and ABL (18.5 µg/L), and higher in the Lake Mead Area (49,100 µg/L in the upstream location and 1,000 µg/kg in the downstream location). Mean concentrations of perchlorate detected in surface water were low at Yuma and Indian Head (4.29 µg/L and 17.9 µg/L, respectively), higher at ABL (212 µg/L) and LHAAP (3,680 µg/L), and were highest in the Lake Mead Area (40,600 µg/L in the upstream location and 13,300 µg/L in the downstream location).

In terrestrial media, perchlorate was not detected above laboratory RLs in terrestrial invertebrates at any of the sites in which they were sampled. However, all other terrestrial media were detected above laboratory RLs at least once.

Perchlorate concentrations detected in terrestrial surface soils are relatively low at Yuma and Indian Head (90 µg/kg and 158 µg/kg, respectively), slightly higher at Holloman (447 µg/kg), higher again at ABL (2,520 µg/kg), and are highest in the Lake Mead Area (188,000 µg/kg). Perchlorate was not detected in surface soils at LHAAP. Terrestrial vegetation sampled at each of the six locations exceeded laboratory RLs. Mean concentrations of perchlorate in terrestrial vegetation appear relatively low at Yuma and Indian Head (686 µg/kg and 477 µg/kg, respectively), with concentrations increasing at Holloman and LHAAP (2,110 µg/kg and 1,230 µg/kg, respectively), ABL (53,800 µg/kg) and in the Lake Mead Area (69,400 µg/kg). Perchlorate in terrestrial insects was not detected above laboratory RLs at Yuma, Indian Head or LHAAP and not collected at Holloman. However, ABL and the Lake Mead Area contained relatively high concentrations (12,600 µg/kg and 4,030 µg/kg, respectively). Terrestrial reptiles were sampled at Yuma, Holloman and LHAAP, but only samples collected at Yuma

contained detectable concentrations of perchlorate (531 µg/kg). Terrestrial bird and terrestrial mammal samples were collected at each of the six sites. However, only ABL and the Lake Mead Area contained detectable concentrations of perchlorate in birds (423 µg/kg and 1,800 µg/kg, respectively) and only LHAAP and the Lake Mead Area contained detectable concentrations of perchlorate in mammals (571 µg/kg and 14,300 µg/kg, respectively).

Based on the information presented in Table 3.2, Figures 3.1a and 3.1b, of the six sites analyzed, the Holloman AFB site appears to contain the lowest frequency and mean concentration of perchlorate across all medium types and medium groups. After Holloman, it appears mean perchlorate concentration and frequency increase at Yuma and Indian Head, increase again slightly at LHAAP then increase sharply at ABL. Furthermore, calculated mean concentrations indicate perchlorate in all medium and matrix types collected in the Lake Mead Area are much higher than at any other site analyzed. To further investigate potential site and media specific perchlorate biotransport, analytical data collected from specific locations and media at each of the six sites is presented and discussed in the following sections.

3.1.1 Yuma, Arizona

Five locations along the Colorado River at the southern California/Arizona border were sampled north of Yuma, Arizona. Various media were collected from each of the five locations and analyzed for perchlorate. Table 3.3 summarizes mean perchlorate concentrations by location, matrix, medium type and medium group in tabular format and Figure 3.2a presents the mean perchlorate concentrations for all samples (using the RL for all samples without detected perchlorate) in graphical format. Figure 3.2b presents the mean concentrations of only detected perchlorate samples. Samples containing values of perchlorate below reporting limits are not used to calculate the mean concentration.

3.1.1.1 Location and Media Specific Analytical Comparisons

As shown in Table 3.3, Figure 3.2a and Figure 3.2b, relatively few samples had perchlorate concentrations detected above the reporting limits. All of the surface water samples at the Palo Verde Lands (Location 1) and Agriculture Lease 1 (Location 4) had detectable perchlorate, with a maximum concentration of 5.6 µg/L. These concentrations are among the lowest detected in surface water among the six sites. Even with the relatively low perchlorate concentrations in the surface water, perchlorate was detected in aquatic vegetation, soil, and terrestrial vegetation from Location 1, terrestrial vegetation from Location 4, and terrestrial vegetation and terrestrial reptile from Location 5 (Agriculture Lease 2). All of these perchlorate concentrations were detected at levels only slightly above the reporting limits. It is possible that perchlorate is present in other samples at concentrations that are below the reporting limits. Samples of aquatic invertebrates, terrestrial invertebrates, aquatic reptiles, and groundwater were not collected at any locations at the Yuma, Arizona site.

Table 3.3 Media and Location Specific Perchlorate Concentrations

Site 1, Yuma, AZ

Location	Medium/ Matrix	No. of Samples	No. Detected	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
1 Palo Verde Lands									
	Fish	1	0	ND	ND	392		392	392
	Aquatic Vegetation	3	1	260	ND	349	260	396	390
	Pore Water	1	1	2.00	2.00	2.00	2.00	4.00	4.00
	Sediment	5	0	ND	ND	80.9		81.6	80.0
	Surface Water	5	5	5.60	4.60	4.98	4.98	4.00	4.00
	Soil	4	1	90.0	ND	82.5	90.0	80.4	79.7
	Terrestrial Vegetation	10	9	1010	ND	568	587	418	380
	Terrestrial Insect	2	0	ND	ND	398		400	396
	Terrestrial Bird	8	0	ND	ND	399		417	388
	Terrestrial Mammal	5	0	ND	ND	401		421	386
2 Palo Verde Drain									
	Aquatic Vegetation	2	0	ND	ND	395		400	390
	Sediment	3	0	ND	ND	80.3		80.6	80.0
	Surface Water	4	0	ND	ND	4.00		4.00	4.00
	Soil	3	0	ND	ND	79.8		79.8	79.7
3 Imperial Dam									
	Surface Water	5	5	4.20	3.00	3.64	3.64	4.00	4.00

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Table 3.3 Media and Location Specific Perchlorate Concentrations

Site 1, Yuma, AZ

Location	Medium/ Matrix	No. of Samples	No. Detected	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
4 Ag Lease 1									
	Aquatic Bird	1	0	ND	ND	400		400	400
	Fish	1	0	ND	ND	400		400	400
	Aquatic Vegetation	3	0	ND	ND	395		408	378
	Pore Water	1	0	ND	ND	4.00		4.00	4.00
	Sediment	4	0	ND	ND	81.0		83.6	79.2
	Surface Water	4	4	4.10	4.00	4.05	4.05	4.00	4.00
	Soil	2	0	ND	ND	79.4		79.9	79.0
	Terrestrial Vegetation	9	1	650	ND	415	650	396	375
	Terrestrial Insect	2	0	ND	ND	415		436	394
	Terrestrial Mammal	8	0	ND	ND	403		417	386

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Table 3.3 Media and Location Specific Perchlorate Concentrations

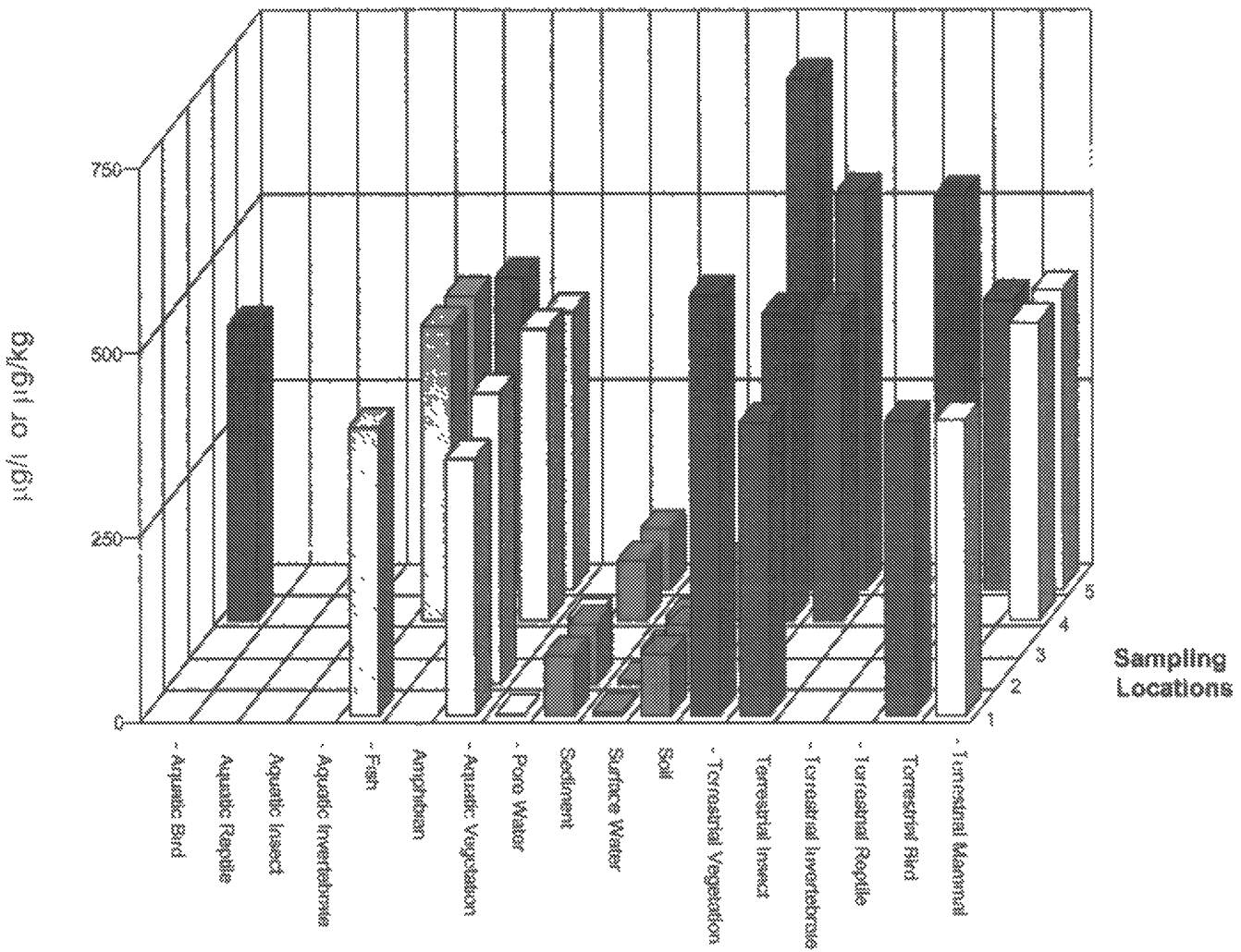
Site 1, Yuma, AZ

Location	Medium/ Matrix	No. of Samples	No. Detected	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
5 Ag Lease 2									
	Fish	5	0	ND	ND	396		400	392
	Amphibian	2	0	ND	ND	421		432	410
	Aquatic Vegetation	3	0	ND	ND	375		384	369
	Sediment	5	0	ND	ND	81.5		81.9	81.0
	Surface Water	6	1	5.00	ND	4.17	5.00	4.00	4.00
	Soil	5	0	ND	ND	80.0		80.3	79.7
	Terrestrial Vegetation	8	5	1790	ND	690	872	418	370
	Terrestrial Insect	3	0	ND	ND	536		822	392
	Terrestrial Reptile	1	1	531	531	531	531	412	412
	Terrestrial Bird	1	0	ND	ND	390		390	390
	Terrestrial Mammal	5	0	ND	ND	404		419	394

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

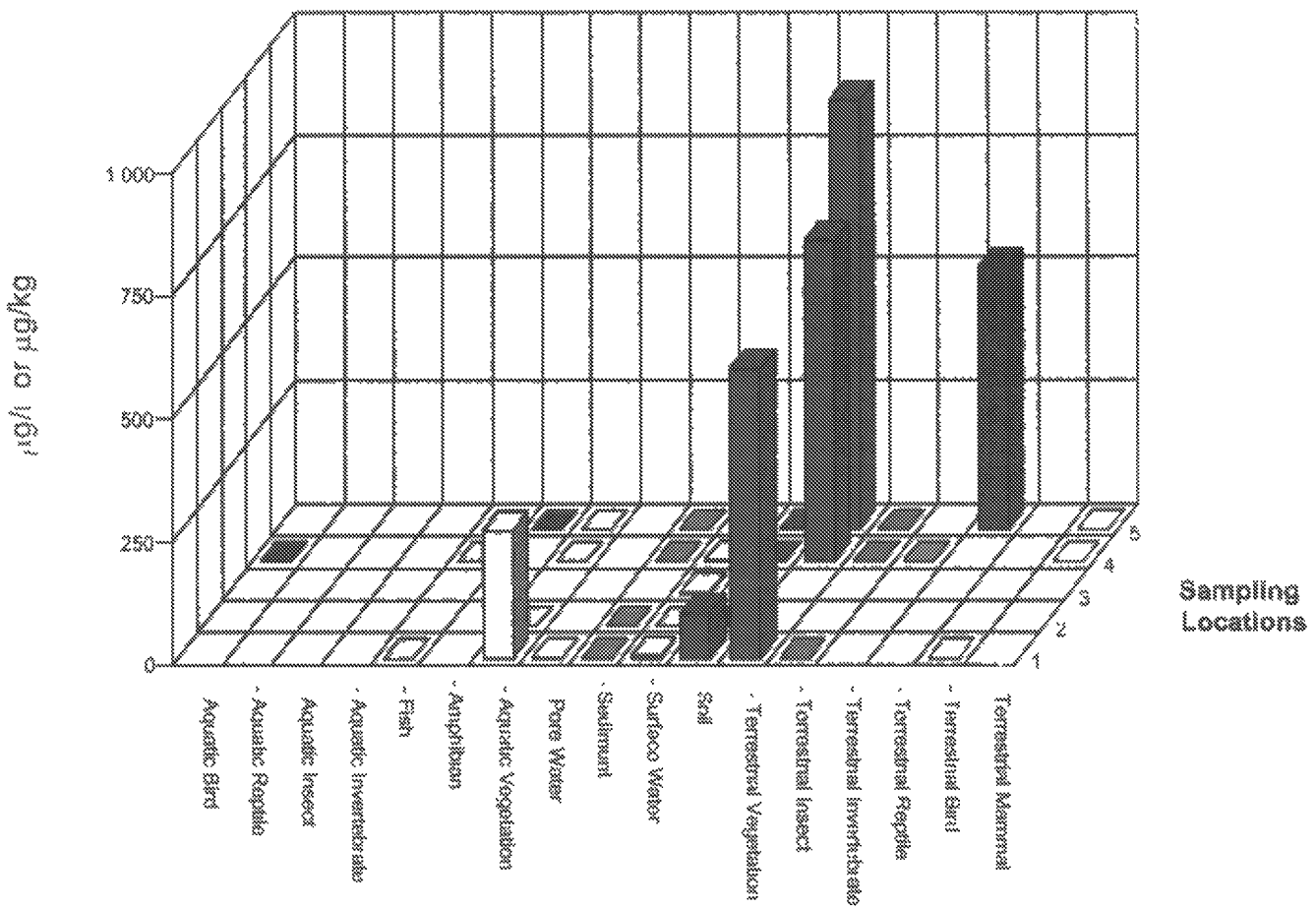
Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Figure 3.2a
Media and Location-Specific Mean Perchlorate Concentrations
Mean of All Results
Yuma, Arizona



In calculation of the mean, the reporting limit is used for non-detected values
Sediment, soil, and tissue sample results are reported in µg/kg
Surface water and pore water results are reported in µg/L

Figure 3.2b
Mean and Location-Specific Mean Perchlorate Concentrations
Mean of Detected Results
Yuma, Arizona



In calculation of the mean, only samples with detectable levels of perchlorate were used
Sediment, soil, and tissue sample results are reported in µg/kg
Surface water and pore water results are reported in µg/L

3.1.2 Lake Mead National Recreation Area and the Las Vegas Wash, Nevada

Three locations along the Las Vegas Wash in the Lake Mead Area west of Las Vegas, Nevada were sampled. Table 3.4 summarizes mean perchlorate concentrations by location, matrix, medium type and medium group in tabular format and Figure 3.3a presents the mean perchlorate concentrations for all samples (using the RL for all samples without detected perchlorate) in graphical format. Figure 3.3b presents the mean concentrations of detected perchlorate samples. Samples containing values of perchlorate below reporting limits are not used to calculate the mean concentration.

Samples collected at Location 1 of this site were actually collected at three sub-locations. Location 1 UP (upstream) is the surface discharge pond of one of the major springs from the Kerr McGee contaminant plume. This is also the location of the intake for the ion exchange treatment plant. This is considered the primary point source for perchlorate in the Wash. The actual sample location is the upstream (west) side of the concrete dam/intake structure for the ion exchange wastewater treatment plant. Included with the data from Location 1 UP are two fish samples and two surface water samples, which were collected at an impoundment located approximately 150-feet upstream of the ion exchange treatment plant intake. The results from these two sub-locations are presented together. Location 1 DN (downstream) is the downstream side (east) of the concrete dam/intake structure for the ion exchange wastewater treatment plant.

The figures and tables use Location 1 UP data unless otherwise noted. This upstream data is used because it is most representative of the concentrations to which plants and animals in the area are subjected. In this sample area, the marshes created by the water of Location 1 UP cover approximately 0.5 acre, while the ponded area of Location 1 DN covers only approximately 500 square feet. In addition, most of the samples collected at this location were obtained closer to Location 1 UP water.

The perchlorate concentration in Location 1 DN would be more indicative of the concentration for this spring discharge into the Wash (after treatment); however, it is likely that there are other groundwater pathways directly into the Wash.

3.1.2.1 Location and Media Specific Analytical Comparisons

As shown in Table 3.4, Figure 3.3a and Figure 3.3b, perchlorate concentrations in the Lake Mead Area are relatively high and vary greatly between sampling locations and media. Media at Location 1 were collected near a spring that discharges into the Las Vegas Wash from a known perchlorate groundwater plume. As expected, all media collected at Location 1 contain much higher concentrations of perchlorate than media collected at Locations 3 or 5. Mean perchlorate concentrations decreased significantly in all media with distance from Location 1 and the known source of perchlorate.

Table 3.4 Media and Location Specific Perchlorate Concentrations

Site 2, Lake Mead Area

Location	Medium/ Matrix	No. of Samples	No. Detected	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
1 Spring									
	Fish	2	2	44300	4310	24300	24300	5740	392
	Aquatic Vegetation	3	3	176000	121000	143000	143000	20900	19800
	Pore Water	1	1	98000	98000	98000	98000	4.00	4.00
	Sediment	6	6	56000	23000	44700	44700	81.2	79.8
	Surface Water	8	8	130000	100000	106000	106000	20000	4.00
	Soil	5	5	1470000	367	450000	450000	80.5	77.0
	Terrestrial Vegetation	9	9	428000	12800	189000	189000	40000	384
	Terrestrial Insect	1	1	4170	4170	4170	4170	407	407
	Terrestrial Bird	5	3	4190	ND	1520	2270	400	381
	Terrestrial Mammal	8	8	53000	8440	25400	25400	6220	406
	Pore Water - downstream	1	1	1000	1000	1000	1000	4.00	4.00
	Sediment - downstream	6	2	21700	ND	4220	12500	82.6	78.0
	Surface Water - downstream	6	6	19000	12000	13300	13300	4.00	4.00

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Table 3.4 Media and Location Specific Perchlorate Concentrations

Site 2, Lake Mead Area

Location	*Medium/ Matrix*	*No. of Samples*	*No. Detected*	*Maximum Adj Result*	*Minimum Adj Result*	*Mean of All Results*	*Mean of Detected Results*	*Maximum Adj RL*	*Minimum Adj RL*
3 ECS3-Upstream of Lake Las Vegas									
	Aquatic Invertebrate	1	0	ND	ND	381		381	381
	Fish	7	0	ND	ND	384		392	381
	Amphibian	5	0	ND	ND	398		400	396
	Aquatic Vegetation	4	1	320	ND	375	320	407	377
	Sediment	5	3	146	ND	101	116	80.4	78.0
	Surface Water	6	6	480	440	458	458	4.00	4.00
	Soil	5	4	121000	ND	39400	49200	80.3	80.0
	Terrestrial Vegetation	15	15	99200	741	37900	37900	416	372
	Terrestrial Insect	2	2	4730	3200	3970	3970	386	385
	Terrestrial Mammal	4	4	4230	1290	2380	2380	419	385

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Table 3.4 Media and Location Specific Perchlorate Concentrations

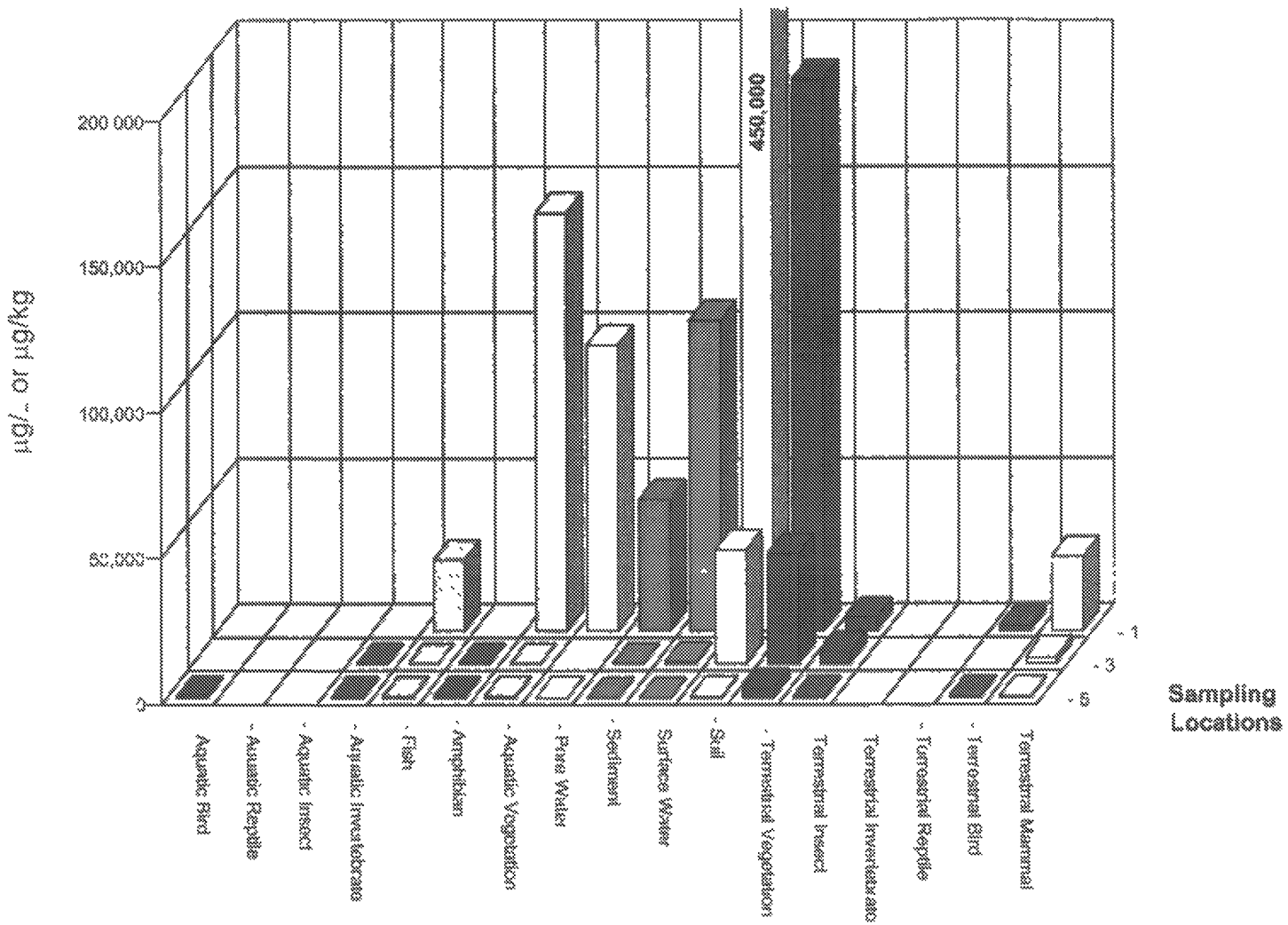
Site 2, Lake Mead Area

Location	*Medium/ Matrix*	*No. of Samples*	*No. Detected*	*Maximum Adj Result*	*Minimum Adj Result*	*Mean of All Results*	*Mean of Detected Results*	*Maximum Adj RL*	*Minimum Adj RL*
5 Lake Mead, Las Vegas Delta Area									
	Aquatic Bird	8	0	ND	ND	397		402	390
	Aquatic Invertebrate	1	0	ND	ND	398		398	398
	Fish	10	0	ND	ND	393		402	385
	Amphibian	1	0	ND	ND	421		421	421
	Aquatic Vegetation	2	0	ND	ND	394		405	383
	Pore Water	1	1	140	140	140	140	4.00	4.00
	Sediment	5	2	81.4	ND	78.8	74.5	83.6	78.0
	Surface Water	9	7	68.0	ND	43.8	55.1	4.00	4.00
	Soil	5	4	672	ND	392	471	80.2	79.2
	Terrestrial Vegetation	9	9	5720	516	2790	2790	416	390
	Terrestrial Insect	1	0	ND	ND	388		388	388
	Terrestrial Bird	2	1	401	ND	397	401	392	385
	Terrestrial Mammal	6	3	447	ND	425	441	417	394

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

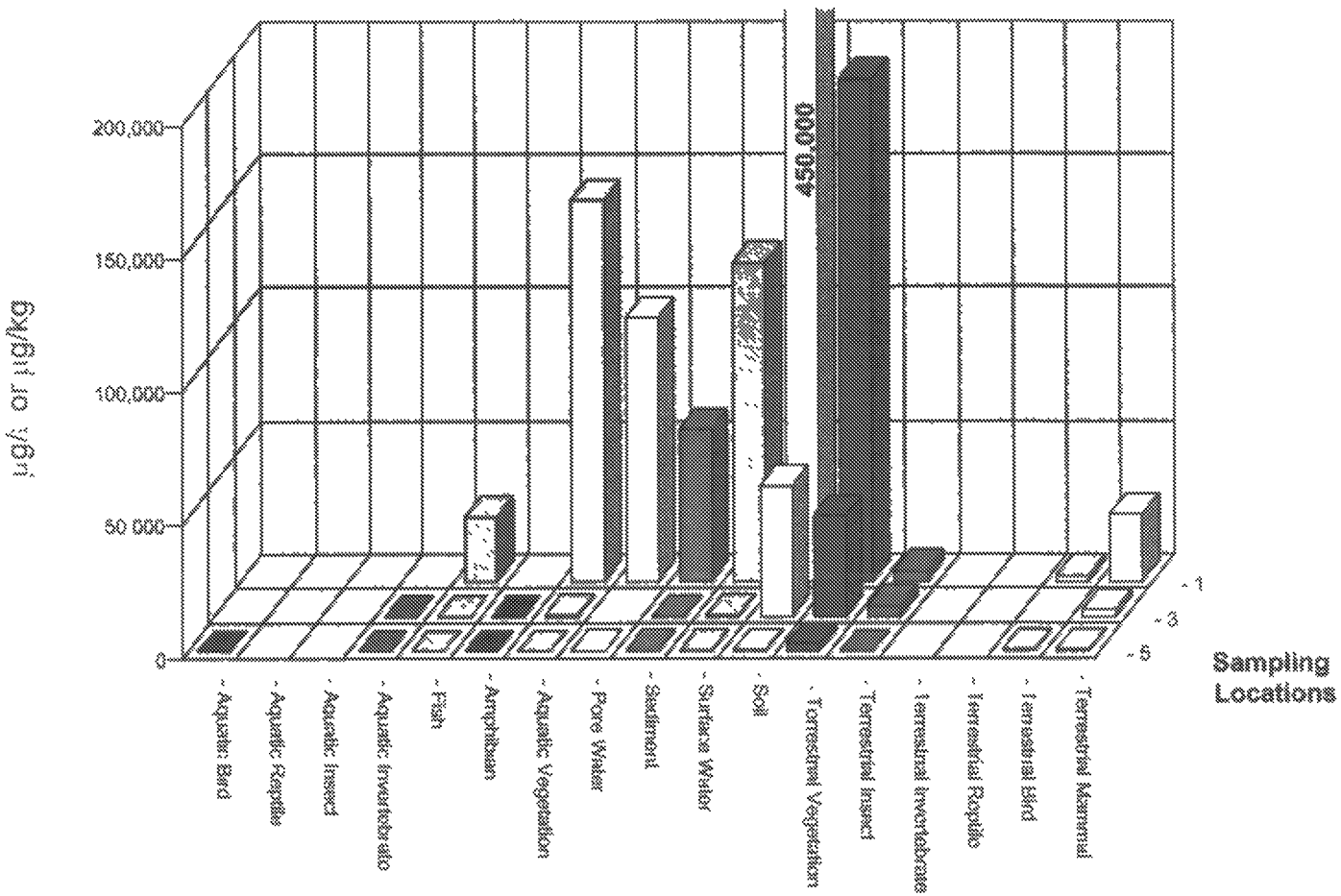
Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Figure 3.3a
Media and Location-Specific Mean Perchlorate Concentrations
Mean of All Results
Lake Mead Area



In calculation of the mean, the reporting limit is used for nondetected values.
Sediment, soil, and tissue sample results are reported in µg/kg.
Surface water and pore water results are reported in µg/L.

Figure 3.3b
Mean and Location-Specific Mean Perchlorate Concentrations
Mean of Detected Results
Lake Mead Area



In calculation of the mean, only samples with detectable levels of perchlorate were used
Sediment, soil, and tissue sample results are reported in µg/kg
Surface water and pore water results are reported in µg/L

Overall, mean concentrations of perchlorate in the Lake Mead Area are highest in soil and terrestrial vegetation. Assuming the comparison value is 1 and the first order of magnitude is 10 µg/kg, each of these media contained concentrations of perchlorate ranging between five orders of magnitude at Location 1 (450,000 µg/kg in soil and 189,000 in vegetation), four orders of magnitude at Location 3 (39,400 µg/kg in soil and 37,900 in vegetation), and between two and three orders of magnitude at Location 5 (376 µg/kg in soil and 2,790 µg/kg in vegetation).

Mean concentrations of perchlorate detected in some media dropped steadily from Location 1 to Location 5, while other media detectable perchlorate concentrations dropped significantly with distance from Location 1. For example, collected mammal samples decrease steadily from Location 1 (25,400 µg/kg) to Location 3 (2,380 µg/kg) to Location 5 (221 µg/kg), while mean perchlorate concentrations in fish dropped from 24,300 µg/kg at Location 1 to not detected at Locations 3 and 5. External lesions were noted on sample 02050503 (largemouth bass) from Las Vegas Bay. However, perchlorate was not detected in this sample.

Terrestrial insects contained relatively consistent mean concentrations of perchlorate at Locations 1 and 3 (4,170 µg/kg and 3,970 µg/kg, respectively), but perchlorate was not detected in terrestrial insects collected at Location 5.

In the Lake Mead Area, aquatic birds, aquatic invertebrates, and amphibians collected for analyses did not contain concentrations of perchlorate that were greater than laboratory RLs. Samples of aquatic reptiles, aquatic insects, terrestrial invertebrates, terrestrial reptiles, and groundwater were not collected.

3.1.3 Allegany Ballistics Laboratory, Rocket Center, West Virginia

Eight locations near the Potomac River at the Maryland/West Virginia border were sampled at ABL. Various media were collected from each of the eight locations and analyzed for perchlorate. Table 3.5 summarizes mean perchlorate concentrations by location, matrix, medium type and medium group in tabular format and Figure 3.4a presents the mean perchlorate concentrations for all samples (using the RL for all samples without detected perchlorate) in graphical format. Figure 3.4b presents the mean concentrations of detected perchlorate samples. Samples containing values of perchlorate below reporting limits are not used to calculate the mean concentration

3.1.3.1 Location and Media Specific Analytical Comparisons

As shown in Table 3.5, Figure 3.4a and Figure 3.4b, perchlorate was detected in various media at most locations at ABL.

Table 3.5 Media and Location Specific Perchlorate Concentrations

Site 3, Allegany Ballistics Laboratory

Location	Medium/ Matrix	No. of Samples	No. Detected	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
1 Burn Area									
	Soil	9	9	22100	117	3500	3500	80.1	78.0
	Terrestrial Vegetation	9	9	300000	9920	83300	83300	39600	2020
	Terrestrial Insect	2	1	12600	ND	6510	12600	2040	419
	Terrestrial Bird	5	1	423	ND	403	423	419	394
	Terrestrial Mammal	2	0	ND	ND	391		392	390
2 Former Bldg 105 (SWMU 37L)									
	Soil	6	0	ND	ND	80.2		83.6	76.8
	Terrestrial Vegetation	6	2	290	ND	360	289	408	384
	Terrestrial Insect	1	0	ND	ND	421		421	421
	Terrestrial Invertebrate	1	0	ND	ND	404		404	404
3 Bldg 446 (SWMU 37E)									
	Soil	3	3	990	88.0	400	400	80.2	80.0
	Terrestrial Vegetation	6	3	1160	ND	639	881	408	384
	Terrestrial Insect	3	0	ND	ND	395		398	390

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Table 3.5 Media and Location Specific Perchlorate Concentrations

Site 3, Allegany Ballistics Laboratory

Location	Medium/ Matrix	No. of Samples	No. Detected	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
4 SSL2									
	Fish	10	0	ND	ND	401		410	388
	Sediment	3	1	104	ND	87.9	104	79.8	78.0
	Surface Water	3	3	6.60	5.75	6.17	6.17	4.00	4.00
	Terrestrial Insect	2	0	ND	ND	396		400	392
5 Upstream of Weir (WWTP)									
	Fish	2	1	451	ND	431	451	410	410
	Amphibian	2	0	ND	ND	410		417	404
	Aquatic Vegetation	9	0	ND	ND	398		408	391
	Sediment	3	1	119	ND	93.5	119	81.6	80.0
	Surface Water	6	6	270	240	258	258	80.0	80.0
	Soil	3	0	ND	ND	79.5		81.4	77.0
	Terrestrial Insect	2	0	ND	ND	414		421	407
	Terrestrial Mammal	3	0	ND	ND	413		417	406

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Table 3.5 Media and Location Specific Perchlorate Concentrations

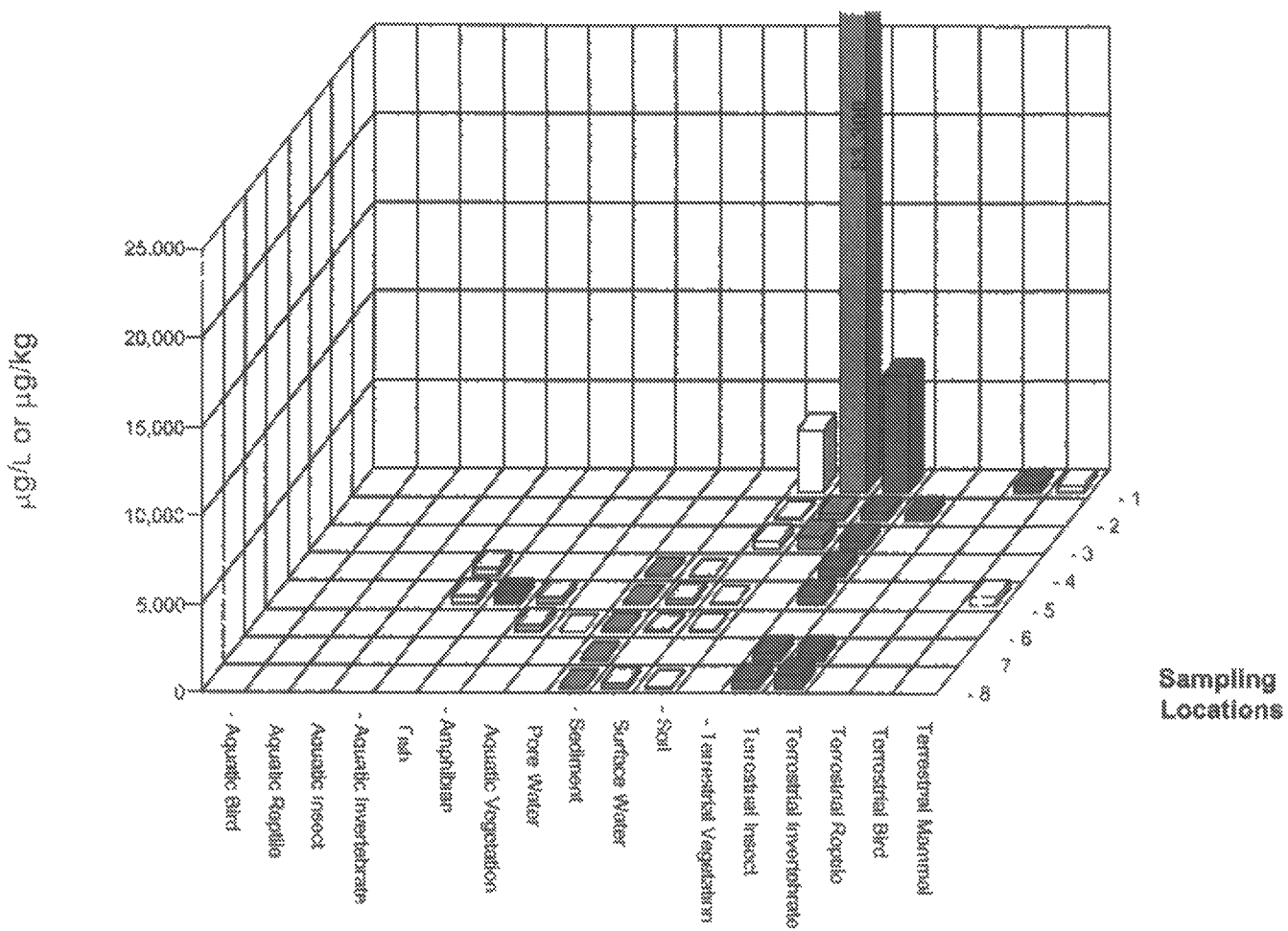
Site 3, Allegany Ballistics Laboratory

Location	Medium/ Matrix	No. of Samples	No. Detected	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
6 Downstream of Weir (WWTP)									
	Aquatic Vegetation	3	0	ND	ND	396		400	390
	Pore Water	2	2	23.0	14.0	18.5	18.5	4.00	4.00
	Sediment	3	0	ND	ND	81.0		81.6	79.8
	Surface Water	6	0	ND	ND	80.0		80.0	80.0
	Soil	3	1	112	ND	90.7	112	80.1	77.0
7 Storm Drain									
	Sediment	3	0	ND	ND	80.3		81.9	79.2
	Terrestrial Insect	1	0	ND	ND	407		407	407
	Terrestrial Invertebrate	2	0	ND	ND	404		417	392
8 CERCLA Site 1 Outfall									
	Sediment	3	0	ND	ND	78.5		79.2	77.0
	Surface Water	6	6	280	250	268	268	80.0	80.0
	Soil	3	0	ND	ND	81.7		83.6	80.1
	Terrestrial Insect	2	0	ND	ND	400		412	388
	Terrestrial Invertebrate	1	0	ND	ND	800		800	800

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

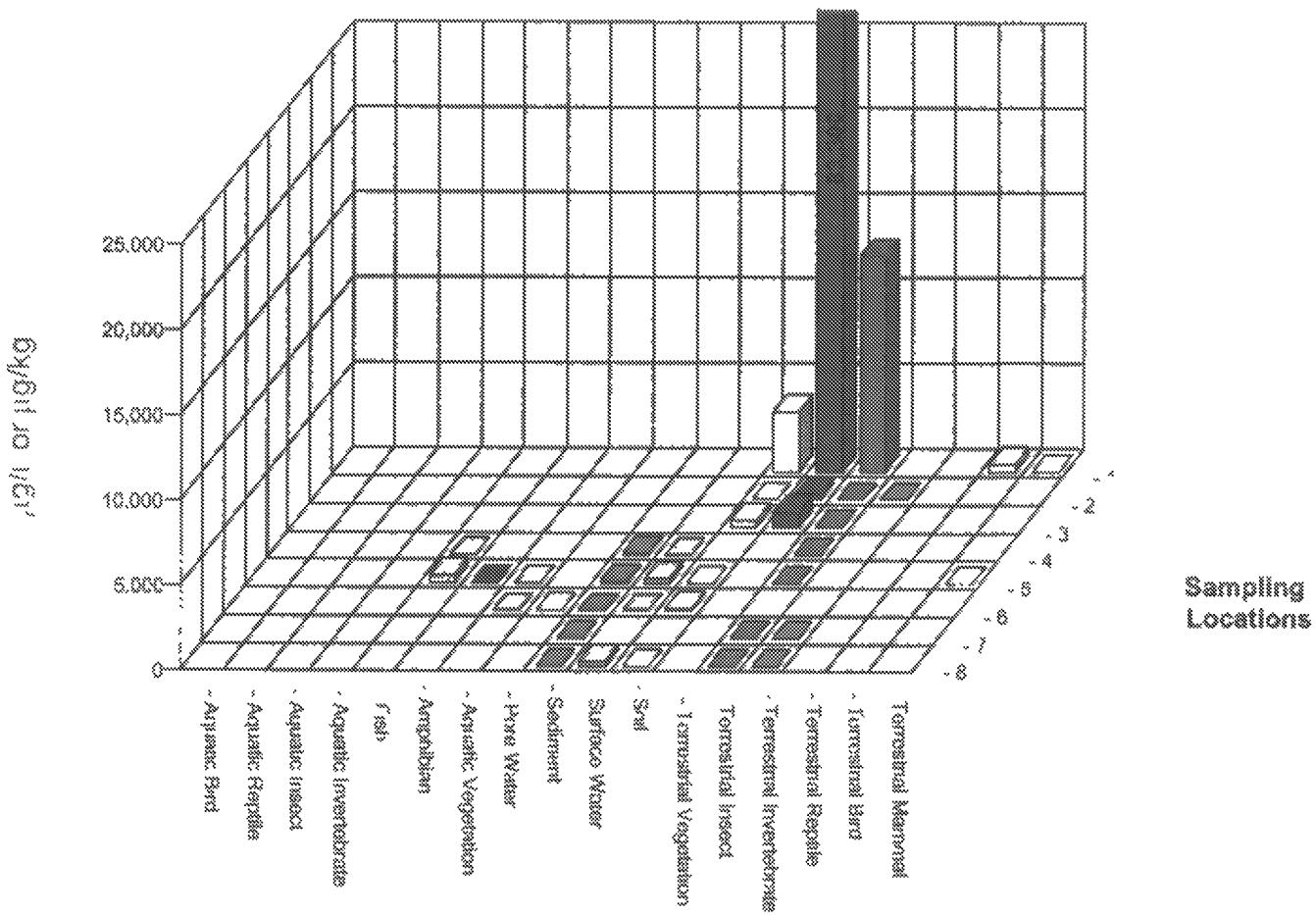
Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Figure 3.4a
Media and Location-Specific Mean Perchlorate Concentrations
Mean of All Results
Allegany Ballistics Laboratory



[illegible] calculation of the mean, the reporting limit is used for nondetected values.
Sediment, soil, and tissue sample results are reported in µg/kg.
Surface water and pore water results are reported in µg/L.

Figure 3.4b
Mean and Location-Specific Mean Perchlorate Concentrations
Mean of Detected Results
Allegany Ballistics Laboratory



In calculation of the mean, only samples with detectable levels of perchlorate were used
Sediment, soil, and tissue sample results are reported in µg/kg
Surface water and pore water results are reported in µg/l

Obvious trends in mean perchlorate concentrations among the media groups were evident only at the Burn Area (Location 1) where mean concentrations increased from terrestrial birds (84.6 µg/kg) to terrestrial insects (6,300 µg/kg) to terrestrial vegetation (83,300 µg/kg). Due to sample collection variability, comparisons of mean perchlorate concentrations between sampling locations at ABL was possible for relatively few media (terrestrial insects and soil). For both terrestrial insects and soil, mean perchlorate concentrations were at least an order of magnitude higher at Location 1 (6,300 µg/kg and 3,500 µg/kg, respectively) than at any other location at ABL. Coincidentally, mean concentrations of perchlorate in terrestrial vegetation, detected above the laboratory reporting limit at all locations where it was sampled, was two orders of magnitude higher at Location 1 (83,300 µg/kg) than at Locations 2 or 3 (93.6 µg/kg and 441 µg/kg, respectively).

Perchlorate was not detected above laboratory RLs in amphibian, aquatic vegetation, terrestrial invertebrate, or mammal samples collected, and it was not detected above laboratory RLs in any media collected at Location 7. Samples of aquatic birds, aquatic reptiles, aquatic insects, aquatic invertebrates, terrestrial reptiles and groundwater were not collected at the ABL site.

3.1.4 Holloman Air Force Base, New Mexico

Six locations near the test track and storage areas at Holloman AFB and along the Lost River were sampled. Various media were collected from each of the six locations and analyzed for perchlorate. Table 3.6 summarizes mean perchlorate concentrations by location, matrix, medium type and medium group in tabular format and Figure 3.5a presents those mean perchlorate concentrations for all samples (using the RL for all samples without detected perchlorate) in graphical format. Figure 3.5b presents the mean concentrations of detected perchlorate samples. Samples containing values of perchlorate below reporting limits are not used to calculate the mean concentration.

3.1.4.1 Location and Media Specific Analytical Comparisons

As shown in Table 3.6, Figure 3.5a and Figure 3.5b, perchlorate was not detected at a high frequency at this site, precluding the ability to describe locations as containing relatively higher or lower perchlorate levels. Comparisons among media are similarly difficult. Nonetheless, perchlorate was present at detectable levels in terrestrial vegetation, but absent in all other biological media, suggesting that terrestrial vegetation may be especially prone to absorbing perchlorate from the environment. For example, south of Building 1176 (Location 1) perchlorate was detected in terrestrial vegetation (238 µg/kg) and in soil (310 µg/kg). However, this relationship was not replicated east of Building 1176 (Location 2), where perchlorate was detected above laboratory reporting limits in terrestrial vegetation (1,030 µg/kg), but not in soil. Perchlorate in groundwater samples collected by base personnel ranged from non-detected to 38 µg/L with a mean of 10.9 µg/L in detected values. However these groundwater samples are not co-located with any of the samples obtained in this study.

Table 3.6 Media and Location Specific Perchlorate Concentrations

Site 4, Holloman AFB

Location	Medium/ Matrix	No. of Samples	No. Detected	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
0 Monitoring Wells									
	Groundwater	19	15	38.0	ND	11.7	13.7	4.00	4.00
1 South Bldg 1176									
	Pore Water	1	0	ND	ND	400		400	400
	Sediment	3	0	ND	ND	80.4		81.6	79.2
	Soil	13	9	2800	ND	334	447	800	78.0
	Terrestrial Vegetation	15	4	1550	ND	526	892	420	370
	Terrestrial Mammal	7	0	ND	ND	403		415	389
2 East Bldg 1176									
	Soil	3	0	ND	ND	320		803	79.2
	Terrestrial Vegetation	15	5	7600	ND	1290	3090	418	372
	Terrestrial Reptile	1	0	ND	ND	408		408	408
	Terrestrial Bird	4	0	ND	ND	405		414	396
	Terrestrial Mammal	10	0	ND	ND	402		417	392
4 First Lost River									
	Sediment	3	0	ND	ND	325		819	78.0

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\73745I\Data\Lab\ABL\Clay_Lab_Data.mdb

Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Table 3.6 Media and Location Specific Perchlorate Concentrations

Site 4, Holloman AFB

Location	Medium/ Matrix	No. of Samples	No. Detected	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
5 Second Lost River									
	Fish	3	0	ND	ND	402		419	388
	Sediment	3	1	105	ND	89.0	105	81.0	80.6
	Surface Water	6	0	ND	ND	400		400	400
	Soil	3	0	ND	ND	78.5		79.3	78.0
	Terrestrial Vegetation	9	0	ND	ND	394		403	380
	Terrestrial Bird	1	0	ND	ND	385		385	385
	Terrestrial Mammal	5	0	ND	ND	403		410	390
6 Third Lost River									
	Fish	3	0	ND	ND	398		404	392
	Sediment	3	0	ND	ND	80.3		80.5	80.0
	Surface Water	6	0	ND	ND	400		400	400
	Soil	3	0	ND	ND	79.3		81.6	78.0
	Terrestrial Vegetation	9	0	ND	ND	397		406	380
7 Fourth Lost River									
	Fish	3	0	ND	ND	396		408	385
	Aquatic Vegetation	3	0	ND	ND	407		416	396
	Sediment	3	0	ND	ND	80.6		81.6	79.2
	Surface Water	6	0	ND	ND	400		400	400
	Soil	3	0	ND	ND	80.5		81.6	78.4
	Terrestrial Vegetation	9	0	ND	ND	397		416	380
	Terrestrial Bird	1	0	ND	ND	414		414	414
	Terrestrial Mammal	6	0	ND	ND	408		421	396

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
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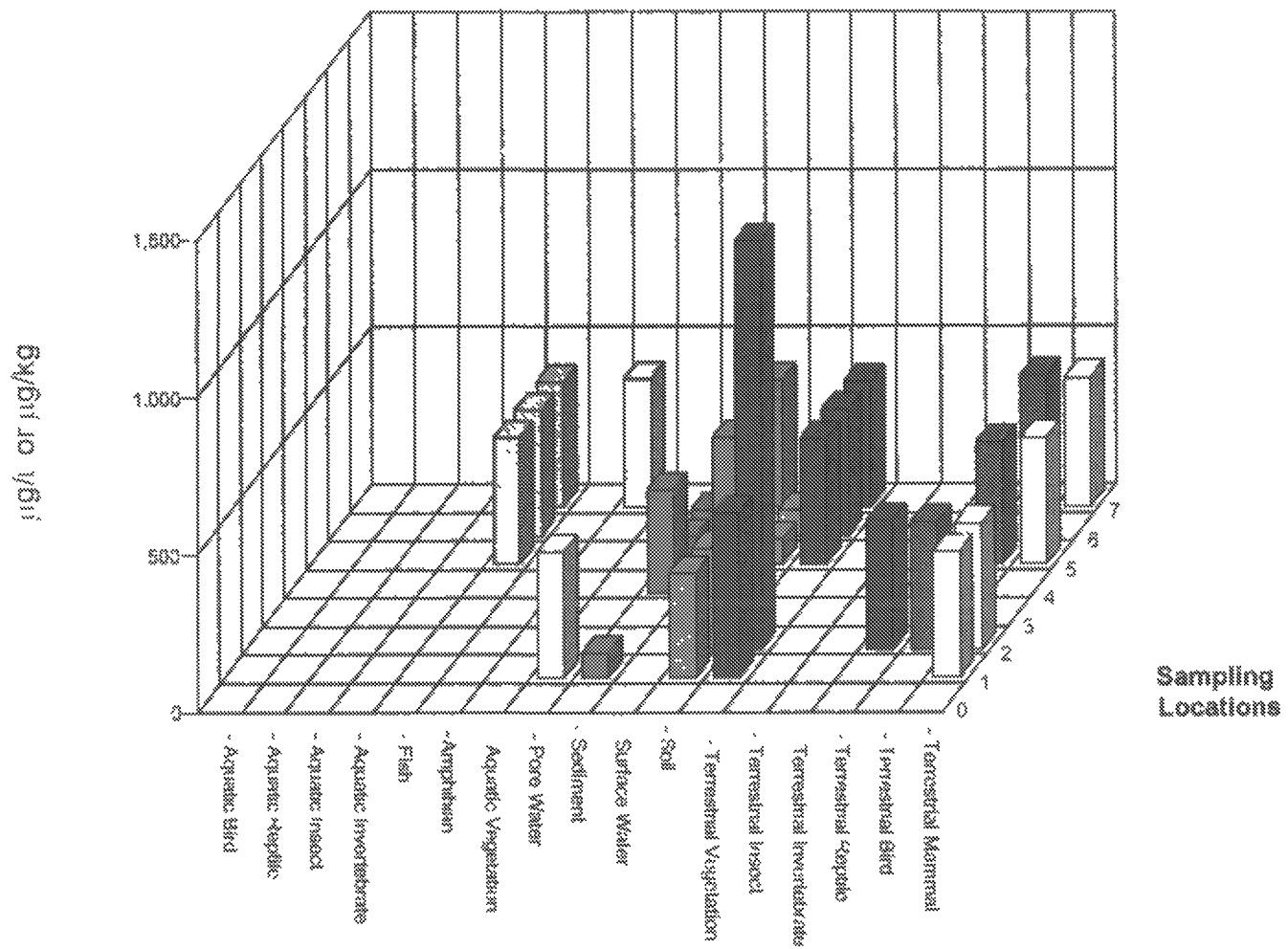
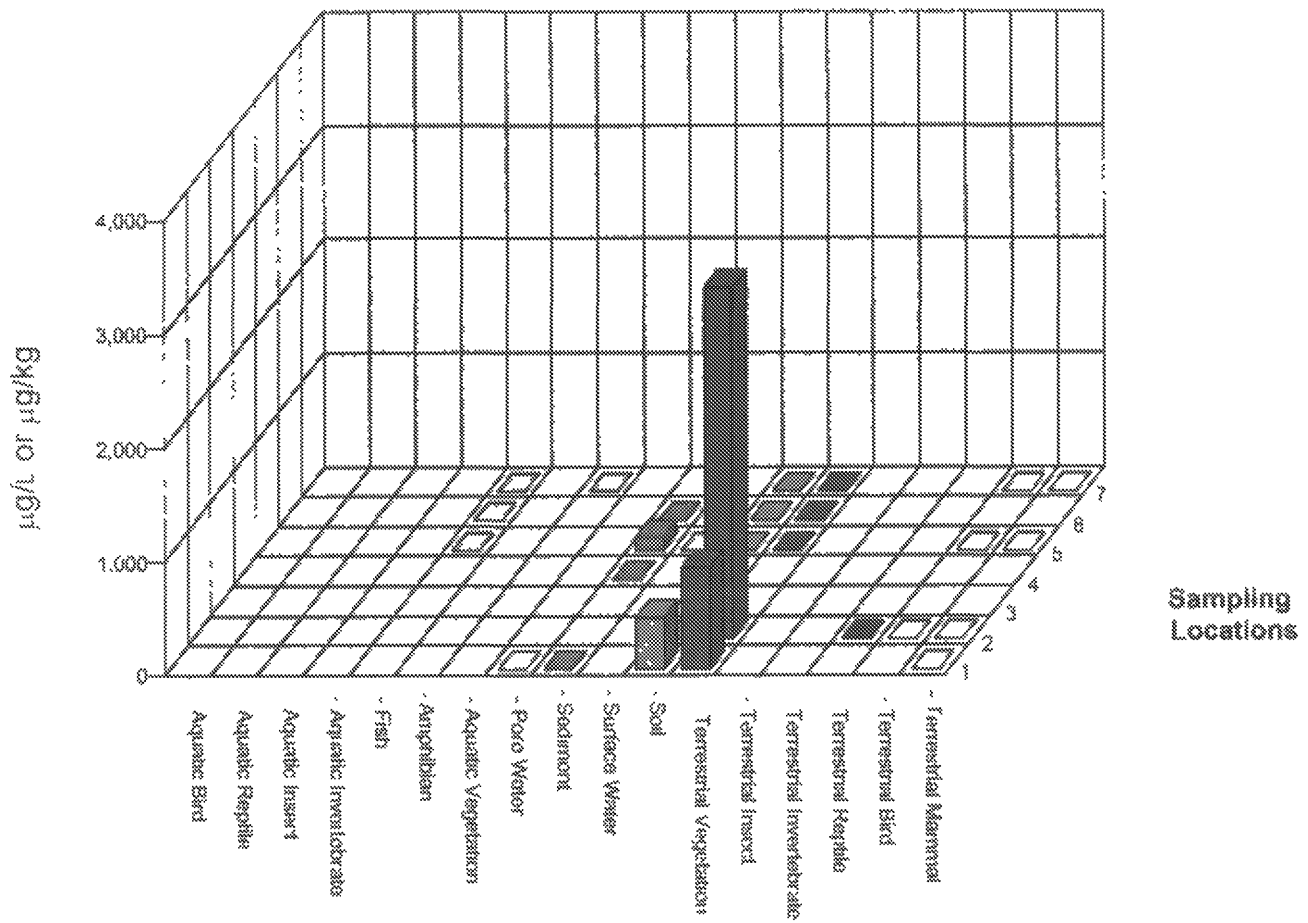
Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Figure 3.5a
Media and Location-Specific Mean Perchlorate Concentrations
Mean of All Results
Holloman Air Force Base



In calculation of the mean, the reporting limit is used for nondetected values.
Sediment, soil, and tissue sample results are reported in µg/kg.
Surface water and pore water results are reported in µg/L.

Figure 3.5b
Mean and Location-Specific Mean Perchlorate Concentrations
Mean of Detected Results
Holloman Air Force Base



In calculation of the mean, only samples with detectable levels of perchlorate were used
Sediment, soil, and tissue sample results are reported in µg/kg
Surface water and pore water results are reported in µg/l.

Perchlorate was not detected above laboratory RLs in fish, aquatic vegetation, pore water, surface water, terrestrial reptile, terrestrial bird or terrestrial mammal samples collected at Holloman. Due to variability in environmental conditions, samples of aquatic birds, aquatic reptiles, aquatic insects, aquatic invertebrates, terrestrial insects or terrestrial invertebrates were not collected at Holloman AFB.

3.1.5 Naval Surface Warfare Center, Indian Head, Maryland

Six locations along the Town Gut Marsh system were sampled at Indian Head. Table 3.7 summarizes mean perchlorate concentrations by location, matrix, medium type and medium group in tabular format and Figure 3.6a presents those mean perchlorate concentrations for all samples (using the RL for all samples without detected perchlorate) in graphical format. Figure 3.6b presents the mean of concentrations of detected perchlorate samples. Samples containing values of perchlorate below reporting limits are not used to calculate the mean concentration.

3.1.5.1 Location and Media Specific Analytical Comparisons

As shown in Table 3.7, Figure 3.6a and Figure 3.6b, perchlorate concentrations at Indian Head are relatively low across each media sampled, and at all six locations. However, sediments, soil, terrestrial vegetation, aquatic vegetation, amphibians and surface water samples collected did contain detectable concentrations of perchlorate. Terrestrial vegetation contains relatively high concentrations of perchlorate compared to other media collected at Indian Head. In addition, terrestrial vegetation appears to contain much higher concentrations of perchlorate (ranging from 36.7 µg/kg at Location 7 to 513 µg/kg at Location 5) when compared to soil collected from the same locations (58.8 µg/kg at Location 7 to not detected at Location 5). In addition, aquatic vegetation collected from Location 7 and Location 4 contain relatively high concentrations of perchlorate (127 µg/kg and 260 µg/kg, respectively) compared to sediment (not detected and 137 µg/kg, respectively) and surface water (not detected and 22.8 µg/L, respectively) collected in the same locations.

At each of the six locations at Indian Head, aquatic reptiles, aquatic insects, aquatic invertebrates, fish, pore water, terrestrial insects, terrestrial invertebrates, terrestrial birds and terrestrial mammals collected for analysis contain concentrations of perchlorate that are lower than laboratory reporting limits. Samples of aquatic birds, terrestrial reptiles, and groundwater were not collected at any locations at Indian Head.

Table 3.7 Media and Location Specific Perchlorate Concentrations

Site 5, Indian Head Area

Location	Medium/ Matrix	No. of Samples	No. Detected	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
3 West Creek of Town Gut Marsh									
	Amphibian	2	0	ND	ND	409		410	408
	Pore Water	1	0	ND	ND	4.00		4.00	4.00
	Sediment	3	1	3.80	ND	56.6	3.80	83.6	82.5
	Surface Water	6	3	4.00	ND	4.00	4.00	4.00	4.00
	Soil	4	3	132	ND	121	107	163	78.0
	Terrestrial Vegetation	3	2	891	ND	572	656	405	399
	Terrestrial Mammal	4	0	ND	ND	395		398	390
4 East Creek of Town Gut Marsh (at IW85)									
	Aquatic Insect	1	0	ND	ND	398		398	398
	Aquatic Invertebrate	2	0	ND	ND	404		406	402
	Fish	1	0	ND	ND	408		408	408
	Amphibian	2	1	465	ND	438	465	410	394
	Aquatic Vegetation	3	2	420	ND	395	390	408	396
	Sediment	3	2	318	ND	164	205	81.4	78.1
	Surface Water	6	6	24.0	22.0	22.8	22.8	4.00	4.00
	Soil	3	2	63.0	ND	75.7	33.0	161	78.0
	Terrestrial Vegetation	6	0	ND	ND	394		418	387
	Terrestrial Insect	1	0	ND	ND	406		406	406
	Terrestrial Mammal	1	0	ND	ND	417		417	417

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
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Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Table 3.7 Media and Location Specific Perchlorate Concentrations

Site 5, Indian Head Area

Location	Medium/ Matrix	No. of Samples	No. Detected	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
5 Town Gut Marsh 1 @ Atkin's Road									
	Aquatic Reptile	6	0	ND	ND	393		398	388
	Fish	13	0	ND	ND	402		415	390
	Amphibian	1	0	ND	ND	396		396	396
	Sediment	3	2	93.6	ND	88.5	91.9	81.7	78.0
	Surface Water	6	6	20.0	17.0	18.7	18.7	4.00	4.00
	Soil	3	0	ND	ND	79.6		81.6	76.8
	Terrestrial Vegetation	3	3	931	200	513	513	400	384
	Terrestrial Insect	2	0	ND	ND	399		400	398
	Terrestrial Bird	3	0	ND	ND	399		402	394
	Terrestrial Mammal	3	0	ND	ND	407		419	388
6 Town Gut Marsh 2 @ Atkin's Spur Road									
	Aquatic Reptile	1	0	ND	ND	412		412	412
	Fish	11	0	ND	ND	399		412	366
	Sediment	3	0	ND	ND	325		810	81.2
	Surface Water	6	6	25.0	24.0	24.8	24.8	4.00	4.00
	Soil	3	1	546	ND	263	546	164	83.6
	Terrestrial Insect	1	0	ND	ND	396		396	396
	Terrestrial Invertebrate	3	0	ND	ND	401		412	394
	Terrestrial Mammal	1	0	ND	ND	392		392	392

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Table 3.7 Media and Location Specific Perchlorate Concentrations

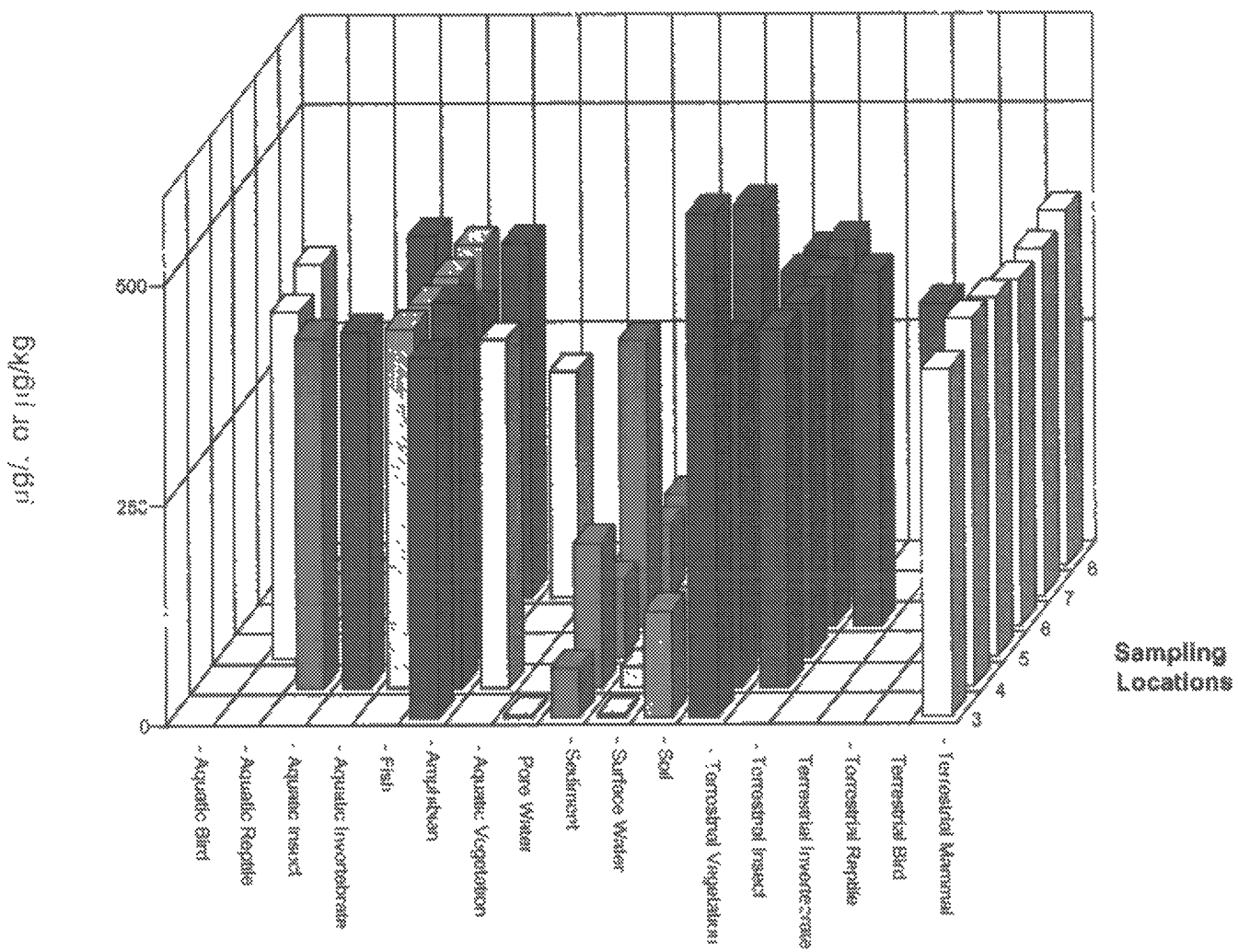
Site 5, Indian Head Area

Location	Medium/ Matrix	No. of Samples	No. Detected	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
7 Town Gut Marsh 3 @ Noble Rd									
	Aquatic Invertebrate	3	0	ND	ND	410		419	404
	Fish	7	0	ND	ND	399		417	385
	Amphibian	1	0	ND	ND	402		402	402
	Aquatic Vegetation	3	2	200	ND	255	190	405	384
	Pore Water	1	0	ND	ND	4.00		4.00	4.00
	Sediment	3	0	ND	ND	81.4		81.6	81.0
	Surface Water	6	6	12.0	12.0	12.0	12.0	4.00	4.00
	Soil	3	1	176	ND	138	176	159	79.0
	Terrestrial Vegetation	6	1	220	ND	367	220	403	385
	Terrestrial Insect	3	0	ND	ND	404		417	394
	Terrestrial Mammal	2	0	ND	ND	393		398	388
8 Creek East of Town Gut Marsh 2									
	Sediment	3	0	ND	ND	80.9		82.8	79.8
	Surface Water	6	0	ND	ND	4.00		4.00	4.00
	Soil	3	0	ND	ND	162		164	158
	Terrestrial Vegetation	3	1	270	ND	356	270	407	392
	Terrestrial Mammal	1	0	ND	ND	402		402	402

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

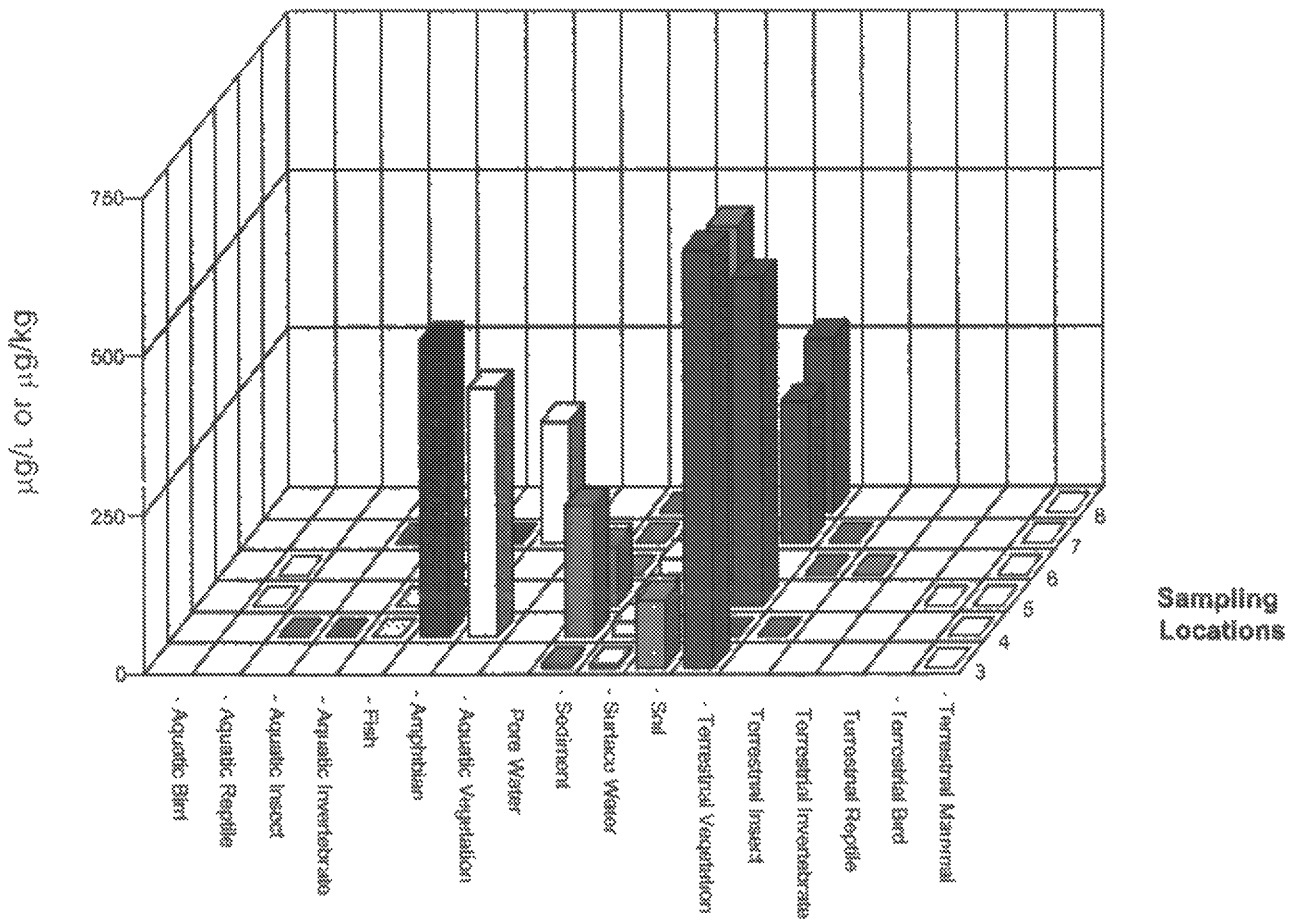
Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Figure 3.6a
Media and Location-Specific Mean Perchlorate Concentrations
Mean of All Results
Naval Surface Weapons Center, Indian Head, MD



In calculation of the mean, the reporting limit is used for nondetected values.
Sediment, soil, and tissue sample results are reported in µg/kg.
Surface water and pore water results are reported in µg/L.

Figure 3.6b
Mean and Location-Specific Mean Perchlorate Concentrations
Mean of Detected Results
Naval Surface Weapons Center, Indian Head, MD



In calculation of the mean, only samples with detectable levels of perchlorate were used.
Sediment, soil, and tissue sample results are reported in µg/kg.
Surface water and pore water results are reported in µg/L.

3.1.6 Longhorn Army Ammunition Plant, Karnack, Texas

Four locations were sampled at LHAAP. Table 3.8 summarizes mean perchlorate concentrations by location, matrix, medium type and medium group in tabular format and Figure 3.7a presents the mean perchlorate concentrations for all samples (using the RL for all samples without detected perchlorate) in graphical format. Figure 3.7b presents the mean concentrations of detected perchlorate samples. Samples containing values of perchlorate below reporting limits are not used to calculate the mean concentration.

3.1.6.1 Location and Media Specific Analytical Comparisons

As shown in Table 3.8, Figure 3.7a and Figure 3.7b, perchlorate was detected at only two locations at LHAAP. Downstream of Harrison Bayou (Location 5) perchlorate was only detected in terrestrial vegetation, with a mean concentration of 510 µg/kg. However, at the INF Pond (Location 2) perchlorate was detected in every biological media sampled except soil, terrestrial insects, and terrestrial birds. Thus, perchlorate appears prevalent at Location 2 but uncommon at the other locations. In a number of Location 2 media (mostly aquatic) mean perchlorate concentrations reached the parts-per-million level (aquatic vegetation, terrestrial vegetation, surface water, and amphibians). Many of the frogs composited for amphibian samples at Location 2 were observed as being in metamorphosis. One frog included in the composite samples had one eye sealed shut. The sample results (06020701 through 06020704) ranged from 867 µg/kg to 1,660 mg/kg perchlorate. No conclusions can be made regarding the relationship between this anomaly and perchlorate, although further study may be warranted. Overall, the biological media were comprehensively sampled at LHAAP with only three media remaining unrepresented (terrestrial invertebrates, aquatic birds, and groundwater).

3.2 SPECIFIC DATA COMPARISONS

3.2.1 Site Relationships

Of the six sites analyzed, Holloman AFB appears to contain the lowest frequency and mean concentration of perchlorate detected across all medium types and medium groups. After Holloman, it appears perchlorate concentration and frequency increase at Yuma and Indian Head, increase again slightly at LHAAP, then increase sharply at ABL. Mean concentrations of perchlorate detected in all medium and matrix types collected in the Lake Mead Area are much higher than at any other site analyzed.

Within the six sites investigated, various media at a total of 32 locations were sampled. Of the 32 sampling locations, the five locations with the overall highest mean perchlorate concentrations were Lake Mead Area, Location 1; Lake Mead Area, Location 3; ABL, Location 1; LHAAP, Location 2; and Lake Mead Area, Location 5.

Table 3.8 Media and Location Specific Perchlorate Concentrations

Site 6, LHAAP

Location	Medium/ Matrix	No. of Samples	No. Detected	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
2 INF Pond									
	Aquatic Insect	3	3	996	603	741	741	412	396
	Amphibian	4	4	1660	867	1200	1200	412	388
	Aquatic Vegetation	3	3	15700	9140	12000	12000	2020	402
	Sediment	3	2	286	ND	169	215	78.0	77.0
	Surface Water	6	6	3800	3500	3680	3680	2000	2000
	Soil	4	0	ND	ND	79.7		79.9	79.5
	Terrestrial Vegetation	6	6	2340	1220	1740	1740	406	390
	Terrestrial Insect	1	0	ND	ND	385		385	385
	Terrestrial Bird	3	0	ND	ND	394		406	384
	Terrestrial Mammal	3	1	571	ND	457	571	410	390
3 Discharge Point on Harrison Bayou									
	Aquatic Reptile	3	0	ND	ND	397		404	388
	Fish	8	0	ND	ND	402		417	387
	Aquatic Vegetation	6	0	ND	ND	394		404	381
	Sediment	3	0	ND	ND	79.7		81.4	78.2
	Surface Water	6	0	ND	ND	4.00		4.00	4.00
	Soil	3	0	ND	ND	78.1		79.2	77.0
	Terrestrial Vegetation	6	0	ND	ND	401		408	392
	Terrestrial Insect	1	0	ND	ND	392		392	392
	Terrestrial Bird	9	0	ND	ND	404		412	385
	Terrestrial Mammal	3	0	ND	ND	393		398	386

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Table 3.8 Media and Location Specific Perchlorate Concentrations

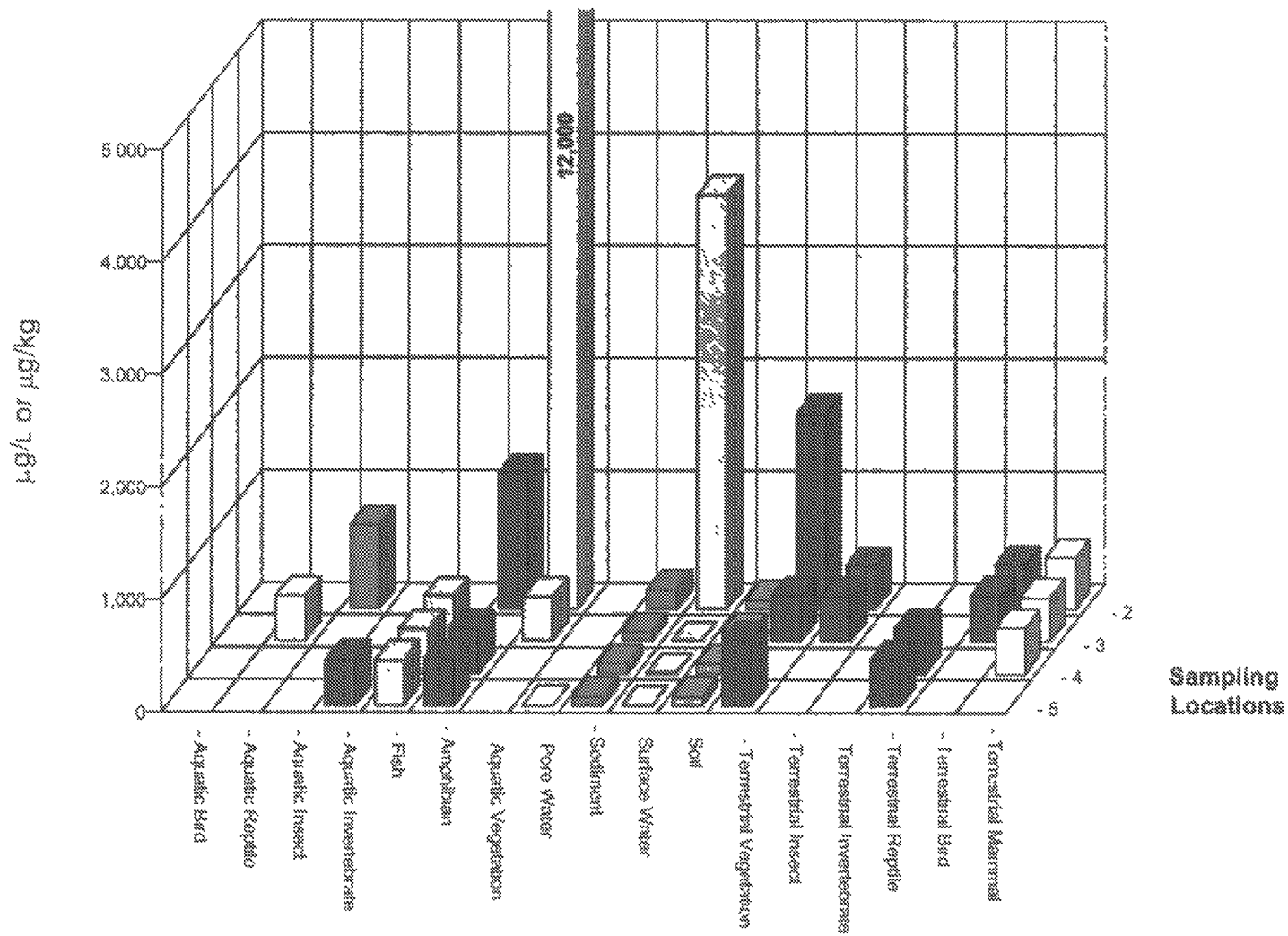
Site 6, LHAAP

Location	Medium/ Matrix	No. of Samples	No. Detected	Maximum Adj Result	Minimum Adj Result	Mean of All Results	Mean of Detected Results	Maximum Adj RL	Minimum Adj RL
4 Goose Prairie Creek (between 51st St and Karnack Ave)									
	Fish	10	0	ND	ND	392		402	385
	Amphibian	1	0	ND	ND	390		390	390
	Sediment	3	0	ND	ND	82.2		82.8	81.4
	Surface Water	6	0	ND	ND	4.00		4.00	4.00
	Soil	3	0	ND	ND	79.8		80.4	79.0
	Terrestrial Reptile	2	0	ND	ND	402		404	400
	Terrestrial Mammal	1	0	ND	ND	404		404	404
5 Harrison Bayou downstream (Sampling Site 9)									
	Aquatic Invertebrate	2	0	ND	ND	401		413	390
	Fish	10	0	ND	ND	399		417	386
	Amphibian	1	0	ND	ND	408		408	408
	Pore Water	1	0	ND	ND	4.00		4.00	4.00
	Sediment	3	0	ND	ND	80.3		80.5	80.0
	Surface Water	7	0	ND	ND	4.00		4.00	4.00
	Soil	3	0	ND	ND	80.8		82.5	79.8
	Terrestrial Vegetation	6	5	905	ND	578	612	406	388
	Terrestrial Reptile	3	0	ND	ND	399		404	394

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

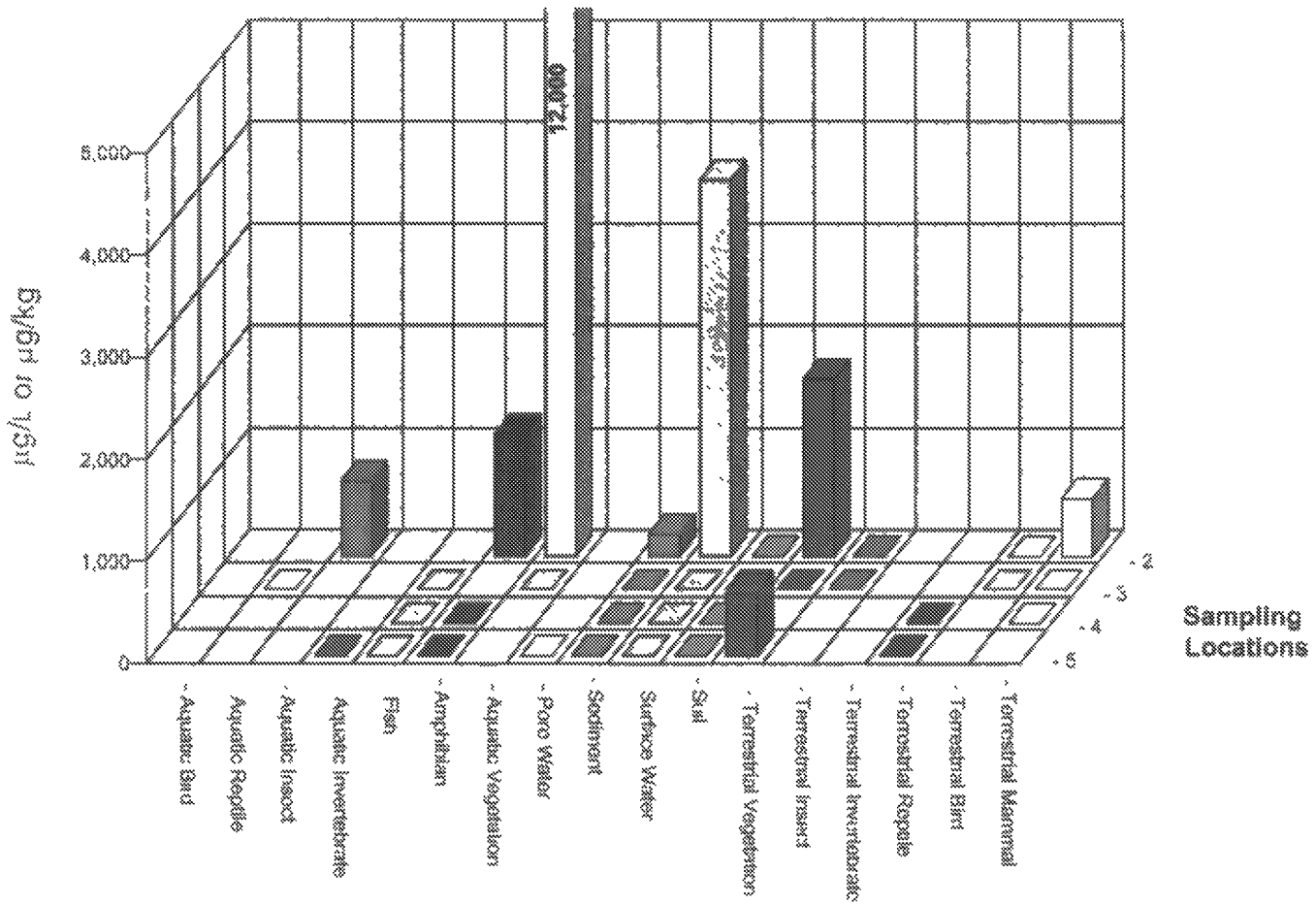
Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Figure 3.7a
Media and Location-Specific Mean Perchlorate Concentrations
Mean of All Results
Longhorn Army Ammunition Plant



In calculation of the mean, the reporting limit is used for nondetected values
Sediment, soil, and tissue sample results are reported in µg/kg
Surface water and pore water results are reported in µg/L

Figure 3.7b
Mean and Location-Specific Mean Perchlorate Concentrations
Mean of Detected Results
Longhorn Army Ammunition Plant



In calculation of the mean, only samples with detectable levels of perchlorate were used.
Sediment, soil, and tissue sample results are reported in µg/kg.
Surface water and pore water results are reported in µg/L.

When comparing potential pathways of perchlorate transfer within functional feeding groups, the Lake Mead Area consistently had the overall highest levels of mean perchlorate within each functional feeding group followed by LHAAP. ABL, Yuma, Indian Head and Holloman had relatively small and sporadic perchlorate concentrations within environmental media and functional feeding groups.

Due to the large number of samples collected and the relatively high concentrations of perchlorate detected in most media and functional feeding groups in the Lake Mead Area, relationships between individual species are presented and discussed in further detail in the following sections.

3.2.2 Inter-Species Relationships

Mean detected perchlorate concentrations for individual species collected at each of the three locations sampled in the Lake Mead Area are presented in Table 3.9. As shown in Table 3.9, abiotic media (terrestrial soils, sediments and surface water) contained mean perchlorate concentrations exceeding laboratory reporting limits at all three sampling locations. However, as previously stated, the mean concentrations of perchlorate decreases significantly with distance from Location 1 and the source of perchlorate.

Samples of several species were collected at all three locations in the Lake Mead Area. A comparison of these species may suggest the propensity of particular organisms to accumulate perchlorate with decreasing distance from the source area. For example, terrestrial invertebrates (misc. insects) were collected at all three locations. At Locations 1 and 3, perchlorate was detected in all insects collected (1 of 1 and 2 of 2, respectively). However, at Location 5, perchlorate was not detected in any insect samples collected (0 of 1).

One species of herbivorous mammal (cactus mouse) was collected at each of the locations in the Lake Mead Area. A mean perchlorate concentration of 16,900 µg/kg was detected in 5 of 5 cactus mouse samples collected from Location 1. At Location 3, the mean perchlorate concentration in 2 of 2 of cactus mouse samples was 1,650 µg/kg, and at Location 5 the mean perchlorate concentration fell to below laboratory RLs in the one sample collected.

In the aquatic regime, algae were collected at all three locations and mosquito fish samples were collected at Locations 1 and 5. At Location 1, perchlorate was detected in 3 of 3 algae samples with a mean perchlorate concentration of 143,000 µg/kg. At Location 3, perchlorate were detected in 1 of 4 (25%) samples with a mean perchlorate concentration of 320 µg/kg, but at Location 5, perchlorate was not detected in either of the two samples collected. In addition, perchlorate was detected in all (2 of 2) Mosquito fish collected at Location 1 with a mean concentration of 24,300 µg/kg, but at Location 5 none of the three mosquito fish samples collected contained perchlorate above laboratory RLs.

Table 3.9 Species and Location Specific Perchlorate Concentrations
Site 2, Lake Mead Area

Location	Medium/ Matrix	No. of Samples	No. of Detects	Scientific Name	Common Name	Max Adj Result	Min of Adj Result	Mean of All Results	Mean of Detected Results	Max Adj RL	Min Adj RL
1 Spring											
Fish		2	2	*Gambusia affinis*	Mosquito fish	44300	4310	24300	24300	5740	392
Aquatic Vegetation		3	3	*Oscillatoria sp.*	Filamentous algae	176000	121000	143000	143000	20900	19800
Pore Water		1	1			98000	98000	98000	98000	4.00	4.00
Sediment		6	6			56000	23000	44700	44700	81.2	79.8
Surface Water		8	8			130000	100000	106000	106000	20000	4.00
Soil		5	5			1470000	367	450000	450000	80.5	77.0
Terrestrial Vegetation		3	3	*Tamarix chinensis*	Salt cedar seed	14200	12800	13400	13400	429	384
Terrestrial Vegetation		3	3	*Tamarix chinensis*	Salt cedar litter	428000	387000	401000	401000	40000	39400
Terrestrial Vegetation		3	3	*Tamarix chinensis*	Salt cedar leaf	207000	53200	151000	151000	38700	418
Terrestrial Insect		1	1	Class Insecta	Terrestrial insect (misc.)	4170	4170	4170	4170	407	407
Terrestrial Bird		2	1	*Chordeiles minor*	Nighthawk	1430	ND	907	1430	383	381
Terrestrial Bird		1	0	*Chordeiles acutipennis*	Lesser nighthawk	ND	ND	381		381	381
Terrestrial Bird		2	2	*Zenaida macroura*	Mourning dove	4190	1200	2690	2690	400	387
Terrestrial Mammal		5	5	*Peromyscus eremicus*	Cactus mouse	23200	8440	16900	16900	4100	406
Terrestrial Mammal		3	3	*Peromyscus maniculatus*	Deer mouse	53000	19500	39500	39500	6220	2050
Pore Water - downstream		1	1			1000	1000	1000	1000	4.00	4.00
Sediment - downstream		6	2			21700	ND	4220	12500	82.6	78.0
Surface Water - downstream		6	6			19000	12000	13300	13300	4.00	4.00

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Table 3.9 Species and Location Specific Perchlorate Concentrations
Site 2, Lake Mead Area

Location	*Medium/ Matrix*	*No. of Samples*	*No. of Detects*	*Scientific Name*	*Common Name*	*Max Adj Result*	*Min of Adj Result*	*Mean of All Results*	*Mean of Detected Results*	*Max Adj RL*	*Min Adj RL*
3 ECS3-Upstream of Lake Las Vegas											
	Aquatic Invertebrate	1	0	Phylum Arthropoda	Aquatic invertebrates	ND	ND	381		381	381
	Fish	1	0	*Ictalurus sp.*	Catfish	ND	ND	381		381	381
	Fish	1	0	*Cyprinus carpio*	Carp	ND	ND	392		392	392
	Fish	3	0	*Lepomis sp.*	Sunfish	ND	ND	383		386	381
	Fish	2	0	*Notropis sp.*	Shiner	ND	ND	382		383	381
	Amphibian	5	0	*Bufo woodhousii*	Woodhouse's toad	ND	ND	398		400	396
	Aquatic Vegetation	4	1	*Oscillatoria sp.*	Filamentous algae	320	ND	375	320	407	377
	Sediment	5	3			146	ND	101	116	80.4	78.0
	Surface Water	6	6			480	440	458	458	4.00	4.00
	Soil	5	4			121000	ND	39400	49200	80.3	80.0
	Terrestrial Vegetation	5	5	*Cynodon dactylon*	Bermuda grass	90000	8370	42900	42900	403	372
	Terrestrial Vegetation	5	5	*Phragmites australis*	Phragmites	99200	52800	68900	68900	416	390
	Terrestrial Vegetation	5	5	*Scirpus sp.*	Bullrush	3080	741	1800	1800	406	384
	Terrestrial Insect	2	2	Class Insecta	Terrestrial insect (misc.)	4730	3200	3970	3970	386	385
	Terrestrial Mammal	2	2	*Peromyscus eremicus*	Cactus mouse	2000	1290	1650	1650	392	385
	Terrestrial Mammal	2	2	*Peromyscus maniculatus*	Deer mouse	4230	2000	3110	3110	419	412

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Table 3.9 Species and Location Specific Perchlorate Concentrations
Site 2, Lake Mead Area

Location	Medium/ Matrix	No. of Samples	No. of Detects	Scientific Name	Common Name	Max Adj Result	Min of Adj Result	Mean of All Results	Mean of Detected Results	Max Adj RL	Min Adj RL
5 Lake Mead, Las Vegas Delta Area											
Aquatic Bird		3	0	*Aechmophorus clarkii*	Grebe	ND	ND	396		398	392
Aquatic Bird		5	0	*Fulica americana*	Coot	ND	ND	398		402	390
Aquatic Invertebrate		1	0	Class Gastropoda	Gastropod	ND	ND	398		398	398
Fish		4	0	*Lepomis sp.*	Sunfish	ND	ND	394		402	390
Fish		3	0	*Gambusia affinis*	Mosquito fish	ND	ND	395		402	388
Fish		3	0	*Micropterus salmoides*	Largemouth bass	ND	ND	391		400	385
Amphibian		1	0	*Bufo woodhousii*	Woodhouse's toad	ND	ND	421		421	421
Aquatic Vegetation		2	0	*Oscillatoria sp.*	Filamentous algae	ND	ND	394		405	383
Pore Water		1	1			140	140	140	140	4.00	4.00
Sediment		5	2			81.4	ND	78.8	74.5	83.6	78.0
Surface Water		9	7			68.0	ND	43.8	55.1	4.00	4.00
Soil		5	4			672	ND	392	471	80.2	79.2
Terrestrial Vegetation		4	4	*Scirpus sp.*	Bullrush	1950	516	1260	1260	396	390
Terrestrial Vegetation		5	5	Family Poaceae	Grass	5720	2420	4020	4020	416	390
Terrestrial Insect		1	0	Class Insecta	Terrestrial insect (misc.)	ND	ND	388		388	388
Terrestrial Bird		2	1	*Callipepla gambelii*	Gambel's quail	401	ND	397	401	392	385
Terrestrial Mammal		3	2	*Perognathus formosus*	Long-tailed pocket mouse	447	ND	431	440	412	394
Terrestrial Mammal		1	1	*Mus musculus*	House mouse	443	443	443	443	394	394
Terrestrial Mammal		1	0	*Pipistrellus hesperus*	Western pipestrelle bat	ND	ND	396		396	396
Terrestrial Mammal		1	0	*Peromyscus eremicus*	Cactus mouse	ND	ND	417		417	417

RL = reporting limit.
ND = not detected above the reporting limit.
Reported results less than RLs are estimated values.
J\737451\Data\Lab\ABL\Clay_Lab_Data.mdb

Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
In calculation of the mean of all results, the RL is used for nondetected values.
13-Jun-01

Based on the above observation of insect and algae samples (direct contact media), as well as cactus mouse (exposed via ingestion) and mosquito fish samples (exposed via direct contact and ingestion), the mean concentrations of perchlorate detected in organisms at the Lake Mead Area appear to decrease in direct correlation to the decrease in perchlorate concentrations detected in soil, sediments, and surface water (environmental media).

3.2.3 Intra-Species Relationships

At Location 1 in the Lake Mead Area, salt cedars were abundant and extensively sampled. Three samples each of salt cedar leaves, seeds and the leaf litter beneath the tree were collected. Analytical results indicate that all (3 of 3) of the leaves, seeds (3 of 3) and leaf litter (3 of 3) collected contain mean perchlorate concentrations exceeding laboratory reporting limits. However, the mean perchlorate concentrations detected in each of the media varied. Mean perchlorate concentrations were the lowest in salt cedar seeds (13,400 µg/kg), higher in the leaves (151,000 µg/kg), and higher again in the leaf litter (401,000 µg/kg). Samples at Location 1 were collected near a spring, but groundwater was not sampled. However, perchlorate was detected in terrestrial soils at Location 1 in all (5 of 5) samples collected at a mean concentration of 450,000 µg/kg.

Based on the information presented above, it appears that salt cedar is taking up perchlorate at concentrations nearly equal to the soil it is in contact with, and accumulating that perchlorate in its seeds and leaves. High concentrations of perchlorate detected in salt cedar leaf litter may result from the mixture of leaf litter and soils prior to analysis or from the loss of moisture upon deposition. When the leaves and seeds fall from the tree (creating litter) they dry out, lose moisture, and their weight decreases. However, perchlorate accumulated in the leaves is retained, and results in an increased concentration of perchlorate detected in litter. It is also possible that the perchlorate found in the soil may come from the degradation of the salt cedar leaf litter.

Table 3.10
Mean Perchlorate Concentrations in Salt Cedar Media at Location 1 (spring), Lake Mead Area

Media	Mean Perchlorate Concentration from Detected Samples (µg/kg)
Salt cedar seeds	13,400
Salt cedar leaves	151,000
Salt cedar leaf litter	401,000
Soil	450,000

3.2.4 Media Relationships

Surface water, sediment, and sediment pore water samples were collected at sub-locations near a spring that discharges at Location 1 in the Lake Mead Area. Samples were collected both upstream (UP) and downstream (DN) of the spring to determine if relationships between surface water and sediment exist and what affect those

relationships may have on other media. The surface water, sediment, and sediment pore water data is presented in Table 3.11 and illustrated in Figure 3.8.

As shown in Table 3.11 and Figure 3.8, surface water, sediment, and sediment pore water samples collected at Location 1 UP exhibited mean perchlorate concentrations of 106,000 µg/L, 44,700 µg/kg, and 98,000 µg/kg, respectively. These same media collected at Location 1 DN exhibited mean perchlorate concentrations of 13,300 µg/L, 12,500 µg/kg, and 1,000 µg/kg, respectively. The data indicate that sediment and pore water contain about the same concentration of perchlorate. Surface water generally contained greater concentrations of perchlorate.

Based on the above data, it appears that surface waters contain the highest levels of perchlorate, with sediments and pore water containing perchlorate at concentrations an order of magnitude smaller. Therefore, it appears that the ion exchange water treatment plant is effective at reducing the amount of perchlorate that flows downstream from the source.

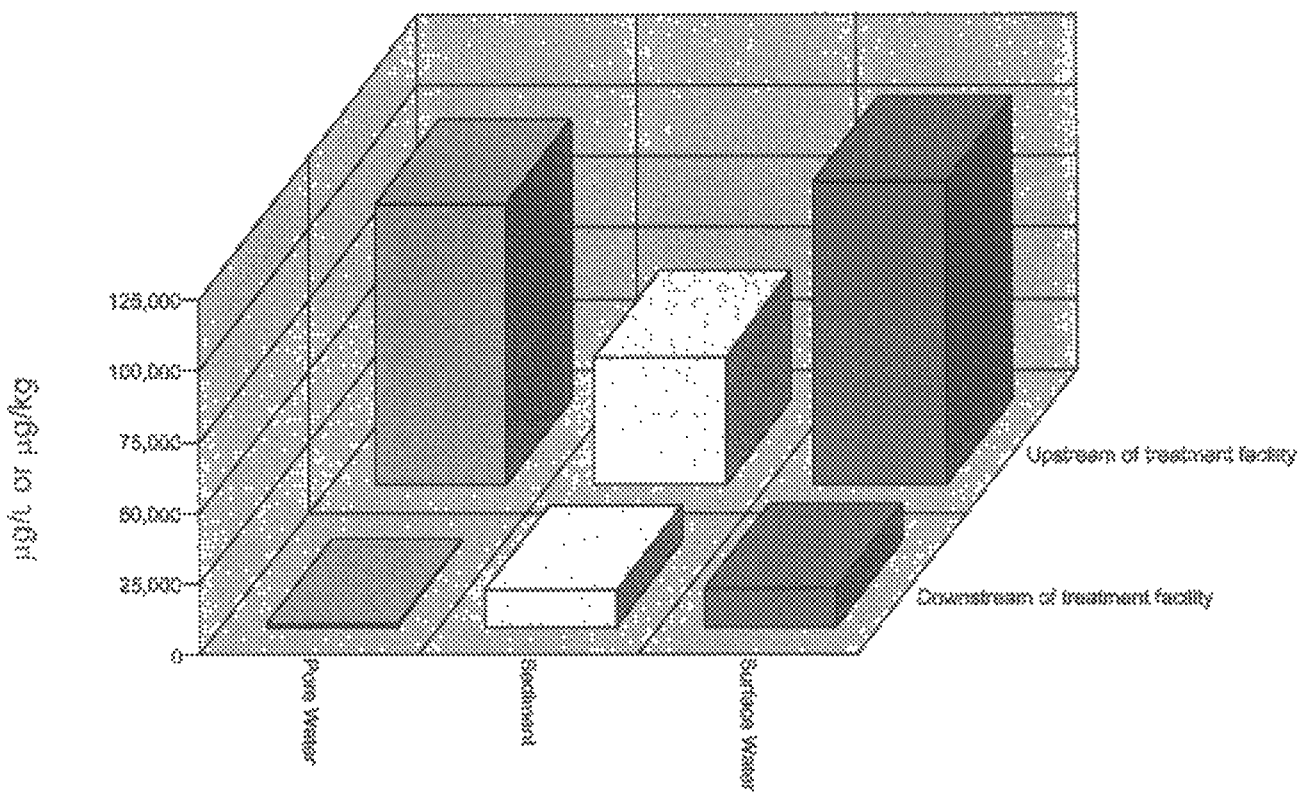
Table 3.11
Mean Perchlorate Concentrations in Upstream and Downstream Samples at Location 1 (spring), Lake Mead Area

	Sediment Pore Water (µg/L)	Sediment (µg/kg)	Surface Water (µg/L)
Upstream of Treatment Facility	98,000	44,700	106,000
Downstream of Treatment Facility	1,000	12,500	13,300

3.3 COMPARISON WITH OTHER DATA

In addition to the samples collected on this project, results were obtained from the Las Vegas Valley Water District (LVVWD) and SNWA for two of the study locations we evaluated at the Lake Mead NRA site during August 2000. Table 3.12 presents the data for comparison. Results of the six samples collected at Location 3 (ECS 3, also named LM-8 and near LW3.85) on August 13, 2000 from this investigation compare very closely with the two samples collected by SNWA at LW3.85 on August 23 and 28, 2000. Similarly, at Location 5 (LVB1.36 and LWLVB) results from four different samples collected by LVVWD/SNWA between August 8, 2000 and August 24, 2000 compare very closely with the six water samples collected on August 14, 2000 for this investigation.

Figure 3.8
Mean Perchlorate Concentrations in Upstream and Downstream Samples at Location 1 (Spring), Lake Mead Area



In calculation of the mean, only samples with detectable levels of perchlorate were used.
Sediment sample results are reported in µg/kg.
Surface water and pore water results are reported in µg/L.

Table 3.12
Comparison of Data With Sampling by Others

Sample Location Description	Sample Location Description for AFIERA Project	Perchlorate (µg/L)	Sample Date	Depth	Sample Identification	Project
LW3.85		480	08/23/00		LM-8	SNWA
LW3.85		408	08/28/00		LW3.85	SNWA LVW ECS
ECS 3 – LW3.85	Location 3 - ECS 3	460	8/13/00	0	02030101	PARSONS
ECS 3 – LW3.85	Location 3 - ECS 3	460	8/13/00	0	02030102	PARSONS
ECS 3 – LW3.85	Location 3 - ECS 3	440	8/13/00	0	02030103	PARSONS
ECS 3 – LW3.85	Location 3 - ECS 3	450	8/13/00	0	02030104	PARSONS
ECS 3 – LW3.85	Location 3 - ECS 3	480	8/13/00	0	02030105	PARSONS
ECS 3 – LW3.85	Location 3 - ECS 3	460	8/13/00	0	02030106	PARSONS
Las Vegas Bay 1.5 Mile		86	08/08/00	0	LVB1.5M	LABOUNTY
Las Vegas Bay 1.5 Mile		57	08/22/00	0	LVB1.5M	LABOUNTY
Las Vegas Bay 1.5 Mile		89	08/10/00	0	LVB1.5M	MONTHLY MODULE
Las Vegas Bay 1.5 Mile		66	08/24/00	0	LVB1.5M	MONTHLY MODULE
Las Vegas Bay 1.5 Mile	Location 5 Las Vegas Bay Delta	58	8/14/00	0	02050101	PARSONS
Las Vegas Bay 1.5 Mile	Location 5 Las Vegas Bay Delta	58	8/14/00	0	02050102	PARSONS
Las Vegas Bay 1.5 Mile	Location 5 Las Vegas Bay Delta	57	8/14/00	0	02050103	PARSONS
Las Vegas Bay 1.5 Mile	Location 5 Las Vegas Bay Delta	60	8/14/00	0	02050104	PARSONS
Las Vegas Bay 1.5 Mile	Location 5 Las Vegas Bay Delta	58	8/14/00	0	02050105	PARSONS
Las Vegas Bay 1.5 Mile	Location 5 Las Vegas Bay Delta	68	8/14/00	0	02050106	PARSONS

Sample location names and reference distance using Lake Mead Water Quality Forum standards (from www.lvwaterquality.org , 2/2001)
ECS = Erosion Control Structure
LVB = Las Vegas Bay
Data Source: Las Vegas Valley Water District (5/7/01)
AFIERA = Air Force Institute for Environment, Safety and Occupational Health Risk Analysis

3.4 UNCERTAINTIES

The goal of this investigation was to report the occurrence of perchlorate in physical media (soil, sediment, and water) and biological media (flora and fauna). This investigation successfully detected the presence of perchlorate in all physical media as

well as some biological media. Due to the breadth and scope of this investigation, there are a number of uncertainties that are introduced into the results. These uncertainties should be considered before interpreting the data presenting the occurrence of perchlorate are used. The following three lists describe the primary uncertainties with this investigation.

Uncertainties related to the sites:

- The samples obtained represent a snapshot in time and do not document the occurrence of perchlorate under any climatic conditions other than those present when sampling occurred,
- Meteorological differences occurred among the sites and no attempt was made to standardize the results for meteorological conditions (i.e., rainfall rates varied among the sites),
- Some of the sample locations are not naturally occurring water bodies (treated water storage ponds),
- Sample locations were not necessarily those expected to have the highest perchlorate concentrations, but the sample locations were chosen for ease of sampling,
- Numbers and types of biological samples at each location vary greatly, and
- All sources of perchlorate may not have been identified (e.g., fertilizer use in irrigated areas).

Biological/ecological uncertainties:

- Specific receptors could not always be targeted – an opportunistic sampling strategy was employed,
- Low sample counts of each medium at each location,
- Life history information on the receptors was not available,
- The modes of transport of perchlorate (direct contact, incidental intake, ingestion) to each receptor are not fully traceable,
- Full investigation of plant physiology was not performed,
- No gut analysis was performed to determine food sources for receptors,
- Performing a trend analysis across the sites requires combining classes of receptors (e.g. small mammals, fish, etc.,), and

- Migration patterns and daily spatial range of mobile biological samples was not studied.

Analytical uncertainties:

- Detection limits for tissues were comparatively higher than basic physical media samples,
- Split sample analyses (separate laboratory) exhibit significant differences for some analyses, and
- Laboratory sample clean up methods for extraction aliquot and dilutions for analysis may greatly affect detection limits.

SECTION 4
SUMMARY AND CONCLUSIONS

This section summarizes the objectives and findings of the perchlorate biotransport investigation and discusses recommendations based on the knowledge gained during this investigation.

4.1 SUMMARY OF LABORATORY METHOD DEVELOPMENT

One of the objectives of this investigation was to develop laboratory procedures for the analysis of soil, sediment, and various plant and animal tissue samples for perchlorate content. The goal of the analytical methods was to develop methodology that allowed for the determination of the perchlorate content at concentrations that were meaningful in the environment (i.e., in the parts per billion range).

Clayton developed standard operating procedures for each of the matrices required. The precision and accuracy observed for each matrix during the method validation procedures demonstrate that the laboratory's method development was successful. While the water matrix exhibited the best recovery, all matrices had accuracy recoveries above 85 percent and a relative standard deviation of less than 30 percent. These results demonstrate the success of the developed methodology in producing high quality (accurate and precise) data. Clayton was able to consistently attain a reporting limit of 4 µg/L for water, 80 µg/kg for soil and sediment, and 400 µg/kg for tissue samples. These reporting limits were sufficient to detect the presence of perchlorate in numerous tissue samples, soil samples, and water samples.

4.2 SUMMARY OF PERCHLORATE BIOTRANSPORT

The remaining objectives of this project were to characterize contaminant exposure pathways for ecological receptors of concern and evaluate the potential for biotransport of perchlorate for these receptors. Simply stated, the question to be answered was: "is there transport of perchlorate into biological receptors?"

In order to answer this question, a total of 965 samples (not including quality control samples) were collected from six perchlorate contaminated sites across the United States and analyzed for perchlorate concentrations. These samples represent 18 different media types to cover a wide range of potential sources, pathways, and receptors.

Perchlorate was detected in abiotic media (e.g., soil and water) and biological media at all six sites investigated. The Lake Mead Area appears to contain the highest mean concentrations of perchlorate across all medium types and medium groups of the six sites investigated with Yuma, Arizona showing the lowest values. Figure 4.1 shows the mean concentration of perchlorate in all media at all sites. The figure uses the reporting limit for all samples with non-detect results. The range of concentrations of

perchlorate in surface water across the sites varied up to approximately six orders of magnitude from Yuma, Arizona (non-detect at a reporting limit of 4.0 µg/L) to Location 1 of the Lake Mead Area (130,000 µg/L). Of the 32 sampling locations, the five locations with the overall highest mean perchlorate concentrations were Lake Mead Area, Location 1; Lake Mead Area, Location 3; ABL, Location 1; LHAAP, Location 2; and Lake Mead Area, Location 5.

The sites and locations with the highest perchlorate concentrations in the surface water also have the highest concentrations in the biological media. Table 4.1 illustrates the relative magnitude of perchlorate found at individual sites for various media. Table 4.2 provides a ranking of perchlorate concentrations in several media groups by site. The highest concentrations of perchlorate were predominately found in vegetation. In aquatic media, vegetation had the highest concentrations of perchlorate at three of the six sites. In terrestrial media, vegetation had the highest concentrations at five of the six sites. Table 4.3 lists the maximum detected perchlorate concentration by media group and gives the site this maximum result was obtained at.

From the results of this investigation we can conclude:

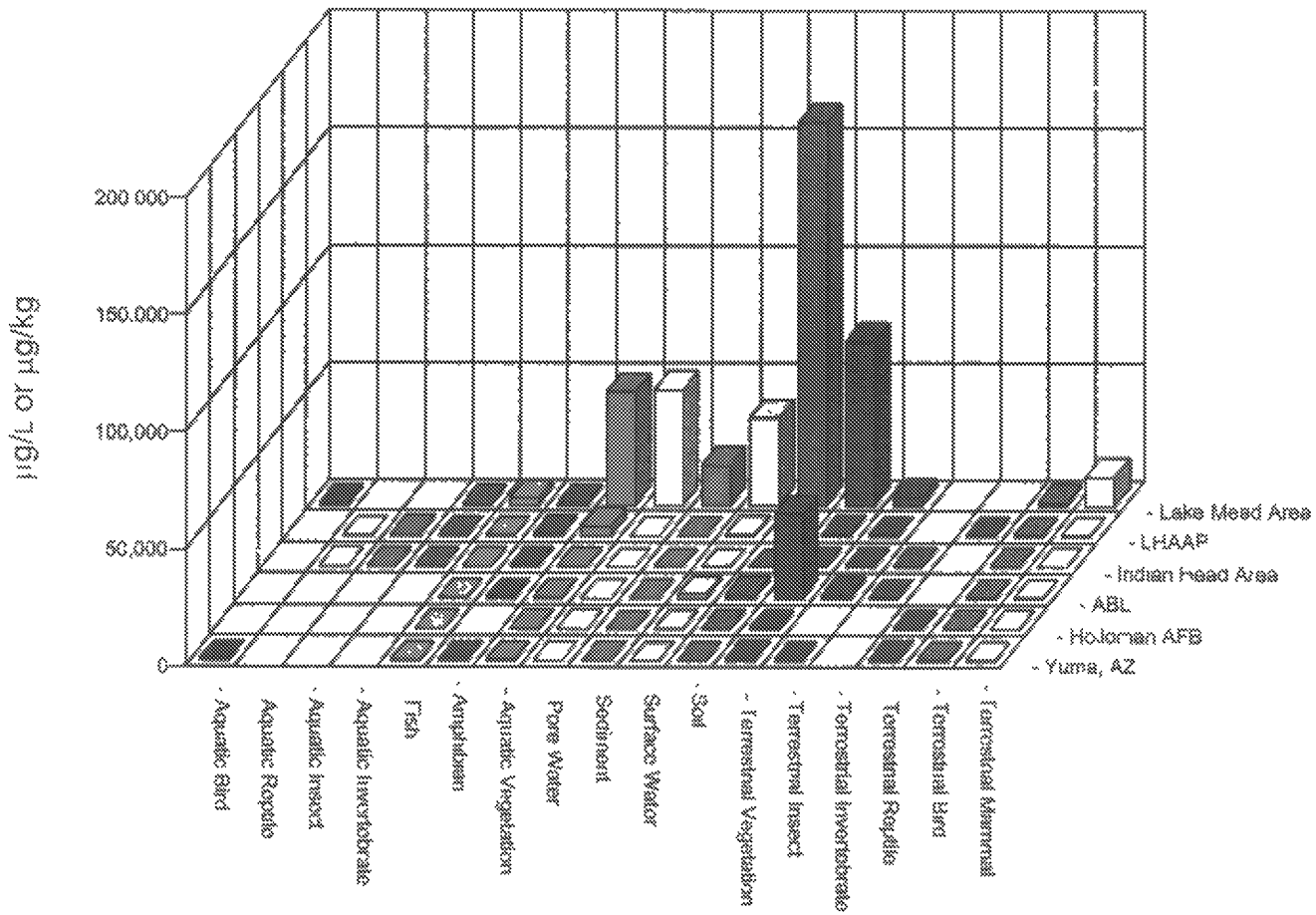
- Biotransport of perchlorate apparently occurs into various biological media,
- Both terrestrial and aquatic vegetation appear to typically exhibit the highest perchlorate concentrations and frequency of detection of all the media sampled,
- There is an apparent correlation between surface water perchlorate concentrations and concentrations in other media, and
- Preliminary investigation of salt cedar leaf, seed, and leaf litter show that different parts of plants accumulate different levels of perchlorate.

4.3 RECOMMENDATIONS FOR FURTHER INVESTIGATION

The goal of this investigation was to determine the potential for perchlorate to enter the food chain. It is evident from the results presented in this report that perchlorate does enter biological receptors through transport from abiotic media and may accumulate in specific biological media. While this report provides significant information for understanding the fate and transport of perchlorate in the environment, additional information is required for a more complete understanding of this issue. Therefore, the following are recommendations for additional investigations that may supplement the information in this report and provide a better understanding of perchlorate in the environment.

- Perform additional field sampling of similar media at the sites and locations with high perchlorate concentrations and good species diversity. This could include more specific co-location of samples collected, performance of field stomach content analysis of collected animals with follow-up sampling of the food sources identified, and some organ specific analysis to determine specific tissue location of stored perchlorate. In addition, this could be designed to address potential seasonal effects.

Figure 4.1
Media and Site-Specific Mean Perchlorate Concentrations



In calculation of the mean, the reporting limit is used for nondetected values.
Sediment, soil, and tissue sample results are reported in µg/kg.
Surface water and pore water results are reported in µg/L.

Table 4.1
Media-Specific Order Among Sites in Mean Detectable Perchlorate Concentrations

Media	highest										lowest
	Perchlorate concentration →										
Soil	Lake Mead Area	>	Allegany Ballistics Laboratory	>	Holloman AFB	>	Yuma, AZ	>	Indian Head Area		
Terrestrial Vegetation	Lake Mead Area	>	Allegany Ballistics Laboratory	>	Holloman AFB	>	Longhorn Army Ammunition Plant	>	Yuma, AZ	>	Indian Head Area
Water	Lake Mead Area	>	Longhorn Army Ammunition Plant	>	Allegany Ballistics Laboratory	>	Indian Head Area	>	Yuma, AZ		
Aquatic Vegetation	Lake Mead Area	>	Longhorn Army Ammunition Plant	>	Indian Head Area	>	Yuma, AZ				
Sediment	Lake Mead Area	>	Longhorn Army Ammunition Plant	>	Indian Head Area	>	Allegany Ballistics Laboratory	>	Holloman AFB		

Table 4.2
Site-Specific Order Among Media in Mean Detectable Perchlorate Concentrations

Site	Medium type	highest										lowest
		Perchlorate concentration →										
Yuma, AZ	Aquatic	vegetation	>	water								
	Terrestrial	vegetation	>	reptile	>	soil						
Lake Mead Area	Aquatic	vegetation	>	water	>	sediment	>	fish				
	Terrestrial	soil	>	vegetation	>	mammal	>	insect	>	bird		
Allegany Ballistics Laboratory	Aquatic	fish	>	water	>	sediment						
	Terrestrial	vegetation	>	insect	>	soil	>	bird				
Holloman AFB	Aquatic	sediment	>	groundwater								
	Terrestrial	vegetation	>	soil								
Indian Head Area	Aquatic	amphibian	>	vegetation	>	sediment	>	water				
	Terrestrial	vegetation	>	soil								
Longhorn Army Ammunition Plant	Aquatic	vegetation	>	water	>	amphibian	>	insect	>	sediment		
	Terrestrial	vegetation	>	mammal								

Table 4.3
Maximum Perchlorate Concentration Detected in Each Medium

Media	Maximum Detected Perchlorate (ppb)	Site
Aquatic Insect	996	Longhorn Army Ammunition Plant
Fish	44,300	Lake Mead Area
Amphibian	1,660	Longhorn Army Ammunition Plant
Aquatic Vegetation	176,000	Lake Mead Area
Sediment	56,000	Lake Mead Area
Pore Water	98,000	Lake Mead Area
Surface Water	130,000	Lake Mead Area
Soil	1,470,000	Lake Mead Area
Terrestrial Vegetation	428,000	Lake Mead Area
Terrestrial Insect	12,600	Allegany Ballistics Laboratory
Terrestrial Bird	4,190	Lake Mead Area
Terrestrial Mammal	53,000	Lake Mead Area

- Conduct further study on the fate and transport of perchlorate among surface water, pore water and various types of sediments.
- Conduct inter-laboratory investigations on sample preparation procedures for biological media to improve reproducibility and documentation of steps to get from field sample to analytical extract.
- Perform an investigation on agricultural crops irrigated with water containing perchlorate to determine the possible occurrence of perchlorate.
- Determination of a water quality standard for perchlorate using microcosm studies on biological species in conjunction with existing environmental effects data.
- Encourage site personnel to continue (or plan and begin) a routine perchlorate sampling program in water (water as a minimum of media collected) at each facility. This would provide data for long-term exposure assessment.

SECTION 5
REFERENCES

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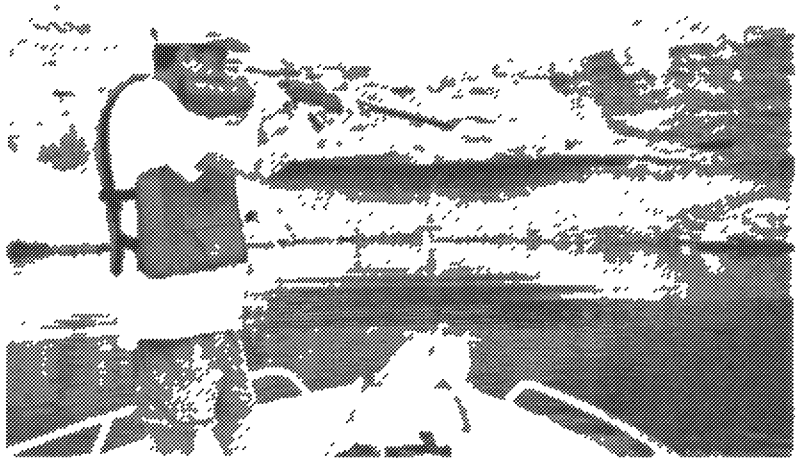
Appendix A
Photo Log

The following photo log documents sample collection procedures, provides examples of biota collected, and provides documentation of the habitat types sampled. The photos are in order by the site number.

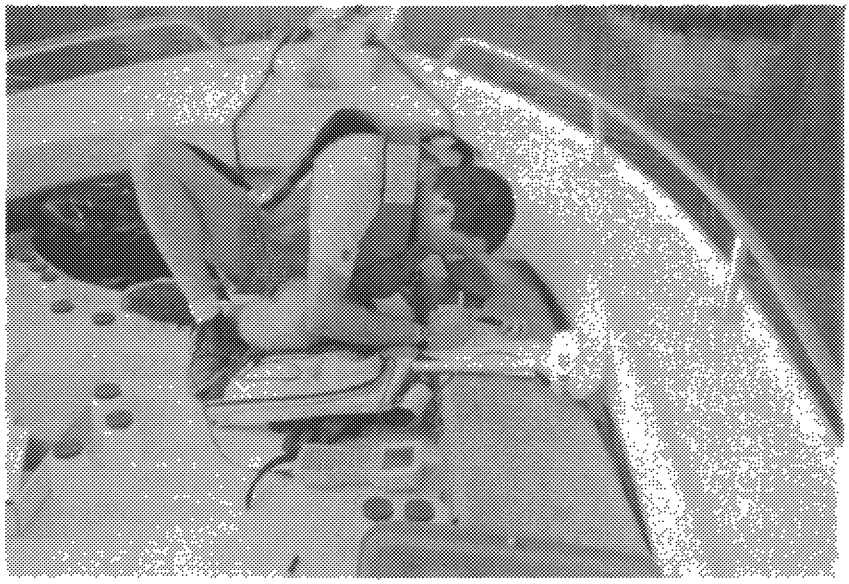
The Colorado River south of Palo Verde, California
Yuma, Arizona site, Location 1 – Palo Verde Irrigated Lands
Tamarisk in foreground at shoreline, irrigated fields in flat stretch across the river.

Largemouth bass in zip-lock type bag with sample label affixed (Sample ID 02050503) from Lake Mead NRA (Site 2), Location 5. Note lesion above pectoral fin and lesions at mouth area. Sample result was non detect for perchlorate for this sample.

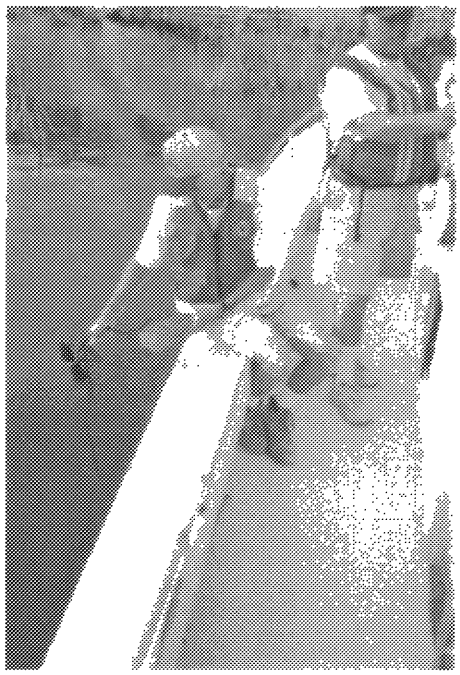


Waterfowl collection with 20-gauge shotgun and steel shot from National Park Service boat at Lake Mead, Nevada, Location 5 - Las Vegas Bay.

Woodhouse toad collected at Las Vegas Wash, Location 3 - FCS 3.

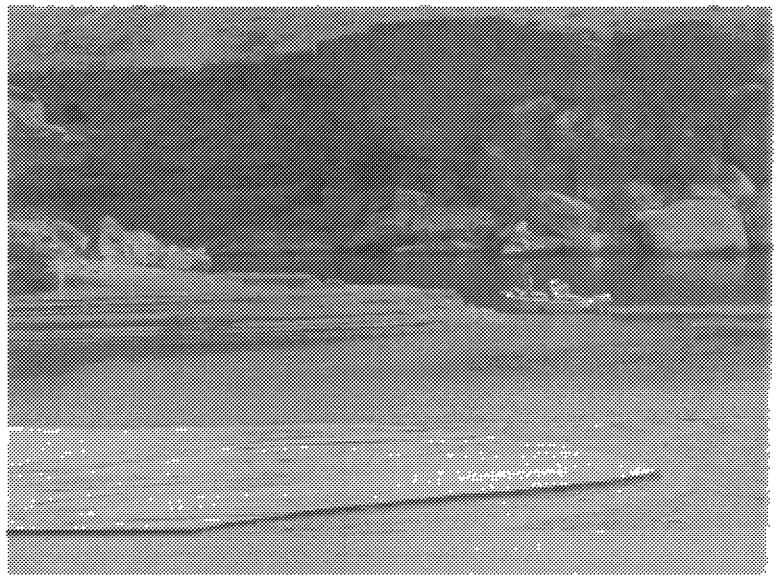


Field team member documenting measurements in field logbook and logsheets at Lake Mead, Nevada, Location 5 – Las Vegas Bay.



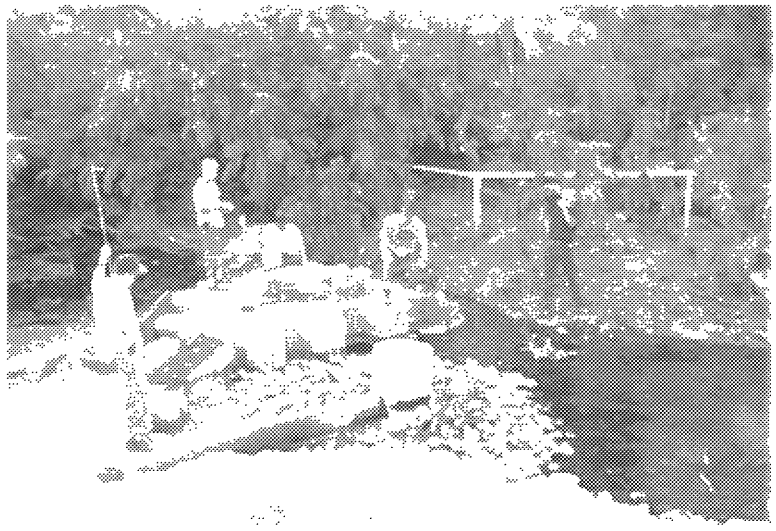
Collection of water quality measurements from National Park Service boat at Lake Mead, Nevada, Location 5 - Las Vegas Bay.

Collection of terrestrial insects using vehicle headlights and portable blacklight on white cotton fabric at night. Lake Mead NRA site, Location 3.

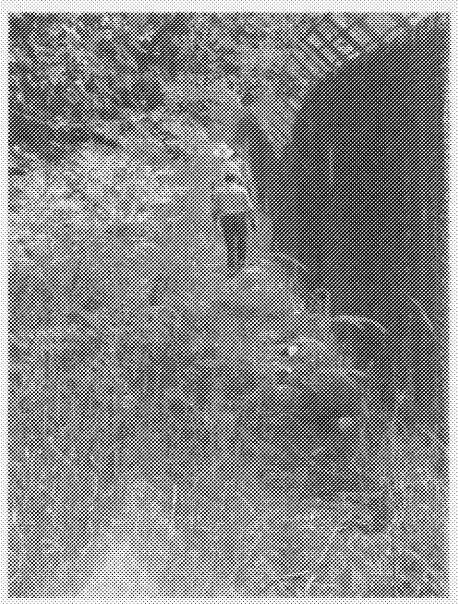


Sampling crew in National Park Service boat heading into Las Vegas Bay for sample collection effort. Lake Mead, Nevada, Location 5 - Las Vegas Bay.

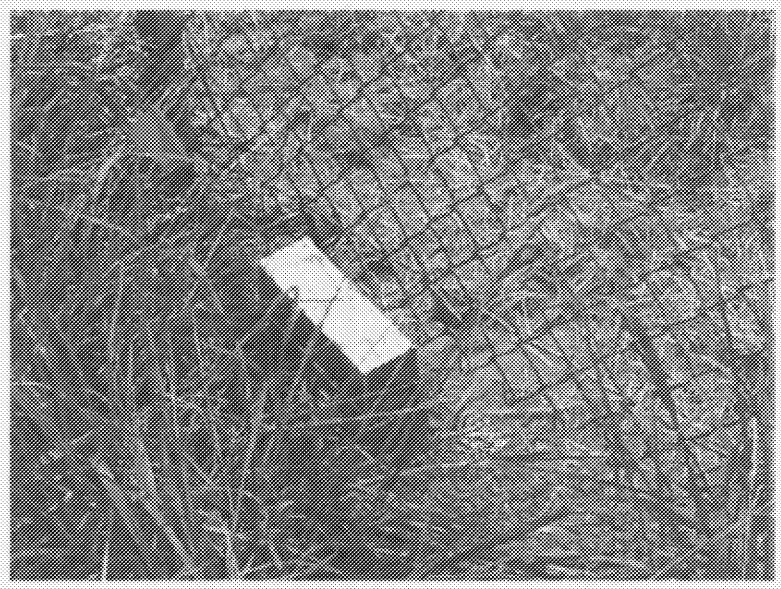
Sediment collection at the delta leading into Las Vegas Bay. Water flow is from top to bottom of picture. Lake Mead, Nevada. Location 5 - Las Vegas Bay.



Field crew processing aquatic vegetation samples after collection effort. Leftmost and rightmost crewmembers are setting up a mist net (note pole in hand) for bat collection at dusk. This is Location 1 – Spring, at the Las Vegas Wash. The concrete slab where sample processing is occurring is part of the cutoff wall dividing untreated groundwater (upstream – on the left of the photo) from treated groundwater (downstream of treatment plant discharge point – on the right of photo).



Small mammal trap line (Sherman aluminum live capture traps) along chain link fence at ABL site, on north edge of Location 1 – Burn Area.



Close-up of baited Sherman aluminum live capture trap used for small mammal collection. Surveyor's pin flag inserted just to the left of trap to assist locating traps by field team. ABL site, Location 1.

Aquatic invertebrate collection with dip net in water canal at ABL site. Note floating mats of filamentous algae and duckweed on water surface. Location 5 – Upstream of weir.

Fish collection with backpack electroshocking unit and hand nets along water canal at ABL site. Note floating mats of filamentous algae and duckweed on water surface. Location 5 – Upstream of weir.

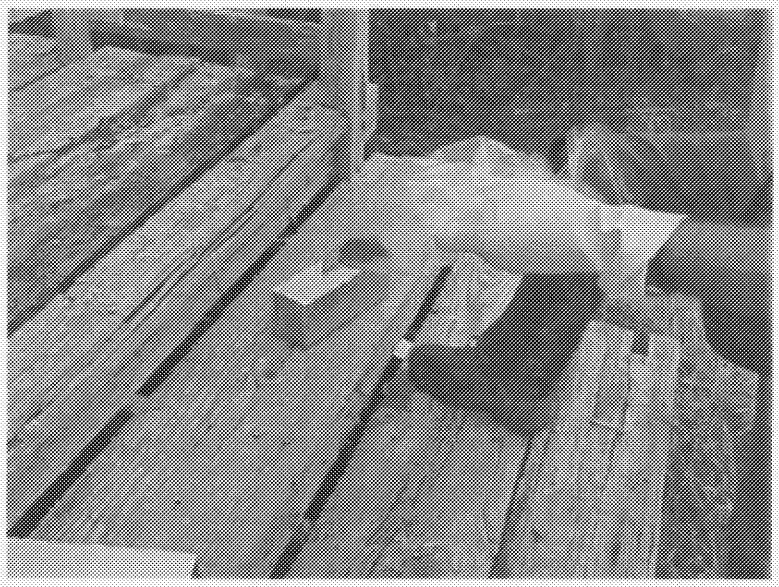
Checking a line of small mammal traps in the Chihuahuan Desert habitat at Holloman AFB. Location 1 – South of Building 1176 near the playa lake bed.

Field team member identifying small mammal (Plains pocket mouse) collected (in zip-lock type bag at left knee). Holloman AFB, Location 5 – second location along the Lost River.

Habitat of Town Gut Marsh system at Naval Surface Warfare Center, Indian Head, Maryland, Location 5 at pond along Atkin's Road. Location 4 is creek entering the pond at top left of photo.

Field team preparing sample jars for shipment. Labeled water, soil, and sediment jars on top of cooler. Attempts were made to maximize sample containers per cooler before completing chain of custody forms, packing with ice, and shipment via overnight courier to analytical laboratory.

Harrison Bayou habitat at Location 3, discharge point for water piped from INF Pond (Location 2). Longhorn Army Ammunition Plant, Karnack, Texas.



Harvest Mouse in zip-lock type bag after being transferred from small mammal trap. At the steps of an abandoned building at Longhorn AAP, Location 2 – INF Pond.

Appendix B

Laboratory Standard Operating Procedures

Determination of Perchlorate by Ion Chromatography in Animal Tissue

STANDARD OPERATING PROCEDURE (SOP): M-CC-71/N, Revision 0, October 2000

Authorization/Approval:		Applicability and Revision History:	
John Spurr, Quality Assurance Director	10/25/00 Date	Office Location:	Novi, Michigan
Scott Caillouette, Supervisor	10/25/00 Date	Effective Date:	R0, October 24, 2000
Robert Lieckfield, Jr., CIH, Laboratory Director	10/26/00 Date	Reference Methods:	EPA 314.0 modified
		Media/Matrices:	Animal Tissue

Distribution:

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1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of the perchlorate anion in animal tissue by ion chromatography after aqueous extraction and solid–phase cleanup procedures.

1.2 Although not specifically tested, this method is potentially applicable to other solid matrixes. When this method is used to analyze unfamiliar samples, perchlorate identification should be supported by the use of matrix spikes. The matrix spike procedure is described in Section 6.5.

1.3 The linear calibration range for perchlorate is approximately 4 to 500 µg/L. The linear calibration may be extended to 1500 µg/L, but accuracy below 50 µg/L may be compromised. Quantitation below 50 µg/L is not recommended with the extended calibration. Sample concentrations higher than the upper calibration limit should be diluted with reagent water to the mid-range of the linear calibration and reanalyzed.

1.4 This method is recommended for use by analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

2.0 SUMMARY OF METHOD

2.1 The analysis is performed using an ion chromatograph. The main components of the ion chromatograph used in this method include an autosampler, pump, guard column, analytical column, ion suppressor device, conductivity detector, and chromatography data system.

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2.2 An aliquot of sample extract is injected into the ion chromatograph, where the perchlorate anion is separated from other sample components, then quantitated. A large volume sample injection, 1 ml, is employed to perform perchlorate measurement at the low µg/L level.

2.3 Perchlorate is a large polarizable anion with hydrophobic properties. To avoid or minimize hydrophobic interaction of perchlorate with the guard and separator columns, care must be exercised in the selection of the ion-exchange column and the eluent composition. Extensive hydrophobic interaction of the perchlorate anion with the ion-exchange column leads to a long retention time and severe peak tailing. The columns and eluents recommended in this method were selected to minimize this hydrophobic interaction.

3.0 INTERFERENCES

3.1 The wide water dip or negative peak that may be observed in the chromatogram is due to the large volume sample injection. The negative response is the result of an imbalance of ions in the sample relative to the eluent and does not interfere with the measurement of perchlorate, since perchlorate elutes well beyond the dip.

3.2 Interferences can be caused by substances with retention times that are similar to and overlap with the anion of interest.

3.3 High concentrations of anions can interfere with the peak resolution of an adjacent anion and/or cause baseline disturbances. When such an interference occurs, it may help to dilute the sample before analysis, although it should be recognized that the sample detection limit increases proportionately with the sample dilution factor.

3.4 Surfactants and detergents may cause fouling of the ion exchange column and membrane suppressor, resulting in an unstable baseline and poor detector response. Other dissolved organic compounds may similarly interfere.

4.0 APPARATUS AND MATERIALS

4.1 Ion chromatograph – Dionex DX120 or equivalent, complete with all required accessories including syringes, analytical columns, compressed gases, and detectors.

4.1.1 Anion guard column: A protector of the separator column. Dionex AG 16, 4 mm.

4.1.2 Anion separator column: This column produces the separation of the anions. Dionex AS 16, 4 mm.

4.1.3 Anion suppressor device: Dionex ASRS II.

4.1.4 Automated Sampler: Dionex ASM 3, or equivalent.

4.1.5 PeakNet IC Software: Dionex.

4.1.6 Conductivity detector: 1.25 µL internal volume.

4.1.7 Sample loop: Approximately 1000 µL (12" x 0.02" I.D. tubing), or larger, as required.

4.2 Polyethylene autosampler vials, 5 mL.

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4.3 Filter caps, 5 mL.

4.4 Volumetric flasks, 50, 100, 1000, and 2000 mL.

4.5 Plastic vials, 10 mL.

4.6 Falcon tubes, 15 ml and 50 ml.

4.7 Laboratory balance, capable of weighing to 0.1 mg.

4.8 Shaker table.

4.9 Vortex

4.10 Disposable syringes, 10 ml.

4.11 Centrifuge(s), capable of accommodating 50 mL Falcon tubes and 16 x 125-mm disposable glass test tubes.

4.12 Kimwipes, delicate task wipes.

4.13 Disposable test tubes, 16 x 125 mm with caps.

4.14 Gelman ion chromatography (IC) Acrodisc or equivalent, 25 mm syringe filter with 0.45 um Supor (PES) membrane, catalog #4585T.

4.15 C18 Solid Phase Extraction (SPE) cartridges or equivalent, Supelco Superclean ENVI-18 SPE Tubes, 20 ml (5 g), catalog #57137.

4.16 Solid phase extraction vacuum manifold.

4.17 Alumina, (Aluminum Oxide), Aldrich catalog #19,944-3, activated, basic, Brockmann l, standard grade, 150 mesh, 58 A, surface area 155 m^2/g.

Note: The alumina is cleaned by mixing one volume of alumina with 10 volumes of reagent water. The fines (cloudy water above the solid) are decanted and replaced with fresh reagent water. The mixing and decanting process is repeated until the water remains clear. The cleaned alumina is then allowed to air dry and stored in a screw cap glass bottle until used.

5.0 REAGENTS

5.1 Reagent water, obtained from the Milli-Q Water System, through the filtered spigot.

5.2 Sodium perchlorate (solid), Aldrich catalog #41,024-1.

5.3 Secondary source standard (1000ppm solution), SPEX catalog #AS-CLO49-2Y.

5.4 50% w/w sodium hydroxide, Fisher Chemical catalog #55254-500, or equivalent.

Note: For 100 mM sodium hydroxide, weigh 8.0 grams of 50% NaOH into a plastic weight boat and transfer into approximately 950 mL of reagent water and bring to 1000 mL volume.

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5.5 Methanol, HPLC grade.

6.0 PREPARATION OF STANDARDS

Note: In all cases where weighing is involved, the exact weight (all decimal places for the balance used) is recorded.

6.1 Primary Stock and Working Standards: All stock standards can be used for up to 12 months. All other standards can be used for up to 6 months.

6.1.1 1000 ppm Primary Perchlorate Stock Standard: Dissolve 1.231 g of sodium perchlorate in 1000 ml of reagent water.

6.1.2 10 ppm Perchlorate Working Standard: Dilute 1.0 ml of 1000 ppm stock perchlorate to 100 ml with reagent water.

6.1.3 1.0 ppm Perchlorate Working Standard: Dilute 10.0 ml of 10 ppm perchlorate to 100 ml with reagent water.

6.1.4 0.1 ppm Perchlorate Working Standard: Dilute 1.0 ml of 10 ppm perchlorate to 100 ml with reagent water.

6.2 Secondary Source Standards

6.2.1 10 ppm Secondary Source Perchlorate Standard: Dilute 1.0 ml of the 1000 ppm secondary source perchlorate standard to 100 ml with reagent water.

6.2.2 1.0 ppm Secondary Source Perchlorate Standard: Dilute 10 ml of 10 ppm secondary source perchlorate standard to 100 ml with reagnt water.

6.3 Mixed Common Anion Stock Solution (containing the anions chloride, sulfate and carbonate each at 25 mg/ml), used to prepare simulated common anion samples in the determination of the MCT: Dissolve the following salts in reagent water to a final volume of 25.0 ml.

2/5/01 2/5/01 0.93

(a) 1.0 g sodium chloride (NaCl, CAS [7647-14-5]) = 0.61 g Cl^-
(b) ~~93~~ g sodium sulfate (Na_2SO_4, CAS [7757-82-6]) = 0.63 g SO_4^{-2}
(c) 1.1 g sodium carbonate (Na_2CO_3, CAS [497-19-8]) = 0.62 g CO_3^{-2}

6.4 Instrument Performance Check (IPC) Standard: Add 2.5 ml of 1.0 ppm perchlorate working standard and 3.2 ml of mixed common anion stock to a 100 ml volumetric flask and dilute to volume with reagent water.

6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD): Prepare by spiking 400 µL of 10 ppm perchlorate working standard or secondary source standard to 2 g of sample. (Note: A smaller volume may have to be used due to limited sample volume)

6.6 Laboratory Control Sample (LCS): Prepare by spiking 400 µL of 10 ppm perchlorate working standard or secondary source standard into 20 ml of reagent water.

6.7 Instrument Calibration Verification Standard (ICV): Prepare by adding 2.0 ml of 1.0 ppm secondary source perchlorate standard to 50 ml of reagent water in a 100 ml volumetric flask and dilute to volume with reagent water.

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7.0 SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

7.1 Samples may be collected in HDPE plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

7.2 Until further preservation and holding time studies are available, there is no maximum holding time. Samples are to be stored at <0°C to minimize decomposition.

8.0 PROCEDURE FOR SAMPLE PREPARATION/EXTRACTION

8.1 Weigh 2.0 g (±0.1 g) of tissue into 50 mL Falcon tube.

8.2 Pipet 20.0 ml of reagent water into tube

8.3 Place on mechanical shaker for approximately 16 hours.

8.4 Centrifuge for 30 minutes.

8.5 Decant top layer (sample extract) into a clean 50 ml Falcon tube.

8.6 Add a 5.0 ml aliquot of this extract to 2.5 g (±0.1g) of Alumina in a disposable 12.5 ml test tube and cap.

8.7 Vortex test tube for approximately 20 seconds.

8.8 Place test tube on a mechanical shaker for 1 hour.

8.9 Centrifuge the test tube for 15 minutes.

8.10 Filter the top layer (sample extract) through a 25 mm Gelman IC Acrodisc 0.45 um into a clean 15 ml Falcon tube using a 10 mL disposable syringe.

8.11 Add 1.0 ml of sample extract to 9.0 ml of reagent water in a clean 15 ml Falcon tube. This results in a 1:10 dilution of the sample extract.

8.12 Solid Phase Extraction (SPE) C18 Cleanup: Pass the diluted sample extract (1:10) through one ENVI-18 SPE cartridge to remove potential organic matrix interference compounds.

8.12.1 Condition each C18 cartridge per the manufacturer's instructions by rinsing with methanol followed by reagent water. The flow rate should not exceed 5.0 ml/minute.

8.12.1.1 Rinse cartridge with 4 ml of methanol.

8.12.1.2 Rinse cartridge with 5 ml of reagent water.

8.12.2 After conditioning with methanol and reagent water, rinse cartridge with 2.0 ml of diluted sample extract.

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8.12.3 To obtain the final extract, pass approximately 5.5 ml of diluted sample extract through the conditioned and rinsed C18 cartridge into a clean disposable test tube. Transfer the final extract to a 15 mL Falcon tube.

8.13 Transfer approximately 2 ml of final extract into an autosampler vial for analysis. The remaining extract is stored refrigerated for any additional analysis.

9.0 HEALTH AND SAFETY

9.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

9.2 The following chemicals have the potential to be highly toxic or hazardous. The MSDS for each chemical should be consulted.
(a) Sodium hydroxide
(b) Sodium perchlorate

10.0 QUALITY CONTROL

10.1 All IC chromatograms are kept for 5 years.

10.2 Perform an initial calibration with standards at 4, 10, 20, 50, and 100 ug/L . The calibration curve must have linear regression coefficient of determination (r^2) of at least 0.995. The 4 ug/L standard, when calculated back to the calibration curve, must be within ±25% of the actual value. The remainder of the standards must be within 15% of the actual value.

10.3 The calibration curve must be verified with the low continuing calibration verification standard (LCCV, 4 ug/L calibration standard) each working day. If the response for perchlorate varies from the expected values by more than ±25%, the test must be repeated, using fresh calibration standard. If the results are still more than ±25%, a new calibration curve must be prepared for the analyte. The shift in retention time must be within ±5% of expected value. If not, a new calibration curve must be prepared.

10.4 A mid-level continuing check verification (MCCV, 20 ug/L), alternating with a high level (HCCV, 100 ug/L) must be run after every 10 client samples. The last analysis of the run sequence is always a check standard. CCV must be within ±15% of expected value.

10.5 The instrument performance check (IPC) should not vary in retention time by more than 5%. The control limits for the perchlorate recovery are ±20%.

10.6 One method blank (MB) is run with every batch of ≤20 samples. The method blank is an aliquot of filtered reagent water that is prepared with samples and carried through the entire process.

10.7 One laboratory control sample (LCS) is run with every batch of ≤20 samples. For the LCS control limits, refer to current control charts.

10.8 One matrix spike (MS) and one matrix spike duplicate (MSD) are run with every batch of ≤20 samples. For the MS/MSD control limits, refer to current control charts.

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10.9 A sample duplicate (SD) must be run for every 20 samples. RPD must be within 15%.

$$RPD = 100 \times \frac{|S - SD|}{(S + SD)/2}$$

11.0 PROCEDURE FOR ANALYSIS

11.1 Start up the IC according to the directions in the operator's manual.

11.2 Suppressor should be set at 300 milliamps.

11.3 Eluent flow rate = 1 ml per minute.

11.4 Injection volume is 1 mL.

Note: The IC injection loop is usually 25 µL. Hence, the injection loop will need to be changed back if any other analysis is to be run.

11.5 The IC must be run in external water mode using reagent water as the regenerant. The regenerate flow rate should be approximately 3 to 5 mL per minute. All reservoirs should be filled at the beginning.

11.6 Allow the system to equilibrate. The conductivity should settle to approximately 2 to 5 µs.

11.7 Prepare a calibration curve using the standards prepared as follows in 100 ml of reagent water as follows:

mL of working STD	µg/L	STD
4.0 mL of 0.1 ppm	4	1
1.0 mL of 1.0 ppm	10	2
2.0 mL of 1.0 ppm	20	3
0.5 mL of 10 ppm	50	4
1.0 mL of 10 ppm	100	5

11.8 Set up a run sheet. Runs 1-5 are the calibration standards, followed by the LCCV, method blank, ICV, IPC, DI water and then samples. Number autosampler vials with the corresponding sample number on the run sheet.

11.9 Remove at least 2 mL of standards, check standards, QC samples, and the client samples and place in the autosampler vials.

11.10 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.0 CORRECTIVE ACTIONS

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12.1 If the samples are more concentrated than the highest standard, dilute with filtered reagent water, preferably to the middle of the calibration curve or extend the calibration range, bracketing the concentration of the sample.

12.2 Method blanks are not subtracted from samples and must be below the reporting limit. Blanks above the reporting limit will require reextraction and analysis of the entire batch. If sufficient sample does not exist to reprepare and analyze, "flag" method blank result as greater than the reporting limit and report "B"- flagged sample data from original batch.

12.3 If the MS/MSD is out of QC limits reanalyze to verify result. If the MS/MSD is still out of limits identify source of problem/contamination and eliminate. Evaluate whether the unspiked sample level (sample) is greater than four times the spike level.

12.3.1 If the sample is greater than four times the spike, flag outlying MS/MSD results as estimated due to the four times rule.

12.3.2 If sample is less than or equal to four times the spike, flag the sample results as matrix effect indicated.

12.4 If the LCS is out of QC limits reanalyze to verify result. If the LCS is still out of limits identify the source of problem/contamination and eliminate. If sufficient sample aliquots exist, reprepare all samples and QC in a new batch and analyze. If sufficient sample does not exist to reprepare and analyze, "flag" LCS result as out and report "J"- flagged (estimated) sample data from original batch.

13.0 CALCULATIONS

13.1 The chromatograms will be labeled with the laboratory sample number.

13.2 Quantitation of analyte concentration is performed automatically by the instrument software.

13.3 The instrument concentration is multiplied by the final extraction volume divided by the mass of sample extracted. This number is multiplied by 10 (prep dilution factor) and divided by the percent (%) solids.

13.4 Multiply this concentration and the Reporting Limit (RL) by any additional dilution factors if necessary.

13.5 Make sure check standards, QC samples, and blanks are within respective acceptance ranges.

13.6 Record all data in the lab book. Report results in µg/kg two significant figures for solids.

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14.0 REAGENT WASTE DISPOSAL

The Health and Safety Officer is responsible for off-site removal of all hazardous materials.

15.0 REFERENCES

15.1 Dionex Application Note 121.

15.2 USEPA. 1999. Determination of Perchlorate in Drinking Water Using Ion Chromatography, Method 314.0, Revision 1.0. Cincinnati: November.

15.3 State of California. Department of Health Services. Sanitation and Radiation Laboratories (SRL) Branch. 1999. Determination of Perchlorate by Ion Chromatography, Revision 1.0 (CLO4RV-1.WPD). Berkeley: CDHS, March.

15.4 USEPA. National Exposure Research Laboratory. Ecosystems Research Division. 2000. Determination of Perchlorate at parts-per-billion levels in plants by Ion Chromatography, Draft. Athens, Georgia: USEPA, April. Jackson Ellington and John J Evans, (Please Note: This draft method was only used for general information).

SOP Author: Mike Wantland & Sharon Johnson	**SOP Reviewer:** Lisa L Quigg for 10/25/00 Kristine Kurtz Date

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Determination of Perchlorate by Ion Chromatography in a Solid Matrix

STANDARD OPERATING PROCEDURE (SOP): M-CC-69/N, Revision 2, October 2000

Authorization/Approval:

Michelle Vermilion for 10/16/00
John Spurr — Date
Quality Assurance Director

10/16/00
Scott Caillouette — Date
Supervisor

10/16/00
Robert Lieckfield Jr., CIH — Date
Laboratory Director

Applicability and Revision History:

Office Location: Novi, Michigan

Effective Date: R0, August 1, 2000
R1, October 3, 2000
R2, October 16, 2000

Reference Methods: EPA 314.0 modified

Media Matrices: Soils & other Solids

Distribution:

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☐ FA Facilities Mngt	☒ GM Govern. Projects	☐ PA Payables/Purch	☐ RE Receivables	☐ MV Method Validation	☐ EX Extractions	☐ ME Metals	☐ PP Pesticides PCBs
☐ FS Field Supplies	☐ HS Health & Safety	☐ QA Quality Assurance	☐ SC Sample Control	☐ SP Special Projects	☐ GC GC/HI	☐ MI Microscopy	☐ XR X-Ray Diffraction
☒ DC Document Control	☒ IC Information Center	☒ OTHERS: (Please List)	Parsons Engineering Science				

1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of the perchlorate anion in solids by ion chromatography after aqueous extraction and soild-phase cleanup procedures.

1.2. When this method is used to analyze unfamiliar samples, perchlorate identification should be supported by the use of matrix spikes. The matrix spike procedure is described in Section 6.5.

1.3 The linear calibration range for perchlorate is approximately 4 to 500 µg/L. The linear calibration may be extended to 1500 µg/L, but accuracy below 50 µg/L may be compromised. Quantitation below 50 µg/L is not recommended with the extended calibration. Sample concentrations higher than the upper calibration limit should be diluted with reagent water to the mid-range of the linear calibration and reanalyzed.

1.4 This method is recommended for use by analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

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2.0 SUMMARY OF METHOD

2.1 The analysis is performed using an ion chromatograph. The main components of the ion chromatograph used in this method include an autosampler, pump, guard column, analytical column, ion suppressor device, conductivity detector, and chromatography data system.

2.2 An aliquot of sample extract is injected into the ion chromatograph, where the perchlorate anion is separated from other sample components, then quantitated. A large volume sample injection, 1 ml, is employed to perform perchlorate measurement at the low µg/L level.

2.3 Perchlorate is a large polarizable anion with hydrophobic properties. To avoid or minimize hydrophobic interaction of perchlorate with the guard and separator columns, care must be exercised in the selection of the ion-exchange column and the eluent composition. Extensive hydrophobic interaction of the perchlorate anion with the ion-exchange column leads to a long retention time and severe peak tailing. The columns and eluents recommended in this method were selected to minimize this hydrophobic interaction.

3.0 INTERFERENCES

3.1 The wide water dip or negative peak that may be observed in the chromatogram is due to the large volume sample injection. The negative response is the result of an imbalance of ions in the sample relative to the eluent and does not interfere with the measurement of perchlorate, since perchlorate elutes well beyond the dip.

3.2 Interferences can be caused by substances with retention times that are similar to and overlap with the anion of interest.

3.3 High concentrations of anions can interfere with the peak resolution of an adjacent anion and/or cause baseline disturbances. When such an interference occurs, it may help to dilute the sample before analysis, although it should be recognized that the sample detection limit increases proportionately with the sample dilution factor.

3.4 Surfactants and detergents may cause fouling of the ion exchange column and membrane suppressor, resulting in an unstable baseline and poor detector response. Other dissolved organic compounds may similarly interfere.

4.0 APPARATUS AND MATERIALS

4.1 Ion chromatograph – Dionex DX120 or equivalent, complete with all required accessories including syringes, analytical columns, compressed gases, and detectors.

4.1.1 Anion guard column: A protector of the separator column. Dionex AG 16, 4 mm.

4.1.2 Anion separator column: This column produces the separation of the anions. Dionex AS 16, 4 mm.

4.1.3 Anion suppressor device: Dionex ASRS II.

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4.1.4 Automated Sampler: Dionex ASM 3, or equivalent.

4.1.5 PeakNet IC Software: Dionex.

4.1.6 Conductivity detector: 1.25 µL internal volume.

4.1.7 Sample loop: Approximately 1000 µL (12" x 0.02" I.D. tubing), or larger, as required.

4.2 Polyethylene autosampler vials, 5 mL.

4.3 Filter caps, 5 mL.

4.4 Volumetric flasks, 50, 100, 1000, and 2000 mL.

4.5 Plastic vials, 10 mL.

4.6 Falcon tubes, 15 ml and 50 ml.

4.7 Laboratory balance, capable of weighing to 0.1 mg.

4.8 Shaker table.

4.9 Vortex

4.10 Disposable syringes, 10 ml.

4.11 Centrifuge(s), capable of accommodating 50 mL Falcon tubes and 16 x 125-mm disposable glass test tubes.

4.12 Kimwipes, delicate task wipes.

4.13 Disposable test tubes, 16 x 125 mm with caps.

4.14 Gelman ion chromatography (IC) Acrodisc or equivalent, 13 mm or 25 mm syringe filter with 0.45 um Supor (PES) membrane, catalog #4485T or #4585T.

4.15 C18 Solid Phase Extraction (SPE) cartridges or equivalent, Supelco Superclean ENVI-18 SPE Tubes, 6 ml (1 g), catalog #505706.

4.16 Solid phase extraction vacuum manifold.

4.17 Alumina, (Aluminum Oxide), Aldrich catalog #19,944-3, activated, basic, Brockmann I, standard grade, 150 mesh, 58 A, surface area 155 m^2/g.

Note: The alumina is cleaned by mixing one volume of alumina with 10 volumes of reagent water. The fines (cloudy water above the solid) are decanted and replaced with fresh reagent water. The mixing and decanting process is repeated until the water remains clear. The cleaned alumina is then allowed to air dry and stored in a screw cap glass bottle until used.

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5.0 REAGENTS

5.1 Reagent water, obtained from the Milli-Q Water System, through the filtered spigot.

5.2 Sodium perchlorate (solid), Aldrich catalog #41,024-1.

5.3 Potassium perchlorate, secondary source standard (1000ppm solution), SPEX catalog #AS-CLO49-2Y.

5.4 50% w/w sodium hydroxide, Fisher Chemical catalog #55254-500, or equivalent.

Note: For 100 mM sodium hydroxide, weigh 8.0 grams of 50% NaOH into a plastic weight boat and transfer into approximately 950 mL of reagent water and bring to 1000 mL volume.

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5.5 Methanol, HPLC grade.

6.0 PREPARATION OF STANDARDS

Note: In all cases where weighing is involved, the exact weight (all decimal places for the balance used) is recorded.

6.1 Primary Stock and Working Standards: All stock standards can be used for up to12 months. All other standards can be used for up to 6 months.

6.1.1 1000 ppm Primary Perchlorate Stock Standard: Dissolve 1.231 g of sodium perchlorate in 1000 mls of reagent water.

6.1.2 10 ppm Perchlorate Working Standard: Dilute 1.0 ml of 1000 ppm stock perchlorate to 100 ml with reagent water.

6.1.3 1.0 ppm Perchlorate Working Standard: Dilute 10.0 ml of 10 ppm perchlorate to 100 ml with reagent water.

6.1.4 0.1 ppm Perchlorate Working Standard: Dilute 1.0 ml of 10 ppm perchlorate to 100 ml with reagent water.

6.2 Secondary Source Standards

6.2.1 10 ppm Secondary Source Perchlorate Standard: Dilute 1.0 ml of the 1000 ppm secondary source perchlorate standard to 100 ml with reagent water.6.2.2 1.0 ppm Secondary Source Perchlorate Standard: Dilute 10 ml of 10 ppm secondary source perchlorate standard to 100 ml with reagent water.

6.3 Mixed Common Anion Stock Solution (containing the anions chloride, sulfate and carbonate each at 25 mg/ml), used to prepare simulated common anion samples in the determination of the MCT: Dissolve the following salts in reagent water to a final volume of 25.0 ml.

(a) 1.0 g sodium chloride (NaCl, CAS [7647-14-5]) = 0.61 g Cl^-
(b) 93 g sodium sulfate (Na_2SO_4, CAS [7757-82-6]) = 0.63 g SO_4^{-2}
(c) 1.1 g sodium carbonate (Na_2CO_3, CAS [497-19-8]) = 0.62 g CO_3^{-2}

6.4 Instrument Performance Check (IPC) Standard: Add 2.5 mls of 1.0 ppm perchlorate working standard and 3.2 mls of mixed common anion stock to a 100 ml volumetric flask and dilute to volume with reagent water.

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6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD): Prepare by spiking 400 µL of 10 ppm perchlorate working standard or secondary source standard to 10 g of sample. (Note: A smaller volume of sample may have to be used for when there is limited sample volume)

6.6 Laboratory Control Sample (LCS): Prepare by spiking 400 µL of 10 ppm perchlorate working standard or secondary source standard into 20 ml of DI water.

6.7 Instrument Calibration Verification Standard (ICV): Prepare by adding 2.0 ml of 1.0 ppm secondary source perchlorate standard to 50 ml of reagent water in a 100 ml volumetric flask and dilute to volume with reagent water.

7.0 SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

7.1 Samples may be collected in HDPE plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

7.2 Until further preservation and holding time studies are available, there is no maximum holding time. Samples will be stored at 4°C only to minimize biological activity.

8.0 PROCEDURE FOR SAMPLE PREPARATION/EXTRACTION

8.1 Weigh 10.0 g (±0.05 g) of solid into 50 mL Falcon tube.

8.2 Pipet 20.0 ml of reagent water into tube

8.3 Place on mechanical shaker for approximately 16 hours.

8.4 Centrifuge for 30 minutes.

8.5 Decant top layer (sample extract) through a Kimwipe into a clean 50 ml Falcon tube.

8.6 Add a 5.0 ml aliquot of this extract to 2.5 g (±0.1g) of Alumina in a disposable 12.5 ml test tube and cap.

8.7 Vortex test tube for approximately 20 seconds.

8.8 Place test tube on a mechanical shaker for 1 hour.

8.9 Centrifuge the test tube for 15 minutes.

8.10 Pour the top layer (sample extract) into a clean 15 ml Falcon tube.

8.11 Add 1.0 ml of sample extract to 9.0 ml of reagent water in a clean 15 ml Falcon tube. This results in a 1:10 dilution of the sample extract.

8.12 Solid Phase Extraction (SPE) C18 Cleanup: Pass the diluted sample extract (1:10) through two ENVI-18 SPE cartridges to remove potential organic matrix interference compounds.

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8.12.1 Condition each C18 cartridge per the manufacturer's instructions by rinsing with methanol followed by reagent water. The flow rate should not exceed 5.0 ml/minute.

8.12.1.1 Rinse cartridges with 2 ml of methanol.

8.12.1.2 Rinse cartridges with 2 ml of reagent water.

8.12.2 After conditioning with methanol and reagent water, rinse cartridge with 0.5 ml of diluted sample extract.

8.12.3 Pass approximately 5.8 ml of diluted sample extract through the conditioned and rinsed C18 cartridge into a clean disposable test tube.

8.12.4 Remove the test tube containing approximately 5.8 ml of sample extract (first C18 extract) and set aside.

8.12.5 Repeat steps in 8.12.1 for a second C18 cartridge.

8.12.6 After conditioning the second C18 cartridge, rinse the cartridge with 0.5 ml of the first C18 extract (extract in test tube from step 8.12.4).

8.12.7 To obtain the final extract, pass 5.0 ml of the first C18 extract (extract in test tube from step 8.12.4) through the second conditioned and rinsed C18 cartridge into a clean disposable test tube.

8.13 Filter approximately 2 ml of final extract through a Gelman IC Acrodisc 0.45 um filter into an autosampler vial for analysis and the remaining extract to a 15 ml Falcon tube to be stored refrigerated for any additional analysis.

9.0 HEALTH AND SAFETY

9.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

9.2 The following chemicals have the potential to be highly toxic or hazardous. The MSDS for each chemical should be consulted.
(a) Sodium hydroxide
(b) Sodium perchlorate
(c) Potassium perchlorate

10.0 QUALITY CONTROL

10.1 All IC chromatograms are kept for 5 years.

10.2 Perform an initial calibration with standards at 4, 10, 20, 50, and 100 ppb. The calibration curve must have linear regression coefficient of determination (r^2) of at least 0.995. The 4 ppb standard, when calculated back to the calibration curve, must be within ±25% of the actual value. The remainder of the standards must be within 15% of the actual value.

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10.3 The calibration curve must be verified with the low continuing calibration verification standard (LCCV, 4ug/l calibration standard) each working day, or whenever the eluent is changed. If the response for perchlorate varies from the expected values by more that ±25%, the test must be repeated, using fresh calibration standard. If the results are still more than ±25%, a new calibration curve must be prepared for the analyte. The shift in retention time must be within ±5% of expected value. If not, a new calibration curve must be prepared.

10.4 A mid-level continuing check verification (MCCV,20 ug/L), alternating with a high level (HCCV,100 ug/L) must be run after every 10 client samples. The last analysis of the run sequence is always a check standard. CCV must be within ±15% of expected value.

10.5 The instrument performance check (IPC) should not vary in retention time by more than 5%. The control limits for the perchlorate recovery are ±20%.

10.6 One method blank (MB) is run with every batch of ≤20 samples. The method blank is an aliquot of filtered DI water that is prepared with samples and carried through the entire process.

10.7 One laboratory control sample (LCS) is run with every batch of ≤20 samples. For the LCS control limits, refer to current control charts.

10.8 One matrix spike (MS) and one matrix spike duplicate (MSD) are run with every batch of ≤20 samples. For the MS/MSD control limits, refer to current control charts.

10.9 A sample duplicate (SD) must be run for every 20 samples. RPD must be within 15%.

$$RPD = 100 \times \frac{|S - SD|}{(S + SD)/2}$$

11.0 PROCEDURE FOR ANALYSIS

11.1 Start up the IC according to the directions in the operator's manual.

11.2 Suppressor should be set at 300 milliamps.

11.3 Eluent flow rate = 1 ml per minute.

11.4 Injection volume is 1 mL.

Note: The IC injection loop is usually 25 µL. Hence, the injection loop will need to be changed back if any other analysis is to be run.

11.5 The IC must be run in external water mode using reagent water as the regenerant. The regenerate flow rate should be approximately 3 to 5 mL per minute. All reservoirs should be filled at the beginning.

11.6 Allow the system to equilibrate. The conductivity should settle to approximately 2 to 5 µs.

11.7 Prepare a calibration curve using the standards prepared as follows in 100 ml of reagent water as follows:

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mL of working STD	µg/L	STD
4 mL of 0.1 ppm	4	1
1.0 mL of 1.0 ppm	10	2
2.0 mL of 1.0 ppm	20	3
0.5 mL of 10 ppm	50	4
1.0 mL of 10 ppm	100	5

11.8 Set up a run sheet. Runs 1-5 are the calibration standards, followed by the LCCV, method blank, ICV, IPC, DI water and then samples. Number autosampler vials with the corresponding sample number on the run sheet.

11.9 Remove at least 2 mL of standards, check standards, QC samples, and the client samples and place in the autosampler vials.

11.10 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.0 CORRECTIVE ACTIONS

12.1 If the samples are more concentrated than the highest standard, dilute with filtered DI water, preferably to the middle of the calibration curve or extend the calibration range, bracketing the concentration of the sample.

12.2 Method blanks are not subtracted from samples and must be below the reporting limit. Blanks above the reporting limit will require reextraction and analysis of the entire batch. If sufficient sample does not exist to reprepare and analyze, "flag" method blank result as greater than the reporting limit and report "B"- flagged sample data from original batch.

12.3 If the MS/MSD is out of QC limits reanalyze to verify result. If the MS/MSD is still out of limits identify source of problem/contamination and eliminate. Evaluate whether the unspiked sample level (sample) is greater than four times the spike level.

12.3.1 If the sample is greater than four times the spike, flag outlying MS/MSD results as estimated due to the four times rule.

12.3.2 If sample is less than or equal to four times the spike, flag the sample results as matrix effect indicated.

12.4 If the LCS is out of QC limits reanalyze to verify result. If the LCS is still out of limits identify the source of problem/contamination and eliminate. If sufficient sample aliquots exist, reprepare all samples and QC in a new batch and analyze. If sufficient sample does not exist to reprepare and analyze, "flag" LCS result as out and report "J"- flagged (estimated) sample data from original batch.

13.0 CALCULATIONS

13.1 The chromatograms will be labeled with the laboratory sample number.

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13.2 Quantitation of analyte concentration is performed automatically by the instrument software.

13.3 The instrument concentration is multiplied by the final extraction volume divided by the amount of samples extracted. This number multiplied by 10 (prep dilution factor) and divided by the percent (%) solids.

13.4 Multiply this concentration and the Reporting Limit (RL) by any additional dilution factors if necessary.

13.5 Make sure check standards, QC samples, and blanks are within respective acceptance ranges.

13.6 Record all data in the lab book. Report results in µg/kg two significant figures for solids.

14.0 REAGENT WASTE DISPOSAL

The Health and Safety Officer is responsible for off-site removal of all hazardous materials.

15.0 REFERENCES

15.1 Dionex Application Note 121.

15.2 USEPA. 1999. Determination of Perchlorate in Drinking Water Using Ion Chromatography, Method 314.0, Revision 1.0. Cincinnati: November.

15.3 State of California. Department of Health Services. Sanitation and Radiation Laboratories (SRL) Branch. 1999. Determination of Perchlorate by Ion Chromatography, Revision 1.0 (CLO4RV-1.WPD). Berkeley: CDHS, March.

15.4 USEPA. National Exposure Research Laboratory. Ecosystems Research Division. 2000. Determination of Perchlorate at parts-per-billion levels in plants by Ion Chromatography, Draft. Athens, Georgia: USEPA, April. Jackson Ellington and John J Evans, (Please Note: This draft method was only used for general information).

SOP Author: Mike Wantland & Sharon Johnson **SOP Reviewer:** Lisa L Ruggle 10/10/00 Date

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Determination of Perchlorate by Ion Chromatography in Vegetation

STANDARD OPERATING PROCEDURE (SOP): M-CC-70/N, Revision 1, November 2000

Authorization/Approval:

John Spurr, Quality Assurance Director	Date 11/29/00
Scott Caillouette, Supervisor	Date 11-29-00
Robert Lieckfield, Jr., CIH, Laboratory Director	Date 11-29-00

Applicability and Revision History:

Office Location:	Novi, Michigan
Effective Date:	R0, October 18, 2000 R1, November 29, 2000
Reference Methods:	EPA 314.0 modified
Media/Matrices:	Vegetation

Distribution:

☐ CS Client Services	☐ GL General Lab Mngt	☐ LI LIMS	☐ RC Report Control	☐ MP cGMP Services	☒ CC Classical Chem	☐ LC HPLC	☐ MS GC/MS
☐ FA Facilities Mngt	☒ GM Govern Projects	☐ PA Payables/Purch	☐ RE Receivables	☐ MV Method Validation	☐ EX Extractions	☐ ME Metals	☐ PP Pesticides/PCBs
☐ FS Field Supplies	☐ HS Health & Safety	☐ QA Quality Assurance	☐ SC Sample Control	☐ SP Special Projects	☐ GC GC/IH	☐ MI Microscopy	☐ XR X-Ray Diffraction
☒ DC Document Control	☒ IC Information Center	☒ OTHERS: (Please List)	Parsons Engineering Service				

1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of the perchlorate anion in vegetation by ion chromatography after aqueous extraction and solid–phase cleanup procedures.

1.2 Although not specifically tested, this method is potentially applicable to other solid matrixes. When this method is used to analyze unfamiliar samples, perchlorate identification should be supported by the use of matrix spikes. The matrix spike procedure is described in Section 6.5.

1.3 The linear calibration range for perchlorate is approximately 4 to 500 µg/L. The linear calibration may be extended to 1500 µg/L, but accuracy below 50 µg/L may be compromised. Quantitation below 50 µg/L is not recommended with the extended calibration. Sample concentrations higher than the upper calibration limit should be diluted with reagent water to the mid-range of the linear calibration and reanalyzed.

1.4 This method is recommended for use by analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

2.0 SUMMARY OF METHOD

2.1 The analysis is performed using an ion chromatograph. The main components of the ion chromatograph used in this method include an autosampler, pump, guard column, analytical column, ion suppressor device, conductivity detector, and chromatography data system.

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2.2 An aliquot of sample extract is injected into the ion chromatograph, where the perchlorate anion is separated from other sample components, then quantitated. A large volume sample injection, 1 ml, is employed to perform perchlorate measurement at the low µg/L level.

2.3 Perchlorate is a large polarizable anion with hydrophobic properties. To avoid or minimize hydrophobic interaction of perchlorate with the guard and separator columns, care must be exercised in the selection of the ion-exchange column and the eluent composition. Extensive hydrophobic interaction of the perchlorate anion with the ion-exchange column leads to a long retention time and severe peak tailing. The columns and eluents recommended in this method were selected to minimize this hydrophobic interaction.

3.0 INTERFERENCES

3.1 The wide water dip or negative peak that may be observed in the chromatogram is due to the large volume sample injection. The negative response is the result of an imbalance of ions in the sample relative to the eluent and does not interfere with the measurement of perchlorate, since perchlorate elutes well beyond the dip.

3.2 Interferences can be caused by substances with retention times that are similar to and overlap with the anion of interest.

3.3 High concentrations of anions can interfere with the peak resolution of an adjacent anion and/or cause baseline disturbances. When such an interference occurs, it may help to dilute the sample before analysis, although it should be recognized that the sample detection limit increases proportionately with the sample dilution factor.

3.4 Surfactants and detergents may cause fouling of the ion exchange column and membrane suppressor, resulting in an unstable baseline and poor detector response. Other dissolved organic compounds may similarly interfere.

4.0 APPARATUS AND MATERIALS

4.1 Ion chromatograph – Dionex DX120 or equivalent, complete with all required accessories including syringes, analytical columns, compressed gases, and detectors.

4.1.1 Anion guard column: A protector of the separator column. Dionex AG 16, 4 mm.

4.1.2 Anion separator column: This column produces the separation of the anions. Dionex AS 16, 4 mm.

4.1.3 Anion suppressor device: Dionex ASRS II.

4.1.4 Automated Sampler: Dionex ASM 3, or equivalent.

4.1.5 PeakNet IC Software: Dionex.

4.1.6 Conductivity detector: 1.25 µL internal volume.

4.1.7 Sample loop: Approximately 1000 µL (12" x 0.02" I.D. tubing), or larger, as required.

4.2 Polyethylene autosampler vials, 5 mL.

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4.3 Filter caps, 5 mL.

4.4 Volumetric flasks, 50, 100, 1000, and 2000 mL.

4.5 Plastic vials, 10 mL.

4.6 Falcon tubes, 15 ml and 50 ml.

4.7 Laboratory balance, capable of weighing to 0.1 mg.

4.8 Shaker table.

4.9 Vortex

4.10 Disposable syringes, 10 ml.

4.11 Centrifuge(s), capable of accommodating 50 mL Falcon tubes and 16 x 125-mm disposable glass test tubes.

4.12 Kimwipes, delicate task wipes.

4.13 Disposable test tubes, 16 x 125 mm with caps.

4.14 Gelman ion chromatography (IC) Acrodisc or equivalent, 25 mm syringe filter with 0.45 um Supor (PES) membrane, catalog #4585T.

4.15 C18 Solid Phase Extraction (SPE) cartridges or equivalent, Supelco Superclean ENVI-18 SPE Tubes, 20 ml (5 g), catalog #57137.

4.16 Solid phase extraction vacuum manifold.

4.17 Alumina, (Aluminum Oxide), Aldrich catalog #19,944-3, activated, basic, Brockmann I, standard grade, 150 mesh, 58 A, surface area 155 m^2/g.

Note: The alumina is cleaned by mixing one volume of alumina with 10 volumes of reagent water. The fines (cloudy water above the solid) are decanted and replaced with fresh reagent water. The mixing and decanting process is repeated until the water remains clear. The cleaned alumina is then allowed to air dry and stored in a screw cap glass bottle until used.

5.0 REAGENTS

5.1 Reagent water, obtained from the Milli-Q Water System, through the filtered spigot.

5.2 Sodium perchlorate (solid), Aldrich catalog #41,024-1.

5.3 Secondary source standard (1000ppm solution), SPEX catalog #AS-CLO49-2Y.

5.4 50% w/w sodium hydroxide, Fisher Chemical catalog #55254-500, or equivalent.

Note: For 50 mM sodium hydroxide, weigh 4.0 grams of 50% NaOH into a plastic weight boat and transfer into approximately 950 mL of reagent water and bring to 1000 mL volume.

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5.5 Methanol, HPLC grade.

6.0 PREPARATION OF STANDARDS

Note: In all cases where weighing is involved, the exact weight (all decimal places for the balance used) is recorded.

6.1 Primary Stock and Working Standards: All stock standards can be used for up to 12 months. All other standards can be used for up to 6 months.

6.1.1 1000 ppm Primary Perchlorate Stock Standard: Dissolve 1.231 g of sodium perchlorate in 1000 ml of reagent water.

6.1.2 10 ppm Perchlorate Working Standard: Dilute 1.0 ml of 1000 ppm stock perchlorate to 100 ml with reagent water.

6.1.3 1.0 ppm Perchlorate Working Standard: Dilute 10.0 ml of 10 ppm perchlorate to 100 ml with reagent water.

6.1.4 0.1 ppm Perchlorate Working Standard: Dilute 1.0 ml of 10 ppm perchlorate to 100 ml with reagent water.

6.2 Secondary Source Standards

6.2.1 10 ppm Secondary Source Perchlorate Standard: Dilute 1.0 ml of the 1000 ppm secondary source perchlorate standard to 100 ml with reagent water.

6.2.2 1.0 ppm Secondary Source Perchlorate Standard: Dilute 10 ml of 10 ppm secondary source perchlorate standard to 100 ml with reagnt water.

6.3 Mixed Common Anion Stock Solution (containing the anions chloride, sulfate and carbonate each at 25 mg/ml), used to prepare simulated common anion samples in the determination of the MCT: Dissolve the following salts in reagent water to a final volume of 25.0 ml.
(a) 1.0 g sodium chloride (NaCl, CAS [7647-14-5]) = 0.61 g Cl^-
(b) 93 g sodium sulfate (Na_2SO_4, CAS [7757-82-6]) = 0.63 g SO_4^{-2}
(c) 1.1 g sodium carbonate (Na_2CO_3, CAS [497-19-8]) = 0.62 g CO_3^{-2}

6.4 Instrument Performance Check (IPC) Standard: Add 2.5 ml of 1.0 ppm perchlorate working standard and 3.2 ml of mixed common anion stock to a 100 ml volumetric flask and dilute to volume with reagent water.

6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD): Prepare by spiking 400 µL of 10 ppm perchlorate working standard or secondary source standard to 2 g of sample. (Note: A smaller volume may have to be used due to limited sample volume)

6.6 Laboratory Control Sample (LCS): Prepare by spiking 400 µL of 10 ppm perchlorate working standard or secondary source standard into 20 ml of reagent water.

6.7 Instrument Calibration Verification Standard (ICV): Prepare by adding 2.0 ml of 1.0 ppm secondary source perchlorate standard to 50 ml of reagent water in a 100 ml volumetric flask and dilute to volume with reagent water.

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7.0 SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

7.1 Samples may be collected in HDPE plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

7.2 Until further preservation and holding time studies are available, there is no maximum holding time. Samples are to be stored at <0°C to minimize decomposition.

8.0 PROCEDURE FOR SAMPLE PREPARATION/EXTRACTION

8.1 Weigh 2.0 g (±0.1 g) of vegetation into 50 mL Falcon tube.

8.2 Pipet 20.0 ml of reagent water into tube

8.3 Place on mechanical shaker for approximately 16 hours.

8.4 Centrifuge for 30 minutes.

8.5 Decant top layer (sample extract) into a clean 50 ml Falcon tube.

8.6 Add a 5.0 ml aliquot of this extract to 2.5 g (±0.1g) of Alumina in a disposable 12.5 ml test tube and cap.

8.7 Vortex test tube for approximately 20 seconds.

8.8 Place test tube on a mechanical shaker for 1 hour.

8.9 Centrifuge the test tube for 15 minutes.

8.10 Filter the top layer (sample extract) through a 25 mm Gelman IC Acrodisc 0.45 um into a clean 15 ml Falcon tube using a 10 mL disposable syringe.

8.11 Add 1.0 ml of sample extract to 9.0 ml of reagent water in a clean 15 ml Falcon tube. This results in a 1:10 dilution of the sample extract.

8.12 Solid Phase Extraction (SPE) C18 Cleanup: Pass the diluted sample extract (1:10) through one ENVI-18 SPE cartridge to remove potential organic matrix interference compounds.

8.12.1 Condition each C18 cartridge per the manufacturer's instructions by rinsing with methanol followed by reagent water. The flow rate should not exceed 5.0 ml/minute.

8.12.1.1 Rinse cartridge with 4 ml of methanol.

8.12.1.2 Rinse cartridge with 5 ml of reagent water.

8.12.2 After conditioning with methanol and reagent water, rinse cartridge with 2.0 ml of diluted sample extract.

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8.12.3 To obtain the final extract, pass approximately 5.5 ml of diluted sample extract through the conditioned and rinsed C18 cartridge into a clean disposable test tube. Transfer the final extract to a 15 mL Falcon tube.

8.13 Transfer approximately 2 ml of final extract into an autosampler vial for analysis. The remaining extract is stored refrigerated for any additional analysis.

9.0 HEALTH AND SAFETY

9.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

9.2 The following chemicals have the potential to be highly toxic or hazardous. The MSDS for each chemical should be consulted.
(a) Sodium hydroxide
(b) Sodium perchlorate

10.0 QUALITY CONTROL

10.1 All IC chromatograms are kept for 5 years.

10.2 Perform an initial calibration with standards at 4, 10, 20, 50, and 100 ug/L . The calibration curve must have linear regression coefficient of determination (r^2) of at least 0.995. The 4 ug/L standard, when calculated back to the calibration curve, must adhere to statistical limits. The remainder of the standards must be within 15% of the actual value.

10.3 The calibration curve must be verified with the low continuing calibration verification standard (LCCV, 4 ug/L calibration standard) each working day. If the response for perchlorate varies from the expected statistical limits, the test must be repeated, using fresh calibration standard. If the results are still outside the limits, a new calibration curve must be prepared for the analyte. The shift in retention time must be within ±5% of expected value. If not, a new calibration curve must be prepared.

10.4 A mid-level continuing check verification (MCCV, 20 ug/L), alternating with a high level (HCCV, 100 ug/L) must be run after every 10 client samples. The last analysis of the run sequence is always a check standard. CCV must be within ±15% of expected value.

10.5 The instrument performance check (IPC) should not vary in retention time by more than 5%. The control limits for the perchlorate recovery are ±20%.

10.6 One method blank (MB) is run with every batch of ≤20 samples. The method blank is an aliquot of filtered reagent water that is prepared with samples and carried through the entire process.

10.7 One laboratory control sample (LCS) is run with every batch of ≤20 samples. For the LCS control limits, refer to current control charts.

10.8 One matrix spike (MS) and one matrix spike duplicate (MSD) are run with every batch of ≤20 samples. For the MS/MSD control limits, refer to current control charts.

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10.9 A sample duplicate (SD) must be run for every 20 samples. RPD must be within statisticaly determined limits.

$$RPD = 100 \times \frac{|S - SD|}{(S + SD)/2}$$

11.0 PROCEDURE FOR ANALYSIS

11.1 Start up the IC according to the directions in the operator's manual.

11.2 Suppressor should be set at 300 milliamps.

11.3 Eluent flow rate = 1 ml per minute.

11.4 Injection volume is 1 mL.

Note: The IC injection loop is usually 25 µL. Hence, the injection loop will need to be changed back if any other analysis is to be run.

11.5 The IC must be run in external water mode using reagent water as the regenerant. The regenerate flow rate should be approximately 3 to 5 mL per minute. All reservoirs should be filled at the beginning.

11.6 Allow the system to equilibrate. The conductivity should settle to approximately 2 to 5 µs.

11.7 Prepare a calibration curve using the standards prepared as follows in 100 ml of reagent water as follows:

mL of working STD	µg/L	STD
4.0 mL of 0.1 ppm	4	1
1.0 mL of 1.0 ppm	10	2
2.0 mL of 1.0 ppm	20	3
0.5 mL of 10 ppm	50	4
1.0 mL of 10 ppm	100	5

11.8 Set up a run sheet. Runs 1-5 are the calibration standards, followed by the LCCV, method blank, ICV, IPC, DI water and then samples. Number autosampler vials with the corresponding sample number on the run sheet.

11.9 Remove at least 2 mL of standards, check standards, QC samples, and the client samples and place in the autosampler vials.

11.10 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

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12.0 CORRECTIVE ACTIONS

12.1 If the samples are more concentrated than the highest standard, dilute with filtered reagent water, preferably to the middle of the calibration curve or extend the calibration range, bracketing the concentration of the sample.

12.2 Method blanks are not subtracted from samples and must be below the reporting limit. Blanks above the reporting limit will require reextraction and analysis of the entire batch. If sufficient sample does not exist to reprepare and analyze, "flag" method blank result as greater than the reporting limit and report "B"- flagged sample data from original batch.

12.3 If the MS/MSD is out of QC limits reanalyze to verify result. If the MS/MSD is still out of limits identify source of problem/contamination and eliminate. Evaluate whether the unspiked sample level (sample) is greater than four times the spike level.

12.3.1 If the sample is greater than four times the spike, flag outlying MS/MSD results as estimated due to the four times rule.

12.3.2 If sample is less than or equal to four times the spike, flag the sample results as matrix effect indicated.

12.4 If the LCS is out of QC limits reanalyze to verify result. If the LCS is still out of limits identify the source of problem/contamination and eliminate. If sufficient sample aliquots exist, reprepare all samples and QC in a new batch and analyze. If sufficient sample does not exist to reprepare and analyze, "flag" LCS result as out and report "J"- flagged (estimated) sample data from original batch.

12.5 If the RPD for sample duplicates is outside statistical limits, flag the data and note in the case narrative.

13.0 CALCULATIONS

13.1 The chromatograms will be labeled with the laboratory sample number.

13.2 Quantitation of analyte concentration is performed automatically by the instrument software.

13.3 The instrument concentration is multiplied by the final extraction volume divided by the mass of sample extracted. This number is multiplied by 10 (prep dilution factor) and divided by the percent (%) solids.

13.4 Multiply this concentration and the Reporting Limit (RL) by any additional dilution factors if necessary.

13.5 Make sure check standards, QC samples, and blanks are within respective acceptance ranges.

13.6 Record all data in the lab book. Report results in µg/kg two significant figures for solids.

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14.0 REAGENT WASTE DISPOSAL

The Health and Safety Officer is responsible for off-site removal of all hazardous materials.

15.0 REFERENCES

15.1 Dionex Application Note 121.

15.2 USEPA. 1999. Determination of Perchlorate in Drinking Water Using Ion Chromatography, Method 314.0, Revision 1.0. Cincinnati: November.

15.3 State of California. Department of Health Services. Sanitation and Radiation Laboratories (SRL) Branch. 1999. Determination of Perchlorate by Ion Chromatography, Revision 1.0 (CLO4RV-1.WPD). Berkeley: CDHS, March.

15.4 USEPA. National Exposure Research Laboratory. Ecosystems Research Division. 2000. Determination of Perchlorate at parts-per-billion levels in plants by Ion Chromatography, Draft. Athens, Georgia: USEPA, April. Jackson Ellington and John J Evans, (Please Note: This draft method was only used for general information).

SOP Author: Mike Wantland & Sharon Johnson **SOP Reviewer:** Kristine Kurtz 10/23/00
Kristine Kurtz Date

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Determination of Perchlorate by Ion Chromatography in a Water Matrix

STANDARD OPERATING PROCEDURE (SOP): M-CC-68/N, Revision 2, June 2000

Authorization/Approval:		Applicability and Revision History:	
John Spurr, Quality Assurance Director	11/29/00 Date	Office Location:	Novi, Michigan
Scott Cailouette, Supervisor	11/29/00 Date	Effective Date:	R0, December 21, 1999 R1, March 24, 2000 R2, June 15, 2000
Robert Lieckfield, Jr., CIH, Laboratory Director	11/29/00 Date	Reference Methods:	EPA 314.0 and CaDHS SRL
		Media/Matrices:	Drinking Water and Other Aqueous

A. M-CC-68/N, Revision 2 was in effect and used for analyses of perchlorate in water matrices beginning June 15, 2000. This method was finalized on November 29, 2000. With the incorporation of format edits and editorial clarifications.

Distribution:

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☒ DC Document Control	☒ IC Information Center	☒ OTHERS: (Please List) Parsons Engineering Science					

1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of the perchlorate anion in water by ion chromatography.

1.2. The applicable matrices are:

1.2.1 Drinking water, ground and surface waters.

1.2.2 Although not specifically tested, this method is potentially applicable to industrial wastewaters. See also Section 1.5.

1.3 The linear calibration range for perchlorate is approximately 4 to 500 µg/L. The linear calibration may be extended to 1500 µg/L, but accuracy below 50 µg/L may be compromised. Quantitation below 50 µg/L is not recommended with the extended calibration. Sample concentrations higher than the upper calibration limit should be diluted with reagent water to the mid-range of the linear calibration and reanalyzed.

1.4 This method is recommended for use by analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

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1.5 When this method is used to analyze unfamiliar samples, perchlorate identification should be supported by the use of matrix spikes. The matrix spike procedure is described in Section 6.5.

2.0 SUMMARY OF METHOD

2.1 The analysis is performed using an ion chromatograph. The main components of the ion chromatograph used in this method include an autosampler, pump, guard column, analytical column, ion suppressor device, conductivity detector, and chromatography data system.

2.2 An aliquot of sample is injected into the ion chromatograph, where the perchlorate anion is separated from other sample components, then quantitated. A large volume sample injection, 1 ml, is employed to perform perchlorate measurement at the low µg/L level.

2.3 Perchlorate is a large polarizable anion with hydrophobic properties. To avoid or minimize hydrophobic interaction of perchlorate with the guard and separator columns, care must be exercised in the selection of the ion-exchange column and the eluent composition. Extensive hydrophobic interaction of the perchlorate anion with the ion-exchange column leads to a long retention time and severe peak tailing. The columns and eluents recommended in this method were selected to minimize this hydrophobic interaction.

3.0 INTERFERENCES

3.1 The wide water dip or negative peak that may be observed in the chromatogram is due to the large volume sample injection. The negative response is the result of an imbalance of ions in the sample relative to the eluent and does not interfere with the measurement of perchlorate, since perchlorate elutes well beyond the dip.

3.2 Interferences can be caused by substances with retention times that are similar to and overlap with the anion of interest.

3.3 High concentrations of anions can interfere with the peak resolution of an adjacent anion and/or cause baseline disturbances. When such an interference occurs, it may help to dilute the sample before analysis, although it should be recognized that the sample detection limit increases proportionately with the sample dilution factor.

3.4 Surfactants and detergents may cause fouling of the ion exchange column and membrane suppressor, resulting in an unstable baseline and poor detector response. Other dissolved organic compounds may similarly interfere.

4.0 APPARATUS AND MATERIALS

4.1 Ion chromatograph Dionex DX120 or equivalent, complete with all required accessories including syringes, analytical columns, compressed gases, and detectors.

4.1.1 Anion guard column: A protector of the separator column. Dionex AG 16, 4 mm.

4.1.2 Anion separator column: This column produces the separation of the anions. Dionex AS 16, 4 mm.

4.1.3 Anion suppressor device: Dionex ASRS II

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4.1.4 Automated Sampler: Dionex ASM 3, or equivalent.

4.1.5 PeakNet IC Software: Dionex.

4.1.6 Conductivity detector: 1.25 µL internal volume.

4.1.7 Sample loop: Approximately 1000 µL (12" x 0.02" I.D. tubing), or larger, as required.

4.2 Polyethylene autosampler vials, 5 mL.

4.3 Filter caps, 5 mL.

4.4 Volumetric flasks, 50, 100, 1000, and 2000 mL.

4.5 Plastic vials, 10 mL.

4.6 Laboratory balance, capable of weighing to 0.1 mg.

5.0 REAGENTS

5.1 Reagent water, obtained from the Milli-Q Water System.

5.2 Sodium perchlorate (solid), Aldrich catalog #41,024-1.

5.3 Potassium perchlorate, secondary source standard (1000ppm solution), SPEX catalog #AS-CLO49-2Y.

5.4 50% w/w sodium hydroxide, Fisher Chemical catalog #55254-500, or equivalent.

Note: For 100 mM sodium hydroxide, weigh 16.0 grams of 50% NaOH into a plastic weight boat and transfer into approximately 950 mL of reagent water and bring to 2000 mL volume.

5.5 Methanol, HPLC grade.

6.0 PREPARATION OF STANDARDS

Note: In all cases where weighing is involved, the exact weight (all decimal places for the balance used) is recorded.

6.1 Primary Stock and Working Standards: All stock standards can be used for up to12 months. All other standards can be used for up to 6 months.

6.1.1 1000 ppm Primary Perchlorate Stock Standard: Dissolve 1.231 g of sodium perchlorate in 1000 mls of reagent water.

6.1.2 10 ppm Perchlorate Working Standard: Dilute 1.0 ml of 1000 ppm stock perchlorate to 100 ml with reagent water.

6.1.3 1.0 ppm Perchlorate Working Standard: Dilute 10.0 ml of 10 ppm perchlorate to 100 ml with reagent water.

6.1.4 0.1 ppm Perchlorate Working Standard: Dilute 1.0 ml of 10 ppm perchlorate to 100 ml with reagent water.

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6.2 Secondary Source Standards

6.2.1 10 ppm Secondary Source Perchlorate Standard: Dilute 1.0 ml of the 1000 ppm secondary source perchlorate standard to 100 ml with reagent water.

6.2.2 1.0 ppm Secondary Source Perchlorate Standard: Dilute 10 ml of 10 ppm secondary source perchlorate standard to 100 ml with reagent water.

6.3 Mixed Common Anion Stock Solution (containing the anions chloride, sulfate and carbonate each at 25 mg/ml), used to prepare simulated common anion samples in the determination of the MCT: Dissolve the following salts in reagent water to a final volume of 25.0 ml.
(a) 1.0 g sodium chloride (NaCl, CAS [7647-14-5]) = 0.61 g Cl^-
(b) 93 g sodium sulfate (Na_2SO_4, CAS [7757-82-6]) = 0.63 g SO_4^{-2}
(c) 1.1 g sodium carbonate (Na_2CO_3, CAS [497-19-8]) = 0.62 g CO_3^{-2}

6.4 Instrument Performance Check (IPC) Standard: Add 2.5 mls of 1.0 ppm perchlorate working standard and 3.2 mls of mixed common anion stock to a 100 ml volumetric flask and dilute to volume with reagent water.

6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD): Prepare by spiking 20 µL of 10 ppm perchlorate working standard to 10 ml of sample. (Note: A smaller volume of sample may have to be used for when there is limited sample volume)

6.6 Instrument Calibration Verification Standard (ICV): Prepare by adding 2.0 ml of 1.0 ppm secondary source perchlorate standard to 50 ml of reagent water in a 100 ml volumetric flask and dilute to volume with reagent water.

7.0 SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

7.1 Samples may be collected in HDPE plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

7.2 The following sample preservation and holding time for perchlorate are recommended, until further preservation and holing time studies are available.

Analyte	Preservation	Holding Time
Perchlorate	None required	28 days

8.0 HEALTH AND SAFETY

8.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous material or procedures.

8.2 The following chemicals have the potential to be highly toxic or hazardous. The MSDS for each chemical should be consulted.

8.2.1 Sodium hydroxide

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8.2.2 Potassium perchlorate

8.2.3 Sodium perchlorate

9.0 QUALITY CONTROL

9.1 All IC chromatograms are kept for 5 years.

9.2 Perform an initial calibration with standards at 4, 10, 20, 50, and 100 ppb. The calibration curve must have linear regression coefficient of determination (r^2) of at least 0.995. The 4 ppb standard, when calculated back to the calibration curve, must be within statistical limits of the actual value. The remainder of the standards must be within 15% of the actual value.

9.3 The calibration curve must be verified with the low continuing calibration verification standard (LCCV, 4ug/l calibration standard) each working day. If the response for perchlorate varies from the expected values of the statistical limits, the test must be repeated, using a fresh calibration standard. If the results still fail a new calibration curve must be prepared for the analyte.

9.4 A mid-level continuing check verification (MCCV,20 ug/L), alternating with a high level (HCCV,100 ug/L) must be run after every 10 client samples. The last analysis of the run sequence is always a check standard. CCV must be within ±15% of expected value.

9.5 The instrument performance check (IPC) should not vary in retention time by more than 5%. The control limits for the perchlorate recovery are ±20%.

9.6 One method blank (MB) is run with every batch of ≤20 samples. The method blank is an aliquot of filtered DI water that is prepared with samples and carried through the entire process.

9.7 One matrix spike (MS) and one matrix spike duplicate (MSD) are run with every batch of ≤20 samples. For the MS/MSD control limits, refer to current control charts.

9.8 A sample duplicate (SD) must be run for every 20 samples. RPD must be within statistical limits.

$$RPD = 100 \times \frac{|S - SD|}{(S + SD)/2}$$

9.9 The instrument calibration verification (ICV) should be within the control limits of ±15%.

10.0 PROCEDURE FOR ANALYSIS

10.1 Before the samples are run a conductivity check must be performed on them to determine if the sample conductivity is below the Matrix Conductivity Threshold (MCT) of 5980. (Refer to EPA Method 314.0)

10.2 If sample conductivity is below the MCT it is analyzed directly.

10.3 If the matrix conductance exceeds the MCT, estimate the proportion required for the dilution by dividing the measured matrix conductance by the MCT. Round up to the next

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whole number and dilute the sample by a proportion equivalent to this value. For example, if the established MCT is 6100 uS/cm and a sample reflecting a conductance of 8000 uS/cm was measured, dilute the sample with reagent water by factor of 2.

10.4 Start up the IC according to the directions in the operator's manual.

10.5 Suppressor should be set at 300 milliamps.

10.6 Eluent flow rate = 1 ml per minute.

10.7 Injection volume is 1 mL.

Note: The IC injection loop is usually 25 µL. Hence, the injection loop will need to be changed back if any other analysis is to be run.

10.8 The IC must be run in external water mode using reagent water as the regenerant. The regenerate flow rate should be approximately 3 to 5 mL per minute. All reservoirs should be filled at the beginning.

10.9 Allow the system to equilibrate. The conductivity should settle to approximately 2 to 5 µs.

10.10 Prepare a calibration curve using the standards prepared as follows in 100 ml of reagent water as follows:

mL of working STD	µg/L	STD
4 mL of 0.1 ppm	4	1
1.0 mL of 1.0 ppm	10	2
2.0 mL of 1.0 ppm	20	3
0.5 mL of 10 ppm	50	4
1.0 mL of 10 ppm	100	5

10.11 Set up a run sheet. Runs 1-5 are the calibration standards, followed by the LCCV, method blank, ICV, IPC, DI water and then samples. Number autosampler vials with the corresponding sample number on the run sheet.

10.12 Remove at least 2 mL of standards, check standards, QC samples, and the client samples and place in the autosampler vials.

10.13 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

11.0 CORRECTIVE ACTIONS

11.1 If the samples are more concentrated than the highest standard, dilute with filtered DI water, preferably to the middle of the calibration curve or extend the calibration range, bracketing the concentration of the sample.

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11.2 Method blanks are not subtracted from samples and must be below the reporting limit. Blanks above the reporting limit will require reanalysis of the entire batch. If sufficient sample does not exist to reanalyze, "flag" method blank result as greater than the reporting limit and report "B"- flagged sample data from original batch.

11.3 If the MS/MSD is out of QC limits reanalyze to verify result. If the MS/MSD is still out of limits identify source of problem/contamination and eliminate. Evaluate whether the unspiked sample level (sample) is greater than four times the spike level.

11.3.1 If the sample is greater than four times the spike, flag outlying MS/MSD results as estimated due to the four times rule.

11.3.2 If sample is less than or equal to four times the spike, flag the outlying MS/MSD as effected by matrix interferences.

11.3.3 If the ICV is outside the contol limits a new one must be prepared and run. If the ICV still fails a new calibration curve must be prepared.

12.0 CALCULATIONS

12.1 The chromatograms will be labeled with the laboratory sample number.

12.2 Quantitation of analyte concentration is performed automatically by the instrument software.

12.2.1 Multiply this concentration and the Reporting Limit (RL) by any additional dilution factors if necessary. Multiply the LOD by the dilution factor also, if dilutions were made prior to analysis and the samples read below the LOD.

12.3 Make sure check standards, QC samples, and blanks are within respective acceptance ranges. Determine the cause if this is not the case and rerun the samples if needed.

12.4 Record all data in the lab book. Report results in ug/L two significant figures for waters.

13.0 REAGENT WASTE DISPOSAL

The Health and Safety Officer is responsible for off-site removal of all hazardous materials.

14.0 REFERENCES

14.1 Dionex Application Note 121.

14.2 USEPA. 1999. Determination of Perchlorate in Drinking Water Using Ion Chromatography, Method 314.0, Revision 1.0. Cincinnati: November.

14.3 State of California. Department of Health Services. Sanitation and Radiation Laboratories (SRL) Branch. 1999. Determination of Perchlorate by Ion Chromatography, Revision 1.0 (CLO4RV-1.WPD). Berkeley: CDHS, March.

SOP Author: Scott Caillouette
Wet Chemistry Supervisor

SOP Reviewer: Lisa Blackburn
Environmental Chemist Date

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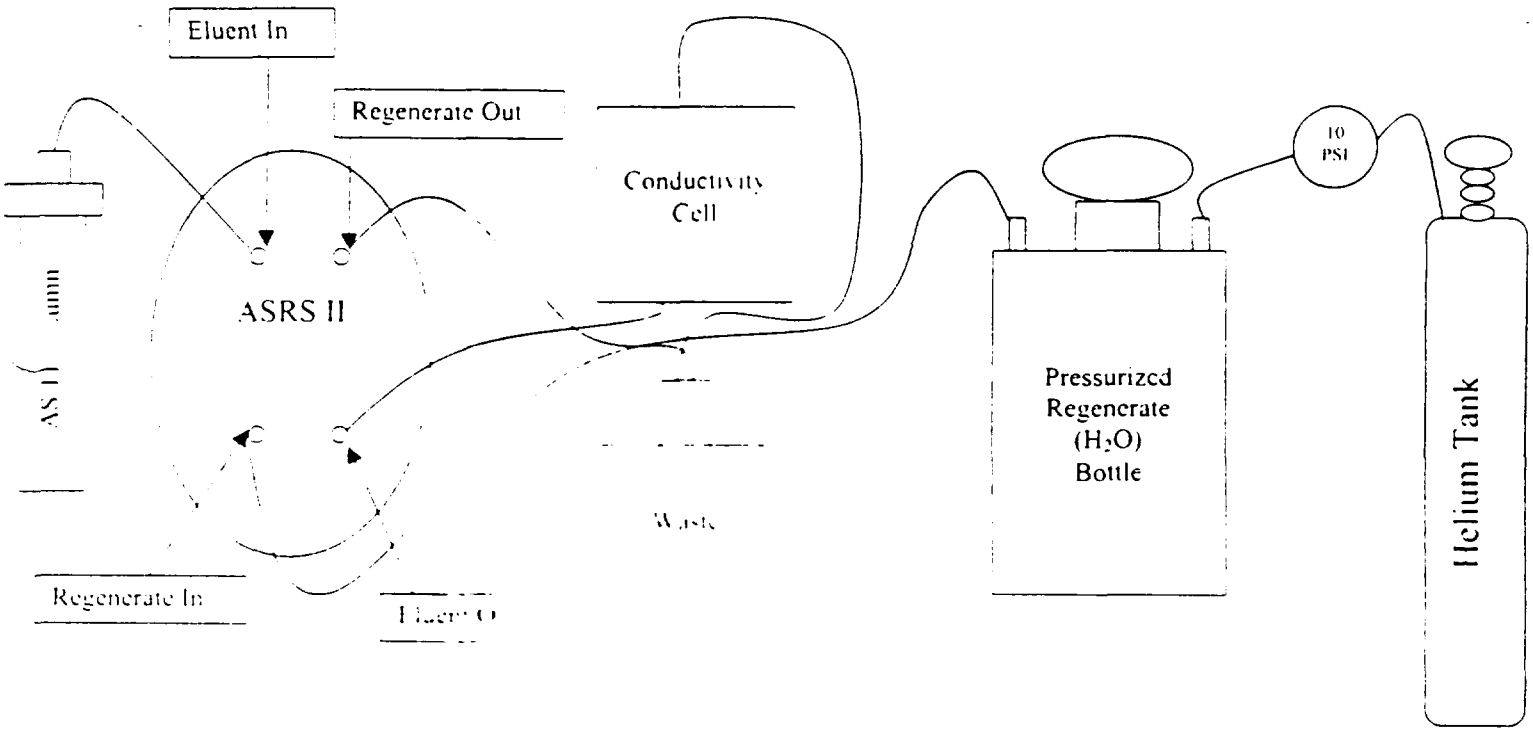
Figure I

External Mode



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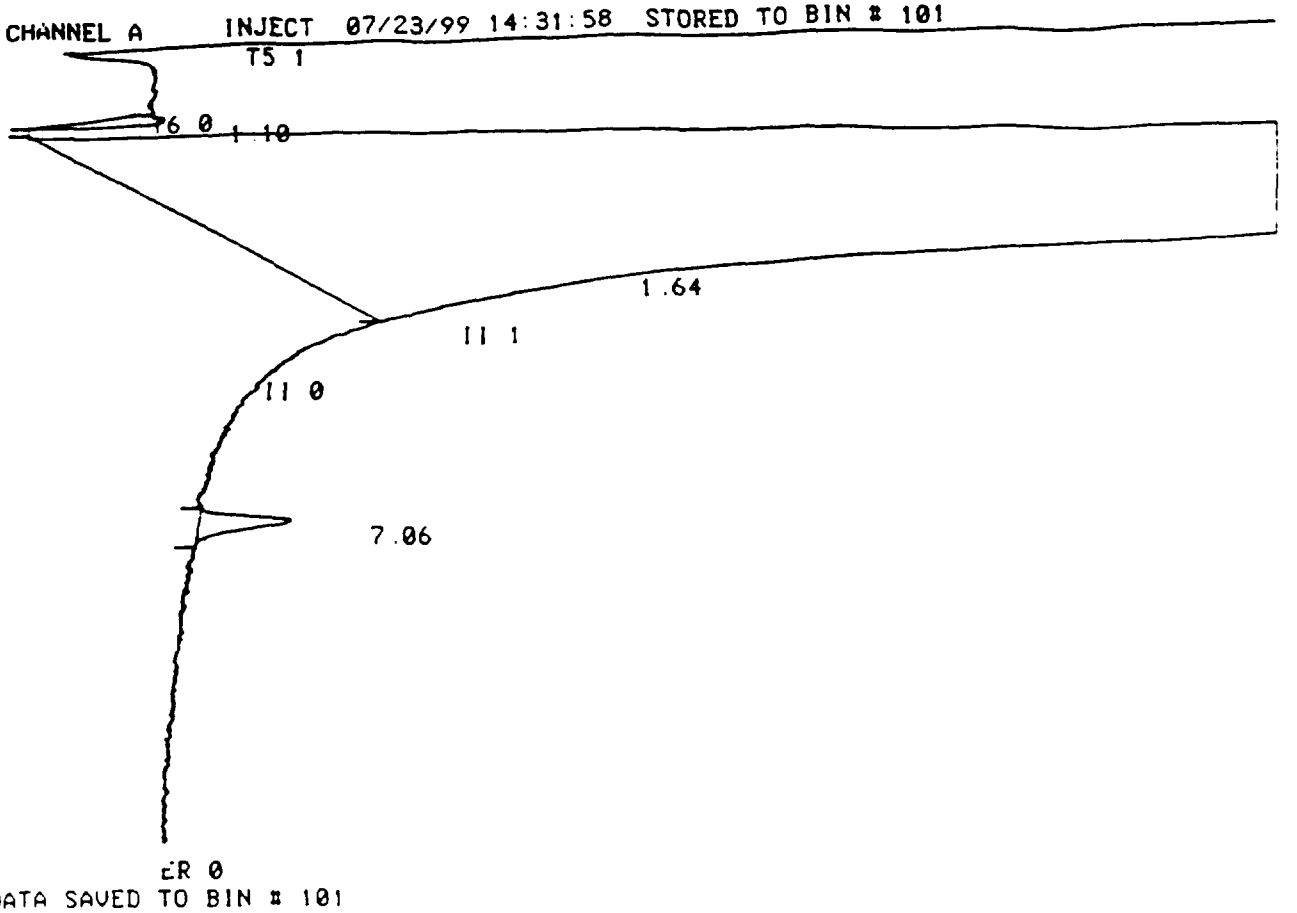
Attachment I



DATA SAVED TO BIN # 101

INPUT OVERRANGE AT RT= 1 36

PERCHLORATE 07/23/99 14 31:58 CH= "A" PS= 1.

FILE 5. METHOD 5 RUN 18 INDEX 1 BIN 101

NAME	PPB	RT	PK HT	BC	RF
1	0	1 1	63249	01	
2	0	1 64	993880	01	
PERCHLORAT	21 007	7 06	81459	01	3877.692
TOTALS	21 007		1138588		

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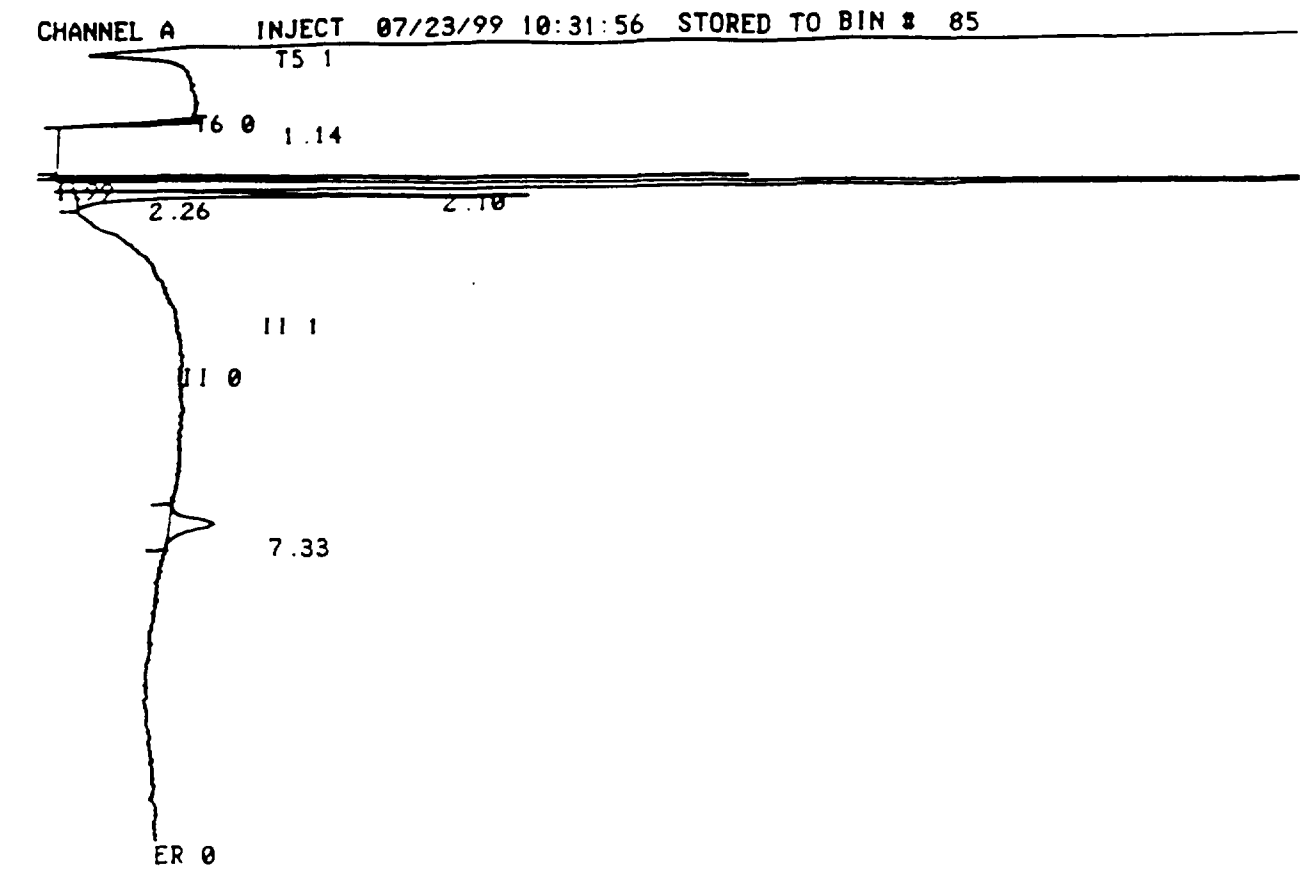
Attachment II



INPUT OVERRANCE AT RT= 2.06 STD-2

PERCHLORATE 07/23/99 10 31:56 CH= "A" PS= 1.

FILE 5. METHOD 5 RUN 2 INDEX 2 CALIB BIN 85

NAME	PPB	RT	PK HT	BC	RF
1	0	1 14	64219	01	
2	0	1 99	602632	01	
3	0	2 1	1135084	02	
4	0	2 26	390091	03	
PERCHLORAT	10	7 33	39418	01	
TOTALS	10		2231443		

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Appendix C

EnChem
Sample Preparation Documents

Preparation of Biota Samples

SPECIFIC TASK PROCEDURE (STP): D-GM-10/N, Revision 0, December 2000

Authorization/Approval:		Revision History:	
John Spurr Quality Assurance Director	12/7/00 Date	Office Location:	Novi, Michigan
Sharon Johnson Program Manager, U.S. Air Force	12-7-00 Date	Effective Date:	R0, December 7, 2000
Robert Lieckfield, Jr., CIH Laboratory Director	12-7-00 Date		

A. D-GM-10/N, Revision 0 was in effect and used for the preparation of biota for the analysis of perchlorate beginning August 1, 2000.

Distribution:

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☐ DC Document Control	☒ IC Information Center	■ OTHERS: (Please List)	Parsons Engineering Science				

1.0 SCOPE AND APPLICATION

This procedure describes the process of proper preparation, processing, bottling and shipping of biota (vegetation, animal, and amphibian) samples.

2.0 SAMPLE PREPARATION: VEGETATION & NON-RODENT ANIMAL

2.1 Samples were removed from the freezer and allowed to thaw.

2.2 All samples are weighed on a top loading balance and the weight is recorded. All samples (except mammals, birds, and aquatic vegetation) are washed with deionized water and allowed to dry.

2.3 Large size samples (>30 grams) were cut into small strips or chunks 1-2 inches in size using a cutting board and knife or clever. Samples were then placed into a commercial grinder. This process is repeated three times to reduce size as much as possible. Additional grinding steps are performed when necessary to obtain proper size reduction. Samples that were small in size or volume were manually ground.

2.4 Ground samples from 2.3 are then placed into a commercial stainless steel blender with liquid nitrogen and blended into a fine powder.

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2.5 Powdered samples are then poured into a glass jar and placed into a freezer until shipment.

2.6 The grinder, blender, knives, cleavers, cutting borar and transfer vessels are then thoroughly cleaned and prepared for the next sample.

3.0 SAMPLE PREPARATION: RODENT

3.1 Due to the possibility of the rodent samples (bats, mice, shrews and rats) containing hunta virus, samples require special handling and treatment to eliminate this threat. All handing and preparation takes place under a Class III biohazard hood at a specially equipped off-site location. The reason for this treatment is to allow dichloromethane to come into contact with the rodent tissues allowing this solvent to dissolve the virus's lipid cell membrane thus deactivating the microbe.

3.2 Samples (rodent bodies) are manually size reduced using knives and cleavers to cut the body into smaller pieces and then placed into a glass container.

3.3 Sufficient dichloromethane is poured into the container to cover all tissue and then thoroughly mixed using a glass stirring rod.

3.4 The dichloromethane was removed via evaporation under a hood.

3.5 Samples were then repackaged and moved to the sample preparation area to complete processing.

3.6 Samples that still are >30 grams in size, are further reduced by cutting into small strips or chunks 1-2 inches in size using a cutting board and a knife or clever. Samples were then placed into a commercial grinder. This process is repeated three times to reduce size as much as possible. Additional grinding steps are performed when necessary to obtain proper size reduction. Samples that were small in size or volume were manually ground using mortar and pestle or other appropriate technique.

3.7 Ground samples from 3.6 are then placed into a commercial stainless steel blender with liquid nitrogen and blended into a fine powder.

3.8 Powdered samples are then poured into a glass jar and placed into a freezer until shipment.

3.9 The grinder, blender, knives, cleavers, cutting board and transfer vessels are then thoroughly cleaned and prepared for the next sample.

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4.0 SAMPLE SHIPMENT

4.1 Samples are removed from the freezer and placed into a cooler with frozen synthetic ice pouches.

4.2 Coolers are sealed and shipped via over night courier.

5.0 REFERENCES

En Chem Inc. In house sample preparation procedures.

STP Author: 12/7/00 Mike Wantland Date	**STP Tester:** 12/7/00 Mike Wantland Date Director, Government Programs

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Appendix D

Parsons ES
Perchlorate Method Development and Validation Report

Perchlorate Method Development and Validation Report

Presented to

Parsons Engineering Sciences, Inc.
Austin, Texas

Parsons Subcontract No. 737451.3000-00, April 2000

Presented by

Clayton Group Services, Inc.
Novi, Michigan

Clayton Work Order No. NV-00046.00

Clayton
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Parsons Engineering Sciences, Inc.
Perchlorate Method Development and Validation Report

INTRODUCTION

Clayton Group Services, Inc., was contracted by Parsons Engineering Science, Inc., to provide method development and validation services associated with the analysis of perchlorate in sand, soils, vegetation, and animal tissue. This work was performed under Parsons Subcontract No. 737451.3000-00, April 2000. The project was performed at Clayton's Novi, Michigan laboratory under Clayton Work Order No. NV-00046.00.

Perchlorate contamination of drinking water supplies is becoming a major concern around the country. The source stems from the use of potassium and ammonium perchlorate as solid fuel rocket propellant, pyrotechnics, and some fertilizers. The subsequent release of perchlorate into the environment has resulted in contamination of surface and ground water supplies. Recent toxicological studies have indicated that consumption of perchlorate contaminated water may reduce thyroid function in humans. The U.S. EPA has recently added perchlorate to the Unregulated Contaminate Monitoring Rule (UCMR).

Parsons was contracted to perform site investigations for perchlorate contamination at six sites across the United States. The objective of the Parsons ES project was to determine the nature and extent of perchlorate concentrations that may be present in soil, water, and various biological media, such as vascular plant tissue and animal tissue. Clayton was contracted to provide services related to development of laboratory analysis methods to assay these matrices for perchlorate.

The primary Clayton participants in the project are listed below.

Participants

- Project Manager: *Mike Wantland*
- Co-Project Manager: *Sharon Johnson*
- Technical Manager: *Scott Caillouette*
- Principal Scientist/Investigator: *Lisa Quiggle, Kristine Kurtz, and Kelli Renaud*
- Project Consultant: *Bob Lieckfield, Jr.*
- Project Accountant: *Dawn Swartzwelter*

The project was organized into six work phases shown below.

- Phase 1 -- Analytical Method Development
- Phase 2 -- Analytical Method Validation Solids (play sand) Matrix
- Phase 3 -- Analytical Method Validation Solids (topsoil) Matrix
- Phase 3A -- Analytical Method Validation Solids Matrix
- Phase 4 -- Analytical Method Validation Vegetation Matrix
- Phase 5 -- Analytical Method Validation Animal Tissue Matrix

Ion chromatography was selected as the method of choice for the analysis of perchlorate in all matrices. The ion chromatographic technique was selected based on the following method sources.

- EPA Method 314.0 – *"Analysis of Perchlorate in Drinking Water Using Ion Chromatography,"* Revision 1.0, November 1999, USEPA Cincinnati.
- Draft Method *"Determination of Perchlorate at Parts-per-Billion Levels in Plants by Ion Chromatography,"* J. Jackson Ellington and John J. Edwards, USEPA, National Exposure Research Laboratory, Ecosystems Division, Athens, GA., April 13, 2000.
- *"Perchlorate by Ion Chromatography,"* Dionex Application Note 121, Dionex Corporation, Inc., undated.
- State of California, Department of Health Services, Sanitation, and Radiation Laboratories (SRL) Branch. *"Determination of Perchlorate By Ion Chromatography,"* Revision 1.0 (CLO4RV-1, WPD). Berkeley, CA, March 1999.
- *"Perchlorate Uptake by Salt Cedar in the Las Vegas Wash Riperian Ecosystem,"* Robert T. Urbansky, Matthew L. Magnuson, Catherine A. Kelty, and Stephanie K. Brown, USEPA National Risk Management Research Laboratory, Water Supply and Water Resources Division, Cincinnati, OH, The Science of the Total Environment, 256 (2000) 227-232, March 25, 2000.
- *"Determination of Ammonium Perchlorate by Ion Chromatography,"* Kenneth E. Basom, Chemical and Physical Analysis Division, Test and Evaluation Department, Indian Head Division, Naval Surface Warfare Center, Indian Head, MD. 1994.
- *"Analysis of Perchlorate in Samples with High Dissolved Solids by Ion Chromatography and AS-16 Column,"* David T. Tsui, Standard Operating Procedure AC-99-41, AL/OET, January 22, 1999.
- *"Analysis of Perchlorate in Soils and Fertilizers by Ion Chromatography,"* David T. Tsui and J. Eric Eldridge, Standard Operating Procedure AC-99-42, AL/OET, March 22, 1999.

SUMMARY OF ACTIVITIES

The ion chromatographic method was established using the operating parameters provided in U.S. EPA Method 314.1. This methodology, used for analysis of perchlorate in drinking water, was operational and tested in the Clayton laboratory prior to the method validation activities undertaken on this project.

Phase I of the work plan, entailing definition of the method performance characteristics, established the ion chromatographic operating conditions for the study. The remaining phases of the project focused on the preparation and clean up of the various matrices, prior to analysis by ion chromatography. The available published literature, listed above, on

analysis of perchlorate all relied on extraction of the matrix in reagent water, initial clean up with Alumina (activated, basic, Brockman 1). Additional clean-up steps were dependent on the specific matrix. Groundwater and surface water could generally be analyzed directly without additional clean-up. Organic based matrices, such as solids, vegetation, and tissue required dilution of the Alumina extract and clean up through a C18 column.

The initial work plan separated matrices into water (aqueous), topsoil, sand, vegetation, and tissue matrices. Representative matrices were evaluated using tap water, commercial grade topsoil, commercial grade sand, carrot greens, and calves liver. The method validation activities were completed on water, topsoil, sand, vegetation, and tissue with acceptable results.

During the course of the validation for sand and topsoil, actual field samples, collected by Parsons' staff, were submitted. Applying the methods validated at Clayton to actual field matrices showed significant differences between the matrices validated to actual field samples. This necessitated changing the original work plan to take into consideration these differences. The most troublesome matrix types were the various solids (sands, topsoils, aggregates, and lake and stream bottoms). All contained significantly more organic material than the commercial grade matrices of the validation protocol. The preparation and clean up was modified to incorporate a C18 clean up on all solid matrices.

Using the experience from the validation of solid matrices, it was decided to abandon the validation of vegetation matrices using common vegetation greens (carrot greens) and tissue (calves liver). The work plan was changed and focused on the validation protocols on actual field matrices. In the case of vegetation, "arrow weed and alfalfa" were selected from the field samples collected by Parsons' staff. For tissue validation, fish and rabbit tissues were selected because they represented the majority of the collected field tissue samples.

Initial work on the validation of vegetation showed the presence of numerous sample artifacts after Alumina and C18 clean up steps. One of these artifacts was a potential chromatographic interferent to the perchlorate ion. The main difficulty was the inconsistent separation of the artifact from perchlorate. This was most likely caused by the subtle differences in the ion exchange characteristics within the ion chromatographic column.

Initial studies to resolve or remove these interfering artifacts consisted of the following steps.

- Applying standard-additions quantitative analysis principles to compensate for the artifact. Standard-additions were attempted on a single sample matrix. While feasible on a research scale, it was decided to abandon this approach as a routine process because of the time-consuming nature of the standard-addition process.

- Varying the extract acidity attempting to protonate any organic acids making them more amenable to trapping by C18 sorbent. Acidifying the sample extract resulted in a lower recovery of perchlorate without any appreciable removal of matrix artifacts.
- Employing an ENVI-ChromP clean-up cartridge. The ENVI-ChromP cartridge did not provide sufficient improvement in the removal of matrix artifacts.
- Varying the eluent flow rate. Changing the eluent flow rate did not provide any improvement in the resolution of sample matrix background.
- Applying a gradient dilution program to the IC eluent concentration. Setting up an elution gradient was unsuccessful because of the IC pumping system. We were unable to attain a reproducible elution gradient with the NaOH eluent. An elution gradient may be feasible on other ion chromatographic systems.
- Varying the ratio of C18 sorbent mass to liquid extract volume from 20:1 (weight to volume) to 2:1 (weight to volume). Increasing the ratio of C18 sorbent mass to sample extract volume produced the most noticeable improvement in removing sample matrix artifacts that impinged upon the chromatographic resolution of perchlorate.
- Changing the eluent concentration from 100 mM to 50 mM NaOH. Incorporating a more dilute NaOH eluent increased the resolution of perchlorate from the sample artifacts while having little effect on the column retention time of the matrix artifacts.

These studies showed that the interferent present in arrow weed and alfalfa could be separated from the perchlorate ion consistently through the use of the 50-mM NaOH eluent at 1.0 mL/minute flowrate combined with the Alumina and C18 clean-up steps. This procedure was also used successfully on the preparation of tissue matrices.

PHASE 1 -- ANALYTICAL METHOD DEVELOPMENT

The first phase of the method validation established operating parameters and defined the operating characteristics of the ion chromatographic method. This work plan consisted of defining the following ion chromatographic operating conditions.

1. Analytical Column
2. Suppressor Column
3. Eluent Solution
4. Eluent Flow Rate
5. Detector Operating Conditions
6. Instrument Settings

These elements are included in each of the standard operating procedures attached to this report. Once the operating parameters were selected, essential elements of method performance – instrument detection limit (IDL), linear range, and ruggedness were evaluated. Precision, accuracy, and method detection limit were also defined in this aspect of the work plan for water matrix.

A summary of the basic operating conditions and method performance is shown below.

Instrument Settings	
Analytical Column	AS16 (Dionex Part No. 55376) with AG16 Guard Column (Dionex Part No. 55377)
Suppressor Column	Dionex ASRS-Ultra II 4 mm (Dionex Part No. 53946)
Suppressor	External wastewater mode @ 5.0 mL/min, 300 mA
Eluent	100 mM NaOH
Eluent Flow Rate	1.0 mL/Min
Detector	Electroconductivity Cell
Detector Background Signal	3 to 4 µS
Method Parameters	
Instrument detection limit	0.5 µg/L
Linear Range	4 µg/L to 1,000 µg/L; Correlation coefficient (r^2) over this range was 0.998
Ruggedness – Retention Time Stability – Response Stability (mean area count) – Response Stability (RPD from Day 0)	 9.99 ± 0.17 minutes; RSD: 1.8% RSD: 15% low level (4 µg/L) 8.8% high level (100 µg/L) Day 2 RPD: 8.3% (overall) Day 5 RPD: 6.2% (overall) Day 7 RPD: 17.9% (overall) Day 14 RPD: 28.3% (overall)
Method Detection Limit (de-ionized water)	1.03 µg/L perchlorate
Precision (de-ionized water)	11.6% RSD_{95}
Accuracy (de-ionized water)	105.5%

The specifics of each work plan element are described below. The resulting data is provided in the Attachments.

Instrument Detection Limit

The instrument detection limit (IDL) is defined as the value of the one-sided 99% confidence interval of a low-level standard. The IDL was determined as follows.

1. Seven (7) injections of a 2 µg/L standard.
2. Calculation of mean and standard deviation of the area response.
3. Calculation of the one-sided 99% confidence interval.

Using the ion chromatographic operating parameters shown above, the instrument detection limit was determined to be 0.5 μg/L. Supporting data is provided in Attachment 1.

Linear Range

The linear range of the ion chromatographic method was determined by analysis of a series of perchlorate standards over the expected range of the analytical method. The linear range was defined over a range of 4 μg/L to 1,000 μg/L. The correlation coefficient (r^2) over this range was 0.998. Under normal operating conditions, we recommend that instrument calibration be performed over the range of 4 μg/L to 100 μg/L, since this approximates the most common range of perchlorate concentrations in samples received. The correlation coefficient (r^2) over this range was also 0.998. Data supporting the linear range study is provided in Attachment 2.

Ruggedness

The ruggedness of the method was defined to determine the stability of the ion chromatographic system over a ten-day period. Defining the ruggedness of a method provides information on the reliability and stability of an instrumental method over a determined time period under normal operating conditions. The normal operating conditions for the ion chromatographic method included addition of eluent, stability of instrument background signal including correction of background signal through manual adjustment of the instrument zero point, and stability of the standard preparations.

The procedure encompassed the preparation and analysis of a 6-point calibration curve representing the typical calibration range of the ion chromatograph – 4 μg/L to 100 μg/L. The calibration curve was analyzed 2, 5, 7, and 14 days after initial calibration. Both the retention time and area response was determined over this range.

The results of the ruggedness study are summarized below.

Parameter	**Performance Measurement**
Retention Time Stability	9.99 ± 0.17 minutes RSD: 1.8%
Response Stability (mean area count)	RSD: 15% low level (4 μg/L) 8.8% high level (100 μg/L)
Response Stability (relative percent difference (RPD) from Day 0)	Day 2 RPD: 8.3% (overall) Day 5 RPD: 6.2% (overall) Day 7 RPD: 17.9% (overall) Day 14 RPD: 28.3% (overall)

The results of the ruggedness study indicate that perchlorate retention time is stable over a 14-day period. The mean area response is relatively stable within a given concentration level. However, the changes in response over a 14-day time period indicates that the effective time period for using a continuing calibration rather than a full calibration may be limited to a 14-day period. Overall, the ion chromatographic analysis of perchlorate is stable over a 5-day period. After 5 days, instrument performance, as measured by response variability, appears to increase from a relative percent difference of approximately 7% to a relative percent difference of 18% when compared to the initial calibration response. While it may be possible to maintain instrument calibration in excess of 7 days, the analyst should monitor the instrument performance carefully as the time period reaches the 7-day period.

Supporting data is provided in Attachment 3.

Overall Method Detection Limit (water extract)

The Method Detection Limit (MDL) of perchlorate in a water matrix was determined by preparation and analysis of eight water samples spiked with perchlorate at 4 µg/L. The prepared samples were analyzed under the operating conditions established during Phase 1 of the work plan. The results show a statistical MDL of 1.03 µg/L perchlorate.

Please note that the chromatographic resolution and response of perchlorate is limited to a concentration of 4 µg/L. We have established 4 µg/L as the reporting limit (RL) of the method. The use of MDL is inappropriate for this method because of a nonlinear response at levels below 4 µg/L. The supporting data for the water MDL study is provided in Attachment 4.

Precision and Accuracy

Precision and accuracy of the ion chromatographic method was determined in a water matrix. Sets of six perchlorate spike samples were prepared in water at 4, 10, and 20-µg/L perchlorate, for a total of 18 samples. All of the prepared samples were analyzed according to method parameters.

The results show that the ion chromatographic method for perchlorate, in water, has a precision of 11.6% RSD at the 95% confidence level and an accuracy of 105.5%.

The supporting data for the precision and accuracy studies is provided in Attachment 5.

PHASE 2, 3, 3A – METHOD VALIDATION PROTOCOLS FOR SAND, TOPSOIL, AND SOLIDS

Summary

The initial work plan focused on sand and topsoil matrices. Representative matrices were evaluated using commercial grade sand and topsoil. The initial method validation activities were completed on sand and topsoil using a three level validation – 1, 2, and 5x the expected reporting limit of 1.3µg/Kg with acceptable results. The fourth level – 10x the reporting limit – was to be incorporated after review of the initial studies.

Using the methods previously published, we incorporated a clean up procedure using Alumina (basic washed) to remove inorganic ions from the sample extract. The literature suggested that Alumina might retain perchlorate in addition to other inorganic ions. Prior to incorporating this Alumina clean up step, studies were performed to determine the recovery of perchlorate through Alumina. A series of six water samples were spiked at 4, 20, and 50-ppb perchlorate (18 total). Five-milliliter aliquots were passed through 0.25 grams of Alumina (activated, basic, Brockman 1).

The results of this study showed the overall recovery of perchlorate from the Alumina was 90.5% with a $\%RSD_{95}$ of 16.7%. The lower recovery at the 4-ppb perchlorate spike level was most likely due to the inherent variability of measuring perchlorate at the detection limit concentration. The supporting data is provided in Attachment 6.

During the course of the validation, actual field samples, collected by Parsons' staff, were submitted. Applying the validated sand and topsoil methods (Phase 2 and 3) to actual field matrices showed significant differences in the matrices validated (sand and topsoil) to actual field samples. This necessitated a change in the analytical preparation method to incorporate an additional dilution prior to the C18 clean-up step and the use of two 1-gram C18 cartridges in series. This more aggressive C18 clean-up step was added and the sand/topsoil methods combined into a single "Solids" methods. The full validation (Phase 3A) was performed using the modified procedure.

Phase 2 and 3 -- Analytical Method Validation on Sand and Topsoil Matrices

A brief summary of the sand and topsoil methods validations is provided for background information only. These methods, although validated as part of the present scope of work, were replaced by the analysis of perchlorate using the "Solids" method.

Sand and topsoil were chosen as a typical matrix based on information provided by the Parsons sampling coordinators. The validation of the sand method used commercially available "play sand" purchased at a local garden center. Topsoil was validated using commercially available "topsoil" also purchased from a local garden center.

Sand

The sand preparation procedure included extraction of a 45-gram aliquot of sand in 15 mL of deionized water for 16 hours. A 5-mL aliquot of the first extract was placed in 0.25 grams of Alumina (activated, basic, Brockman 1). Contact time with the Alumina was one hour. The Alumina/water solution was then centrifuged and the water layer decanted. The decanted water was filtered through a 0.45 micron, 13-mm Acrodisc filter prior to injection on the IC.

The initial validation of the sand method consisted of preparation of a set of six-perchlorate spike samples in sand at 1.3, 3.3, and 6.7 µg/Kg (24 samples total). Control samples included six water spikes at 20 µg/L (5 times the reporting limit) and six sand blanks as required by the method. The prepared samples were analyzed under the operating conditions established during Phase 1 of the work plan.

The results show that the ion chromatographic method for perchlorate, in sand, has a precision of 24.4% RSD at the 95% confidence level and an accuracy of 89.5%.

Topsoil

The preparation procedure for topsoil included extraction of a 10-gram aliquot of topsoil in 20 mL of deionized water for 16 hours. A 5-mL aliquot of the first extract was placed in 0.25 grams of Alumina (basic washed). Contact time with the Alumina was one hour. The Alumina/water solution was then centrifuged and the water layer decanted. The decanted water was then filtered through a 0.45-micron Acrodisc filter prior to injection on the ion chromatograph.

The validation of the topsoil method consisted of preparation of a set of eight-perchlorate spike samples in topsoil at 8, 20, 40, and 100 µg/Kg (32 samples total). Control samples included six water spikes at 20 µg/L (5 times the reporting limit) and six topsoil blanks. Both the Control and topsoil blanks were processed in the same manner as the spike samples. The prepared samples were analyzed under the operating conditions established during Phase 1 of the work plan.

The results show that the ion chromatographic method for perchlorate, in topsoil, has a precision of 14.7% RSD at the 95% confidence level and an accuracy of 78.6%.

Phase 3A -- Analytical Method Validation Solid Matrices

Because of numerous chromatographic interferences present after preparation and analysis of actual field matrices using the sand and topsoil methods, it was determined that the matrices chosen for validation were not indicative of field matrices. Additional modifications were required to remove the interfering sample artifacts. After researching various options, a new preparation procedure was proposed increasing the ratio of sample extract to Alumina and incorporating an additional C18 clean-up procedure. The new

clean-up procedure was defined using a 2:1 ratio of sample extract to Alumina (activated, basic, Brockman 1) and a 1:10 dilution prior to passing through two 1-gram C18 cartridges.

The instrument specifications for determination of perchlorate in solids followed the same IC operating parameters. These are repeated below.

Instrument Settings -- Solids	
Analytical Column	AS16 (Dionex Part No. 55376) with AG16 Guard Column (Dionex Part No. 55377)
Suppressor Column	Dionex ASRS-Ultra II 4 mm (Dionex Part No. 53946)
Suppressor	External wastewater mode @ 5.0 mL/min, 300 mA
Eluent	100 mM NaOH
Eluent Flow Rate	1.0 mL/Min
Detector	Electroconductivity Cell
Detector Background Signal	3 to 4 µS

The preparation procedure for solids included extraction of a 10-gram aliquot of sample in 20 mL of deionized water for 16 hours. A 5-mL aliquot of the first extract was placed in 2.5 grams of Alumina (basic washed). Contact time with the Alumina was one hour. The Alumina/water solution was then centrifuged and the water layer decanted. The decanted water was diluted 1:10 with deionized water. Approximately 5 mLs of diluted sample extract is passed through two 1-gram C18 Solid Phase Extraction (SPE) cartridges (Supelco Superclean ENVI-18 SPE Tubes, Part No. 505706). The eluent was then filtered through a 0.45-micron Acrodisc filter prior to injection on the IC.

A summary of the preparation procedure for solids is provided below.

Sample Weight	10 grams
Extraction Solvent	Deionized water
Extraction Volume	20 mLs
Clean-up Aliquot	5 mLs
Inorganic Clean-up Procedure – Media – Amount – Sample/Alumina Ratio – Contact Time Organic Clean-up Procedure – Media – Amount	 Alumina (activated, basic, Brockman 1, standard grade 150 mesh, 58A, surface area 155 m^2/g) 2.5 grams 2:1 1 Hour C18 Solid Phase Extraction cartridge Two 1-gram cartridges in series
Pre-filter	0.45 micron Acrodisc, 13 mm
Ion Chromatograph Injection Volume	1 mL

The validation of the solids method consisted of preparation of a set of eight perchlorate spike samples in actual field matrix, collected by Parsons' staff, at 80, 200, 400, and 1000 µg/Kg (32 samples total). The four validation spike concentrations were prepared from a representative field matrix, using a separate extraction aliquot for each concentration level. Each spike level grouping was prepared from the same sample extract to ensure consistency within a validation level. Control samples included six water spikes at 20 µg/L (5 times the reporting limit) to monitor recovery independent of matrix and six water blanks to monitor equipment contamination. The prepared samples were analyzed under the operating conditions established during Phase 1 of the work plan.

The results show that the ion chromatographic method for perchlorate, in solids, has a precision of 26.9% RSD at the 95% confidence level and an accuracy of 88.0%.

The method Standard Operating Procedure and supporting data for the precision and accuracy studies is provided in Attachment 7.

PHASE 4 -- ANALYTICAL METHOD VALIDATION VEGETATION MATRIX

Based on our experience with the validation of perchlorate methods in commercial sand and topsoil, the use of surrogate vegetation (carrot greens) was not performed. The development and validation of a perchlorate method in vegetation was based on actual field matrices submitted by Parsons' staff. Both arrowweed and alfalfa were selected because these matrices represented the majority of the actual field samples submitted.

The first step in the preparation of the vegetation samples was ensuring that the cellular structure of the vegetation samples was sufficiently destroyed to release perchlorate. EnChem Laboratories in Madison, Wisconsin performed this procedure. The entire vegetation field sample was manually reduced in size by cutting into strips approximately one inch wide. The sample was then homogenized by passing through a commercial style meat grinder three times. This step is referred to as the "coarse grind." Aliquots of the "coarse grind" sample were placed in a commercial stainless steel blender containing liquid nitrogen. The liquid nitrogen and "coarse grind" sample was blended for one minute. The liquid nitrogen blended sample or "prepared vegetation" was then poured into a glass container and sealed with a plastic screw-cap for storage. The blending of vegetation with liquid nitrogen was repeated until the entire vegetation sample was prepared. The processed vegetation samples were stored at 10 °F until analysis.

Each "prepared vegetation" matrix was initially extracted for perchlorate according to the Solids method previously validated. The extracts were then analyzed by ion chromatography using the same operating conditions.

The results of these preliminary studies showed insufficient resolution of perchlorate from matrix artifacts. The arrowweed presented the most difficulties and was selected as the representative matrix for further development. Method development trials on vegetation

included the following changes to the Solids method in an attempt to resolve the vegetation interferences.

- Applying standard-additions quantitative analysis principles to compensate for the artifact. Standard-additions were attempted on a single sample matrix. While feasible on a research scale, it was decided to abandon this approach as a routine process because of the time-consuming nature of the standard-addition process.
- Varying the extract acidity attempting to protonate any organic acids making them more amenable to trapping by C18 sorbent. Acidifying the sample extract resulted in a lower recovery on perchlorate without any appreciable removal of matrix artifacts.
- Employing an ENVI-ChromP clean-up cartridge. The ENVI-ChromP cartridge did not provide sufficient improvement in the removal of matrix artifacts.
- Varying the eluent flow rate. Changing the eluent flow rate did not provide any improvement in the resolution of sample matrix background.
- Applying a gradient dilution program to the IC eluent concentration. Setting up an elution gradient was unsuccessful because of the IC pumping system. We were unable to attain a reproducible elution gradient with the NaOH eluent. An elution gradient may be feasible on other ion chromatographic systems.
- Adding a boiling step to the sample extraction. The addition of a boiling step did not change the extraction efficiency of perchlorate and had no effect on the extraction of sample matrix artifacts. This procedure was not pursued because it did not improve the chromatographic resolution of perchlorate form the matrix artifacts.
- Varying the ratio of C18 sorbent mass to liquid extract volume from 20:1 (weight to volume) to 2:1 (weight to volume). Increasing the ratio of C18 sorbent mass to sample extract volume produced the most noticeable improvement in removing sample matrix artifacts that impinged upon the chromatographic resolution of perchlorate.
- Changing the eluent concentration from 100 mM to 50 mM NaOH. Incorporating a more dilute NaOH eluent increased the resolution of perchlorate from the sample artifacts while having little effect on the column retention time of the matrix artifacts.

The resulting preparation procedure for vegetation included extraction of a 2-gram aliquot of sample in 20 mL of deionized water for 16 hours. A 5-mL aliquot of the first extract was placed in 2.5 grams of Alumina (activated, basic, Brockman 1). Contact time with the Alumina was one hour. The Alumina/water solution was then centrifuged and the water layer decanted and filtered. The decanted/filtered water was diluted 1:10 with deionized water. A 5-mL volume was placed onto one 5-gram C18 cartridge. The eluent was then filtered through a 0.45-micron Acrodisc filter prior to injection on the IC.

A summary of the preparation procedure for vegetation is provided below.

Sample Weight	2 grams
Extraction Solvent	Deionized water
Extraction Volume	20 mLs
Clean-up Aliquot	5 mLs
Inorganic Clean-up Procedure – Media – Amount – Sample/Alumina Ratio – Contact Time Organic Clean-up Procedure – Media – Amount	 Alumina (activated, basic, Brockman 1, standard grade 150 mesh, 58A, surface area 155 m^2/g) 2.5 grams 2:1 1 Hour C18 Solid Phase Extraction cartridge One 5-gram cartridge
Pre-filter	0.45 micron Acrodisc, 13 mm
Ion Chromatograph Injection Volume	1 mL

The instrument specifications for determination of perchlorate in vegetation followed the same IC operating parameters with the exception of the eluent concentration. This was changed from 100 mM NaOH to 50 mM NaOH. The operating parameters for vegetation are listed below.

Instrument Settings -- Vegetation	
Analytical Column	AS16 (Dionex Part No. 55376) with AG16 Guard Column (Dionex Part No. 55377)
Suppressor Column	Dionex ASRS-Ultra II 4 mm (Dionex Part No. 53946)
Suppressor	External wastewater mode @ 5.0 mL/min, 300 mA
Eluent	50 mM NaOH
Eluent Flow Rate	1.0 mL/Min
Detector	Electroconductivity Cell
Detector Background Signal	3 to 4 µS

The validation of the vegetation method consisted of preparation of a set of eight perchlorate spike samples in actual field matrix, collected by Parsons' staff, at 400, 800, 2000, and 4000 µg/Kg (32 samples total). The four validation spike concentrations were prepared from a representative field matrix, using a separate extraction aliquot for each concentration level. Each spike level grouping was prepared from the same sample extract to ensure consistency within a validation level. Control samples included six water spikes at 20 µg/L (5 times the reporting limit) to monitor recovery independent of matrix and six water blanks to monitor any equipment and contamination. The prepared samples were analyzed under the operating conditions established during Phase 1 of the work plan.

The results show that the ion chromatographic method for perchlorate, in vegetation, has a precision of 22.5% RSD at the 95% confidence level and an accuracy of 95.5%.

The method Standard Operating Procedure and supporting data for the precision and accuracy studies is provided in Attachment 8.

PHASE 5 -- ANALYTICAL METHOD VALIDATION ANIMAL TISSUE MATRIX

The development and validation of a perchlorate method in tissue was based on actual field matrices submitted by Parsons' staff. Both sunfish and cottontail were selected because of the presumed amount of fats and oils present and the resulting potential difficulties these matrices would present in the analysis of perchlorate.

The first step in the preparation of the tissue samples was ensuring sample homogeneity and that the cellular structure of the tissue sample was sufficiently destroyed to release perchlorate. EnChem Laboratories in Madison, Wisconsin performed this procedure. The entire animal specimen was homogenized by passing through a commercial style meat grinder three times. This step is referred to as the "tissue coarse grind." Aliquots of the "tissue coarse grind" sample were placed in a commercial stainless steel blender containing liquid nitrogen. The liquid nitrogen and "tissue coarse grind" sample was blended for one minute. The liquid nitrogen blended sample or "prepared tissue" was then poured into a glass container with a plastic screw cap top for storage. The blending of tissue with liquid nitrogen was repeated until the entire animal specimen sample was prepared. The "prepared tissue" samples were stored at 10 °F until analysis.

Each matrix was initially prepared according to the vegetation method previously validated. The extracts were then analyzed by ion chromatography using the same operating conditions. The results of these preliminary studies showed sufficient resolution of perchlorate from matrix artifacts.

The resulting preparation procedure for tissue included extraction of a 2-gram aliquot of tissue sample in 20 mL of deionized water for 16 hours. A 5-mL aliquot of the first extract was placed in 2.5 grams of Alumina (activated, basic, Brockman 1). Contact time with the Alumina was one hour. The Alumina/water solution was then centrifuged and the water layer decanted and filtered. The decanted/filtered water was diluted 1:10 with deionized water. A 5-mL volume was placed onto one 5-gram C18 cartridge. The eluent was then filtered through a 0.45-micron Acrodisc filter prior to injection on the IC.

A summary of the preparation procedure for tissue is provided below.

Sample Weight	2 grams
Extraction Solvent	Deionized water
Extraction Volume	20 mLs
Clean-up Aliquot	5 mLs
Inorganic Clean-up Procedure – Media – Amount – Sample/Alumina Ratio – Contact Time Organic Clean-up Procedure – Media – Amount	 Alumina (activated, basic, Brockman 1) 2.5 grams 2:1 1 Hour C18 Solid Phase Extraction cartridge One 5 gram cartridge
Pre-filter	0.45 micron Acrodisc
Ion Chromatograph Injection Volume	1 mL

The instrument specifications for determination of perchlorate in tissue followed the same IC operating parameters as used for water and solids -- 100 mM NaOH. The operating parameters for tissue are listed below.

Instrument Settings -- Tissue	
Analytical Column	AS16 (Dionex Part No. 55376) with AG16 Guard Column (Dionex Part No. 55377)
Suppressor Column	Dionex ASRS-Ultra II 4 mm (Dionex Part No. 53946)
Suppressor	External wastewater mode @ 5.0 mL/min, 300 mA
Eluent	100 mM NaOH
Eluent Flow Rate	1.0 mL/Min
Detector	Electroconductivity Cell
Detector Background Signal	3 to 4 µS

The validation of the tissue method consisted of preparation of a set of eight perchlorate spike samples in actual field matrix, collected by Parsons' staff, at 400, 800, 2000, and 10,000 µg/Kg (32 samples total). The four validation spike concentrations were prepared from a representative field matrix, using a separate extraction aliquot for each concentration level. Each spike level grouping was prepared from the same sample extract to ensure consistency within a validation level. Control samples included six water spikes at 20 µg/L (5 times the reporting limit) to monitor recovery independent of matrix and six water blanks to monitor any equipment and reagent contamination. The prepared samples were analyzed under the operating conditions established during Phase 1 of the work plan.

The results show that the ion chromatographic method for perchlorate, in tissue, has a precision of 8.74 % RSD at the 95% confidence level and an accuracy of 86.4 %. The method Standard Operating Procedure and supporting data for the precision and accuracy studies is provided in Attachment 9.

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ATTACHMENT 1

INSTRUMENT DETECTION LIMIT DATA

Instrument Detection Limit (IDL)	
Anticipated IDL	1 ppb perchlorate in water
IDL Concentration	2 ppb
Trial	Concentration
1	2.01
2	2.09
3	1.94
4	2.31
5	1.84
6	2.23
7	2.00
Mean	2.06
SD	0.16
t-statistic @ 95%	3.143
One-sided Confidence Interval	0.515
IDL	0.515

Instrument Detection Limit (IDL) Study

Clayton Group Services, Inc.
22345 Roethel Drive, Novi, MI

IDL

LIMS Matrix: Drinking Water
LIMS Test Codes: WC_DX3C
LIMS Instrument Number: 314.0_W & PERC_CA_W

SAMPLE PREPARATION INFORMATION

Prep. Date:	05/02/2000
Prep. Person:	John Collins
Prep. Method:	EPA 314.0 & SRL, Rev.1.0, 3/3/99
Initial Weight/Volume:	NA
Final Volume:	NA
Matrix Used:	Deionized Water

Students' "t" Values

n	t99
7	3.143
8	2.998
9	2.896
10	2.821

SAMPLE ANALYSIS INFORMATION

Analysis Date:	05/02/2000
Analyst:	John Collins
Analytical Method:	EPA 314.0 & SRL, Rev.1.0, 3/3/99
Instrument/Detector:	Dionex DX-120 IC
Column Supplier/Part No.:	Dionex IonPac AS-16 (4x250mm) #55376
Column Serial No.:	#1308

Analyte	Spike Level	Conc. Units	Blank 1 Result	Blank 2 Result	Spike 1 Result	Spike 2 Result	Spike 3 Result	Spike 4 Result	Spike 5 Result	Spike 6 Result	Spike 7 Result	Spike 8 Result	Spike 9 Result	Spike 10 Result	Mean Blank Result	Mean Spike Result	n	t	Mean Recov. (%)	Std Dev. (s)	Stat. IDL (t*s)	(10*s)	Spike Check	Stat. IDL Check	LIMS PQL Check	Agency Req'd IDL	LIMS PQL (=RL)
Perchlorate (ClO_4)	2.0	ug/l	0	0	2.01	2.09	1.94	2.31	1.84	2.23	2.00				0.0000	2.0600	7	3.143	103.0%	0.1639	0.5152	1.6391	OK	OK	OK	NA	4.0

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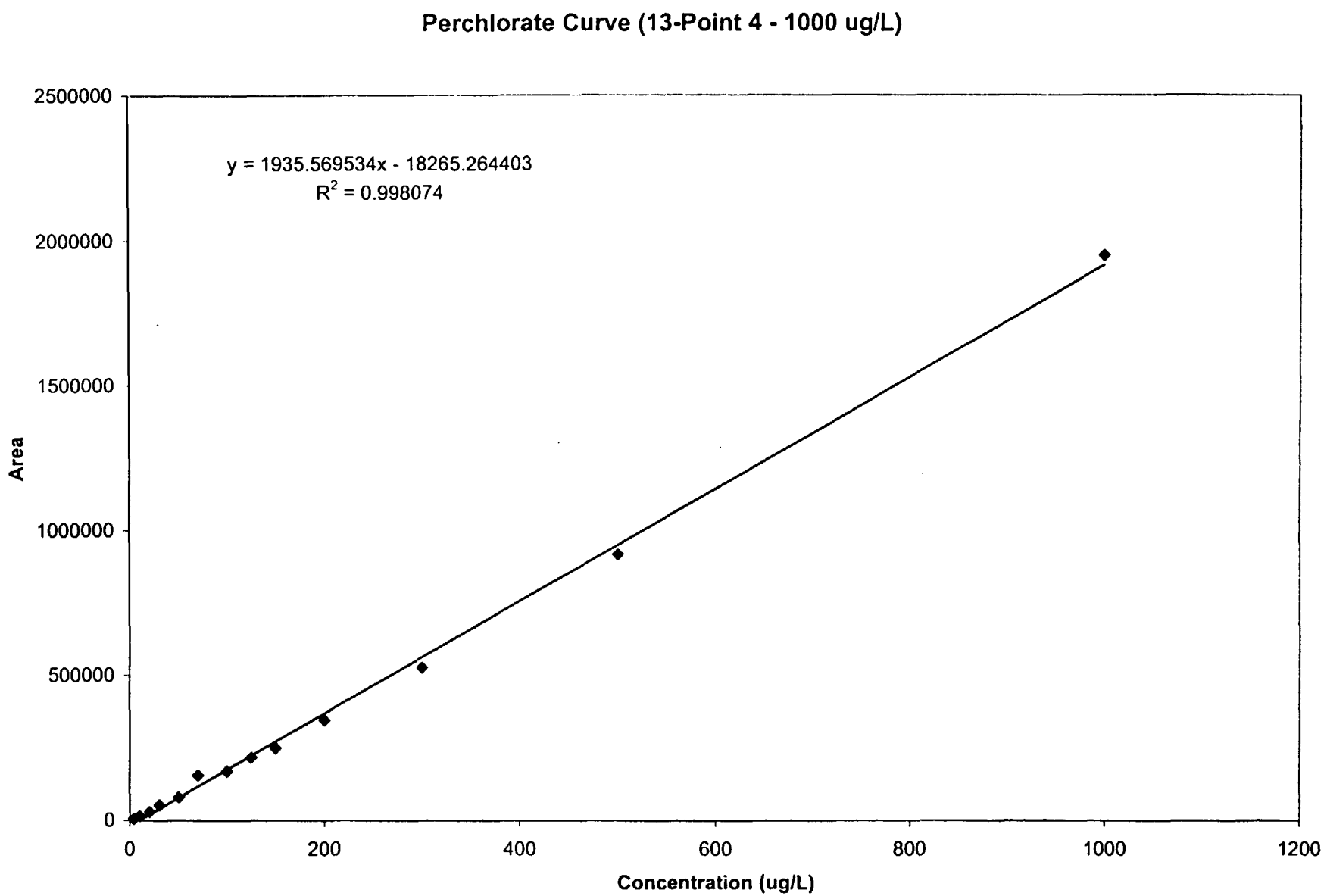
ATTACHMENT 2

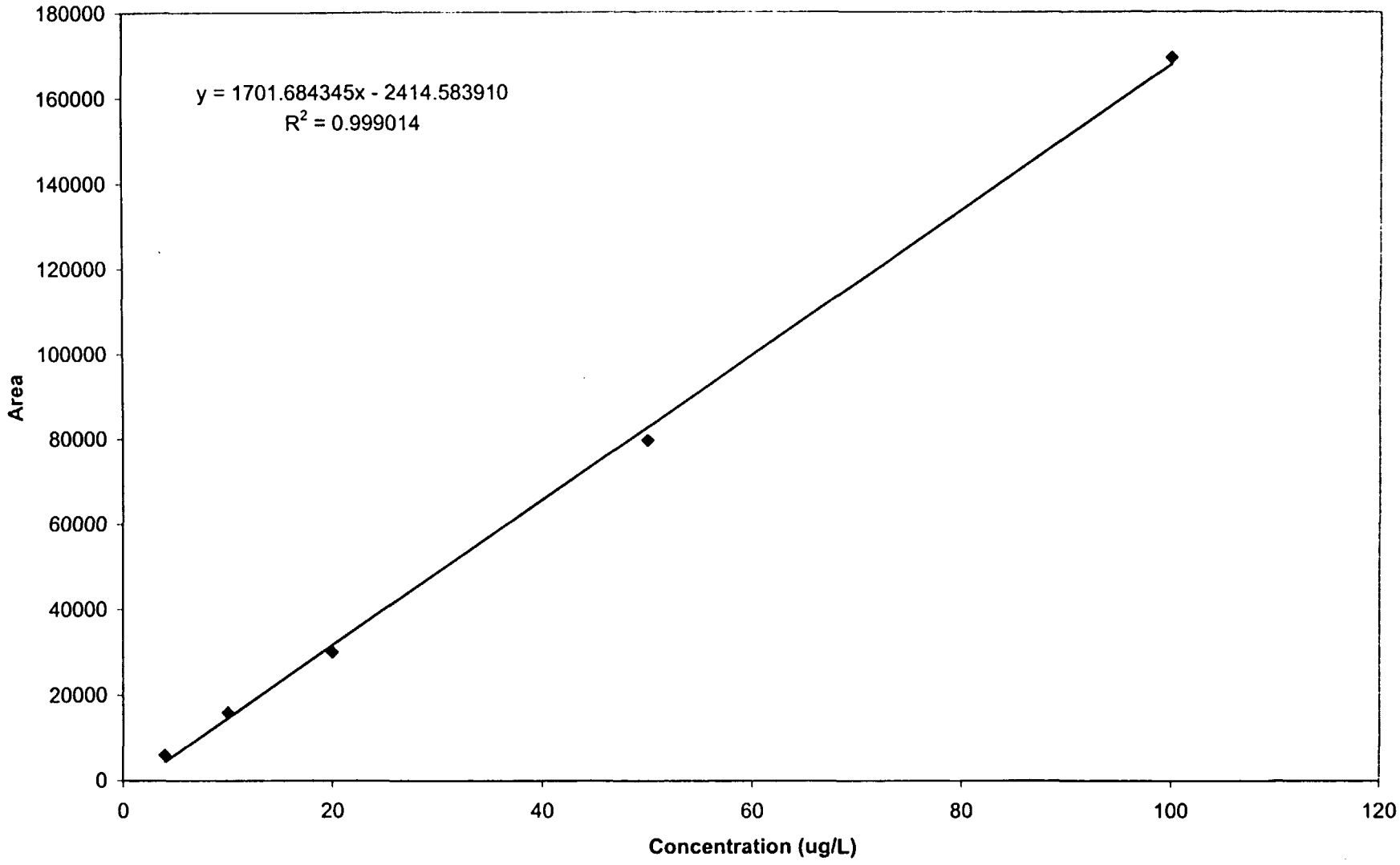
ION CHROMATOGRAPH LINEAR RANGE DATA

Linear Range Data

Expanded 13-point		**SOP Calibration**	
Concentration (ppb)	Area	Concentration (ppb)	Area
4	5990	4	5990
10	15917	10	15917
20	30058	20	30058
30	53329	30	53329
50	79733	50	79733
70	155290	100	169339
100	169339	*Intercept*	-1481
125	216798	*Slope*	1697.5
150	249523	r^2	0.998
200	345639		
300	526923		
500	916969		
1000	1950166		
Intercept	-18265		
Slope	1935.6		
r^2	0.998		


Perchlorate Curve (13-Point 4 - 1000 ug/L)
y = 1935.569534x - 18265.264403
R2 = 0.998074
Area
2500000
2000000
1500000
1000000
500000
0
0
200
400
600
800
1000
1200
Concentration (ug/L)

Perchlorate Curve (SOP Calibration Standard Levels)



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ATTACHMENT 3

ION CHROMATOGRAPH RUGGEDNESS DATA

Perchlorate Study Write-up

Ruggedness Study

Instrument Conditions

Column	AS16 w/guard column
Eluent	100 mM NaOH @ 1.0 mL/min
Suppressor	External wastewater mode @ 5.0 mL/min, 300 mA
Background Signal	3.93 uS

Retention Time Stability (minutes) Over Time

Calibration Level	Day 0	Day 2	Day 5	Day 7	Day 14	Mean	SD	RSD %		
4 ppb	9.88	10.18	9.80	10.32	9.95	10.03	0.22	2.2%		
10 ppb	9.93	9.80	9.82	10.30	9.97	9.96	0.20	2.0%	Overall	9.99
20 ppb	9.93	10.13	9.82	10.30	9.97	10.03	0.19	1.9%	SD	0.18
50 ppb	9.92	9.98	9.82	10.27	9.97	9.99	0.17	1.7%	RSD%	1.8%
70 ppb	9.93	9.90	9.85	10.30	9.98	9.99	0.18	1.8%		
100 ppb	9.92	9.85	9.80	10.28	9.97	9.96	0.19	1.9%		
Mean	9.92	9.97	9.82	10.30	9.97					
SD	0.02	0.15	0.02	0.02	0.01					
RSD%	0.20%	1.54%	0.19%	0.17%	0.10%					
Date	12-Apr-00	14-Apr-00	17-Apr-00	19-Apr-00	26-Apr-00					

Response Stability (area counts) Mean Response over Time

Calibration Level	Day 0	Day 2	Day 5	Day 7	Day 14	Mean	SD	RSD%
4 ppb	5622	5764	5227	6444	7564	6124	917	15.0%
10 ppb	13407	15834	14537	17249	19065	16018	2227	13.9%
20 ppb	30134	33445	30171	35118	37722	33318	3267	9.8%
50 ppb	78369	82527	82320	90262	95788	85853	7030	8.2%
70 ppb	111624	116193	122814	127640	134807	122616	9165	7.5%
100 ppb	154579	168226	165132	182619	193056	172722	15153	8.8%
Date	12-Apr-00	14-Apr-00	17-Apr-00	19-Apr-00	26-Apr-00			

Response Stability (area counts) Relative Percent Difference from Day 0

Calibration Level	Day 0	Day 2	Day 0-2 % Diff.	Day 5	Day 0-5 % Diff.	Day 7	Day 0-7 % Diff.	Day 14	Day 0-14 % Diff.
4 ppb	5622	5764	2.5%	5227	-7.0%	6444	14.6%	7564	34.5%
10 ppb	13407	15834	18.1%	14537	8.4%	17249	28.7%	19065	42.2%
20 ppb	30134	33445	11.0%	30171	0.1%	35118	16.5%	37722	25.2%
50 ppb	78369	82527	5.3%	82320	5.0%	90262	15.2%	95788	22.2%
70 ppb	111624	116193	4.1%	122814	10.0%	127640	14.3%	134807	20.8%
100 ppb	154579	168226	8.8%	165132	6.8%	182619	18.1%	193056	24.9%
Date	12-Apr-00	14-Apr-00		17-Apr-00		19-Apr-00		26-Apr-00	

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ATTACHMENT 4

WATER METHOD DETECTION LIMIT DATA

Water MDL Data			
Concentration	4 ppb	Concentration	10 ppb
Trial	Result	Trial	Result
1	4.54	1	10.07
2	4.27	2	9.83
3	4.54	3	9.99
4	4.49	4	9.99
5	4.74	5	10.05
6	4.60	6	10.11
Mean	4.53	Mean	10.01
SD	0.15	SD	0.10
t-Statistic	3.365	t-statistic	3.365
MDL	0.52	MDL	0.33

Method Detection Limit (MDL) Study

Clayton Group Services, Inc.
22345 Roethel Drive, Novi, MI

MDL

LIMS Matrix: **Matrix or Media**
LIMS Test Codes: **314.0w**
LIMS Instrument Number:

SAMPLE PREPARATION INFORMATION

Prep. Date:	**08/16/2000**
Prep. Person:	**Kellie Fuller**
Prep. Method:	**314.0W**
Initial Weight/Volume:	**NA**
Final Volume:	**NA**
Matrix Used:	**Deionized Water?**

Students' "t" Values

n	t99
7	3.143
8	2.998
9	2.896
10	2.821

SAMPLE ANALYSIS INFORMATION

Analysis Date:	**08/17/2000**
Analyst:	**John Collins**
Analytical Method:	**314.0W**
Instrument/Detector:	**DX600**
Column Supplier/Part No.:	**AS16**
Column Serial No.:	

Analyte	Spike Level	Conc. Units	Blank 1 Result	Blank 2 Result	Spike 1 Result	Spike 2 Result	Spike 3 Result	Spike 4 Result	Spike 5 Result	Spike 6 Result	Spike 7 Result	Spike 8 Result	Spike 9 Result	Spike 10 Result	Mean Blank Result	Mean Spike Result	n	t	Mean Recov. (%)	Std Dev (s)	Stat. MDL (t*s)	Stat. PQL (10*s)	Spike Check	Stat. MDL Check	LIMS PQL Check	Agency Req'd MDL	LIMS PQL (=RL)
Perchlorate	4	ug/L	0	0	3.97	4.55	4.69	4.24	4.08	4.39	3.71	4.62			0.0000	4.2813	8	2.998	107.0%	0.3446	1.0331	3.4461	OK	HIGHER	OK	≥ 0.7	NA

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ATTACHMENT 5

WATER PRECISION AND ACCURACY DATA

Matrix	Water
Clean-up	None
Sample Size	Not Applicable
Final Volume (mL)	Not Applicable

Analysis Date	Study Series	Spike Concentration (ug/L)	Measured Concentration (ug/L)	Recovery (%)	Mean	SD	RSD_{95} (%)
16-May-00	Method Blank	n/a	<4	n/a			
16-May-00	Method Blank	n/a	<4	n/a			
16-May-00	Method Blank	n/a	<4	n/a			
16-May-00	Method Blank	n/a	<4	n/a			
16-May-00	Method Blank	n/a	<4	n/a	n/a	n/a	n/a
16-May-00	1x LOD	4	4.54	113.5%			
16-May-00	1x LOD	4	4.27	106.8%			
16-May-00	1x LOD	4	4.54	113.5%			
16-May-00	1x LOD	4	4.49	112.3%			
16-May-00	1x LOD	4	4.74	118.5%			
16-May-00	1x LOD	4	4.6	115.0%	113.3%	3.8%	6.7%
16-May-00	2.5x LOD	10	10.07	100.7%			
16-May-00	2.5x LOD	10	9.83	98.3%			
16-May-00	2.5x LOD	10	9.99	99.9%			
16-May-00	2.5x LOD	10	9.99	99.9%			
16-May-00	2.5x LOD	10	10.05	100.5%			
16-May-00	2.5x LOD	10	10.11	101.1%	100.1%	1.0%	1.9%
16-May-00	5x LOD	20	20.78	103.9%			
16-May-00	5x LOD	20	20.82	104.1%			
16-May-00	5x LOD	20	20.81	104.1%			
16-May-00	5x LOD	20	20.54	102.7%			
16-May-00	5x LOD	20	20.34	101.7%			
16-May-00	5x LOD	20	20.39	102.0%	103.1%	1.1%	2.1%
	10x LOD			#DIV/0!			
	10x LOD			#DIV/0!			
	10x LOD			#DIV/0!			
	10x LOD			#DIV/0!			
	10x LOD			#DIV/0!			
	10x LOD			#DIV/0!			
	10x LOD			#DIV/0!			
	10x LOD			#DIV/0!			
			Overall Performance		105.5%	6.2%	11.6%

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ATTACHMENT 6

ALUMINA CLEAN UP RECOVERY DATA

Matrix	Water Alumina Control
Clean-up	Alumina
Final Volume (mL)	Not Applicable
Preparation Method	Clayton SOP

Analysis Date	Study Series	Spike Concentration (ug/L)	Measured Concentration (ug/L)	Recovery (%)	Mean	SD	RSD_{95} (%)
	Matrix Blank	0	<4	n/a			
	Matrix Blank	0	<4	n/a			
	Matrix Blank	0	<4	n/a			
	Matrix Blank	0	<4	n/a			
	Matrix Blank	0	<4	n/a			
	Matrix Blank	0	<4	n/a	n/a	n/a	n/a
	Control Water Mid-level Spike	20	19.24	96.2%			
	Control Water Mid-level Spike	20	18.73	93.7%			
	Control Water Mid-level Spike	20	18.66	93.3%			
	Control Water Mid-level Spike	20	19.20	96.0%			
	Control Water Mid-level Spike	20	19.27	96.4%			
	Control Water Mid-level Spike	20	18.19	91.0%	94.4%	2.2%	4.5%
	Control Water Low-level Spike	4	3.08	77.0%			
	Control Water Low-level Spike	4	3.28	82.0%			
	Control Water Low-level Spike	4	3.48	87.0%			
	Control Water Low-level Spike	4	2.92	73.0%			
	Control Water Low-level Spike	4	3.32	83.0%			
	Control Water Low-level Spike	4	3.31	82.8%	80.8%	5.0%	12.1%
	Control Water High-level Spike	50	49.26	98.5%			
	Control Water High-level Spike	50	47.30	94.6%			
	Control Water High-level Spike	50	48.65	97.3%			
	Control Water High-level Spike	50	48.21	96.4%			
	Control Water High-level Spike	50	47.92	95.8%			
	Control Water High-level Spike	50	47.15	94.3%	96.2%	1.6%	3.3%
					90.5%	7.7%	16.7%

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ATTACHMENT 7

PERCHLORATE IN SOLIDS METHOD PRECISION AND ACCURACY DATA

Matrix	Solids
Clean-up	2 1 Extract/Alumina, 1 10 dilution, Two C18 cartridges
Sample Weight (grams)	10 grams
Final Volume (mL)	20 ml

Analysis Date	Study Series	Spike Concentration (ug/L)	Spike Concentration (ug/Kg)	Measured Concentration (ug/L)	Measured Concentration (ug/Kg)	Recovery (%)	Mean	SD	RSD_{95} (%)
	Matrix Blank	n/a		<4		n/a			
	Matrix Blank	n/a		<4		n/a			
	Matrix Blank	n/a		<4		n/a			
	Matrix Blank	n/a		<4		n/a			
	Matrix Blank	n/a		<4		n/a			
	Matrix Blank	n/a		<4		n/a	n/a	n/a	n/a
23-Jul-00	Control Water Mid-level Spike	20		19 24		96 2%			
23-Jul-00	Control Water Mid-level Spike	20		18 73		93 7%			
23-Jul-00	Control Water Mid-level Spike	20		18 66		93 3%			
23-Jul-00	Control Water Mid-level Spike	20		19 20		96 0%			
23-Jul-00	Control Water Mid-level Spike	20		19 27		96 4%			
23-Jul-00	Control Water Mid-level Spike	20		18 19		91 0%	94 4%	2 2%	4 5%
24-Aug-00	1x LOD	4	80	4 43	88.6	110 8%			
24-Aug-00	1x LOD	4	80	4 39	87.8	109 8%			
24-Aug-00	1x LOD	4	80	4 31	86.2	107 8%			
24-Aug-00	1x LOD	4	80	4 37	87.4	109 3%			
24-Aug-00	1x LOD	4	80	4 43	88.6	110 8%			
24-Aug-00	1x LOD	4	80	4.73	94.5	118 1%			
24-Aug-00	1x LOD	4	80	3 61	72.2	90 3%			
24-Aug-00	1x LOD	4	80	3 40	68	85 0%	105 2%	11 4%	21 2%
24-Aug-00	2x LOD	10	200	6 96	139.2	69 6%			
24-Aug-00	2x LOD	10	200	8 52	170.4	85 2%			
24-Aug-00	2x LOD	10	200	8 42	168.4	84 2%			
24-Aug-00	2x LOD	10	200	8 23	164.6	82 3%			
24-Aug-00	2x LOD	10	200	7 97	159.4	79 7%			
24-Aug-00	2x LOD	10	200	8 35	167	83 5%			
24-Aug-00	2x LOD	10	200	8 81	176.2	88 1%			
24-Aug-00	2x LOD	10	200		Void	Void	81 8%	6 0%	14 3%
24-Aug-00	5x LOD	20	400	16 43	328.6	82 2%			
24-Aug-00	5x LOD	20	400	16 37	327.4	81 9%			
24-Aug-00	5x LOD	20	400	16 36	327.2	81 8%			
24-Aug-00	5x LOD	20	400	17.33	346.6	86 7%			
24-Aug-00	5x LOD	20	400	17 52	350.4	87 6%			
24-Aug-00	5x LOD	20	400	16 61	332.2	83 1%			
24-Aug-00	5x LOD	20	400	15 98	319.6	79 9%			
24-Aug-00	5x LOD	20	400	16 14	322.8	80 7%	83 0%	2 7%	6 5%
24-Aug-00	10x LOD	50	1000	40 24	804.8	80 5%			
24-Aug-00	10x LOD	50	1000	41 28	825.6	82 6%			
24-Aug-00	10x LOD	50	1000	41.06	821.2	82 1%			
24-Aug-00	10x LOD	50	1000	40 33	806.6	80 7%			
24-Aug-00	10x LOD	50	1000	39 58	791.6	79 2%			
24-Aug-00	10x LOD	50	1000	39 86	797.2	79 7%			
24-Aug-00	10x LOD	50	1000	41 10	822	82 2%			
24-Aug-00	10x LOD	50	1000	41 45	829	82 9%	81 2%	1 4%	3 4%
					Overall Performance		88 0%	12 1%	26 9%

Determination of Perchlorate by Ion Chromatography in a Solid Matrix

STANDARD OPERATING PROCEDURE (SOP): M-CC-69/N, Revision 2, October 2000

Authorization/Approval:		Applicability and Revision History:	
Michelle Vermillion for 10/16/00		Office Location:	Novi, Michigan
John Spurr, Quality Assurance Director	Date	Effective Date:	R0, August 1, 2000 R1, October 3, 2000 R2, October 16, 2000
10/16/00			
Scott Cailloutte, Supervisor	Date	Reference Methods:	EPA 314.0 modified
10/16/00			
Robert Lieckfield Jr., CIH, Laboratory Director	Date	Media/Matrices:	Soils & other Solids

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☐ DC Document Control	☒ IC Information Center	☒ OTHERS: (Please List) Parsons Engineering Science					

1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of the perchlorate anion in solids by ion chromatography after aqueous extraction and soild-phase cleanup procedures.

1.2. When this method is used to analyze unfamiliar samples, perchlorate identification should be supported by the use of matrix spikes. The matrix spike procedure is described in Section 6.5.

1.3 The linear calibration range for perchlorate is approximately 4 to 500 µg/L. The linear calibration may be extended to 1500 µg/L, but accuracy below 50 µg/L may be compromised. Quantitation below 50 µg/L is not recommended with the extended calibration. Sample concentrations higher than the upper calibration limit should be diluted with reagent water to the mid-range of the linear calibration and reanalyzed.

1.4 This method is recommended for use by analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

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2.0 SUMMARY OF METHOD

2.1 The analysis is performed using an ion chromatograph. The main components of the ion chromatograph used in this method include an autosampler, pump, guard column, analytical column, ion suppressor device, conductivity detector, and chromatography data system.

2.2 An aliquot of sample extract is injected into the ion chromatograph, where the perchlorate anion is separated from other sample components, then quantitated. A large volume sample injection, 1 ml, is employed to perform perchlorate measurement at the low µg/L level.

2.3 Perchlorate is a large polarizable anion with hydrophobic properties. To avoid or minimize hydrophobic interaction of perchlorate with the guard and separator columns, care must be exercised in the selection of the ion-exchange column and the eluent composition. Extensive hydrophobic interaction of the perchlorate anion with the ion-exchange column leads to a long retention time and severe peak tailing. The columns and eluents recommended in this method were selected to minimize this hydrophobic interaction.

3.0 INTERFERENCES

3.1 The wide water dip or negative peak that may be observed in the chromatogram is due to the large volume sample injection. The negative response is the result of an imbalance of ions in the sample relative to the eluent and does not interfere with the measurement of perchlorate, since perchlorate elutes well beyond the dip.

3.2 Interferences can be caused by substances with retention times that are similar to and overlap with the anion of interest.

3.3 High concentrations of anions can interfere with the peak resolution of an adjacent anion and/or cause baseline disturbances. When such an interference occurs, it may help to dilute the sample before analysis, although it should be recognized that the sample detection limit increases proportionately with the sample dilution factor.

3.4 Surfactants and detergents may cause fouling of the ion exchange column and membrane suppressor, resulting in an unstable baseline and poor detector response. Other dissolved organic compounds may similarly interfere.

4.0 APPARATUS AND MATERIALS

4.1 Ion chromatograph – Dionex DX120 or equivalent, complete with all required accessories including syringes, analytical columns, compressed gases, and detectors.

4.1.1 Anion guard column: A protector of the separator column. Dionex AG 16, 4 mm.

4.1.2 Anion separator column: This column produces the separation of the anions. Dionex AS 16, 4 mm.

4.1.3 Anion suppressor device: Dionex ASRS II.

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4.1.4 Automated Sampler: Dionex ASM 3, or equivalent.

4.1.5 PeakNet IC Software: Dionex.

4.1.6 Conductivity detector: 1.25 µL internal volume.

4.1.7 Sample loop: Approximately 1000 µL (12" x 0.02" I.D. tubing), or larger, as required.

4.2 Polyethylene autosampler vials, 5 mL.

4.3 Filter caps, 5 mL.

4.4 Volumetric flasks, 50, 100, 1000, and 2000 mL.

4.5 Plastic vials, 10 mL.

4.6 Falcon tubes, 15 ml and 50 ml.

4.7 Laboratory balance, capable of weighing to 0.1 mg.

4.8 Shaker table.

4.9 Vortex

4.10 Disposable syringes, 10 ml.

4.11 Centrifuge(s), capable of accommodating 50 mL Falcon tubes and 16 x 125-mm disposable glass test tubes.

4.12 Kimwipes, delicate task wipes.

4.13 Disposable test tubes, 16 x 125 mm with caps.

4.14 Gelman ion chromatography (IC) Acrodisc or equivalent, 13 mm or 25 mm syringe filter with 0.45 um Supor (PES) membrane, catalog #4485T or #4585T.

4.15 C18 Solid Phase Extraction (SPE) cartridges or equivalent, Supelco Superclean ENVI-18 SPE Tubes, 6 ml (1 g), catalog #505706.

4.16 Solid phase extraction vacuum manifold.

4.17 Alumina, (Aluminum Oxide), Aldrich catalog #19,944-3, activated, basic, Brockmann l, standard grade, 150 mesh, 58 A, surface area 155 m^2/g.

Note: The alumina is cleaned by mixing one volume of alumina with 10 volumes of reagent water. The fines (cloudy water above the solid) are decanted and replaced with fresh reagent water. The mixing and decanting process is repeated until the water remains clear. The cleaned alumina is then allowed to air dry and stored in a screw cap glass bottle until used.

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5.0 REAGENTS

5.1 Reagent water, obtained from the Milli-Q Water System, through the filtered spigot.

5.2 Sodium perchlorate (solid), Aldrich catalog #41,024-1.

5.3 Potassium perchlorate, secondary source standard (1000ppm solution), SPEX catalog #AS-CLO49-2Y.

5.4 50% w/w sodium hydroxide, Fisher Chemical catalog #55254-500, or equivalent.

Note: For 100 mM sodium hydroxide, weigh 8.0 grams of 50% NaOH into a plastic weight boat and transfer into approximately 950 mL of reagent water and bring to 1000 mL volume.

5.5 Methanol, HPLC grade.

6.0 PREPARATION OF STANDARDS

Note: In all cases where weighing is involved, the exact weight (all decimal places for the balance used) is recorded.

6.1 Primary Stock and Working Standards: All stock standards can be used for up to12 months. All other standards can be used for up to 6 months.

6.1.1 1000 ppm Primary Perchlorate Stock Standard: Dissolve 1.231 g of sodium perchlorate in 1000 mls of reagent water.

6.1.2 10 ppm Perchlorate Working Standard: Dilute 1.0 ml of 1000 ppm stock perchlorate to 100 ml with reagent water.

6.1.3 1.0 ppm Perchlorate Working Standard: Dilute 10.0 ml of 10 ppm perchlorate to 100 ml with reagent water.

6.1.4 0.1 ppm Perchlorate Working Standard: Dilute 1.0 ml of 10 ppm perchlorate to 100 ml with reagent water.

6.2 Secondary Source Standards

6.2.1 10 ppm Secondary Source Perchlorate Standard: Dilute 1.0 ml of the 1000 ppm secondary source perchlorate standard to 100 ml with reagent water.6.2.2 1.0 ppm Secondary Source Perchlorate Standard: Dilute 10 ml of 10 ppm secondary source perchlorate standard to 100 ml with reagent water.

6.3 Mixed Common Anion Stock Solution (containing the anions chloride, sulfate and carbonate each at 25 mg/ml), used to prepare simulated common anion samples in the determination of the MCT: Dissolve the following salts in reagent water to a final volume of 25.0 ml.

(a) 1.0 g sodium chloride (NaCl, CAS [7647-14-5]) = 0.61 g Cl^-
(b) 93 g sodium sulfate (Na_2SO_4, CAS [7757-82-6]) = 0.63 g SO_4^{-2}
(c) 1.1 g sodium carbonate (Na_2CO_3, CAS [497-19-8]) = 0.62 g CO_3^{-2}

6.4 Instrument Performance Check (IPC) Standard: Add 2.5 mls of 1.0 ppm perchlorate working standard and 3.2 mls of mixed common anion stock to a 100 ml volumetric flask and dilute to volume with reagent water.

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6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD): Prepare by spiking 400 µL of 10 ppm perchlorate working standard or secondary source standard to 10 g of sample. (Note: A smaller volume of sample may have to be used for when there is limited sample volume)

6.6 Laboratory Control Sample (LCS): Prepare by spiking 400 µL of 10 ppm perchlorate working standard or secondary source standard into 20 ml of DI water.

6.7 Instrument Calibration Verification Standard (ICV): Prepare by adding 2.0 ml of 1.0 ppm secondary source perchlorate standard to 50 ml of reagent water in a 100 ml volumetric flask and dilute to volume with reagent water.

7.0 SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

7.1 Samples may be collected in HDPE plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

7.2 Until further preservation and holding time studies are available, there is no maximum holding time. Samples will be stored at 4°C only to minimize biological activity.

8.0 PROCEDURE FOR SAMPLE PREPARATION/EXTRACTION

8.1 Weigh 10.0 g (±0.05 g) of solid into 50 mL Falcon tube.

8.2 Pipet 20.0 ml of reagent water into tube

8.3 Place on mechanical shaker for approximately 16 hours.

8.4 Centrifuge for 30 minutes.

8.5 Decant top layer (sample extract) through a Kimwipe into a clean 50 ml Falcon tube.

8.6 Add a 5.0 ml aliquot of this extract to 2.5 g (±0.1g) of Alumina in a disposable 12.5 ml test tube and cap.

8.7 Vortex test tube for approximately 20 seconds.

8.8 Place test tube on a mechanical shaker for 1 hour.

8.9 Centrifuge the test tube for 15 minutes.

8.10 Pour the top layer (sample extract) into a clean 15 ml Falcon tube.

8.11 Add 1.0 ml of sample extract to 9.0 ml of reagent water in a clean 15 ml Falcon tube. This results in a 1:10 dilution of the sample extract.

8.12 Solid Phase Extraction (SPE) C18 Cleanup: Pass the diluted sample extract (1:10) through two ENVI-18 SPE cartridges to remove potential organic matrix interference compounds.

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8.12.1 Condition each C18 cartridge per the manufacturer's instructions by rinsing with methanol followed by reagent water. The flow rate should not exceed 5.0 ml/minute.

8.12.1.1 Rinse cartridges with 2 ml of methanol.

8.12.1.2 Rinse cartridges with 2 ml of reagent water.

8.12.2 After conditioning with methanol and reagent water, rinse cartridge with 0.5 ml of diluted sample extract.

8.12.3 Pass approximately 5.8 ml of diluted sample extract through the conditioned and rinsed C18 cartridge into a clean disposable test tube.

8.12.4 Remove the test tube containing approximately 5.8 ml of sample extract (first C18 extract) and set aside.

8.12.5 Repeat steps in 8.12.1 for a second C18 cartridge.

8.12.6 After conditioning the second C18 cartridge, rinse the cartridge with 0.5 ml of the first C18 extract (extract in test tube from step 8.12.4).

8.12.7 To obtain the final extract, pass 5.0 ml of the first C18 extract (extract in test tube from step 8.12.4) through the second conditioned and rinsed C18 cartridge into a clean disposable test tube.

8.13 Filter approximately 2 ml of final extract through a Gelman IC Acrodisc 0.45 um filter into an autosampler vial for analysis and the remaining extract to a 15 ml Falcon tube to be stored refrigerated for any additional analysis.

9.0 HEALTH AND SAFETY

9.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

9.2 The following chemicals have the potential to be highly toxic or hazardous. The MSDS for each chemical should be consulted.
(a) Sodium hydroxide
(b) Sodium perchlorate
(c) Potassium perchlorate

10.0 QUALITY CONTROL

10.1 All IC chromatograms are kept for 5 years.

10.2 Perform an initial calibration with standards at 4, 10, 20, 50, and 100 ppb. The calibration curve must have linear regression coefficient of determination (r^2) of at least 0.995. The 4 ppb standard, when calculated back to the calibration curve, must be within ±25% of the actual value. The remainder of the standards must be within 15% of the actual value.

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10.3 The calibration curve must be verified with the low continuing calibration verification standard (LCCV, 4ug/l calibration standard) each working day, or whenever the eluent is changed. If the response for perchlorate varies from the expected values by more that ±25%, the test must be repeated, using fresh calibration standard. If the results are still more than ±25%, a new calibration curve must be prepared for the analyte. The shift in retention time must be within ±5% of expected value. If not, a new calibration curve must be prepared.

10.4 A mid-level continuing check verification (MCCV,20 ug/L), alternating with a high level (HCCV,100 ug/L) must be run after every 10 client samples. The last analysis of the run sequence is always a check standard. CCV must be within ±15% of expected value.

10.5 The instrument performance check (IPC) should not vary in retention time by more than 5%. The control limits for the perchlorate recovery are ±20%.

10.6 One method blank (MB) is run with every batch of ≤20 samples. The method blank is an aliquot of filtered DI water that is prepared with samples and carried through the entire process.

10.7 One laboratory control sample (LCS) is run with every batch of ≤20 samples. For the LCS control limits, refer to current control charts.

10.8 One matrix spike (MS) and one matrix spike duplicate (MSD) are run with every batch of ≤20 samples. For the MS/MSD control limits, refer to current control charts.

10.9 A sample duplicate (SD) must be run for every 20 samples. RPD must be within 15%.

$$RPD = 100 \times \frac{|S - SD|}{(S + SD)/2}$$

11.0 PROCEDURE FOR ANALYSIS

11.1 Start up the IC according to the directions in the operator's manual.

11.2 Suppressor should be set at 300 milliamps.

11.3 Eluent flow rate = 1 ml per minute.

11.4 Injection volume is 1 mL.

Note: The IC injection loop is usually 25 µL. Hence, the injection loop will need to be changed back if any other analysis is to be run.

11.5 The IC must be run in external water mode using reagent water as the regenerant. The regenerate flow rate should be approximately 3 to 5 mL per minute. All reservoirs should be filled at the beginning.

11.6 Allow the system to equilibrate. The conductivity should settle to approximately 2 to 5 µs.

11.7 Prepare a calibration curve using the standards prepared as follows in 100 ml of reagent water as follows:

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mL of working STD	µg/L	STD
4 mL of 0.1 ppm	4	1
1.0 mL of 1.0 ppm	10	2
2.0 mL of 1.0 ppm	20	3
0.5 mL of 10 ppm	50	4
1.0 mL of 10 ppm	100	5

11.8 Set up a run sheet. Runs 1-5 are the calibration standards, followed by the LCCV, method blank, ICV, IPC, DI water and then samples. Number autosampler vials with the corresponding sample number on the run sheet.

11.9 Remove at least 2 mL of standards, check standards, QC samples, and the client samples and place in the autosampler vials.

11.10 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.0 CORRECTIVE ACTIONS

12.1 If the samples are more concentrated than the highest standard, dilute with filtered DI water, preferably to the middle of the calibration curve or extend the calibration range, bracketing the concentration of the sample.

12.2 Method blanks are not subtracted from samples and must be below the reporting limit. Blanks above the reporting limit will require reextraction and analysis of the entire batch. If sufficient sample does not exist to reprepare and analyze, "flag" method blank result as greater than the reporting limit and report "B"- flagged sample data from original batch.

12.3 If the MS/MSD is out of QC limits reanalyze to verify result. If the MS/MSD is still out of limits identify source of problem/contamination and eliminate. Evaluate whether the unspiked sample level (sample) is greater than four times the spike level.

12.3.1 If the sample is greater than four times the spike, flag outlying MS/MSD results as estimated due to the four times rule.

12.3.2 If sample is less than or equal to four times the spike, flag the sample results as matrix effect indicated.

12.4 If the LCS is out of QC limits reanalyze to verify result. If the LCS is still out of limits identify the source of problem/contamination and eliminate. If sufficient sample aliquots exist, reprepare all samples and QC in a new batch and analyze. If sufficient sample does not exist to reprepare and analyze, "flag" LCS result as out and report "J"- flagged (estimated) sample data from original batch.

13.0 CALCULATIONS

13.1 The chromatograms will be labeled with the laboratory sample number.

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13.2 Quantitation of analyte concentration is performed automatically by the instrument software.

13.3 The instrument concentration is multiplied by the final extraction volume divided by the amount of samples extracted. This number multiplied by 10 (prep dilution factor) and divided by the percent (%) solids.

13.4 Multiply this concentration and the Reporting Limit (RL) by any additional dilution factors if necessary.

13.5 Make sure check standards, QC samples, and blanks are within respective acceptance ranges.

13.6 Record all data in the lab book. Report results in µg/kg two significant figures for solids.

14.0 REAGENT WASTE DISPOSAL

The Health and Safety Officer is responsible for off-site removal of all hazardous materials.

15.0 REFERENCES

15.1 Dionex Application Note 121.

15.2 USEPA. 1999. Determination of Perchlorate in Drinking Water Using Ion Chromatography, Method 314.0, Revision 1.0. Cincinnati: November.

15.3 State of California. Department of Health Services. Sanitation and Radiation Laboratories (SRL) Branch. 1999. Determination of Perchlorate by Ion Chromatography, Revision 1.0 (CLO4RV-1.WPD). Berkeley: CDHS, March.

15.4 USEPA. National Exposure Research Laboratory. Ecosystems Research Division. 2000. Determination of Perchlorate at parts-per-billion levels in plants by Ion Chromatography, Draft. Athens, Georgia: USEPA, April. Jackson Ellington and John J Evans, (Please Note: This draft method was only used for general information).

SOP Author: Mike Wantland & Sharon Johnson	**SOP Reviewer:** Lisa L Ruggle 10/16/00 Date

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ATTACHMENT 8

PERCHLORATE IN VEGETATION METHOD PRECISION AND ACCURACY DATA

Matrix	Vegetation
Clean-up	2.5 grams Alumina/1.10 C18 5 grams
Sample Weight (grams)	2 grams
Final Volume (mL)	20 mLs

Analysis Date	Study Series	Spike Concentration (ug/L)	Spike Concentration (ug/Kg)	Measured Concentration (ug/L)	Measured Concentration (ug/Kg)	Recovery (%)	Mean	SD	RSD_{95} (%)
06-Oct-00	Matrix Blank	n/a		<4		n/a			
06-Oct-00	Matrix Blank	n/a		<4		n/a			
06-Oct-00	Matrix Blank	n/a		<4		n/a			
06-Oct-00	Matrix Blank	n/a		<4		n/a			
06-Oct-00	Matrix Blank	n/a		<4		n/a			
06-Oct-00	Matrix Blank	n/a		<4		n/a	n/a	n/a	n/a
06-Oct-00	Control Water Mid-level Spike	20		16.98		84.9%			
06-Oct-00	Control Water Mid-level Spike	20		16.57		82.9%			
06-Oct-00	Control Water Mid-level Spike	20		16.40		82.0%			
06-Oct-00	Control Water Mid-level Spike	20		17.00		85.0%			
06-Oct-00	Control Water Mid-level Spike	20		17.16		85.8%			
06-Oct-00	Control Water Mid-level Spike	20		17.34		86.7%	84.5%	1.78%	2.11%
06-Oct-00	1x LOD	3.98	398	4.32	432	108.5%			
06-Oct-00	1x LOD	3.98	398	4.32	432	108.5%			
06-Oct-00	1x LOD	3.98	398	4.73	473	118.8%			
06-Oct-00	1x LOD	3.98	398	4.60	460	115.6%			
06-Oct-00	1x LOD	3.98	398	4.54	454	114.1%			
06-Oct-00	1x LOD	3.98	398	3.53	353	88.7%			
06-Oct-00	1x LOD	3.98	398	3.96	396	99.5%	107.7%	10.46%	9.72%
06-Oct-00	1x LOD	3.98	398	9.04	904	227.1%	Not used in statistical calculation		
06-Oct-00	2x LOD	7.98	798	8.43	843	105.6%			
06-Oct-00	2x LOD	7.98	798	8.52	852	106.8%			
06-Oct-00	2x LOD	7.98	798	7.89	789	98.9%			
06-Oct-00	2x LOD	7.98	798	8.25	825	103.4%			
06-Oct-00	2x LOD	7.98	798	8.04	804	100.8%			
06-Oct-00	2x LOD	7.98	798	8.32	832	104.3%			
06-Oct-00	2x LOD	7.98	798	8.24	824	103.3%			
06-Oct-00	2x LOD	7.98	798	7.62	762	95.5%	102.3%	3.73%	3.65%
06-Oct-00	5x LOD	20	2000	17.28	1728	86.4%			
06-Oct-00	5x LOD	20	2000	17.78	1778	88.9%			
06-Oct-00	5x LOD	20	2000	17.29	1729	86.5%			
06-Oct-00	5x LOD	20	2000	17.22	1722	86.1%			
06-Oct-00	5x LOD	20	2000	16.78	1678	83.9%			
06-Oct-00	5x LOD	20	2000	16.79	1679	84.0%			
06-Oct-00	5x LOD	20	2000	18.00	1800	90.0%			
06-Oct-00	5x LOD	20	2000	17.42	1742	87.1%	86.6%	2.13%	2.46%
06-Oct-00	10x LOD	40	4000	35.27	3527	88.2%			
06-Oct-00	10x LOD	40	4000	34.02	3402	85.1%			
06-Oct-00	10x LOD	40	4000	34.54	3454	86.4%			
06-Oct-00	10x LOD	40	4000	37.25	3725	93.1%			
06-Oct-00	10x LOD	40	4000	34.09	3409	85.2%			
06-Oct-00	10x LOD	40	4000	33.50	3350	83.8%			
06-Oct-00	10x LOD	40	4000	35.50	3550	88.8%			
06-Oct-00	10x LOD	40	4000	34.38	3438	86.0%	87.0%	2.95%	3.39%
Notes									
					Overall Performance		95.5%	10.7%	11.2%

Determination of Perchlorate by Ion Chromatography in Vegetation

STANDARD OPERATING PROCEDURE (SOP): M-CC-70/N, Revision 0, October 2000

Authorization/Approval:		Applicability and Revision History:	
John Spurr, Quality Assurance Director	10/23/00 Date	Office Location:	Novi, Michigan
Scott Caillouette, Supervisor	10/25/00 Date	Effective Date:	R0, October 18, 2000
Robert Lieckfield, Jr., CIH, Laboratory Director	10/26/00 Date	Reference Methods:	EPA 314.0 modified
		Media/Matrices:	Vegetation

Distribution:

☐ CS Client Services	☐ GL General Lab Mngt.	☐ LI LIMS	☐ RC Report Control	☐ MP cGMP Services	☒ CC Classical Chem	☐ LC HPLC	☐ MS GC/MS
☐ FA Facilities Mngt	☒ GM Govern. Projects	☐ PA Payables/Purch	☐ RE Receivables	☐ MV Method Validation	☐ EX Extractions	☐ ME Metals	☐ PP Pesticides/PCBs
☐ FS Field Supplies	☐ HS Health & Safety	☐ QA Quality Assurance	☐ SC Sample Control	☐ SP Special Projects	☐ GC GC/IH	☐ MI Microscopy	☐ XR X-Ray Diffraction
☒ DC Document Control	☒ IC Information Center	☒ OTHERS: (Please List) Parsons Engineering Service					

1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of the perchlorate anion in vegetation by ion chromatography after aqueous extraction and solid–phase cleanup procedures.

1.2 Although not specifically tested, this method is potentially applicable to other solid matrixes. When this method is used to analyze unfamiliar samples, perchlorate identification should be supported by the use of matrix spikes. The matrix spike procedure is described in Section 6.5.

1.3 The linear calibration range for perchlorate is approximately 4 to 500 µg/L. The linear calibration may be extended to 1500 µg/L, but accuracy below 50 µg/L may be compromised. Quantitation below 50 µg/L is not recommended with the extended calibration. Sample concentrations higher than the upper calibration limit should be diluted with reagent water to the mid-range of the linear calibration and reanalyzed.

1.4 This method is recommended for use by analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

2.0 SUMMARY OF METHOD

2.1 The analysis is performed using an ion chromatograph. The main components of the ion chromatograph used in this method include an autosampler, pump, guard column, analytical column, ion suppressor device, conductivity detector, and chromatography data system.

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2.2 An aliquot of sample extract is injected into the ion chromatograph, where the perchlorate anion is separated from other sample components, then quantitated. A large volume sample injection, 1 ml, is employed to perform perchlorate measurement at the low µg/L level.

2.3 Perchlorate is a large polarizable anion with hydrophobic properties. To avoid or minimize hydrophobic interaction of perchlorate with the guard and separator columns, care must be exercised in the selection of the ion-exchange column and the eluent composition. Extensive hydrophobic interaction of the perchlorate anion with the ion-exchange column leads to a long retention time and severe peak tailing. The columns and eluents recommended in this method were selected to minimize this hydrophobic interaction.

3.0 INTERFERENCES

3.1 The wide water dip or negative peak that may be observed in the chromatogram is due to the large volume sample injection. The negative response is the result of an imbalance of ions in the sample relative to the eluent and does not interfere with the measurement of perchlorate, since perchlorate elutes well beyond the dip.

3.2 Interferences can be caused by substances with retention times that are similar to and overlap with the anion of interest.

3.3 High concentrations of anions can interfere with the peak resolution of an adjacent anion and/or cause baseline disturbances. When such an interference occurs, it may help to dilute the sample before analysis, although it should be recognized that the sample detection limit increases proportionately with the sample dilution factor.

3.4 Surfactants and detergents may cause fouling of the ion exchange column and membrane suppressor, resulting in an unstable baseline and poor detector response. Other dissolved organic compounds may similarly interfere.

4.0 APPARATUS AND MATERIALS

4.1 Ion chromatograph – Dionex DX120 or equivalent, complete with all required accessories including syringes, analytical columns, compressed gases, and detectors.

4.1.1 Anion guard column: A protector of the separator column. Dionex AG 16, 4 mm.

4.1.2 Anion separator column: This column produces the separation of the anions. Dionex AS 16, 4 mm.

4.1.3 Anion suppressor device: Dionex ASRS II.

4.1.4 Automated Sampler: Dionex ASM 3, or equivalent.

4.1.5 PeakNet IC Software: Dionex.

4.1.6 Conductivity detector: 1.25 µL internal volume.

4.1.7 Sample loop: Approximately 1000 µL (12" x 0.02" I.D. tubing), or larger, as required.

4.2 Polyethylene autosampler vials, 5 mL.

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4.3 Filter caps, 5 mL.

4.4 Volumetric flasks, 50, 100, 1000, and 2000 mL.

4.5 Plastic vials, 10 mL.

4.6 Falcon tubes, 15 ml and 50 ml.

4.7 Laboratory balance, capable of weighing to 0.1 mg.

4.8 Shaker table.

4.9 Vortex

4.10 Disposable syringes, 10 ml.

4.11 Centrifuge(s), capable of accommodating 50 mL Falcon tubes and 16 x 125-mm disposable glass test tubes.

4.12 Kimwipes, delicate task wipes.

4.13 Disposable test tubes, 16 x 125 mm with caps.

4.14 Gelman ion chromatography (IC) Acrodisc or equivalent, 25 mm syringe filter with 0.45 um Supor (PES) membrane, catalog #4585T.

4.15 C18 Solid Phase Extraction (SPE) cartridges or equivalent, Supelco Superclean ENVI-18 SPE Tubes, 20 ml (5 g), catalog #57137.

4.16 Solid phase extraction vacuum manifold.

4.17 Alumina, (Aluminum Oxide), Aldrich catalog #19,944-3, activated, basic, Brockmann I, standard grade, 150 mesh, 58 A, surface area 155 m^2/g.

Note: The alumina is cleaned by mixing one volume of alumina with 10 volumes of reagent water. The fines (cloudy water above the solid) are decanted and replaced with fresh reagent water. The mixing and decanting process is repeated until the water remains clear. The cleaned alumina is then allowed to air dry and stored in a screw cap glass bottle until used.

5.0 REAGENTS

5.1 Reagent water, obtained from the Milli-Q Water System, through the filtered spigot.

5.2 Sodium perchlorate (solid), Aldrich catalog #41,024-1.

5.3 Secondary source standard (1000ppm solution), SPEX catalog #AS-CLO49-2Y.

5.4 50% w/w sodium hydroxide, Fisher Chemical catalog #55254-500, or equivalent.

Note: For 50 mM sodium hydroxide, weigh 4.0 grams of 50% NaOH into a plastic weight boat and transfer into approximately 950 mL of reagent water and bring to 1000 mL volume.

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5.5 Methanol, HPLC grade.

6.0 PREPARATION OF STANDARDS

Note: In all cases where weighing is involved, the exact weight (all decimal places for the balance used) is recorded.

6.1 Primary Stock and Working Standards: All stock standards can be used for up to 12 months. All other standards can be used for up to 6 months.

6.1.1 1000 ppm Primary Perchlorate Stock Standard: Dissolve 1.231 g of sodium perchlorate in 1000 ml of reagent water.

6.1.2 10 ppm Perchlorate Working Standard: Dilute 1.0 ml of 1000 ppm stock perchlorate to 100 ml with reagent water.

6.1.3 1.0 ppm Perchlorate Working Standard: Dilute 10.0 ml of 10 ppm perchlorate to 100 ml with reagent water.

6.1.4 0.1 ppm Perchlorate Working Standard: Dilute 1.0 ml of 10 ppm perchlorate to 100 ml with reagent water.

6.2 Secondary Source Standards

6.2.1 10 ppm Secondary Source Perchlorate Standard: Dilute 1.0 ml of the 1000 ppm secondary source perchlorate standard to 100 ml with reagent water.

6.2.2 1.0 ppm Secondary Source Perchlorate Standard: Dilute 10 ml of 10 ppm secondary source perchlorate standard to 100 ml with reagnt water.

6.3 Mixed Common Anion Stock Solution (containing the anions chloride, sulfate and carbonate each at 25 mg/ml), used to prepare simulated common anion samples in the determination of the MCT: Dissolve the following salts in reagent water to a final volume of 25.0 ml.
(a) 1.0 g sodium chloride (NaCl, CAS [7647-14-5]) = 0.61 g Cl^-
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(c) 1.1 g sodium carbonate (Na_2CO_3, CAS [497-19-8]) = 0.62 g CO_3^{-2}

6.4 Instrument Performance Check (IPC) Standard: Add 2.5 ml of 1.0 ppm perchlorate working standard and 3.2 ml of mixed common anion stock to a 100 ml volumetric flask and dilute to volume with reagent water.

6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD): Prepare by spiking 400 µL of 10 ppm perchlorate working standard or secondary source standard to 2 g of sample. (Note: A smaller volume may have to be used due to limited sample volume)

6.6 Laboratory Control Sample (LCS): Prepare by spiking 400 µL of 10 ppm perchlorate working standard or secondary source standard into 20 ml of reagent water.

6.7 Instrument Calibration Verification Standard (ICV): Prepare by adding 2.0 ml of 1.0 ppm secondary source perchlorate standard to 50 ml of reagent water in a 100 ml volumetric flask and dilute to volume with reagent water.

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7.0 SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

7.1 Samples may be collected in HDPE plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

7.2 Until further preservation and holding time studies are available, there is no maximum holding time. Samples are to be stored at <0°C to minimize decomposition.

8.0 PROCEDURE FOR SAMPLE PREPARATION/EXTRACTION

8.1 Weigh 2.0 g (±0.1 g) of vegetation into 50 mL Falcon tube.

8.2 Pipet 20.0 ml of reagent water into tube

8.3 Place on mechanical shaker for approximately 16 hours.

8.4 Centrifuge for 30 minutes.

8.5 Decant top layer (sample extract) into a clean 50 ml Falcon tube.

8.6 Add a 5.0 ml aliquot of this extract to 2.5 g (±0.1g) of Alumina in a disposable 12.5 ml test tube and cap.

8.7 Vortex test tube for approximately 20 seconds.

8.8 Place test tube on a mechanical shaker for 1 hour.

8.9 Centrifuge the test tube for 15 minutes.

8.10 Filter the top layer (sample extract) through a 25 mm Gelman IC Acrodisc 0.45 um into a clean 15 ml Falcon tube using a 10 mL disposable syringe.

8.11 Add 1.0 ml of sample extract to 9.0 ml of reagent water in a clean 15 ml Falcon tube. This results in a 1:10 dilution of the sample extract.

8.12 Solid Phase Extraction (SPE) C18 Cleanup: Pass the diluted sample extract (1:10) through one ENVI-18 SPE cartridge to remove potential organic matrix interference compounds.

8.12.1 Condition each C18 cartridge per the manufacturer's instructions by rinsing with methanol followed by reagent water. The flow rate should not exceed 5.0 ml/minute.

8.12.1.1 Rinse cartridge with 4 ml of methanol.

8.12.1.2 Rinse cartridge with 5 ml of reagent water.

8.12.2 After conditioning with methanol and reagent water, rinse cartridge with 2.0 ml of diluted sample extract.

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8.12.3 To obtain the final extract, pass approximately 5.5 ml of diluted sample extract through the conditioned and rinsed C18 cartridge into a clean disposable test tube. Transfer the final extract to a 15 mL Falcon tube.

8.13 Transfer approximately 2 ml of final extract into an autosampler vial for analysis. The remaining extract is stored refrigerated for any additional analysis.

9.0 HEALTH AND SAFETY

9.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

9.2 The following chemicals have the potential to be highly toxic or hazardous. The MSDS for each chemical should be consulted.
(a) Sodium hydroxide
(b) Sodium perchlorate

10.0 QUALITY CONTROL

10.1 All IC chromatograms are kept for 5 years.

10.2 Perform an initial calibration with standards at 4, 10, 20, 50, and 100 ug/L . The calibration curve must have linear regression coefficient of determination (r^2) of at least 0.995. The 4 ug/L standard, when calculated back to the calibration curve, must be within ±25% of the actual value. The remainder of the standards must be within 15% of the actual value.

10.3 The calibration curve must be verified with the low continuing calibration verification standard (LCCV, 4 ug/L calibration standard) each working day. If the response for perchlorate varies from the expected values by more than ±25%, the test must be repeated, using fresh calibration standard. If the results are still more than ±25%, a new calibration curve must be prepared for the analyte. The shift in retention time must be within ±5% of expected value. If not, a new calibration curve must be prepared.

10.4 A mid-level continuing check verification (MCCV, 20 ug/L), alternating with a high level (HCCV, 100 ug/L) must be run after every 10 client samples. The last analysis of the run sequence is always a check standard. CCV must be within ±15% of expected value.

10.5 The instrument performance check (IPC) should not vary in retention time by more than 5%. The control limits for the perchlorate recovery are ±20%.

10.6 One method blank (MB) is run with every batch of ≤20 samples. The method blank is an aliquot of filtered reagent water that is prepared with samples and carried through the entire process.

10.7 One laboratory control sample (LCS) is run with every batch of ≤20 samples. For the LCS control limits, refer to current control charts.

10.8 One matrix spike (MS) and one matrix spike duplicate (MSD) are run with every batch of ≤20 samples. For the MS/MSD control limits, refer to current control charts.

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10.9 A sample duplicate (SD) must be run for every 20 samples. RPD must be within 15%.

$$RPD = 100 \times \frac{|S - SD|}{(S + SD)/2}$$

11.0 PROCEDURE FOR ANALYSIS

11.1 Start up the IC according to the directions in the operator's manual.

11.2 Suppressor should be set at 300 milliamps.

11.3 Eluent flow rate = 1 ml per minute.

11.4 Injection volume is 1 mL.

Note: The IC injection loop is usually 25 µL. Hence, the injection loop will need to be changed back if any other analysis is to be run.

11.5 The IC must be run in external water mode using reagent water as the regenerant. The regenerate flow rate should be approximately 3 to 5 mL per minute. All reservoirs should be filled at the beginning.

11.6 Allow the system to equilibrate. The conductivity should settle to approximately 2 to 5 µs.

11.7 Prepare a calibration curve using the standards prepared as follows in 100 ml of reagent water as follows:

mL of working STD	µg/L	STD
4.0 mL of 0.1 ppm	4	1
1.0 mL of 1.0 ppm	10	2
2.0 mL of 1.0 ppm	20	3
0.5 mL of 10 ppm	50	4
1.0 mL of 10 ppm	100	5

11.8 Set up a run sheet. Runs 1-5 are the calibration standards, followed by the LCCV, method blank, ICV, IPC, DI water and then samples. Number autosampler vials with the corresponding sample number on the run sheet.

11.9 Remove at least 2 mL of standards, check standards, QC samples, and the client samples and place in the autosampler vials.

11.10 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.0 CORRECTIVE ACTIONS

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12.0 CORRECTIVE ACTIONS

12.1 If the samples are more concentrated than the highest standard, dilute with filtered reagent water, preferably to the middle of the calibration curve or extend the calibration range, bracketing the concentration of the sample.

12.2 Method blanks are not subtracted from samples and must be below the reporting limit. Blanks above the reporting limit will require reextraction and analysis of the entire batch. If sufficient sample does not exist to reprepare and analyze, "flag" method blank result as greater than the reporting limit and report "B"- flagged sample data from original batch.

12.3 If the MS/MSD is out of QC limits reanalyze to verify result. If the MS/MSD is still out of limits identify source of problem/contamination and eliminate. Evaluate whether the unspiked sample level (sample) is greater than four times the spike level.

12.3.1 If the sample is greater than four times the spike, flag outlying MS/MSD results as estimated due to the four times rule.

12.3.2 If sample is less than or equal to four times the spike, flag the sample results as matrix effect indicated.

12.4 If the LCS is out of QC limits reanalyze to verify result. If the LCS is still out of limits identify the source of problem/contamination and eliminate. If sufficient sample aliquots exist, reprepare all samples and QC in a new batch and analyze. If sufficient sample does not exist to reprepare and analyze, "flag" LCS result as out and report "J"- flagged (estimated) sample data from original batch.

13.0 CALCULATIONS

13.1 The chromatograms will be labeled with the laboratory sample number.

13.2 Quantitation of analyte concentration is performed automatically by the instrument software.

13.3 The instrument concentration is multiplied by the final extraction volume divided by the mass of sample extracted. This number is multiplied by 10 (prep dilution factor) and divided by the percent (%) solids.

13.4 Multiply this concentration and the Reporting Limit (RL) by any additional dilution factors if necessary.

13.5 Make sure check standards, QC samples, and blanks are within respective acceptance ranges.

13.6 Record all data in the lab book. Report results in µg/kg two significant figures for solids.

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14.0 REAGENT WASTE DISPOSAL

The Health and Safety Officer is responsible for off-site removal of all hazardous materials.

15.0 REFERENCES

15.1 Dionex Application Note 121.

15.2 USEPA. 1999. Determination of Perchlorate in Drinking Water Using Ion Chromatography, Method 314.0, Revision 1.0. Cincinnati: November.

15.3 State of California. Department of Health Services. Sanitation and Radiation Laboratories (SRL) Branch. 1999. Determination of Perchlorate by Ion Chromatography, Revision 1.0 (CLO4RV-1.WPD). Berkeley: CDHS, March.

15.4 USEPA. National Exposure Research Laboratory. Ecosystems Research Division. 2000. Determination of Perchlorate at parts-per-billion levels in plants by Ion Chromatography, Draft. Athens, Georgia: USEPA, April. Jackson Ellington and John J Evans, (Please Note: This draft method was only used for general information).

SOP Author: Mike Wantland & Sharon Johnson	**SOP Reviewer:** Kristine Kurtz 10/23/00 Kristine Kurtz Date

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Calibration Update Report

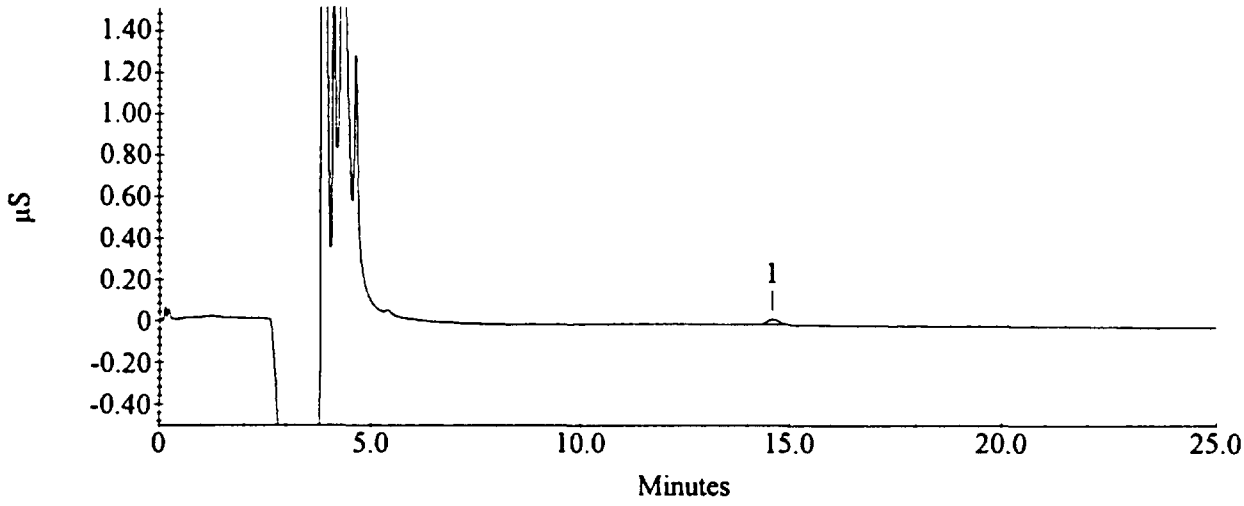
Sample Name : Std 4 ppb Perchlorate ICR-1555
Data File Name : C:\PEAKNET\DATA\100500_002.DXD

Method File Name : C:\PEAKNET\METHOD\perchlorate.met
Schedule File Name : c:\peaknet\schedule\100500 vegitation method validation.sch
Date Time Collected : 10/05/2000 11:40:28 AM
Calibration Date : 10/05/2000 12:43:44 PM
System Operator : KK

Peak Information : All Components

Peak #	Component Name	Retention Time	Cal Response (Previous)	Cal Response (Measured)	Cal Response (New)
1	Perchlorate	14.58	67067	4713	4713

Std 4 ppb Perchlorate ICR-1555



Calibration Update Report

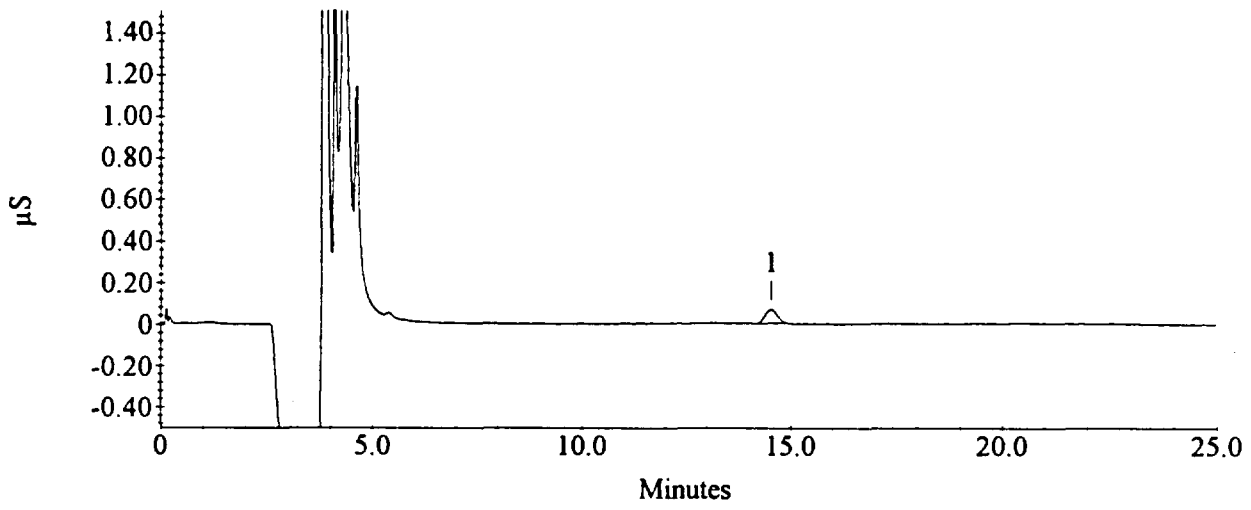
Sample Name : Std 10 ppb Perchlorate ICR-155
Data File Name : C:\PEAKNET\DATA\100500_003.DXD

Method File Name : C:\PeakNet\method\perchlorate.met
Schedule File Name : c:\peaknet\schedule\100500 vegitation method validation.sch
Date Time Collected : 10/05/2000 12:07:46 PM
Calibration Date : 10/05/2000 12:44:29 PM
System Operator : KK

Peak Information : All Components

Peak #	Component Name	Retention Time	Cal Response (Previous)	Cal Response (Measured)	Cal Response (New)
1	Perchlorate	14.53	10171	13006	13006

Std 10 ppb Perchlorate ICR-155



Calibration Update Report

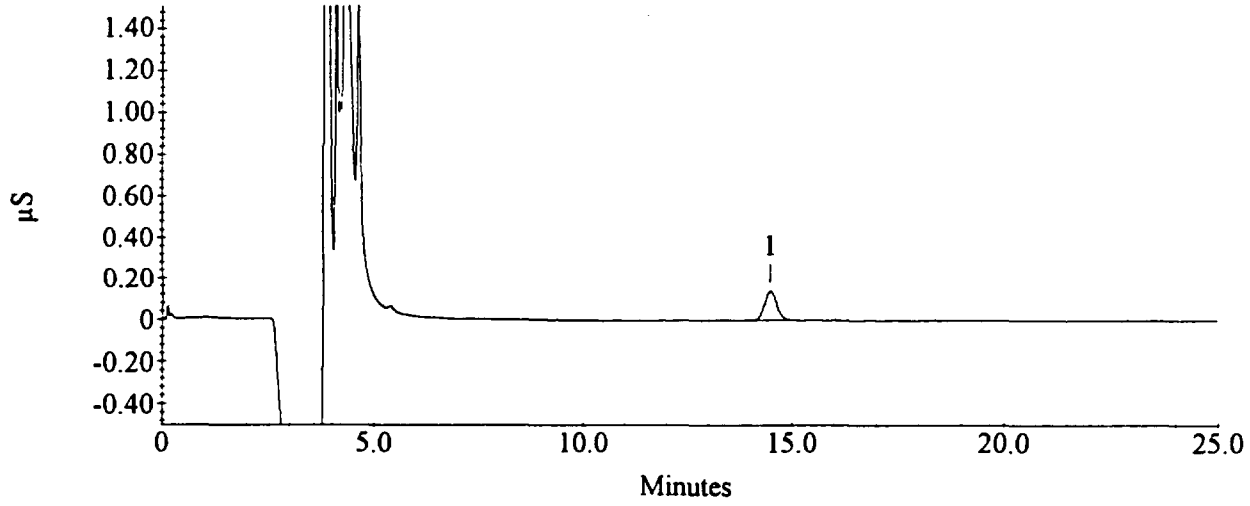
Sample Name : Std 20 ppb Perchlorate ICR-1557
Data File Name : C:\PEAKNET\DATA\100500_004.DXD

Method File Name : C:\PeakNet\method\perchlorate.met
Schedule File Name : c:\peaknet\schedule\100500 vegitation method validation.sch
Date Time Collected : 10/05/2000 12:35:03 PM
Calibration Date : 10/05/2000 1:09:41 PM
System Operator : KK

Peak Information : All Components

Peak #	Component Name	Retention Time	Cal Response (Previous)	Cal Response (Measured)	Cal Response (New)
1	Perchlorate	14.47	22364	27423	27423

Std 20 ppb Perchlorate ICR-1557



Calibration Update Report

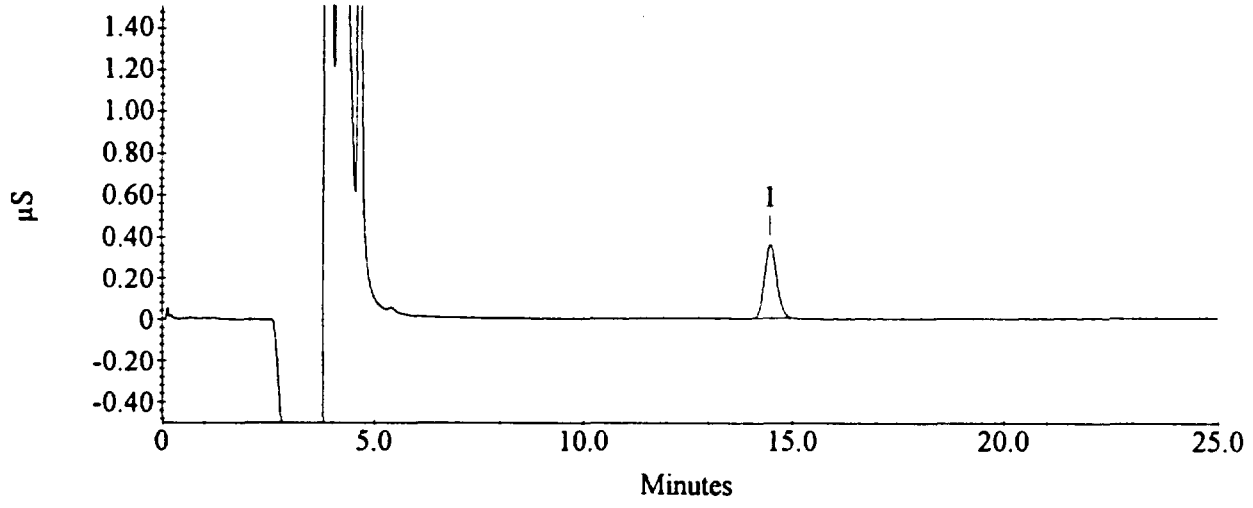
Sample Name : Std 50 ppb Perchlorate ICR-155δ
Data File Name : C:\PEAKNET\DATA\100500_005.DXD

Method File Name : C:\PeakNet\method\perchlorate.met
Schedule File Name : c:\peaknet\schedule\100500 vegitation method validation.sch
Date Time Collected : 10/05/2000 1:02:19 PM
Calibration Date : 10/05/2000 1:36:37 PM
System Operator : KK

Peak Information : All Components

Peak #	Component Name	Retention Time	Cal Response (Previous)	Cal Response (Measured)	Cal Response (New)
1	Perchlorate	14.47	57434	71498	71498

Std 50 ppb Perchlorate ICR-155δ



Calibration Update Report

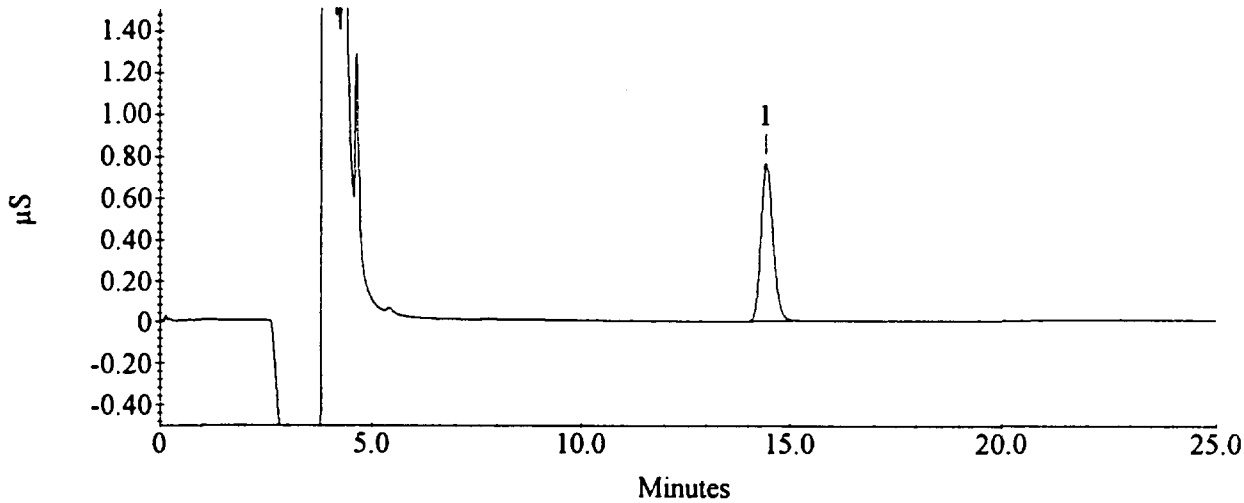
Sample Name : Std 100ppb Perchlorate ICR-1559
Data File Name : C:\PEAKNET\DATA\100500_006.DXD

Method File Name : C:\PeakNet\method\perchlorate.met
Schedule File Name : c:\peaknet\schedule\100500 vegitation method validation.sch
Date Time Collected : 10/05/2000 1:29:36 PM
Calibration Date : 10/05/2000 2:00:01 PM
System Operator : KK

Peak Information : All Components

Peak #	Component Name	Retention Time	Cal Response (Previous)	Cal Response (Measured)	Cal Response (New)
1	Perchlorate	14.42	143935	152480	152480

Std 100ppb Perchlorate ICR-1559



Sample Analysis Report

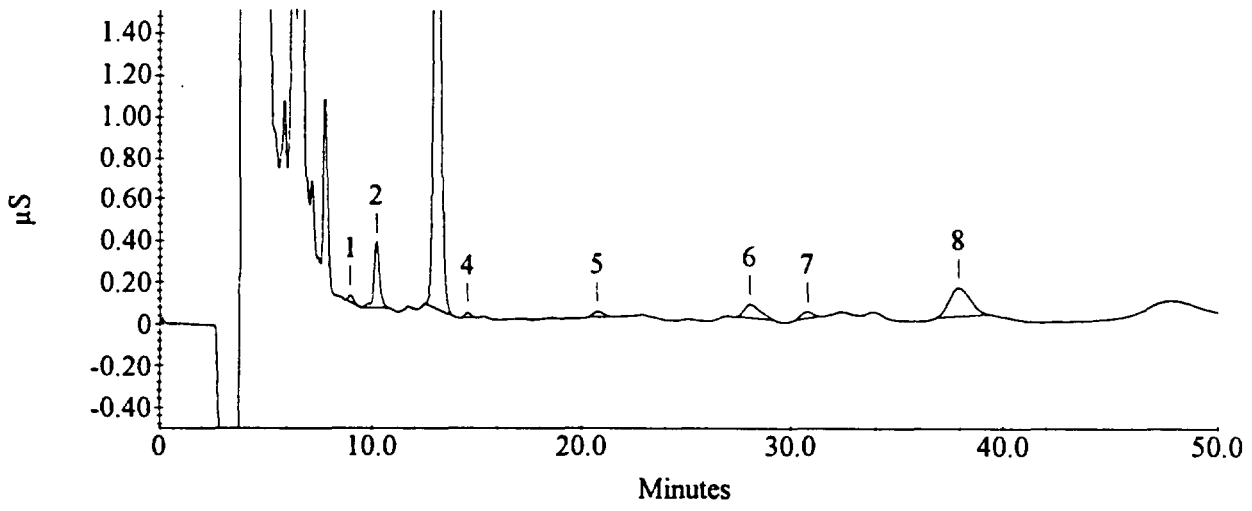
Sample Name : 1x MS #2 (3.984 ppb)
Data File Name : C:\PEAKNET\DATA\100500_028.DXD

Method File Name : c:\peaknet\method\perchlorate.met
Date Time Collected : 10/06/2000 6:32:44 AM
System Operator : KK

Peak Information : All Peaks

Peak #	Component Name	Retention Time	Amount (ug/L)	Peak Area	Peak Height
1		8.97	0.0000	5210	342
2		10.20	0.0000	57371	3119
3		13.07	0.0000	653476	29866
4	Perchlorate	14.55	4.3193	4552	231
5		20.75	0.0000	8291	247
6		28.03	0.0000	33901	632
7		30.78	0.0000	12537	311
8		37.90	0.0000	92206	1359

1x MS #2 (3.984 ppb)



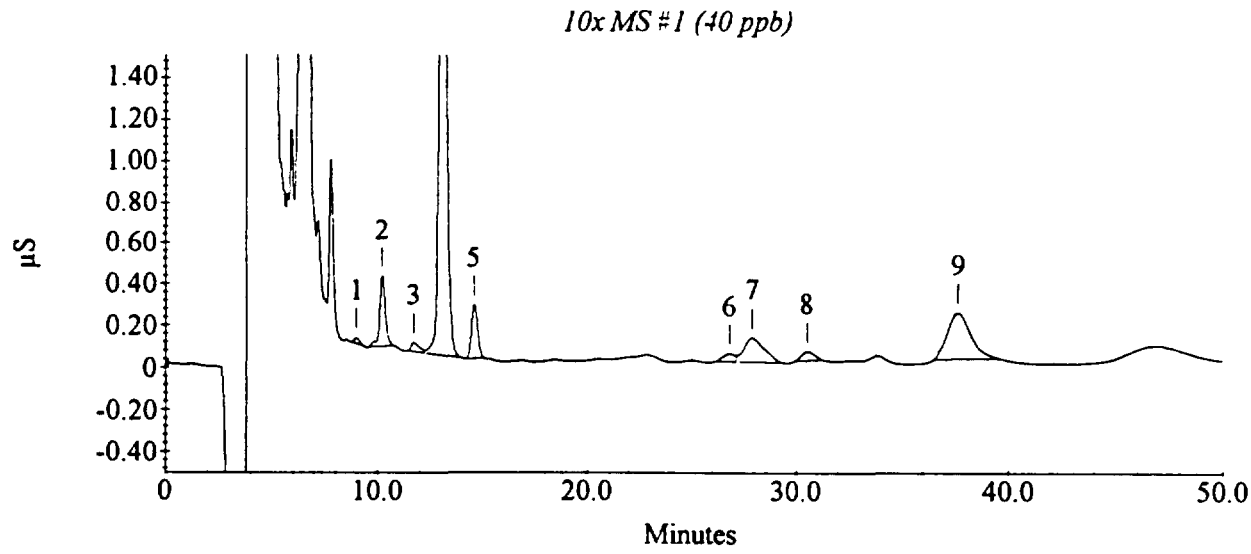
Sample Analysis Report

Sample Name : 10x MS #1 (40 ppb)
Data File Name : C:\PEAKNET\DATA\100500_055.DXD

Method File Name : c:\peaknet\method\perchlorate.met
Date Time Collected : 10/07/2000 8:03:26 PM
System Operator : KK

Peak Information : All Peaks

Peak #	Component Name	Retention Time	Amount (ug/L)	Peak Area	Peak Height
1		9.00	0.0000	4374	276
2		10.22	0.0000	62083	3361
3		11.72	0.0000	11487	427
4		13.07	0.0000	726675	32247
5	Perchlorate	14.60	35.2747	51902	2541
6		26.82	0.0000	13662	349
7		27.90	0.0000	72968	1135
8		30.52	0.0000	16903	396
9		37.65	0.0000	164814	2190



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ATTACHMENT 9

PERCHLORATE IN TISSUE METHOD PRECISION AND ACCURACY DATA

Matrix	Tissue (Cotton Tail)
Clean-up	2.5 grams Alumina/1.10 C18 5 grams
Sample Weight (grams)	2 grams
Final Volume (mL)	20 mLs

Analysis Date	Study Series	Spike Concentration (ug/L)	Spike Concentration (ug/Kg)	Measured Concentration (ug/L)	Measured Concentration (ug/Kg)	Recovery (%)	Mean	SD	RSD_{95} (%)
19-Oct-00	Matrix Blank	n/a		<4		n/a			
19-Oct-00	Matrix Blank	n/a		<4		n/a			
19-Oct-00	Matrix Blank	n/a		<4		n/a			
19-Oct-00	Matrix Blank	n/a		<4		n/a			
19-Oct-00	Matrix Blank	n/a		<4		n/a			
19-Oct-00	Matrix Blank	n/a		<4		n/a	n/a	n/a	n/a
19-Oct-00	Control Water Mid-level Spike	20		17.36		86.8%			
19-Oct-00	Control Water Mid-level Spike	20		16.49		82.5%			
19-Oct-00	Control Water Mid-level Spike	20		17.25		86.3%			
19-Oct-00	Control Water Mid-level Spike	20		17.41		87.1%			
19-Oct-00	Control Water Mid-level Spike	20		18.23		91.2%			
19-Oct-00	Control Water Mid-level Spike	20		17.16		85.8%	86.6%	2.79%	6.32%
19-Oct-00	1x LOD	4	400	3.62	362	90.5%			
19-Oct-00	1x LOD	4	400	3.85	385	96.3%			
19-Oct-00	1x LOD	4	400	3.99	399	99.8%			
19-Oct-00	1x LOD	4	400	3.97	397	99.3%			
19-Oct-00	1x LOD	4	400	3.84	384	96.0%			
19-Oct-00	1x LOD	4	400	3.52	352	88.0%			
19-Oct-00	1x LOD	4	400	3.89	389	97.3%			
19-Oct-00	1x LOD	4	400	3.82	382	95.5%	95.3%	4.09%	8.41%
19-Oct-00	2x LOD	7.98	798	8.18	818	102.5%			
19-Oct-00	2x LOD	7.98	798	7.00	700	87.7%			
19-Oct-00	2x LOD	7.98	798	7.06	706	88.5%			
19-Oct-00	2x LOD	7.98	798	7.06	706	88.5%			
19-Oct-00	2x LOD	7.98	798	6.92	692	86.7%			
19-Oct-00	2x LOD	7.98	798	6.69	669	83.8%			
19-Oct-00	2x LOD	7.98	798	7.01	701	87.8%			
19-Oct-00	2x LOD	7.98	798	6.96	696	87.2%	89.1%	5.62%	12.36%
19-Oct-00	5x LOD	20	2,000	15.6	1,560	78.0%			
19-Oct-00	5x LOD	20	2,000	15.54	1,554	77.7%			
19-Oct-00	5x LOD	20	2,000	17.53	1,753	87.7%			
19-Oct-00	5x LOD	20	2,000	15.66	1,566	78.3%			
19-Oct-00	5x LOD	20	2,000	15.38	1,538	76.9%			
19-Oct-00	5x LOD	20	2,000	15.61	1,561	78.1%			
19-Oct-00	5x LOD	20	2,000	17.76	1,776	88.8%			
19-Oct-00	5x LOD	20	2,000	15.99	1,599	80.0%	80.7%	4.75%	11.54%
19-Oct-00	25x LOD	100	10,000	81.61	8,161	81.6%			
19-Oct-00	25x LOD	100	10,000	78.78	7,878	78.8%			
19-Oct-00	25x LOD	100	10,000	82.82	8,282	82.8%			
19-Oct-00	25x LOD	100	10,000	81.32	8,132	81.3%			
19-Oct-00	25x LOD	100	10,000	82.27	8,227	82.3%			
19-Oct-00	25x LOD	100	10,000	80.45	8,045	80.5%			
19-Oct-00	25x LOD	100	10,000	79.25	7,925	79.3%			
19-Oct-00	25x LOD	100	10,000	76.90	7,690	76.9%	80.4%	1.99%	4.86%
					Overall Performance		86.4%	7.5%	17.1%

Determination of Perchlorate by Ion Chromatography in Animal Tissue

STANDARD OPERATING PROCEDURE (SOP): M-CC-71/N, Revision 0, October 2000

Authorization/Approval:		Applicability and Revision History:	
John Spurr, Quality Assurance Director	10/25/00 Date	Office Location:	Novi, Michigan
Scott Caillouette, Supervisor	10/25/00 Date	Effective Date:	R0, October 24, 2000
Robert Leckfield, Jr., CIH, Laboratory Director	10/26/00 Date	Reference Methods:	EPA 314.0 modified
		Media/Matrices:	Animal Tissue

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☒ DC Document Control	☒ IC Information Center	☒ OTHERS: (Please List)	Parsons Engineering Service				

1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of the perchlorate anion in animal tissue by ion chromatography after aqueous extraction and solid–phase cleanup procedures.

1.2 Although not specifically tested, this method is potentially applicable to other solid matrixes. When this method is used to analyze unfamiliar samples, perchlorate identification should be supported by the use of matrix spikes. The matrix spike procedure is described in Section 6.5.

1.3 The linear calibration range for perchlorate is approximately 4 to 500 µg/L. The linear calibration may be extended to 1500 µg/L, but accuracy below 50 µg/L may be compromised. Quantitation below 50 µg/L is not recommended with the extended calibration. Sample concentrations higher than the upper calibration limit should be diluted with reagent water to the mid-range of the linear calibration and reanalyzed.

1.4 This method is recommended for use by analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

2.0 SUMMARY OF METHOD

2.1 The analysis is performed using an ion chromatograph. The main components of the ion chromatograph used in this method include an autosampler, pump, guard column, analytical column, ion suppressor device, conductivity detector, and chromatography data system.

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2.2 An aliquot of sample extract is injected into the ion chromatograph, where the perchlorate anion is separated from other sample components, then quantitated. A large volume sample injection, 1 ml, is employed to perform perchlorate measurement at the low µg/L level.

2.3 Perchlorate is a large polarizable anion with hydrophobic properties. To avoid or minimize hydrophobic interaction of perchlorate with the guard and separator columns, care must be exercised in the selection of the ion-exchange column and the eluent composition. Extensive hydrophobic interaction of the perchlorate anion with the ion-exchange column leads to a long retention time and severe peak tailing. The columns and eluents recommended in this method were selected to minimize this hydrophobic interaction.

3.0 INTERFERENCES

3.1 The wide water dip or negative peak that may be observed in the chromatogram is due to the large volume sample injection. The negative response is the result of an imbalance of ions in the sample relative to the eluent and does not interfere with the measurement of perchlorate, since perchlorate elutes well beyond the dip.

3.2 Interferences can be caused by substances with retention times that are similar to and overlap with the anion of interest.

3.3 High concentrations of anions can interfere with the peak resolution of an adjacent anion and/or cause baseline disturbances. When such an interference occurs, it may help to dilute the sample before analysis, although it should be recognized that the sample detection limit increases proportionately with the sample dilution factor.

3.4 Surfactants and detergents may cause fouling of the ion exchange column and membrane suppressor, resulting in an unstable baseline and poor detector response. Other dissolved organic compounds may similarly interfere.

4.0 APPARATUS AND MATERIALS

4.1 Ion chromatograph – Dionex DX120 or equivalent, complete with all required accessories including syringes, analytical columns, compressed gases, and detectors.

4.1.1 Anion guard column: A protector of the separator column. Dionex AG 16, 4 mm.

4.1.2 Anion separator column: This column produces the separation of the anions. Dionex AS 16, 4 mm.

4.1.3 Anion suppressor device: Dionex ASRS II.

4.1.4 Automated Sampler: Dionex ASM 3, or equivalent.

4.1.5 PeakNet IC Software: Dionex.

4.1.6 Conductivity detector: 1.25 µL internal volume.

4.1.7 Sample loop: Approximately 1000 µL (12" x 0.02" I.D. tubing), or larger, as required.

4.2 Polyethylene autosampler vials, 5 mL.

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4.3 Filter caps, 5 mL.

4.4 Volumetric flasks, 50, 100, 1000, and 2000 mL.

4.5 Plastic vials, 10 mL.

4.6 Falcon tubes, 15 ml and 50 ml.

4.7 Laboratory balance, capable of weighing to 0.1 mg.

4.8 Shaker table.

4.9 Vortex

4.10 Disposable syringes, 10 ml.

4.11 Centrifuge(s), capable of accommodating 50 mL Falcon tubes and 16 x 125-mm disposable glass test tubes.

4.12 Kimwipes, delicate task wipes.

4.13 Disposable test tubes, 16 x 125 mm with caps.

4.14 Gelman ion chromatography (IC) Acrodisc or equivalent, 25 mm syringe filter with 0.45 um Supor (PES) membrane, catalog #4585T.

4.15 C18 Solid Phase Extraction (SPE) cartridges or equivalent, Supelco Superclean ENVI-18 SPE Tubes, 20 ml (5 g), catalog #57137.

4.16 Solid phase extraction vacuum manifold.

4.17 Alumina, (Aluminum Oxide), Aldrich catalog #19,944-3, activated, basic, Brockmann l, standard grade, 150 mesh, 58 A, surface area 155 m^2/g.

Note: The alumina is cleaned by mixing one volume of alumina with 10 volumes of reagent water. The fines (cloudy water above the solid) are decanted and replaced with fresh reagent water. The mixing and decanting process is repeated until the water remains clear. The cleaned alumina is then allowed to air dry and stored in a screw cap glass bottle until used.

5.0 REAGENTS

5.1 Reagent water, obtained from the Milli-Q Water System, through the filtered spigot.

5.2 Sodium perchlorate (solid), Aldrich catalog #41,024-1.

5.3 Secondary source standard (1000ppm solution), SPEX catalog #AS-CLO49-2Y.

5.4 50% w/w sodium hydroxide, Fisher Chemical catalog #55254-500, or equivalent.

Note: For 100 mM sodium hydroxide, weigh 8.0 grams of 50% NaOH into a plastic weight boat and transfer into approximately 950 mL of reagent water and bring to 1000 mL volume.

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5.5 Methanol, HPLC grade.

6.0 PREPARATION OF STANDARDS

Note: In all cases where weighing is involved, the exact weight (all decimal places for the balance used) is recorded.

6.1 Primary Stock and Working Standards: All stock standards can be used for up to 12 months. All other standards can be used for up to 6 months.

6.1.1 1000 ppm Primary Perchlorate Stock Standard: Dissolve 1.231 g of sodium perchlorate in 1000 ml of reagent water.

6.1.2 10 ppm Perchlorate Working Standard: Dilute 1.0 ml of 1000 ppm stock perchlorate to 100 ml with reagent water.

6.1.3 1.0 ppm Perchlorate Working Standard: Dilute 10.0 ml of 10 ppm perchlorate to 100 ml with reagent water.

6.1.4 0.1 ppm Perchlorate Working Standard: Dilute 1.0 ml of 10 ppm perchlorate to 100 ml with reagent water.

6.2 Secondary Source Standards

6.2.1 10 ppm Secondary Source Perchlorate Standard: Dilute 1.0 ml of the 1000 ppm secondary source perchlorate standard to 100 ml with reagent water.

6.2.2 1.0 ppm Secondary Source Perchlorate Standard: Dilute 10 ml of 10 ppm secondary source perchlorate standard to 100 ml with reagnt water.

6.3 Mixed Common Anion Stock Solution (containing the anions chloride, sulfate and carbonate each at 25 mg/ml), used to prepare simulated common anion samples in the determination of the MCT: Dissolve the following salts in reagent water to a final volume of 25.0 ml.
(a) 1.0 g sodium chloride (NaCl, CAS [7647-14-5]) = 0.61 g Cl^-
(b) 93 g sodium sulfate (Na_2SO_4, CAS [7757-82-6]) = 0.63 g SO_4^{-2}
(c) 1.1 g sodium carbonate (Na_2CO_3, CAS [497-19-8]) = 0.62 g CO_3^{-2}

6.4 Instrument Performance Check (IPC) Standard: Add 2.5 ml of 1.0 ppm perchlorate working standard and 3.2 ml of mixed common anion stock to a 100 ml volumetric flask and dilute to volume with reagent water.

6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD): Prepare by spiking 400 µL of 10 ppm perchlorate working standard or secondary source standard to 2 g of sample. (Note: A smaller volume may have to be used due to limited sample volume)

6.6 Laboratory Control Sample (LCS): Prepare by spiking 400 µL of 10 ppm perchlorate working standard or secondary source standard into 20 ml of reagent water.

6.7 Instrument Calibration Verification Standard (ICV): Prepare by adding 2.0 ml of 1.0 ppm secondary source perchlorate standard to 50 ml of reagent water in a 100 ml volumetric flask and dilute to volume with reagent water.

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7.0 SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

7.1 Samples may be collected in HDPE plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

7.2 Until further preservation and holding time studies are available, there is no maximum holding time. Samples are to be stored at <0°C to minimize decomposition.

8.0 PROCEDURE FOR SAMPLE PREPARATION/EXTRACTION

8.1 Weigh 2.0 g (±0.1 g) of tissue into 50 mL Falcon tube.

8.2 Pipet 20.0 ml of reagent water into tube

8.3 Place on mechanical shaker for approximately 16 hours.

8.4 Centrifuge for 30 minutes.

8.5 Decant top layer (sample extract) into a clean 50 ml Falcon tube.

8.6 Add a 5.0 ml aliquot of this extract to 2.5 g (±0.1g) of Alumina in a disposable 12.5 ml test tube and cap.

8.7 Vortex test tube for approximately 20 seconds.

8.8 Place test tube on a mechanical shaker for 1 hour.

8.9 Centrifuge the test tube for 15 minutes.

8.10 Filter the top layer (sample extract) through a 25 mm Gelman IC Acrodisc 0.45 um into a clean 15 ml Falcon tube using a 10 mL disposable syringe.

8.11 Add 1.0 ml of sample extract to 9.0 ml of reagent water in a clean 15 ml Falcon tube. This results in a 1:10 dilution of the sample extract.

8.12 Solid Phase Extraction (SPE) C18 Cleanup: Pass the diluted sample extract (1:10) through one ENVI-18 SPE cartridge to remove potential organic matrix interference compounds.

8.12.1 Condition each C18 cartridge per the manufacturer's instructions by rinsing with methanol followed by reagent water. The flow rate should not exceed 5.0 ml/minute.

8.12.1.1 Rinse cartridge with 4 ml of methanol.

8.12.1.2 Rinse cartridge with 5 ml of reagent water.

8.12.2 After conditioning with methanol and reagent water, rinse cartridge with 2.0 ml of diluted sample extract.

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8.12.3 To obtain the final extract, pass approximately 5.5 ml of diluted sample extract through the conditioned and rinsed C18 cartridge into a clean disposable test tube. Transfer the final extract to a 15 mL Falcon tube.

8.13 Transfer approximately 2 ml of final extract into an autosampler vial for analysis. The remaining extract is stored refrigerated for any additional analysis.

9.0 HEALTH AND SAFETY

9.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

9.2 The following chemicals have the potential to be highly toxic or hazardous. The MSDS for each chemical should be consulted.
(a) Sodium hydroxide
(b) Sodium perchlorate

10.0 QUALITY CONTROL

10.1 All IC chromatograms are kept for 5 years.

10.2 Perform an initial calibration with standards at 4, 10, 20, 50, and 100 ug/L . The calibration curve must have linear regression coefficient of determination (r^2) of at least 0.995. The 4 ug/L standard, when calculated back to the calibration curve, must be within ±25% of the actual value. The remainder of the standards must be within 15% of the actual value.

10.3 The calibration curve must be verified with the low continuing calibration verification standard (LCCV, 4 ug/L calibration standard) each working day. If the response for perchlorate varies from the expected values by more than ±25%, the test must be repeated, using fresh calibration standard. If the results are still more than ±25%, a new calibration curve must be prepared for the analyte. The shift in retention time must be within ±5% of expected value. If not, a new calibration curve must be prepared.

10.4 A mid-level continuing check verification (MCCV, 20 ug/L), alternating with a high level (HCCV, 100 ug/L) must be run after every 10 client samples. The last analysis of the run sequence is always a check standard. CCV must be within ±15% of expected value.

10.5 The instrument performance check (IPC) should not vary in retention time by more than 5%. The control limits for the perchlorate recovery are ±20%.

10.6 One method blank (MB) is run with every batch of ≤20 samples. The method blank is an aliquot of filtered reagent water that is prepared with samples and carried through the entire process.

10.7 One laboratory control sample (LCS) is run with every batch of ≤20 samples. For the LCS control limits, refer to current control charts.

10.8 One matrix spike (MS) and one matrix spike duplicate (MSD) are run with every batch of ≤20 samples. For the MS/MSD control limits, refer to current control charts.

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10.9 A sample duplicate (SD) must be run for every 20 samples. RPD must be within 15%.

$$RPD = 100 \times \frac{|S - SD|}{(S + SD)/2}$$

11.0 PROCEDURE FOR ANALYSIS

11.1 Start up the IC according to the directions in the operator's manual.

11.2 Suppressor should be set at 300 milliamps.

11.3 Eluent flow rate = 1 ml per minute.

11.4 Injection volume is 1 mL.

Note: The IC injection loop is usually 25 µL. Hence, the injection loop will need to be changed back if any other analysis is to be run.

11.5 The IC must be run in external water mode using reagent water as the regenerant. The regenerate flow rate should be approximately 3 to 5 mL per minute. All reservoirs should be filled at the beginning.

11.6 Allow the system to equilibrate. The conductivity should settle to approximately 2 to 5 µs.

11.7 Prepare a calibration curve using the standards prepared as follows in 100 ml of reagent water as follows:

mL of working STD	µg/L	STD
4.0 mL of 0.1 ppm	4	1
1.0 mL of 1.0 ppm	10	2
2.0 mL of 1.0 ppm	20	3
0.5 mL of 10 ppm	50	4
1.0 mL of 10 ppm	100	5

11.8 Set up a run sheet. Runs 1-5 are the calibration standards, followed by the LCCV, method blank, ICV, IPC, DI water and then samples. Number autosampler vials with the corresponding sample number on the run sheet.

11.9 Remove at least 2 mL of standards, check standards, QC samples, and the client samples and place in the autosampler vials.

11.10 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.0 CORRECTIVE ACTIONS

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12.1 If the samples are more concentrated than the highest standard, dilute with filtered reagent water, preferably to the middle of the calibration curve or extend the calibration range, bracketing the concentration of the sample.

12.2 Method blanks are not subtracted from samples and must be below the reporting limit. Blanks above the reporting limit will require reextraction and analysis of the entire batch. If sufficient sample does not exist to reprepare and analyze, "flag" method blank result as greater than the reporting limit and report "B"- flagged sample data from original batch.

12.3 If the MS/MSD is out of QC limits reanalyze to verify result. If the MS/MSD is still out of limits identify source of problem/contamination and eliminate. Evaluate whether the unspiked sample level (sample) is greater than four times the spike level.

12.3.1 If the sample is greater than four times the spike, flag outlying MS/MSD results as estimated due to the four times rule.

12.3.2 If sample is less than or equal to four times the spike, flag the sample results as matrix effect indicated.

12.4 If the LCS is out of QC limits reanalyze to verify result. If the LCS is still out of limits identify the source of problem/contamination and eliminate. If sufficient sample aliquots exist, reprepare all samples and QC in a new batch and analyze. If sufficient sample does not exist to reprepare and analyze, "flag" LCS result as out and report "J"- flagged (estimated) sample data from original batch.

13.0 CALCULATIONS

13.1 The chromatograms will be labeled with the laboratory sample number.

13.2 Quantitation of analyte concentration is performed automatically by the instrument software.

13.3 The instrument concentration is multiplied by the final extraction volume divided by the mass of sample extracted. This number is multiplied by 10 (prep dilution factor) and divided by the percent (%) solids.

13.4 Multiply this concentration and the Reporting Limit (RL) by any additional dilution factors if necessary.

13.5 Make sure check standards, QC samples, and blanks are within respective acceptance ranges.

13.6 Record all data in the lab book. Report results in µg/kg two significant figures for solids.

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14.0 REAGENT WASTE DISPOSAL

The Health and Safety Officer is responsible for off-site removal of all hazardous materials.

15.0 REFERENCES

15.1 Dionex Application Note 121.

15.2 USEPA. 1999. Determination of Perchlorate in Drinking Water Using Ion Chromatography, Method 314.0, Revision 1.0. Cincinnati: November.

15.3 State of California. Department of Health Services. Sanitation and Radiation Laboratories (SRL) Branch. 1999. Determination of Perchlorate by Ion Chromatography, Revision 1.0 (CLO4RV-1.WPD). Berkeley: CDHS, March.

15.4 USEPA. National Exposure Research Laboratory. Ecosystems Research Division. 2000. Determination of Perchlorate at parts-per-billion levels in plants by Ion Chromatography, Draft. Athens, Georgia: USEPA, April. Jackson Ellington and John J Evans, (Please Note: This draft method was only used for general information).

SOP Author: Mike Wantland & Sharon Johnson	**SOP Reviewer** Lisa L Quigg for 10/25/00 Kristine Kurtz Date

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Calibration Update Report

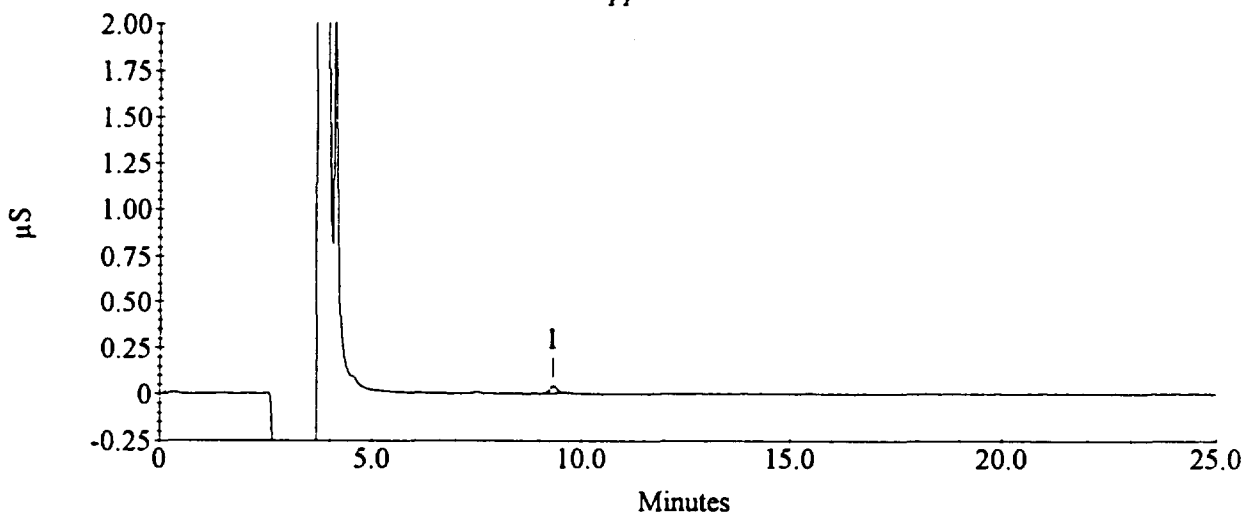
Sample Name : Std 4 ppb Perchlorate
Data File Name : C:\PEAKNET\DATA\101900a_002.DXD

Method File Name : c:\peaknet\method\perchlorate.met
Schedule File Name : c:\peaknet\schedule\101900 tissue method validation2.sch
Date Time Collected : 10/19/2000 5:16:08 PM
Calibration Date : 10/19/2000 5:41:09 PM
System Operator : KK

Peak Information : All Components

Peak #	Component Name	Retention Time	Cal Response (Previous)	Cal Response (Measured)	Cal Response (New)
1	Perchlorate	9.32	5373	4616	4616

Std 4 ppb Perchlorate



Calibration Update Report

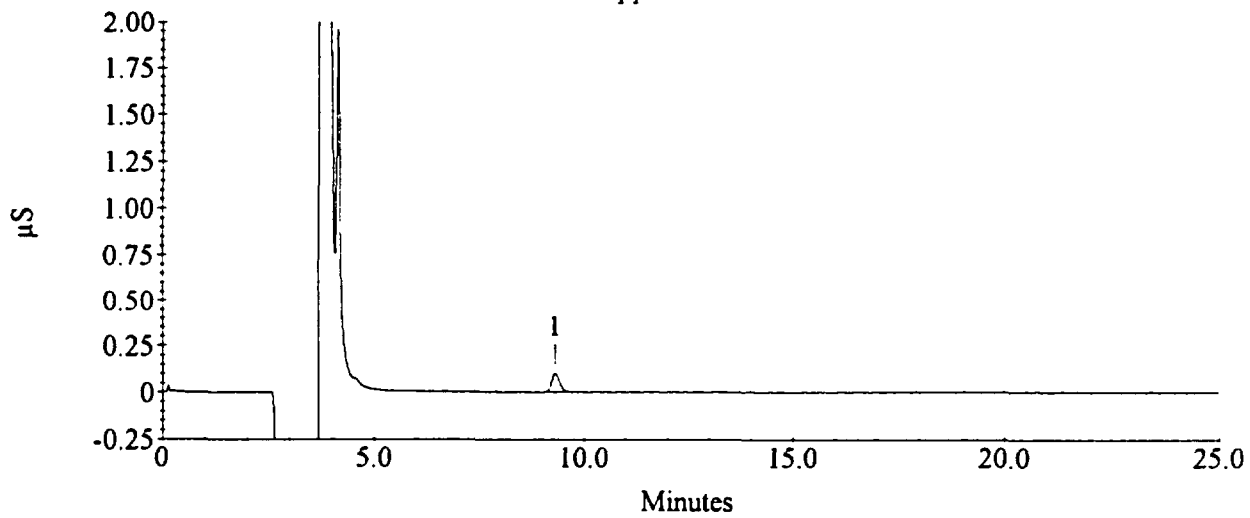
Sample Name : Std 10 ppb Perchlorate
Data File Name : C:\PEAKNET\DATA\101900a_003.DXD

Method File Name : c:\peaknet\method\perchlorate.met
Schedule File Name : c:\peaknet\schedule\101900 tissue method validation2.sch
Date Time Collected : 10/19/2000 5:43:24 PM
Calibration Date : 10/19/2000 6:08:25 PM
System Operator : KK

Peak Information : All Components

Peak #	Component Name	Retention Time	Cal Response (Previous)	Cal Response (Measured)	Cal Response (New)
1	Perchlorate	9.32	20786	13319	13319

Std 10 ppb Perchlorate



Calibration Update Report

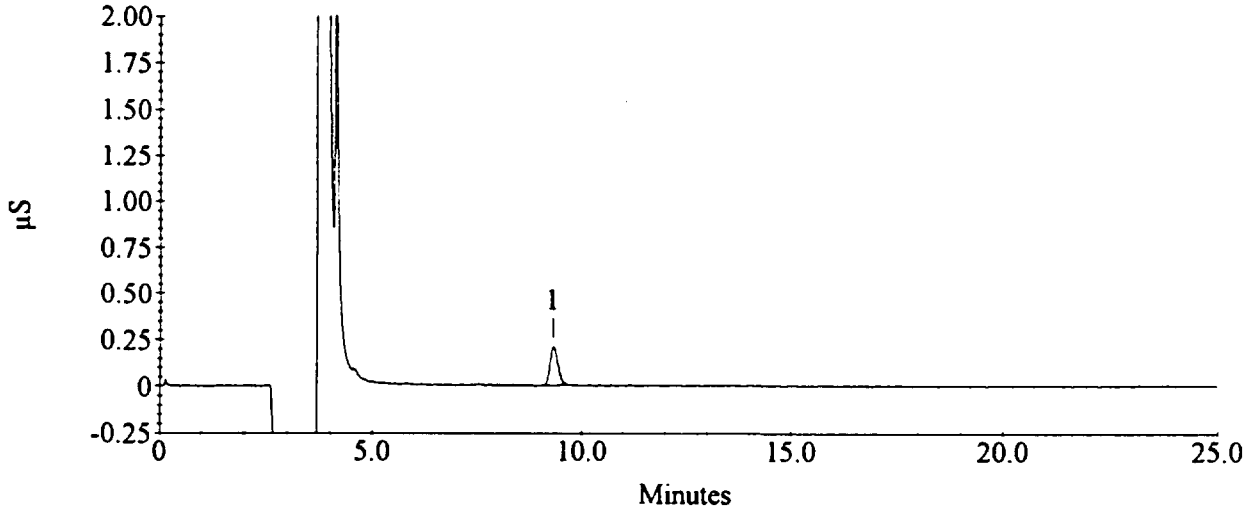
Sample Name : Std 20 ppb Perchlorate
Data File Name : C:\PEAKNET\DATA\101900a_004.DXD

Method File Name : c:\peaknet\method\perchlorate.met
Schedule File Name : c:\peaknet\schedule\101900 tissue method validation2.sch
Date Time Collected : 10/19/2000 6:10:41 PM
Calibration Date : 10/19/2000 6:35:42 PM
System Operator : KK

Peak Information : All Components

Peak #	Component Name	Retention Time	Cal Response (Previous)	Cal Response (Measured)	Cal Response (New)
1	Perchlorate	9.32	27099	27613	27613

Std 20 ppb Perchlorate



Calibration Update Report

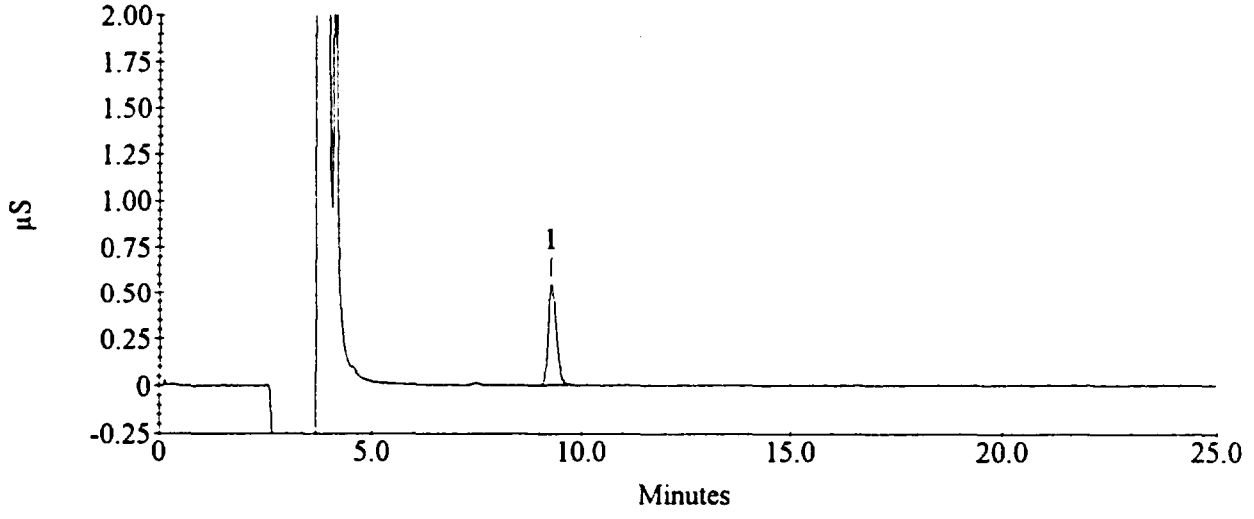
Sample Name : Std 50 ppb Perchlorate
Data File Name : C:\PEAKNET\DATA\101900a_005.DXD

Method File Name : c:\peaknet\method\perchlorate.met
Schedule File Name : c:\peaknet\schedule\101900 tissue method validation2.sch
Date Time Collected : 10/19/2000 6:37:56 PM
Calibration Date : 10/19/2000 7:02:58 PM
System Operator : KK

Peak Information : All Components

Peak #	Component Name	Retention Time	Cal Response (Previous)	Cal Response (Measured)	Cal Response (New)
1	Perchlorate	9.28	81263	71314	71314

Std 50 ppb Perchlorate



Calibration Update Report

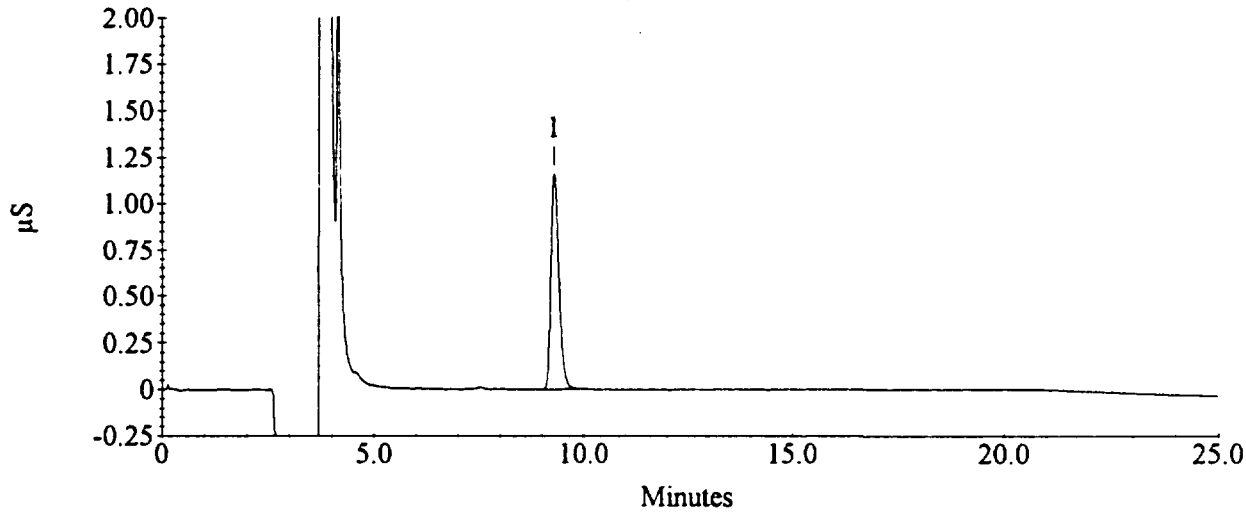
Sample Name : Std 100 ppb Perchlorate
Data File Name : C:\PEAKNET\DATA\101900a_006.DXD

Method File Name : c:\peaknet\method\perchlorate.met
Schedule File Name : c:\peaknet\schedule\101900 tissue method validation2.sch
Date Time Collected : 10/19/2000 7:05:14 PM
Calibration Date : 10/19/2000 7:30:15 PM
System Operator : KK

Peak Information : All Components

Peak #	Component Name	Retention Time	Cal Response (Previous)	Cal Response (Measured)	Cal Response (New)
1	Perchlorate	9.30	173122	153269	153269

Std 100 ppb Perchlorate



Sample Analysis Report

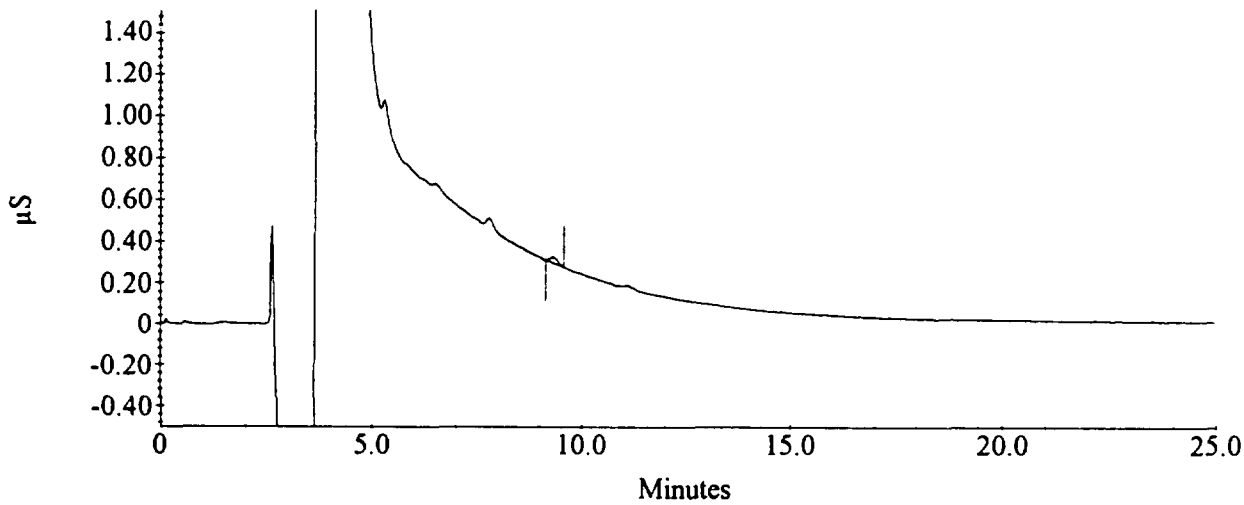
Sample Name : 1x MS #1 (4 ppb)
Data File Name : C:\PEAKNET\DATA\101900a_027rev.DXD

Method File Name : c:\peaknet\method\perchlorate.met
Date Time Collected : 10/20/2000 4:37:54 AM
System Operator : KK

Peak Information : All Peaks

Peak #	Component Name	Retention Time	Amount (ug/L)	Peak Area	Peak Height
1	Perchlorate	9.30	3.6251	3485	259

1x MS #1 (4 ppb)



Manual integration performed because Perchlorate peak was not detected by software.

KKuth 23 Oct - 00

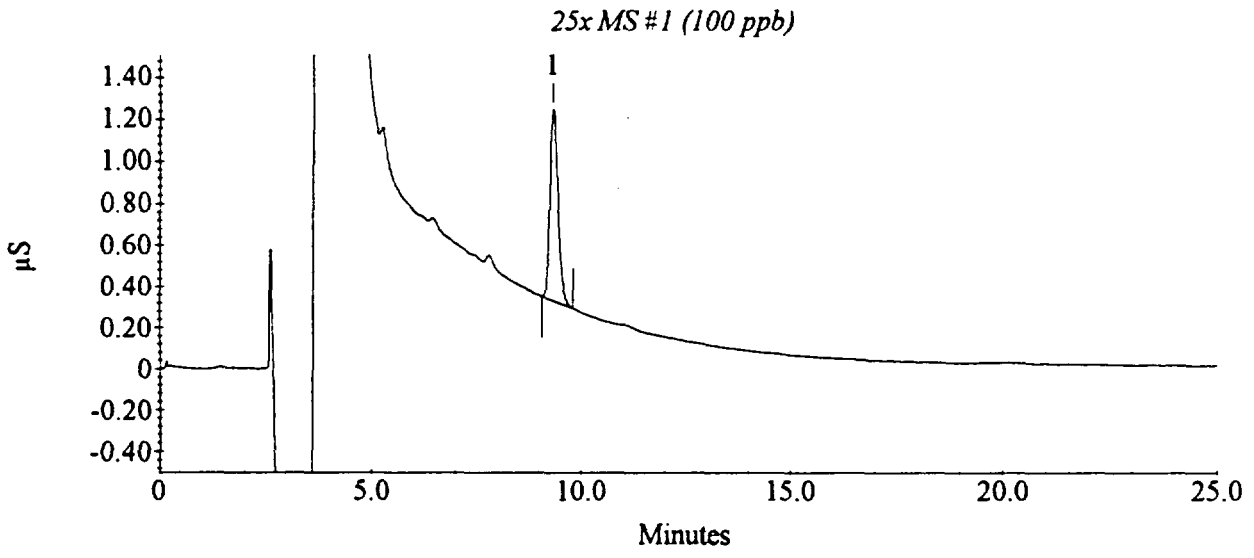
Sample Analysis Report

Sample Name : 25x MS #1 (100 ppb)
Data File Name : C:\peaknet\data\101900a_053rev.DXD

Method File Name : c:\peaknet\method\perchlorate.met
Date Time Collected : 10/20/2000 4:27:05 PM
System Operator : KK

Peak Information : All Peaks

Peak #	Component Name	Retention Time	Amount (ug/L)	Peak Area	Peak Height
1	Perchlorate	9.32	81.6094	123272	8982



Manual integration performed due to incorrect baseline extension.

KKguk 23-Oct-00

Appendix E

Data Validation Summary Reports

PERCHLORATE ANALYSIS BY CLAYTON GROUP SERVICES

DATA VALIDATION SUMMARY REPORTS
AND
DATA VERIFICATION REPORTS

Part I

The following Sample Delivery Groups (SDGs) were validated by scientists at Parsons ES.

Yuma Site: 00080031, 00080042, 00080118 and 00100980
Lake Mead Site: 00080562, 00080672, 00100347 and 00101001
Holloman Site: 00090666 and 00101117
ABL Site: 00060511, 00081023 and 00100349
Indian Head Site: 00081123 and 00100350
Longhorn Site: 00090393

The results are included in the Data Validation Reports provided. Data validation includes an extensive review of Chain of Custody (COC), case narrative, analytical sample results reports, quality control reports, preparation summaries, calibration curves, instrument sequence files, raw data and manual integrations.

All data are considered usable.

Part II

The following Sample Delivery Groups (SDGs) were verified by scientists at Parsons ES.

Yuma Site: 00100344, 00100345 and 00100983
Lake Mead Site: 00100987 and 00100999
Holloman Site: 00100679 and 00101115
ABL Site: 00101003
Indian Head Site: 00101005, 00101007 and 00101009
Longhorn Site: 00110360

Verification includes a general overview of the SDGs including review of the Chain of Custody (COC), case narrative, analytical sample results reports, quality control reports and calibration curves. A Verification Checklist is completed and stored with each the SDG data package.

All data are considered usable.

PART I

DATA VALIDATION SUMMARY REPORT
for samples collected from

YUMA SITE
YUMA, TEXAS

Data Validators: Karen Wong and Sandra Dover

INTRODUCTION

The following data validation summary report covers environmental soil and aqueous samples collected from Yuma Site on July 31, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00080031

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD), field duplicates and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000 for soil samples, and Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000 for water samples.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00080031 (soil/sediment)

General

This SDG consisted of twenty five (25) environmental soil samples, including seventeen (17) soils, two (2) field duplicate samples, two (2) laboratory duplicate samples and two (2) sets of MS/MSDs. The samples were collected on July 31, 2000 and analyzed for perchlorate. Sediment sample 01040302 contained an excessive amount of water. The laboratory decanted the water portion of the sample and analyzed it separately from the sediment portion.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 1, October 2000. There is no maximum holding time required by the method for soil samples.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. No samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on samples 01050302 and 01050904. There were two LCSs analyzed with this SDG.

All MS/MSD and LCS %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD values, and the laboratory duplicate values. Samples 01040305 and 01040306 were both the field duplicates of 01040304. Samples 01040303 and 01050901 were analyzed in duplicate as laboratory duplicates for this SDG.

All MS/MSD, field duplicate and laboratory duplicate RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 1, October 2000;
- Comparing actual analytical procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 1, October 2000; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 1, October 2000.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

Perchlorate SDG 00080031 (aqueous)

General

This SDG consisted of one (1) environmental aqueous sample. Sediment sample 01040302 contained an excessive amount of water. The laboratory decanted the water portion of the sample and analyzed it separately from the sediment portion. The sample was collected on July 31, 2000 and analyzed for perchlorate.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. The sample collected was prepared and analyzed within the holding times required by the method.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. No samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on samples 01010106 and 01010101 from another SDG (00080118). There were two LCSs analyzed with this SDG.

All MS/MSD and LCS %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD values, and the laboratory duplicate values. Sample 01010105 from another SDG (00080118) was analyzed in duplicate as a laboratory duplicate for this SDG.

All MS/MSD and laboratory duplicate RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;

- Comparing actual analytical procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Evaluating holding times; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. All samples were prepared and analyzed within the holding times required for the analysis.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT
for samples collected from

YUMA SITE
YUMA, TEXAS

Data Validator: Tammy Chang

INTRODUCTION

The following data validation summary report covers environmental aqueous samples collected from Yuma Site on July 31, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00080042

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD), field duplicates and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AIERA). Samples were analyzed by Clayton Group Services following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00080042 (aqueous)

General

This SDG consisted of twenty-three (23) environmental aqueous samples including sixteen (16) field samples, one (1) field duplicate sample, two (2) laboratory duplicate samples and two (2) sets of MS/MSD. All samples were collected on July 31, 2000 and analyzed for perchlorate.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. The sample collected was prepared and analyzed within the holding times required by the method.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. Clayton randomly selected sample 01040101 and 01030104 for MS/MSD analyses. There were four LCSs analyzed with this SDG.

All MS/MSD and LCS %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD values, field duplicate values and the laboratory duplicate values. Sample 01040105 and 01040106 were field duplicate of sample 01040101. Sample 01030102 and 01030105 were analyzed in duplicate as the laboratory duplicates for this SDG.

All MS/MSD and laboratory duplicate RPDs were within acceptance criteria. Sample 01040101 (parent sample) was detected with 4.1 μg/L of perchlorate and its duplicates, 01040105 and 01040106 were both non-detected with reporting limit of 4.0 μg/L. The RPD calculation could not be performed.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Comparing actual analytical procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Evaluating holding times; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. All samples were prepared and analyzed within the holding times required for the analysis.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT
for samples collected from

YUMA SITE
YUMA, TEXAS

Data Validators: Karen Wong and Sandra Dover

INTRODUCTION

The following data validation summary report covers environmental soil and aqueous samples collected from Yuma Site on August 1 and 2, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00080118

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD), field duplicates and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000 for soil samples, and Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000 for water samples.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00080118 (soil)

General

This SDG consisted of eighteen (24) environmental soil samples including fifteen (15) soil/sediment samples, three (3) field duplicates, two (2) laboratory duplicates, and two (2) sets of MS/MSD. The samples were collected on July 31, 2000 and analyzed for perchlorate. Sediment sample 01010301 contained an excessive amount of water. The laboratory decanted the water portion of the sample and analyzed it separately from the sediment portion. The results for the top portion of the samples were reported with the other aqueous field samples.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 1, October 2000. There is no maximum holding time required by the method for soil samples.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. No samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on samples 01010306 and 01010903. There were two LCSs analyzed with this SDG.

All MS/MSD and LCS %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD values, field duplicates and laboratory duplicate values. Sample 01010302 was the field duplicate of 01010301. Sample 01010902 was the field duplicate of 01010901. Sample 01010904 was the field duplicate of 01010903. Samples 01010901 and 01010902 were analyzed in duplicate as the laboratory duplicates for this SDG.

All MS/MSD, field duplicate and laboratory duplicate RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 1, October 2000;
- Comparing actual analytical procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 1, October 2000; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 1, October 2000.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

Perchlorate SDG 00080118 (aqueous)

General

This SDG consisted of thirteen (20) environmental aqueous samples including ten (10) environmental aqueous samples, three (3) field duplicates, two (2) laboratory duplicates, two (2) sets of MS/MSDs and one (1) aqueous "pour-off" layer from a sediment sample. The samples were collected on August 1 and 2, 2000 and analyzed for perchlorate. Sediment sample 01010301 contained an excessive amount of water. The laboratory decanted the water portion of the sample and analyzed it separately from the sediment portion.

The perchlorate analyses were performed following Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. All samples collected were prepared and analyzed within the holding times required by the method.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. No samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on samples 10101060 and 01010101. There were four LCSs analyzed with this SDG.

All MS/MSD and LCS %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD values, and the laboratory duplicate values. Samples 01020105 and 01020106 were both field duplicates of 01020104. Sample 01010102 was the field duplicate of 01010101. Samples 01010105 and 01020105 were analyzed in duplicate as the laboratory duplicates for this SDG.

All MS/MSD, field duplicate and laboratory duplicate RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Comparing actual analytical procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Evaluating holding times; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. All samples were prepared and analyzed within the holding times required for the analysis.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT
for samples collected from

YUMA SITE
YUMA, TEXAS

Data Validators: Michelle Wolfe and Sandra Dover

INTRODUCTION

The following data validation summary report covers environmental tissue samples collected from YUMA Site on July 31, August 1 and 2, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00100980

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD) and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000 for tissue samples.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00100980 (vegetation)

General

This SDG consisted of thirty-two (32) samples, including twenty-six (26) confirmation environmental vegetation samples and two (2) sets of matrix spike/matrix spike duplicate samples and two (2) laboratory duplicates. The samples were collected on July 31, August 1 and 2, 2000, 2000 and analyzed for perchlorate.

The perchlorate analyses were performed using Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000. There is no maximum holding time required by the method for the tissue samples.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples and two LCS samples. Samples 01041101 and 01041102 were used as the MS/MSD samples in this SDG.

All MS/MSD and LCS %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD results and the laboratory duplicate analyte values. Samples 01041101 and 01041102 were used as the MS/MSD samples in this SDG. Sample 01011201 and 01051103 were analyzed in duplicate as the laboratory duplicates for this SDG.

All MS/MSD and laboratory duplicate RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100.0% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000;

- Comparing actual analytical procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000; and
- Examining laboratory blanks for cross contamination of samples during the analysis.

All samples in this SDG were analyzed following chain-of-custody forms (COCs) and analytical procedures described in Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were two method blanks associated with the perchlorate analyses in this SDG. The method blanks were free of perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT
for samples collected from

LAKE MEAD
LAKE MEAD, NEVADA

Data Validator: Sandra Dover

INTRODUCTION

The following Data Validation Summary Report covers environmental soil and aqueous samples collected from Lake Mead Site on August 12, 13 and 14, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00080562

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD), field duplicates and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000 for soil samples, and Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000 for water samples.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00080562 (soil)

General

This SDG consisted of thirty-nine (48) environmental soil samples, including thirty seven (37) soil samples, two (2) field duplicates, three (3) laboratory duplicates and three (3) sets of MS/MSDs. The samples were collected on August 12, 13 and 14, 2000 and were analyzed for perchlorate.

Sediment samples 02050306, 02010307 UP and 02010301 DN contained an excessive amount of water. The laboratory decanted the water portion of the samples and the soil and water portions were then analyzed separately. The water portions were designated as 02050306 (TOP), 02010307 UP (TOP) and 02010301 DN (TOP) respectively to differentiate them from the associated soil 02050306, 02010307 UP and 02010301 DN respectively. The results for the top portion of the samples were reported with the other aqueous field samples

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000. There is no maximum holding time required by the method for soil samples.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. No samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on 02030905, 02010302 DN and 02010305. There were multiple LCSs analyzed with this SDG.

All LCS %Rs were within acceptance criteria.

All MS/MSD %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from the MS/MSD concentrations, and the field and laboratory duplicate values. Sample 02050306 was the field duplicate of sample 02050305. Sample 02010307 was the field duplicate of 02010306. Samples 02050905, 02010304 and 02030301 were analyzed in duplicate as the laboratory duplicates for this SDG.

All MS/MSD RPDs were within acceptance criteria.

Field duplicate sample 02050307 and the parent sample 02050306 were below detection limits for perchlorate. Field duplicate sample 02010307 and the parent sample 02010306 were outside of acceptable RPD limits. The perchlorate concentration for 02010307 was 120,000 ug/Kg and the concentration for 02010306 was 190,000 ug/Kg. The calculated % RPD was 45%, therefore the associated results are flagged "J".

All laboratory duplicate samples RPDs were within acceptance criteria except for the laboratory duplicate of 02050905 and 02030301. The %RPD for the duplicate analyses of sample 02050905 was 150%, which exceeds the specified tolerance. No corrective actions were required since the samples contained large amounts of rocks,

which likely contributed to the high %RPD noted. The %RPD for the duplicate analysis of sample 02030301 outside of acceptance limits. The parent sample concentration was below detection limits and the duplicate sample was 710 ug/Kg. The evaluation criteria used when comparing a below detection limit (ND) value and a value above the detection limit is any value more than twice the RL is considered outside of %RPD acceptance limits. The associated data for both the laboratory duplicate samples are flagged "J".

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000;
- Comparing actual analytical procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

Perchlorate SDG 00080562 (aqueous)

General

The aqueous portion of this SDG consisted of forty (40) environmental aqueous samples including twenty seven (27) aqueous samples, two (2) field duplicates, two (2) laboratory duplicates, three (3) sets of MS/MSDs and three (3) "pour-offs" from soil samples. The samples were collected on August 12,13 and 14, 2000 and were analyzed for perchlorate.

Sediment samples 02050306, 02010307 UP and 02010301 DN contained an excessive amount of water. The laboratory decanted the water portion of the samples and the soil and water portions were then analyzed separately. The water portions were designated as 02050306 (TOP), 02010307 UP (TOP) and 02010301 DN (TOP) respectively to differentiate them from the associated soil 02050306, 02010307 UP and 02010301 DN respectively.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. The samples were prepared and analyzed within hold time required by the method.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. No samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on sample 02030105, 02050121 and 02010103 DN. There were multiple LCSs analyzed with this SDG.

All LCS %Rs were within acceptance criteria.

All MS/MSD %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from the MS/MSD concentrations, and the field and laboratory duplicate values. Sample 02050107 was analyzed as the field duplicate of sample 02050106. Sample 02010107 was analyzed as the field duplicate of 02010106. Samples 02050104 and 02010104 UP were analyzed in duplicate as the laboratory duplicates for this SDG.

All MS/MSD RPDs were within acceptance criteria.

All field duplicate samples RPDs were within acceptance criteria.

All laboratory duplicate samples RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Comparing actual analytical procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Evaluating holding times; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. The samples were prepared and analyzed within the hold time required by the method.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the aqueous perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT
for samples collected from

LAKE MEAD
LAKE MEAD, NEVADA

Data Validators: Karen Wong and Sandra Dover

INTRODUCTION

The following data validation summary report covers aqueous samples collected from Lake Mead Site on August 15, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00080672

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD) and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000 for water samples.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00080672 (aqueous)

General

This SDG consisted of five (5) environmental aqueous samples, including two (2) aqueous samples, one (1) lab duplicate and one (1) set of MS/MSD. The samples were collected on August 15, 2000 and analyzed for perchlorate.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. The samples collected were prepared and analyzed within the holding times required by the method.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. The laboratory performed MS/MSD analyses on sample 02010131. Both samples and the MS/MSD required a 5000x dilution, and the laboratory spiked the MS/MSD samples after the dilutions were made. There were two LCSs analyzed with this SDG.

All MS/MSD and LCS %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD values, and the laboratory duplicate values. Sample 02010130 was analyzed in duplicate as the laboratory duplicate for this SDG.

All MS/MSD and laboratory duplicate RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Comparing actual analytical procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;

- Evaluating holding times; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. All samples were prepared and analyzed within the holding times required for the analysis.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT
for samples collected from

LAKE MEAD
LAKE MEAD, NEVADA

Data Validators: Michelle Wolfe and Sandra Dover

INTRODUCTION

The following data validation summary report covers environmental vegetation samples collected from Lake Mead Site on August 12, 13 and 14, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00100347

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD), field duplicates and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-70/N, Revision 1, November 2000 for vegetation samples.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00100347 (vegetation)

General

This SDG consisted of fifty-three (53) samples, including forty-two (42) confirmation environmental vegetation samples, three (3) sets of matrix spike/matrix spike duplicate samples, two (2) field duplicate samples and three (3) laboratory duplicate samples. The samples were collected on August 12, 13 and 14, 2000 and analyzed for perchlorate.

The perchlorate analyses were performed using Clayton Standard Operating Procedure (SOP), M-CC-70/N, Revision 1, November 2000. All samples for this SDG were analyzed following the procedures outlined in the SOP.

Claytons' SOP requires a LCCV at a concentration of 4 ppb be analyzed at the beginning of each run. Samples analyzed on 11/06/00 and 11/17/00 did not include a LCCV. All non-detected sample results are flagged with "UJ". All sample results with extract concentrations between 4 ppb and 10 ppb are flagged with "J".

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples and three LCS samples. Samples 02031006, 02051005 and 020110TA07 were used as the MS/MSD samples in this SDG.

The MS/MSD %Rs could not be calculated due to the high perchlorate concentration in the unspiked sample.

All LCS %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD results and the laboratory duplicate analyte values. Samples 02031006, 02051005 and 020110TA07 were used as the MS/MSD samples in this SDG. Samples 02031001, 02051002 and 020110TA04 were analyzed in duplicate as the laboratory duplicates for this SDG. Sample 02030404 was the field duplicate of sample 02030403. Sample 02010404 was the field duplicate of sample 02010403

The MS/MSD RPDs could not be calculated due to the high perchlorate concentration in the unspiked sample.

All laboratory duplicate RPDs were within acceptance criteria.

The field duplicate RPD was within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100.0% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-70/N, Revision 1, November 2000;
- Comparing actual analytical procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-70/N, Revision 1, November 2000; and
- Examining laboratory blanks for cross contamination of samples during the analysis.

All samples in this SDG were analyzed following chain-of-custody forms (COCs) and analytical procedures described in Clayton Standard Operating Procedure (SOP), M-CC-70/N, Revision 1, November 2000.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were three method blanks associated with the perchlorate analyses in this SDG. The method blanks were free of perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT
for samples collected from

LAKE MEAD
LAKE MEAD, NEVADA

Data Validators: Michelle Wolfe and Sandra Dover

INTRODUCTION

The following data validation summary report covers environmental tissue samples collected from Lake Mead Site on August 13, 14 and 15, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00101001

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD) and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000 for tissue samples.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00101001 (tissue)

General

This SDG consisted of twenty one (21) samples, including eighteen (18) environmental tissue samples one (1) set of matrix spike/matrix spike duplicate and one (1) laboratory duplicate sample. The samples were collected on August 13, 14 and 15, 2000 and analyzed for perchlorate.

The perchlorate analyses were performed using SOP M-CC-71/N. All samples for this SDG were analyzed following the procedures outlined in the SOP.

Claytons' SOP requires a LCCV at a concentration of 4 ppb be analyzed at the beginning of each run. Samples analyzed on 12/28/00, 01/03/01 and 01/10/01 did not include a LCCV. All non-detected sample results are flagged with "UJ". All sample results with extract concentrations between 4 ppb and 10 ppb are flagged with "J".

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD sample and one LCS sample. Sample 02031102 were used as the MS/MSD sample in this SDG.

The MS/MSD %Rs were within acceptance criteria

The LCS %R was within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD result and the laboratory duplicate analyte value. Sample 02031102 was used as the MS/MSD sample in this SDG. Sample 02011105 was analyzed in duplicate as the laboratory duplicate for this SDG.

The MS/MSD RPD was within acceptance criteria.

The laboratory duplicate RPD was within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100.0% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000;
- Comparing actual analytical procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000; and
- Examining laboratory blanks for cross contamination of samples during the analysis.

All samples in this SDG were analyzed following chain-of-custody forms (COCs) and analytical procedures described in Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There was one method blank associated with the perchlorate analyses in this SDG. The method blank was free of perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT
for samples collected from

HOLLOMAN SITE

Data Validator: Sandra Dover

INTRODUCTION

The following Data Validation Summary Report covers environmental soil and aqueous samples collected from Holloman Site on August 18 and 19, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00090666

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD), field duplicates and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000 for soil samples, and Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000 for water samples.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00090666 (soil)

General

This SDG consisted of seventy-five (75) environmental soil samples including fifty five (55) soil samples, five (5) field duplicates, three (3) laboratory duplicates and six (6) sets of MS/MSDs. The samples were collected on August 18 and 19, 2000 and were analyzed for perchlorate.

Sediment sample 04070301 contained an excessive amount of water. The laboratory decanted the water portion of the samples and the soil and water portions were then analyzed separately. The water portion was designated as 04070301 TOP to differentiate them from the associated soil portions. The results for the top portion of the samples were reported with the other aqueous field samples.

The calibration curve for the soil analysis performed on 10/07/00 includes only 4 calibration data points (4 ppb, 10 ppb, 50 ppb and 100 ppb). Clayton dropped the 20 ppb ICAL standard from the curve, due to possible preparation error. The medium level calibration check verification (MCCV) used for this batch was the 50 ppb CCV instead of the 20 ppb CCV.

Sample 04020902 was re-prepared and re-analyzed on April 2, 2001. The original data was rejected during data validation due to a possible sample identification error. The sample was analyzed undiluted.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000. There is no maximum holding time required by the method for soil samples.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. There were no samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on samples 04010303, 04020904 and 04010913. There were multiple LCSs analyzed with this SDG.

The LCS %Rs were within acceptance criteria. The MS/MSD for 04010303 and 04020904 were within acceptance criteria. After analyzing the extracts for the MS/MSD on sample 04010913, it was noticed that there was no perchlorate added by mistake. The batch was accepted based on the results for the LCS.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from the MS/MSD concentrations, the field duplicates and the laboratory duplicate values. Sample 04050304 was the field duplicate of 04050303. Sample 04070303 was the field duplicate of 04070304. Sample 04050904 was the field duplicate

of 04050903. Sample 04020904 was the field duplicate of 04020903. Sample 04070904 was the field duplicate of 04070903. Samples 04070303, 04060902 and 04010909 were analyzed in duplicate as the laboratory duplicates for this SDG.

The MS/MSDs RPDs for 04010303 and 04020904 were within acceptance criteria.

All field duplicate samples and their parent samples were ND, except for 04050304 had a slight perchlorate concentration of 340 µg/Kg-dry.

The laboratory duplicate sample and its parent sample for 04070303 were ND. For 04010909, the %RPD was 30.8%, which exceeds the laboratory specified tolerance. No data are flagged since the National Functional Guidelines (NFG) for Data Review require only duplicate samples with a %RPD greater than 70% to be flagged. The laboratory duplicate results for sample 04060902 was 1300 µg/Kg-dry and the sample duplicated was ND. No corrective actions were required since the samples appeared to have a high clay content with other debris (i.e. twigs, stones) present, which likely contributed to the high %RPD noted. The perchlorate results for the laboratory duplicates are flagged "J".

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in the Clayton SOP;
- Comparing actual analytical procedures to those described in the Clayton SOP; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in the Clayton SOP.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

Perchlorate SDG 00090666 (aqueous)

General

The aqueous portion of this SDG consisted of twenty-two (22) environmental aqueous samples including eighteen (18) aqueous samples, two (2) field duplicates, one (1) laboratory duplicate, one (1) set of MS/MSD (not analyzed, see below) and one (1) "pour-off" from a soil sample. The samples were collected on August 19, 2000 and were analyzed for perchlorate.

Sediment sample 04070301 contained an excessive amount of water. The laboratory decanted the water portion of the samples and the soil and water portions were then analyzed separately. The water portion was designated as 04070301 TOP to differentiate them from the associated soil portions.

All water samples were analyzed at a 1:100 dilution due to the high conductivity of the sample matrix. The reporting limits were raised accordingly to 400 µg/L on the report for the water samples.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. The samples were prepared and analyzed outside of the hold time required by the method. Due to the nature of this project (investigative), Parsons made the decision to consider the data acceptable even though the recommended hold time of 28 days was exceeded.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. No samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on sample 04060106, which was spiked with perchlorate and extracted although not analyzed (see explanation below). There were multiple LCSs analyzed with this SDG.

All LCS %Rs were within acceptance criteria.

Based on the necessary 1:20 dilution due to sample matrix for sample 04060106. The associated MS/MSD was not run. The laboratory determined based on the spike concentration that at a 1:20 dilution, the concentration of perchlorate would not have been above the reporting limit.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from the MS/MSD concentrations, and the field duplicate values. Sample 04050107 was the field duplicate of 04050106 and sample 04070107 was the field duplicate of 04070106 for this SDG. Sample 04070104 was analyzed in duplicate as the laboratory duplicate for this SDG. The laboratory performed MS/MSD analyses on sample 04060106, although they were not analyzed (see explanation above).

All field duplicate samples and their parent samples were ND.

The laboratory duplicate sample and it's parent sample were ND.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in the Clayton SOP;
- Comparing actual analytical procedures to those described in the Clayton SOP;
- Evaluating holding times; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in the Clayton SOP. The samples were prepared and analyzed outside of the hold time required by the method. Due to the nature of this project, Parsons made the decision to consider the data acceptable even though the recommended hold time of 28 days was exceeded.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the aqueous perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT
for samples collected from

HOLLOMAN SITE

Data Validators: Michelle Wolfe and Sandra Dover

INTRODUCTION

The following data validation summary report covers environmental tissue samples collected from Holloman Site during the period of September 14, 18, 19 and 20, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00101117

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD) and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000 for tissue samples.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00101117 (tissue)

General

This SDG consisted of fifty-seven (57) samples, including forty-eight (48) confirmation environmental tissue samples, three (3) laboratory duplicates and three (3) sets of matrix spike/matrix spike duplicate samples. The samples were collected on September 14, 18, 19 and 20 2000 and analyzed for perchlorate.

The perchlorate analyses were performed using SOP M-CC-71/N. All samples for this SDG were analyzed following the procedures outlined in the SOP.

Claytons' SOP requires a LCCV at a concentration of 4 ppb be analyzed at the beginning of each run. Samples analyzed on 01/09/01 and 01/12/01 did not include a LCCV. All non-detected sample results are flagged with "UJ". All sample results with extract concentrations between 4 ppb and 10 ppb are flagged with "J".

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD sample and three LCS samples. Samples 04060501, 04071103 and 06030508 were used as the MS/MSD sample in this SDG.

The MS/MSD %Rs were within acceptance criteria

The LCS %R was within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD result and the laboratory duplicate analyte values. Samples 04060501, 04071103 and 06030508 were used as the MS/MSD samples in this SDG. Samples 04050503, 04011104 and 06030507 were analyzed in duplicate as the laboratory duplicates.

The MS/MSD RPDs were within acceptance criteria.

The laboratory duplicate RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100.0% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000;
- Comparing actual analytical procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000; and
- Examining laboratory blanks for cross contamination of samples during the analysis.

All samples in this SDG were analyzed following chain-of-custody forms (COCs) and analytical procedures described in Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were three method blanks associated with the perchlorate analyses in this SDG. The method blanks were free of perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT
for samples collected from

ABL SITE
MARYLAND

Data Validator: Sandra Dover

INTRODUCTION

The following Data Validation Summary Report covers fish tissue and aqueous samples collected from ABL Site on June 13, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

000605111

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD) and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000 for water samples and Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00060511 (tissue)

General

This SDG consisted of ten (11) fish tissue samples including nine (9) fish tissue samples, one (1) lab duplicate and one (1) fish egg composite. The tissues were sent to Covance, Inc. on June 27, 2000 for sample preparation and percent moisture determination. Clayton received the prepared tissue samples on July 29, 2000 for perchlorate analysis.

Fish eggs were removed by Covance, Inc. from samples 03040502, 03040506, 03040507 and 03040509 and were composited to comprise sample, Fish Egg Composite.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000. There is no maximum holding time required by the method for tissue samples.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. There were no samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on sample 03040505. There was one LCS analyzed with this SDG.

The MS/MSD and LCS %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from the MS/MSD concentrations, and the laboratory duplicate values. Sample 03040509 was analyzed in duplicate as the laboratory duplicate for this SDG.

The laboratory duplicate RPD was within acceptance criteria.

The MS/MSD RPD was outside laboratory specified tolerance. The %RPD for the MS/MSD on sample 03040505 was 37%. The parent sample, MS and MSD were all reanalyzed for confirmation of the original results. The MS %R was within acceptance limits, although the MSD fell outside of acceptance limits. The RPD for the reanalysis remained outside of laboratory tolerance. The original data was reported. Since the %RPD was within NFG guidelines of 70%, no data were flagged.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000;
- Comparing actual analytical procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in Clayton Standard Operating Procedure (SOP), M-CC-71/N, Revision 0, October 2000.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There was one method blank associated with the perchlorate analyses in this SDG. The blank was free of any perchlorate above the RL.

Perchlorate SDG 00060511 (aqueous)

General

The aqueous portion of this SDG consisted six (6) environmental aqueous samples including three (3) aqueous samples, one (1) laboratory duplicate and one (1) set of MS/MSDs. The samples were collected on June 13, and were analyzed for perchlorate.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. The samples were prepared and analyzed within the hold time required by the method.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. No samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on sample 03040102. There were multiple LCSs analyzed with this SDG.

All LCS %Rs were within acceptance criteria.

The MS/MSD %R was within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from the MS/MSD concentrations, and the lab duplicate value. Sample 03040103 was analyzed in duplicate as the laboratory duplicate for this SDG. The laboratory performed MS/MSD analyses on sample 03040102.

The laboratory duplicate sample RPD was within acceptance criteria.

The MS/MSD RPD was within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Comparing actual analytical procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Evaluating holding times; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. The samples were prepared and analyzed within the hold time required by the method.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the aqueous perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT
for samples collected from

ABL SITE
MARYLAND

Data Validators: Karen Wong and Sandra Dover

INTRODUCTION

The following Data Validation Summary Report covers environmental soil and aqueous samples collected from ABL Site on August 22 and 23, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00081023

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD), field duplicates and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000 for soil samples, and Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000 for water samples.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Appendix E

Data Validation Summary Reports

PERCHLORATE ANALYSIS BY CLAYTON GROUP SERVICES

DATA VALIDATION SUMMARY REPORTS
AND
DATA VERIFICATION REPORTS

Part I

The following Sample Delivery Groups (SDGs) were validated by scientists at Parsons ES.

Yuma Site: 00080031, 00080042, 00080118 and 00100980
Lake Mead Site: 00080562, 00080672, 00100347 and 00101001
Holloman Site: 00090666 and 00101117
ABL Site: 00060511, 00081023 and 00100349
Indian Head Site: 00081123 and 00100350
Longhorn Site: 00090393

The results are included in the Data Validation Reports provided. Data validation includes an extensive review of Chain of Custody (COC), case narrative, analytical sample results reports, quality control reports, preparation summaries, calibration curves, instrument sequence files, raw data and manual integrations.

All data are considered usable.

Part II

The following Sample Delivery Groups (SDGs) were verified by scientists at Parsons ES.

Yuma Site: 00100344, 00100345 and 00100983
Lake Mead Site: 00100987 and 00100999
Holloman Site: 00100679 and 00101115
ABL Site: 00101003
Indian Head Site: 00101005, 00101007 and 00101009
Longhorn Site: 00110360

Verification includes a general overview of the SDGs including review of the Chain of Custody (COC), case narrative, analytical sample results reports, quality control reports and calibration curves. A Verification Checklist is completed and stored with each the SDG data package.

All data are considered usable.

PART I

DATA VALIDATION SUMMARY REPORT
for samples collected from

YUMA SITE
YUMA, TEXAS

Data Validators: Karen Wong and Sandra Dover

INTRODUCTION

The following data validation summary report covers environmental soil and aqueous samples collected from Yuma Site on July 31, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00080031

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD), field duplicates and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000 for soil samples, and Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000 for water samples.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00080031 (soil/sediment)

General

This SDG consisted of twenty five (25) environmental soil samples, including seventeen (17) soils, two (2) field duplicate samples, two (2) laboratory duplicate samples and two (2) sets of MS/MSDs. The samples were collected on July 31, 2000 and analyzed for perchlorate. Sediment sample 01040302 contained an excessive amount of water. The laboratory decanted the water portion of the sample and analyzed it separately from the sediment portion.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 1, October 2000. There is no maximum holding time required by the method for soil samples.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. No samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on samples 01050302 and 01050904. There were two LCSs analyzed with this SDG.

All MS/MSD and LCS %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD values, and the laboratory duplicate values. Samples 01040305 and 01040306 were both the field duplicates of 01040304. Samples 01040303 and 01050901 were analyzed in duplicate as laboratory duplicates for this SDG.

All MS/MSD, field duplicate and laboratory duplicate RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 1, October 2000;
- Comparing actual analytical procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 1, October 2000; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 1, October 2000.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

Perchlorate SDG 00081023 (soil)

General

This SDG consisted of sixty-one (61) environmental soil samples including forty two (42) soil samples, four (4) field duplicates, five (5) laboratory duplicates and five (5) sets of MS/MSDs. The samples were collected on August 22 and 23, 2000 and were analyzed for perchlorate.

Sediment samples 03060301 and 03060303 contained an excessive amount of water. The laboratory decanted the water portion of the samples and the soil and water portions were then analyzed separately. The water portions were designated as 03060301 TOP and 03060303 TOP respectively to differentiate them from the associated soil portions. The results for the top portion of the samples were reported with the other aqueous field samples.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000. There is no maximum holding time required by the method for soil samples.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. There were no samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on samples 03050901, 03080903, 03060301, 03080304, and 03010910. There were multiple LCSs analyzed with this SDG.

All MS/MSD and LCS %Rs were within acceptance criteria. It should be noted that the MS of sample 03010910 was spiked twice with spiking solution. The percent recovery calculation for the MS was modified to take the double-spike into account.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from the MS/MSD concentrations, and the laboratory duplicate values. Sample 03060304 was the field duplicate for 03060303. Sample 03080304 was the field duplicate for 03080303. Sample 03010910 was the field duplicate of 0310909. Sample 03060904 was the field duplicate of 03060903. Samples 03050302, 03080302, 03010905, 03020905 and 03060903 were analyzed as in duplicate as the laboratory duplicates for this SDG.

All MS/MSDs RPDs were within acceptance criteria.

All field duplicate samples RPDs were within acceptance criteria.

All laboratory duplicate samples RPDs were within acceptance criteria except for the laboratory duplicate of 03010905. The %RPD for the duplicate analyses of sample 03010905 was 30%, which exceeds the specified tolerance. No corrective actions were required since the samples contained large amounts of rocks, which likely contributed to

the high %RPD noted. The perchlorate results for the laboratory duplicates are flagged "J".

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000;
- Comparing actual analytical procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

Perchlorate SDG 00081023 (aqueous)

General

The aqueous portion of this SDG consisted of twenty-five (25) environmental aqueous samples including eighteen (18) aqueous samples, one (1) field duplicate, two (2) laboratory duplicates, one (1) set of MS/MSD and two (2) "pour-offs" from soil samples. The samples were collected on August 22 and 23, 2000 and were analyzed for perchlorate.

Sediment samples 03060301 and 03060303 contained an excessive amount of water. The laboratory decanted the water portion of the samples and the soil and water portions were then analyzed separately. The water portions were designated as 03060301 TOP and 03060303 TOP respectively to differentiate them from the associated soil portions.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. The samples were prepared and analyzed outside of the hold time required by the method. Due to the nature of this project (investigative), Parsons made the decision to consider the data acceptable even though the recommended hold time of 28 days was exceeded.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. No samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on sample 03060301 TOP. Samples 03080106, and 03060104 were spiked with perchlorate and extracted although not analyzed (see explanation below). There were multiple LCSs analyzed with this SDG.

All LCS %Rs were within acceptance criteria.

The MS/MSD was within acceptance criteria for %Rs for sample 03060301 TOP. Based on the necessary 1:20 dilution due to sample matrix for samples 03080106 and 03060104, the associated MS/MSD were not run. The laboratory determined based on the spike concentration that at a 1:20 dilution, the concentration of perchlorate would not have been above the reporting limit.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from the MS/MSD concentrations, and the field duplicate values. Sample 03060107 was the field duplicate of 03060106. Samples 03050105 and 03080102 were analyzed in duplicate as the laboratory duplicates for this SDG. The laboratory performed MS/MSD analyses on sample 03060301 TOP.

All MS/MSD RPDs were within acceptance criteria.

The field duplicate sample RPDs were within acceptance criteria.

All laboratory duplicate samples RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Comparing actual analytical procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Evaluating holding times; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. The samples were prepared and analyzed outside of the hold time required by the method. Due to the nature of this project, Parsons made the decision to consider the data acceptable even though the recommended hold time of 28 days was exceeded.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the aqueous perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT
for samples collected from

ABL SITE
MARYLAND

Data Validators: Karen Wong and Sandra Dover

INTRODUCTION

The following data validation summary report covers environmental vegetation samples collected from ABL Site on August 22 and 23, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00100349

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD) and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-70/N, Revision 1, November 2000 for vegetation samples.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00100349 (vegetation)

General

This SDG consisted of thirty-seven (39) samples, including thirty-three (33) confirmation environmental vegetation samples and two (2) sets of matrix spike/matrix spike duplicate samples, and two (2) laboratory duplicates. The samples were collected on August 22 and 23, 2000 and analyzed for perchlorate.

The perchlorate analyses were performed using Clayton Standard Operating Procedure (SOP), M-CC-70/N, Revision 1, November 2000. All samples for this SDG were analyzed following the procedures outlined in the SOP.

Claytons' SOP requires a LCCV at a concentration of 4 ppb be analyzed at the beginning of each run. Samples analyzed on 11/27/00 did not include a LCCV. All non-detected sample results are flagged with "UJ". All sample results with extract concentrations between 4 ppb and 10 ppb are flagged with "J".

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples and two LCS samples. Samples 030504099 and 03031004 were used as the MS/MSD samples in this SDG.

All MS/MSD and LCS %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD results and the laboratory duplicate analyte values. Samples 030504099 and 03031004 were used as the MS/MSD samples in this SDG. Samples 03011004 and 03050406 were analyzed in duplicate as the laboratory duplicates for this SDG.

All MS/MSD and laboratory duplicate RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100.0% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-70/N, Revision 1, November 2000;
- Comparing actual analytical procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-70/N, Revision 1, November 2000; and
- Examining laboratory blanks for cross contamination of samples during the analysis.

All samples in this SDG were analyzed following chain-of-custody forms (COCs) and analytical procedures described in Clayton Standard Operating Procedure (SOP), M-CC-70/N, Revision 1, November 2000.

- All instrument performance check criteria were met.
- All initial calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a secondary source standard.
- Most of the continuing calibration criteria were met except for as follows:

Date	CCV ID	Analyte	%D	QC
11/9/00	MCCV	perchlorate	118.6	85-115

No action was taken since the LCS and MS/MSD results were compliant. The non-compliant calibration was the ending calibration and all other calibrations were compliant. All the sample results were non-detect.

There were two method blanks associated with the perchlorate analyses in this SDG. The method blanks were free of perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT
for samples collected from

INDIAN HEAD SITE

Data Validators: Karen Wong and Sandra Dover

INTRODUCTION

The following data validation summary report covers environmental soil/sediment and aqueous samples collected from Indian Head on August 26, 27 and 28, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00081123

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD), field duplicates and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000 for soil samples, and Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000 for water samples.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00081123 (soil/sediment)

General

This SDG consisted of fifty-two (52) environmental soil/sediment samples including forty (36) soil/sediment samples, four (4) field duplicates, four (4) laboratory duplicates and four (4) sets of MS/MSDs. The samples were collected on August 26, 27 and 28, 2000 and analyzed for perchlorate. Sediment samples 05030303 and 05070302 contained an excessive amount of water. The laboratory decanted the water portion of the samples and analyzed them separately from the sediment portion. The results for the top portion of the samples were reported with the other aqueous field samples.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000 for soil samples. There is no maximum holding time required by the method for soil samples.

Sample 05030303 BOTTOM was first analyzed straight and contained a large peak. Sample 05030305 was run immediately following and contained a peak within the perchlorate window. Sample 05030303 BOTTOM was reanalyzed at a 100x dilution and perchlorate was not detected. Sample 05030305 was reanalyzed at a 10x dilution and then reanalyzed straight again. Perchlorate was not detected in either of the runs. According to the laboratory if the peak in the original analysis of 05030303 BOTTOM had been perchlorate, it would have been detected in the 100x dilution. Therefore the peak in the original analyses of 05030303 BOTTOM can be attributed to matrix affect and the peak in the original analyses of 05030305 was due to the carryover from the previous injection (05030303 BOTTOM).

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. No samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on samples 05070902, 05050302, 05080303, and 05080903. There were multiple LCSs analyzed with this SDG. One LCS, with the id of "LCS filtered", associated with filtered one extra time at the end of the preparation procedure. The extra filtration was incorporated due to a visible particulate and/or coloration present in the associated samples.

All MS/MSD and LCS %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD values, field duplicate values and the laboratory duplicate values. Samples 05070902, 05050302, 05080303 and 05080903 were analyzed as the MS/MSDs for this SDG. Sample 05040905 was the field duplicate of 05040903. Sample 05030305 was the field duplicate of 05030303 (bottom). Sample 05040304 was the field

duplicate of 05040303. Samples 05060301, 05040901, 05050901, and 05040301 were analyzed in duplicate as the laboratory duplicates for this SDG.

All MS/MSD, field duplicate and laboratory duplicate RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000;
- Comparing actual analytical procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a secondary source standard.

There were multiple method blanks associated with the perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

Perchlorate SDG 00081123 (aqueous)

General

This SDG consisted of fifty one (51) environmental aqueous samples including thirty six (36) environmental samples, three (3) field duplicates, four (4) laboratory duplicates, three (3) sets of MS/MSDs and two (2) aqueous "pour-off" layers from sediment samples. The samples were collected on August 26, 27 and 28, 2000 and analyzed for perchlorate. Sediment samples 05030303 and 05070302 contained an excessive amount of water. The laboratory decanted the water portion of the sampled and analyzed them separately from the sediment portion.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000 for the water samples. All samples collected were prepared and analyzed within the holding times required by the method.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. No samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on samples 05050103, 05080101, and 05040108. There were four LCSs analyzed with this SDG.

All MS/MSD and LCS %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD values, field duplicate values and the field duplicate values. Samples 05050103, 05080101 and 05040108 were analyzed as the MS/MSDs for this SDG. Sample 05070108 was the field duplicate of 05070106. Sample 05030107 was the field duplicate of 05030106. Sample 05040108 was the field duplicate of 05040106. Samples 05030101, 05040103, 05060102, and 05070105 were analyzed in duplicate as the laboratory duplicates for this SDG.

All MS/MSD, field duplicate and lab duplicate RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Comparing actual analytical procedures to those described in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Evaluating holding times; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. All samples were prepared and analyzed within the holding times required for the analysis.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a secondary source standard.

There were four method blanks associated with the perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT
for samples collected from

INDIAN HEAD SITE

Data Validators: Karen Wong and Sandra Dover

INTRODUCTION

The following data validation summary report covers environmental vegetation samples collected from Indian Head Site on August 26, 27 and 28, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00100350

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD) and laboratory duplicates. The Quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-70/N, Revision 1, November 2000 for vegetation samples.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00100350 (vegetation)

General

This SDG consisted of thirty-three (33) samples, including twenty-seven (27) confirmation environmental vegetation samples and two (2) sets of matrix spike/matrix spike duplicate samples and two (2) laboratory duplicates. The samples were collected on August 26, 27 and 28, 2000 and analyzed for perchlorate.

The perchlorate analyses were performed using SOP M-CC-69/N. All samples for this SDG were analyzed following the procedures outlined in the SOP.

Claytons' SOP requires a LCCV at a concentration of 4 ppb be analyzed at the beginning of each run. Samples analyzed on 11/14/00 did not include a LCCV. All non-detected sample results are flagged with "UJ". All sample results with extract concentrations between 4 ppb and 10 ppb are flagged with "J".

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples and two LCS samples. Samples 05031001 and 05040403 were used as the MS/MSD samples in this SDG.

All MS/MSD and LCS %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD results and the laboratory duplicate analyte values. Samples 05031001 and 05040403 were used as the MS/MSD samples in this SDG. Samples 05071001 and 05040401 were analyzed in duplicate as the laboratory duplicates for this SDG.

All MS/MSD and laboratory duplicate RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100.0% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-70/N, Revision 1, November 2000;
- Comparing actual analytical procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-70/N, Revision 1, November 2000; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following chain-of-custody forms (COCs) and analytical procedures described in Clayton Standard Operating Procedure (SOP), M-CC-70/N, Revision 1, November 2000.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a secondary source standard.

There were two method blanks associated with the perchlorate analyses in this SDG. The method blanks were free of perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT
for samples collected from

LONGHORN SITE
TEXAS

Data Validator: Tammy Chang

INTRODUCTION

The following Data Validation Summary Report covers environmental soil/sediment and aqueous samples collected from Longhorn, Texas on September 11 and 12, 2000. The samples in the following laboratory Sample Delivery Group (SDG) were analyzed for perchlorate:

00090393

Quality control samples collected were matrix spike/matrix spike duplicates (MS/MSD), field duplicates and laboratory duplicates. The quality control samples were analyzed for the same parameters as their associated samples.

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by Clayton Group Services laboratory following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000 for soil samples, and Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000 for water samples.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate SDG 00090393 (soil/sediment)

General

This SDG consisted of thirty-four (34) environmental soil samples including twenty-five (25) soil/sediment samples, three (3) field duplicates, two (2) laboratory duplicates and two (2) sets of MS/MSDs. The samples were collected on September 11 and 12, 2000 and were analyzed for perchlorate.

Sediment samples 06050304 contained an excessive amount of water. The laboratory decanted the water portion of the samples and the soil and water portions were then analyzed separately. The water portion was designated as 06050304 TOP and the solid portion was designated as 06050304 BOTTOM to differentiate them. The results for the top portion of the samples were reported with the other aqueous field samples.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000. There is no maximum holding time required by the method for soil samples.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. There were no samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on samples 06040903 and 06020303. There were two LCSs analyzed with this SDG.

All MS/MSD and LCS %Rs were within acceptance criteria

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from the MS/MSD values, field duplicate values, and the laboratory duplicate values. Sample 06050904 is the field duplicate of sample 06050903. Sample 06050304 BOTTOM is the field duplicate of sample 06050303. Sample 06040304 is the field duplicate of sample 06040303. Samples 06030301 and 06030901 were analyzed in duplicate as laboratory duplicates for this SDG.

All MS/MSDs RPDs were within acceptance criteria.

All field duplicate samples RPDs were within acceptance criteria.

All laboratory duplicate samples RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000;
- Comparing actual analytical procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

Perchlorate SDG 00090393 (aqueous)

General

The aqueous portion of this SDG consisted of thirty-three (33) environmental aqueous samples including twenty-five (25) aqueous samples, two (2) field duplicate, two (2) laboratory duplicates, two (2) set of MS/MSD. The samples were collected on September 11 and 12, 2000 and were analyzed for perchlorate.

Sediment samples 06050304 contained an excessive amount of water. The laboratory decanted the water portion of the samples and the soil and water portions were then analyzed separately. The water portion was designated as 06050304 TOP and the solid portion was designated as 06050304 BOTTOM to differentiate them.

The perchlorate analyses were performed following the Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. Due to the nature of this project (investigative), Parsons made the decision to consider the data acceptable even though the recommended hold time of 28 days was exceeded.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD samples, and LCS samples. No samples were designated by the sampling team for MS/MSD analyses. The laboratory performed MS/MSD analyses on sample 06030102 and 06040101. There were six LCSs analyzed with this SDG.

All LCS %Rs were within acceptance criteria.

All MS/MSD %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from the MS/MSD concentrations, and the field duplicate values. Sample 06050108 is the field duplicate of sample 06050106. Sample 06020104 is the field duplicate of sample 06040107 for this SDG. Samples 06020104 and 06050105 were analyzed in duplicate as laboratory duplicates for this SDG.

All MS/MSD RPDs were within acceptance criteria.

The field duplicate sample RPDs were within acceptance criteria.

All laboratory duplicate samples RPDs were within acceptance criteria.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Comparing actual analytical procedures to those described in Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000;
- Evaluating holding times; and
- Examining laboratory blanks for cross contamination of samples during analysis.

All samples in this SDG were analyzed following the chain-of-custody (COC) and analytical procedures described in Clayton Standard Operating Procedure (SOP), M-CC-68/N, Revision 2, June 2000. The samples were prepared and analyzed outside of the hold time required by the method. All initial and continuing calibration criteria were met.

- All instrument performance check criteria were met.
- All initial and continuing calibration criteria were met.
- All second source calibration criteria were met. The initial calibration verification (ICV) was prepared from a second source standard.

There were multiple method blanks associated with the aqueous perchlorate analyses in this SDG. All blanks were free of any perchlorate above the RL.

DATA VALIDATION SUMMARY REPORT

FOR PERCHLORATE SAMPLES SPLIT

WITH CLAYTON GROUP SERVICES

Data Validator: Sandra Dover

INTRODUCTION

The following data validation summary report covers soils, sediments, tissues, environmental vegetation and water samples collected from Yuma, Lake Mead, Holloman, ABL, Indian Head and Longhorn Sites between August 31, 2000 through September 19, 2000. The samples in the following Analytical Request Form (ARF) were analyzed for perchlorate:

34274

All samples were collected by Parsons Engineering Science (Parsons ES) and Air Force Institute for Environment, Safety, and Occupational Health Risk Analysis (AFIERA). Samples were analyzed by APPL, INC. following the procedures outlined in the SOP.

EVALUATION CRITERIA

The data submitted by the laboratory has been reviewed and validated following the guidelines outlined in the Unites States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994). Information reviewed in the data packages include sample results; the summary of laboratory quality control results; case narrative; raw data; and chain-of-custody forms. The analyses and findings presented in this report are based on the reviewed information, and whether guidelines in the United States Environmental Protection Agency (USEPA) National Functional Guidelines (NFG) for Data Review (February 1994) were met.

Perchlorate ARF 34274
General

This ARF consisted of forty-eight (48) samples, including twenty-two (22) tissue samples, ten (10) environmental vegetation samples, six (6) water samples, six (6) sediment samples and four (4) soil samples. The laboratory assigned one matrix spike/matrix spike duplicate sample. The samples were collected between August 31 and September 19, 2000 and analyzed for perchlorate.

The perchlorate analyses were performed using USEPA Method 310.0 Revision 1.0 (November 1999), Determination of Perchlorate in Drinking Water Using Ion Chromatography. All samples for this ARF were analyzed following the procedures outlined in this SOP.

Accuracy

Accuracy was evaluated using the %R results for the MS/MSD sample and four LCS samples. Aqueous sample 06020101 was used as the MS/MSD sample in this ARF.

The MS/MSD %Rs were within acceptance criteria.

All LCS %Rs were within acceptance criteria.

Precision

Precision was evaluated using the Relative Percent Difference (RPD) results obtained from MS/MSD results. Sample 06020101 was used as the MS/MSD sample in this ARF. All samples analyzed were splits shared with Clayton Group Services. The %RPD between the duplicate field samples are provided in a table format attached to this report.

The MS/MSD %RPD was within acceptance criteria.

Comparisons between APPLs' and Claytons' data are listed following this report.

Completeness

Completeness has been evaluated by comparing the total number of samples collected with the total number of samples with valid analytical data.

All results were considered usable. The completeness for this SDG is 100.0% compared to the minimum acceptance limit of 90%.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents actual site conditions. Representativeness has been evaluated by:

- Comparing the chain-of-custody procedures to those described in the SOP;
- Comparing actual analytical procedures to those described in the SOP; and
- Evaluation of hold time; samples were not delivered to APPL until the sample hold times (28 days for waters, none for all other matrices) had expired. The expiration of the water samples is not considered significant due to the research nature of the project.
- Examining laboratory blanks for cross contamination of samples during the analysis.

All samples in this SDG were analyzed following chain-of-custody forms (COCs) and analytical procedures described in the SOP.

- All instrument performance check criteria were met.
- All initial calibration criteria were met.
- All continuing calibration criteria were met.

There were three solid method blanks and one water method blank associated with the perchlorate analyses in this ARF. The method blanks were free of perchlorate above the RLs.

Split Sample Comparison Table

Parson's Sample ID	APPL's Results	Clayton's Results	Units	% RPD APPL and Clayton wet wt
6020903	<5	<4.85	ug/kg	
3050301	<5	<80	ug/kg	
3080301	<5	<79.2	ug/kg	
2010902	82900	83300	ug/kg	0.48
02010302 (UP)	20800	23000	ug/kg	10
1050901	<10	<79.7	ug/kg	
5050303	<5	<190	ug/kg	
6021102	<100	<410	ug/kg	
2010502	4220	4310	ug/kg	2.1
4070502	5450	<408	ug/kg	
6020101	3130	3600	ug/L	14
1050101	<5	<4	ug/L	
3080101	210	270	ug/L	25
3050101	204	240	ug/L	16
02010102 (UP)	81200	100000	ug/L	21

Parson's Sample ID	APPL's Results	Clayton's Results	Units	% RPD APPL and Clayton wet wt
1050301	<5	<81	ug/kg	
1010903	96	90	ug/kg	6.5
6020301	9.1	286	ug/kg	188
4070401	<100	<416	ug/kg	
3050404	<100	<396	ug/kg	
6020401	17700	11000	ug/kg	47
010410AF01	<500	650	ug/kg	
4071001	<50	<416	ug/kg	
020110TA05	18800	14200	ug/kg	28
2010402	137000	121000	ug/kg	12
6021001	3310	2460	ug/kg	29
6020404	2470	2160	ug/kg	13
010504CT01	<50	<371	ug/kg	
5050702	<50	<388	ug/kg	
1051102	<50	<400	ug/kg	
3051101	<50	<417	ug/kg	
6021301	<50	<385	ug/kg	
1041102	<50	<408	ug/kg	
1011103	<50	<386	ug/kg	
6020601	<100	624	ug/kg	
2011204	<50	<383	ug/kg	
1051201	<50	<390	ug/kg	
010408CT01	785	<400	ug/kg	
5050508	<50	<394	ug/kg	
5050514	<50	<412	ug/kg	
3081302	<50	<388	ug/kg	
6021202	<50	<384	ug/kg	
2031302	1900	3200	ug/kg	51
3050702	721	<417	ug/kg	
6020703	900	1660	ug/kg	59
1051302	296	<392	ug/kg	
2011101	12400	15100	ug/kg	20

* For calculation comparison purposes only, all results for perchlorate are reported by wet weight.
%RPD was only calculated when both results are greater than the RL.
Note: Highlighted rows represent sample results with high variability; although %RPD wasn't calculated.

PART II

GENERAL OVERVIEW OF DATA VERIFICATION

Data packages for the following twelve SDGs were verified by Parsons ES:

Yuma Site: 00100344, 00100345 and 00100983
Lake Mead Site: 00100987 and 00100999
Holloman Site: 00100679 and 00101115
ABL Site: 00101003
Indian Head Site: 00101005, 00101007 and 00101009
Longhorn Site: 00110360

All samples were collected by Parsons Engineering Science (Parsons ES) and analyzed by Clayton Group Services following procedures outlined in the Clayton Standard Operating Procedure (SOP), M-CC-69/N, Revision 2, October 2000 for soil samples, M-CC-71/N, Revision 0, October 2000 for animal tissue samples, M-CC-70/N, Revision 1, November 2000 for vegetation samples and M-CC-68/N, Revision 2, June 2000 for water samples.

Information verified in the data packages include Chain of Custody (COC), case narrative, analytical sample results reports, quality control reports and calibration curves.

All method blanks (MB) were free of any perchlorate above the RL. All laboratory control samples (LCS) were within acceptance criteria. All initial and continuing calibration criteria were met for all the verified SDGs. All initial calibration verifications (ICVs), prepared from a secondary source standard, were met.

Claytons' SOPs require a low –level continuing calibration verification (LCCV) at a concentration of 4 ppb be analyzed at the beginning of each run. The following SDGs contained sample analysis on daily analytical runs in which the LCCV was not analyzed.

Yuma Site: 00100983
Lake Mead Site: 00100987
Holloman Site: 00100679 and 00101115
ABL Site: 00101003
Indian Head Site: 00101005, 00101007 and 00101009
Longhorn Site: 00110360

All non-detected sample results are flagged with "UJ". All sample results with extract concentrations between 4 ppb and 10 ppb are flagged with "J".

No additional flags were applied as a result of Parsons' verification, except for the "UJ" and "J". All data are considered usable.

Appendix F

Analytical Results

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
1 Yuma, AZ												
	/	01010102	Surface Water	FD				5.60		4.00		
	/	01010102	Surface Water	N				4.80		4.00		
	/	01010103	Surface Water	N				4.90		4.00		
	/	01010104	Surface Water	N				4.60		4.00		
	/	01010105	Surface Water	N				4.90		4.00		
	/	01010106	Surface Water	N				4.90		4.00		
	/	01010301 (TOP)	Pore Water	N				2.00		4.00		J
	/	01010302	Sediment	N			ND	ND	80.3	110	27	
	/	01010302	Sediment	FD			ND	ND	81.4	110	26	
	/	01010303	Sediment	N			ND	ND	80.0	100	20	
	/	01010304	Sediment	N			ND	ND	80.3	110	27	
	/	01010305	Sediment	N			ND	ND	81.6	120	32	
	/	01010306	Sediment	N			ND	ND	81.4	110	26	
	/	01010401	Plant Tissue	N	Sago	*Potamogeton pectinatus*	260	2000	390	3000	87	
	/	01010402	Plant Tissue	N	Sago	*Potamogeton pectinatus*	ND	ND	390	3000	87	
	/	01010403	Plant Tissue	N	Sago	*Potamogeton pectinatus*	ND	ND	396	3300	88	
	/	01010501	Animal Tissue	N	Minnows	Family Cyprinidae	ND	ND	392	1510	74	UJ
	/	01010901	Soil	FD			ND	ND	80.4	81.0	0.8	
	/	01010901	Soil	N			ND	ND	79.9	81.0	1.4	
	/	01010903	Soil	FD			ND	ND	79.8	81.0	1.5	
	/	01010903	Soil	N			90.0	93.0	80.3	83.0	3.2	
	/	01010905	Soil	N			ND	ND	79.7	80.0	0.4	
	/	01010906	Soil	N			ND	ND	79.8	80.0	0.3	
	/	01011001	Plant Tissue	N	Alfalfa	*Medicago sativa*	1010	4400	391	1700	77	
	/	01011002	Plant Tissue	N	Arrowweed	*Pluchea sericea*	342	900	380	1000	62	
	/	01011003	Plant Tissue	N	Arrowweed	*Pluchea sericea*	333	900	407	1100	63	
	/	01011004	Plant Tissue	N	Arrowweed	*Pluchea sericea*	456	1200	418	1100	62	
	/	01011005	Plant Tissue	N	Honey mesquite	*Prosopis glandulosa*	333	900	407	1100	63	
	/	01011006	Plant Tissue	N	Honey mesquite	*Prosopis glandulosa*	ND	ND	397	640	38	

RL = reporting limit.
ND = not detected above the reporting limit.
N = normal sample, FD = field duplicate, and FT = field triplicate
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Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
Reported results less than RLs are estimated values.
13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	1	01011007	Plant Tissue	N	Salt cedar leaf	*Tamarix chinensis*	320	800	400	1000	60	
	1	01011007	Plant Tissue	FD	Salt cedar leaf	*Tamarix chinensis*	555	1500	407	1100	63	
	1	01011009	Plant Tissue	N	Salt cedar leaf	*Tamarix chinensis*	528	1600	396	1200	67	
	1	01011010	Plant Tissue	N	Salt cedar leaf	*Tamarix chinensis*	840	2000	391	930	58	
	1	01011011	Plant Tissue	N	Quail bush	*Atriplex lentiformis*	884	2600	408	1200	66	
	1	01011101	Animal Tissue	N	Cactus mouse	*Peromyscus eremicus*	ND	ND	400	1330	70	
	1	01011102	Animal Tissue	N	Cactus mouse	*Peromyscus eremicus*	ND	ND	421	1450	71	
	1	01011103	Animal Tissue	N	Rock pocket mouse	*Chaetodipus intermedius*	ND	ND	386	1490	74	
	1	01011104	Animal Tissue	N	Rock pocket mouse	*Chaetodipus intermedius*	ND	ND	400	1430	72	
	1	01011105	Animal Tissue	N	Rock pocket mouse	*Chaetodipus intermedius*	ND	ND	396	1410	72	
	1	01011201	Animal Tissue	N	Mourning dove	*Zenaida macroura*	ND	ND	417	1260	67	
	1	01011202	Animal Tissue	N	Mourning dove	*Zenaida macroura*	ND	ND	388	1080	64	UJ
	1	01011203	Animal Tissue	N	Tree swallow	*Tachycineta bicolor*	ND	ND	400	1080	63	UJ
	1	01011204	Animal Tissue	N	Roughwing swallow	*Stelgidopteryx serripennis*	ND	ND	396	1020	61	UJ
	1	01011205	Animal Tissue	N	Roughwing swallow	*Stelgidopteryx serripennis*	ND	ND	398	995	60	UJ
	1	01011206	Animal Tissue	N	Roughwing swallow	*Stelgidopteryx serripennis*	ND	ND	400	930	57	UJ
	1	01011207	Animal Tissue	N	Roughwing swallow	*Stelgidopteryx serripennis*	ND	ND	392	1010	61	UJ
	1	01011291	Animal Tissue	N	Mourning dove	*Zenaida macroura*	ND	ND	400	1180	66	UJ
	1	01011301	Animal Tissue	N	Moths	Order Lepidoptera	ND	ND	396	966	59	UJ
	1	01011302	Animal Tissue	N	Moths	Order Lepidoptera	ND	ND	400	889	55	UJ
	2	01020101	Surface Water	N				ND		4.00		
	2	01020102	Surface Water	N				ND		4.00		
	2	01020103	Surface Water	N				ND		4.00		
	2	01020104	Surface Water	FD				ND		4.00		
	2	01020104	Surface Water	FT				ND		4.00		
	2	01020104	Surface Water	N				ND		4.00		
	2	01020301	Sediment	N			ND	ND	80.0	160	50	
	2	01020302	Sediment	N			ND	ND	80.4	98.0	18	
	2	01020303	Sediment	N			ND	ND	80.6	130	38	
	2	01020401	Plant Tissue	N	Sago	*Potamogeton pectinatus*	ND	ND	400	4000	90	

RL = reporting limit.
ND = not detected above the reporting limit.
N = normal sample, FD = field duplicate, and FT = field triplicate
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Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
Reported results less than RLs are estimated values.
13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	2	01020402	Plant Tissue	N	Sago	*Potamogeton pectinatus*	ND	ND	390	2600	85	
	2	01020901	Soil	N			ND	ND	79.8	81.0	1.5	
	2	01020902	Soil	N			ND	ND	79.7	82.0	2.8	
	2	01020903	Soil	N			ND	ND	79.8	84.0	5	
	3	01030101	Surface Water	N				3.00		4.00		J
	3	01030102	Surface Water	N				3.00		4.00		J
	3	01030103	Surface Water	N				4.20		4.00		
	3	01030104	Surface Water	N				4.00		4.00		
	3	01030105	Surface Water	N				4.00		4.00		J
	4	01040101	Surface Water	FD				4.00		4.00		J
	4	01040101	Surface Water	FT				4.00		4.00		J
	4	01040101	Surface Water	N				4.10		4.00		
	4	01040102	Surface Water	N				4.10		4.00		
	4	01040103	Surface Water	N				4.00		4.00		J
	4	01040104	Surface Water	N				4.00		4.00		J
	4	01040301	Sediment	N			ND	ND	80.0	100	20	
	4	01040302	Sediment	N			ND	ND	81.2	140	42	
	4	01040302 (TOP)	Pore Water	N				ND		4.00		
	4	01040303	Sediment	N			ND	ND	79.2	110	28	
	4	01040304	Sediment	N			ND	ND	76.8	120	36	
	4	01040304	Sediment	FT			ND	ND	83.6	110	24	
	4	01040304	Sediment	FD			ND	ND	78.0	130	40	
	4	01040401	Plant Tissue	N	Rush	*Juncus sp.*	ND	ND	408	1700	76	
	4	01040402	Plant Tissue	N	Rush	*Juncus sp.*	ND	ND	400	2000	80	
	4	01040403	Plant Tissue	N	Rush	*Juncus sp.*	ND	ND	378	1400	73	
	4	01040501	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	400	1330	70	UJ
	4	01040801	Animal Tissue	N	American coot	*Fulica americana*	ND	ND	400	1210	67	UJ
	4	01040901	Soil	FD			ND	ND	83.6	110	24	
	4	01040901	Soil	N			ND	ND	79.0	100	21	
	4	01040903	Soil	N			ND	ND	79.9	82.0	2.6	

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ND = not detected above the reporting limit.
N = normal sample, FD = field duplicate, and FT = field triplicate
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Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
Reported results less than RLs are estimated values.
13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	4	01041001	Plant Tissue	N	Honey mesquite bean	*Prosopis glandulosa*	650	1000	390	600	35	
	4	01041002	Plant Tissue	N	Honey mesquite bean	*Prosopis glandulosa*	ND	ND	395	590	33	
	4	01041003	Plant Tissue	N	Honey mesquite bean	*Prosopis glandulosa*	ND	ND	380	550	31	
	4	01041004	Plant Tissue	N	Arrowweed	*Pluchea sericea*	ND	ND	392	1400	72	
	4	01041005	Plant Tissue	N	Arrowweed	*Pluchea sericea*	ND	ND	385	1100	65	
	4	01041006	Plant Tissue	N	Arrowweed	*Pluchea sericea*	ND	ND	396	1100	64	
	4	01041007	Plant Tissue	N	Alfalfa	*Medicago sativa*	ND	ND	380	2000	81	
	4	01041008	Plant Tissue	N	Alfalfa	*Medicago sativa*	ND	ND	380	1900	80	
	4	01041009	Plant Tissue	N	Alfalfa	*Medicago sativa*	ND	ND	375	1500	75	
	4	01041101	Animal Tissue	N	Audoban's cottontail	*Sylvilagus audubonii*	ND	ND	386	1840	79	
	4	01041102	Animal Tissue	N	Audoban's cottontail	*Sylvilagus audubonii*	ND	ND	408	1570	74	
	4	01041103	Animal Tissue	N	Audoban's cottontail	*Sylvilagus audubonii*	ND	ND	404	2020	80	
	4	01041104	Animal Tissue	N	Rock pocket mouse	*Chaetodipus intermedius*	ND	ND	404	1500	73	
	4	01041105	Animal Tissue	N	Rock pocket mouse	*Chaetodipus intermedius*	ND	ND	406	1620	75	
	4	01041151	Animal Tissue	N	Rock pocket mouse	*Chaetodipus intermedius*	ND	ND	410	1470	72	
	4	01041152	Animal Tissue	N	Rock pocket mouse	*Chaetodipus intermedius*	ND	ND	417	1540	73	
	4	01041153	Animal Tissue	N	Rock pocket mouse	*Chaetodipus intermedius*	ND	ND	392	1450	73	
	4	01041301	Animal Tissue	N	Dragonfly (mature)	Suborder Anisoptera	ND	ND	436	970	55	UJ
	4	01041301	Animal Tissue	N	Dragonfly (mature)	Suborder Anisoptera	ND	ND	436	970	60	UJ
	4	01041301	Animal Tissue	N	Dragonfly (mature)	Suborder Anisoptera	ND	ND	436	970	55	UJ
	4	01041301	Animal Tissue	N	Dragonfly (mature)	Suborder Anisoptera	ND	ND	394	985	60	
	4	01041301	Animal Tissue	N	Dragonfly (mature)	Suborder Anisoptera	ND	ND	436	970	60	UJ
	4	01041301	Animal Tissue	N	Dragonfly (mature)	Suborder Anisoptera	ND	ND	394	985	55	
	4	01041301	Animal Tissue	N	Dragonfly (mature)	Suborder Anisoptera	ND	ND	394	985	55	
	4	01041301	Animal Tissue	N	Dragonfly (mature)	Suborder Anisoptera	ND	ND	394	985	60	
	5	01050101	Surface Water	N				ND		4.00		
	5	01050102	Surface Water	N				ND		4.00		
	5	01050103	Surface Water	N				ND		4.00		
	5	01050104	Surface Water	N				ND		4.00		
	5	01050105	Surface Water	N				ND		4.00		

RL = reporting limit.
ND = not detected above the reporting limit.
N = normal sample, FD = field duplicate, and FT = field triplicate
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Units are in µg/L or µg/kg.
Adjusted Result = Result[1-(%moisture/100)].*
Reported results less than RLs are estimated values.
13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	5	01050106	Surface Water	N				5.00		4.00		
	5	01050301	Sediment	N			ND	ND	81.0	270	70	
	5	01050302	Sediment	N			ND	ND	81.9	130	37	
	5	01050303	Sediment	N			ND	ND	81.6	240	66	
	5	01050304	Sediment	N			ND	ND	81.4	220	63	
	5	01050305	Sediment	N			ND	ND	81.6	120	32	
	5	01050401	Plant Tissue	N	Coontail	*Ceratophyllum demersum*	ND	ND	371	5300	93	
	5	01050402	Plant Tissue	N	Coontail	*Ceratophyllum demersum*	ND	ND	384	4800	92	
	5	01050403	Plant Tissue	N	Coontail	*Ceratophyllum demersum*	ND	ND	369	4100	91	
	5	01050501	Animal Tissue	N	Sunfish and largemouth bass	Family Centrarchidae	ND	ND	392	1450	73	UJ
	5	01050502	Animal Tissue	N	Sailfin molly	*Poecilia latipinna*	ND	ND	400	1210	67	UJ
	5	01050503	Animal Tissue	N	Mullet	*Mugil cephalus*	ND	ND	396	1520	74	UJ
	5	01050504	Animal Tissue	N	Mullet	*Mugil cephalus*	ND	ND	396	1280	69	UJ
	5	01050505	Animal Tissue	N	Mullet	*Mugil cephalus*	ND	ND	396	1370	71	UJ
	5	01050601	Animal Tissue	N	Damselfly (mature)	Suborder Zygoptera	ND	ND	822	2930	72	UJ
	5	01050701	Animal Tissue	N	Woodhouse's toad	*Bufo woodhousii*	ND	ND	410	2410	83	
	5	01050702	Animal Tissue	N	Woodhouse's toad	*Bufo woodhousii*	ND	ND	432	2160	80	
	5	01050901	Soil	N			ND	ND	79.7	80.0	0.4	
	5	01050902	Soil	N			ND	ND	80.0	100	20	
	5	01050903	Soil	N			ND	ND	80.3	81.0	0.9	
	5	01050904	Soil	N			ND	ND	79.9	80.0	0.1	
	5	01050905	Soil	N			ND	ND	80.2	99.0	19	
	5	01051001	Plant Tissue	N	Quail bush	*Atriplex lentiformis*	1630	4400	370	1000	63	
	5	01051002	Plant Tissue	N	Quail bush	*Atriplex lentiformis*	1790	5100	385	1100	65	
	5	01051003	Plant Tissue	N	Arrowweed	*Pluchea sericea*	190	500	380	1000	62	
	5	01051004	Plant Tissue	N	Arrowweed	*Pluchea sericea*	570	1500	418	1100	62	
	5	01051005	Plant Tissue	N	Arrowweed	*Pluchea sericea*	185	500	407	1100	63	
	5	01051006	Plant Tissue	N	Screw mesquite	*Prosopis pubescens*	ND	ND	387	450	14	
	5	01051007	Plant Tissue	N	Screw mesquite	*Prosopis pubescens*	ND	ND	387	440	12	
	5	01051008	Plant Tissue	N	Screw mesquite	*Prosopis pubescens*	ND	ND	387	450	14	

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13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	5	01051101	Animal Tissue	N	Cactus mouse	*Peromyscus eremicus*	ND	ND	404	1390	71	
	5	01051102	Animal Tissue	N	Cactus mouse	*Peromyscus eremicus*	ND	ND	400	1540	74	
	5	01051103	Animal Tissue	N	Cactus mouse	*Peromyscus eremicus*	ND	ND	419	1400	70	
	5	01051104	Animal Tissue	N	Cactus mouse	*Peromyscus eremicus*	ND	ND	394	1310	70	
	5	01051105	Animal Tissue	N	Cactus mouse	*Peromyscus eremicus*	ND	ND	402	1260	68	
	5	01051201	Animal Tissue	N	Mourning dove	*Zenaida macroura*	ND	ND	390	1180	67	
	5	01051301	Animal Tissue	N	Terrestrial insect (misc.)	Class Insecta	ND	ND	393	1040	62	
	5	01051302	Animal Tissue	N	Terrestrial insect (misc.)	Class Insecta	ND	ND	392	912	57	
	5	01051401	Animal Tissue	N	Brush lizard	*Urosaurus graciosus*	531	1710	412	1330	69	

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Data Summary-Clayton Laboratory

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2 Lake Mead Area												
	/	02010101 DN	Surface Water	N				13000		4.00		
	/	02010101 UP	Surface Water	N				100000		4.00		
	/	02010102 UP	Surface Water	N				100000		4.00		
	/	02010102 DN	Surface Water	N				19000		4.00		
	/	02010103 UP	Surface Water	N				100000		4.00		
	/	02010103 DN	Surface Water	N				12000		4.00		
	/	02010104 UP	Surface Water	N				100000		4.00		
	/	02010104 DN	Surface Water	N				12000		4.00		
	/	02010105 UP	Surface Water	N				100000		4.00		
	/	02010105 DN	Surface Water	N				12000		4.00		
	/	02010106 UP	Surface Water	N				100000		4.00		
	/	02010106 UP	Surface Water	FD				100000		4.00		
	/	02010106 DN	Surface Water	N				12000		4.00		
	/	02010130	Surface Water	N				120000		20000		
	/	02010131	Surface Water	N				130000		20000		
	/	02010301 DN	Sediment	N			ND	ND	78.0	130	40	
	/	02010301 DN (TO	Pore Water	N				1000		4.00		
	/	02010301 UP	Sediment	N			53200	140000	79.8	210	62	
	/	02010302 DN	Sediment	N			3320	4200	79.0	100	21	
	/	02010302 UP	Sediment	N			23000	49000	79.9	170	53	
	/	02010303 DN	Sediment	N			ND	ND	81.6	120	32	
	/	02010303 UP	Sediment	N			43200	160000	81.0	300	73	
	/	02010304 UP	Sediment	N			56000	200000	81.2	290	72	
	/	02010305 UP	Sediment	N			45500	130000	80.5	230	65	
	/	02010306 UP	Sediment	FD			39600	120000	79.2	240	67	J
	/	02010306 UP	Sediment	N			47500	190000	80.0	320	75	J
	/	02010307 UP (TO	Pore Water	N				98000		4.00		
	/	02010401	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	132000	1200000	19800	80000	89	
	/	02010402	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	121000	1100000	20900	90000	89	

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Data Summary-Clayton Laboratory

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	1	02010403	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	140000	1400000	21000	10000	90	
	1	02010403	Plant Tissue	FD	Filamentous algae	*Oscillatoria sp.*	176000	1600000	19800	80000	89	
	1	02010501	Animal Tissue	N	Mosquito fish	*Gambusia affinis*	44300	202000	5740	26100	78	
	1	02010502	Animal Tissue	N	Mosquito fish	*Gambusia affinis*	4310	18700	392	1710	77	
	1	02010901	Soil	N			1470000	2100000	77.0	110	30	
	1	02010902	Soil	N			83300	87000	80.5	84.0	4.2	
	1	02010903	Soil	N			507000	570000	80.1	90.0	11	
	1	02010904	Soil	N			367	470	78.0	100	22	
	1	02010905	Soil	N			187000	200000	79.6	85.0	6.4	
	1	020110TA01	Plant Tissue	N	Salt cedar leaf	*Tamarix chinensis*	53200	140000	418	1100	62	
	1	020110TA02	Plant Tissue	N	Salt cedar leaf	*Tamarix chinensis*	194000	510000	20100	53000	62	
	1	020110TA03	Plant Tissue	N	Salt cedar leaf	*Tamarix chinensis*	207000	470000	38700	88000	56	
	1	020110TA04	Plant Tissue	N	Salt cedar seed	*Tamarix chinensis*	12800	40000	384	1200	68	
	1	020110TA05	Plant Tissue	N	Salt cedar seed	*Tamarix chinensis*	14200	43000	429	1300	67	
	1	020110TA06	Plant Tissue	N	Salt cedar seed	*Tamarix chinensis*	13300	39000	408	1200	66	
	1	020110TA07	Plant Tissue	N	Salt cedar litter	*Tamarix chinensis*	387000	530000	39400	54000	27	
	1	020110TA08	Plant Tissue	N	Salt cedar litter	*Tamarix chinensis*	390000	410000	39900	42000	5	
	1	020110TA09	Plant Tissue	N	Salt cedar litter	*Tamarix chinensis*	428000	460000	40000	43000	7	
	1	02011101	Animal Tissue	N	Cactus mouse	*Peromyscus eremicus*	15100	48600	1990	6420	69	
	1	02011102	Animal Tissue	N	Cactus mouse	*Peromyscus eremicus*	16000	55200	2010	6930	71	
	1	02011103	Animal Tissue	N	Cactus mouse	*Peromyscus eremicus*	21900	66500	2020	6120	67	
	1	02011104	Animal Tissue	N	Deer mouse	*Peromyscus maniculatus*	53000	151000	6220	17800	65	
	1	02011105	Animal Tissue	N	Cactus mouse	*Peromyscus eremicus*	23200	70200	4100	12400	67	
	1	02011106	Animal Tissue	N	Deer mouse	*Peromyscus maniculatus*	46100	136000	5910	17400	66	
	1	02011107	Animal Tissue	N	Cactus mouse	*Peromyscus eremicus*	8440	26400	406	1270	68	
	1	02011108	Animal Tissue	N	Deer mouse	*Peromyscus maniculatus*	19500	61000	2050	6410	68	
	1	02011201	Animal Tissue	N	Mourning dove	*Zenaida macroura*	1200	3330	400	1110	64	
	1	02011202	Animal Tissue	N	Mourning dove	*Zenaida macroura*	4190	11600	387	1070	64	
	1	02011203	Animal Tissue	N	Lesser nighthawk	*Chordeiles acutipennis*	ND	ND	381	1120	66	
	1	02011204	Animal Tissue	N	Nighthawk	*Chordeiles minor*	ND	ND	383	1040	63	

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Data Summary-Clayton Laboratory

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	1	02011205	Animal Tissue	N	Nighthawk	*Chordeiles minor*	1430	4610	381	1230	69	
	1	02011301	Animal Tissue	N	Terrestrial insect (misc.)	Class Insecta	4170	11600	407	1130	64	
	3	02030101	Surface Water	N				460		4.00		
	3	02030102	Surface Water	N				460		4.00		
	3	02030103	Surface Water	N				440		4.00		
	3	02030104	Surface Water	N				450		4.00		
	3	02030105	Surface Water	N				480		4.00		
	3	02030106	Surface Water	N				460		4.00		
	3	02030301	Sediment	N			ND	ND	79.2	110	28	J
	3	02030302	Sediment	N			ND	ND	78.0	150	48	
	3	02030303	Sediment	N			80.4	120	80.4	120	33	
	3	02030304	Sediment	N			146	170	80.0	93.0	14	
	3	02030305	Sediment	N			122	150	80.2	99.0	19	
	3	02030401	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	ND	ND	377	2900	87	UJ
	3	02030402	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	ND	ND	407	3700	89	UJ
	3	02030403	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	ND	ND	396	4400	91	UJ
	3	02030403	Plant Tissue	FD	Filamentous algae	*Oscillatoria sp.*	ND	ND	392	5600	93	UJ
	3	02030501	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	383	1370	72	
	3	02030502	Animal Tissue	N	Carp	*Cyprinus carpio*	ND	ND	392	2180	82	
	3	02030503	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	386	1430	73	
	3	02030504	Animal Tissue	N	Catfish	*Ictalurus sp.*	ND	ND	381	2010	81	
	3	02030505	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	381	1470	74	
	3	02030506	Animal Tissue	N	Shiner	*Notropis sp.*	ND	ND	381	1360	72	
	3	02030507	Animal Tissue	N	Shiner	*Notropis sp.*	ND	ND	383	1370	72	
	3	02030601	Animal Tissue	N	Aquatic invertebrates	Phylum Arthropoda	ND	ND	381	1150	67	
	3	02030701	Animal Tissue	N	Woodhouse's toad	*Bufo woodhousii*	ND	ND	396	1980	80	UJ
	3	02030702	Animal Tissue	N	Woodhouse's toad	*Bufo woodhousii*	ND	ND	400	2500	84	UJ
	3	02030703	Animal Tissue	N	Woodhouse's toad	*Bufo woodhousii*	ND	ND	400	2000	80	UJ
	3	02030704	Animal Tissue	N	Woodhouse's toad	*Bufo woodhousii*	ND	ND	396	1650	76	UJ
	3	02030705	Animal Tissue	N	Woodhouse's toad	*Bufo woodhousii*	ND	ND	396	1890	79	UJ

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Data Summary-Clayton Laboratory

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	3	02030901	Soil	N			1070	1100	80.0	82.0	2.4	
	3	02030902	Soil	N			684	700	80.1	82.0	2.3	
	3	02030903	Soil	N			ND	ND	80.3	81.0	0.9	
	3	02030904	Soil	N			121000	130000	80.3	86.0	6.6	
	3	02030905	Soil	N			73800	80000	80.2	87.0	7.8	
	3	02031001	Plant Tissue	N	Bullrush	*Scirpus sp.*	741	5700	390	3000	87	J
	3	02031002	Plant Tissue	N	Bullrush	*Scirpus sp.*	1020	7300	392	2800	86	
	3	02031003	Plant Tissue	N	Bullrush	*Scirpus sp.*	2160	18000	384	3200	88	
	3	02031004	Plant Tissue	N	Bullrush	*Scirpus sp.*	3080	22000	406	2900	86	
	3	02031005	Plant Tissue	N	Bullrush	*Scirpus sp.*	2000	20000	400	4000	90	
	3	02031006	Plant Tissue	N	Bermuda grass	*Cynodon dactylon*	90000	200000	396	880	55	
	3	02031007	Plant Tissue	N	Bermuda grass	*Cynodon dactylon*	8370	27000	372	1200	69	
	3	02031008	Plant Tissue	N	Bermuda grass	*Cynodon dactylon*	37200	120000	403	1300	69	
	3	02031009	Plant Tissue	N	Bermuda grass	*Cynodon dactylon*	27500	110000	400	1600	75	
	3	02031010	Plant Tissue	N	Bermuda grass	*Cynodon dactylon*	51300	190000	378	1400	73	
	3	02031011	Plant Tissue	N	Phragmites	*Phragmites australis*	99200	310000	416	1300	68	
	3	02031012	Plant Tissue	N	Phragmites	*Phragmites australis*	55800	180000	403	1300	69	
	3	02031013	Plant Tissue	N	Phragmites	*Phragmites australis*	76800	240000	416	1300	68	
	3	02031014	Plant Tissue	N	Phragmites	*Phragmites australis*	52800	220000	408	1700	76	
	3	02031015	Plant Tissue	N	Phragmites	*Phragmites australis*	60000	200000	390	1300	70	
	3	02031101	Animal Tissue	N	Cactus mouse	*Peromyscus eremicus*	1290	4030	392	1230	68	
	3	02031102	Animal Tissue	N	Cactus mouse	*Peromyscus eremicus*	2000	6260	385	1200	68	
	3	02031103	Animal Tissue	N	Deer mouse	*Peromyscus maniculatus*	2000	5710	412	1180	65	
	3	02031104	Animal Tissue	N	Deer mouse	*Peromyscus maniculatus*	4230	12100	419	1200	65	
	3	02031301	Animal Tissue	N	Terrestrial insect (misc.)	Class Insecta	4730	12400	385	1010	62	
	3	02031302	Animal Tissue	N	Terrestrial insect (misc.)	Class Insecta	3200	11400	386	1380	72	
	3	02050410	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	320	8000	400	10000	96	UJ
	5	02050101	Surface Water	N				58.0		4.00		
	5	02050102	Surface Water	N				58.0		4.00		
	5	02050103	Surface Water	N				57.0		4.00		

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Data Summary-Clayton Laboratory

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	5	02050104	Surface Water	N				60.0		4.00		
	5	02050105	Surface Water	N				58.0		4.00		
	5	02050106	Surface Water	N				68.0		4.00		
	5	02050106	Surface Water	FD				60.0		4.00		
	5	02050120	Surface Water	N				ND		4.00		
	5	02050121	Surface Water	N				ND		4.00		
	5	02050122	Surface Water	N				27.0		4.00		
	5	02050301	Sediment	N			ND	ND	78.0	100	22	
	5	02050302	Sediment	N			ND	ND	83.6	110	24	
	5	02050303	Sediment	N			ND	ND	83.6	110	24	
	5	02050304	Sediment	N			81.4	110	81.4	110	26	
	5	02050305	Sediment	N			67.5	90.0	82.5	110	25	J
	5	02050305	Sediment	FD			ND	ND	79.0	100	21	
	5	02050306 (TOP)	Pore Water	N				140		4.00		
	5	02050411	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	ND	ND	383	850	55	UJ
	5	02050412	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	ND	ND	405	920	56	UJ
	5	02050501	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	388	1620	76	UJ
	5	02050502	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	385	1430	73	UJ
	5	02050503	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	400	1670	76	UJ
	5	02050504	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	390	1560	75	UJ
	5	02050505	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	394	1710	77	UJ
	5	02050506	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	390	1700	77	UJ
	5	02050507	Animal Tissue	N	Mosquito fish	*Gambusia affinis*	ND	ND	394	1640	76	UJ
	5	02050508	Animal Tissue	N	Mosquito fish	*Gambusia affinis*	ND	ND	388	1690	77	UJ
	5	02050509	Animal Tissue	N	Mosquito fish	*Gambusia affinis*	ND	ND	402	1910	79	UJ
	5	02050510	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	402	1750	77	UJ
	5	02050601	Animal Tissue	N	Gastropod	Class Gastropoda	ND	ND	398	1730	77	UJ
	5	02050701	Animal Tissue	N	Woodhouse's toad	*Bufo woodhousii*	ND	ND	421	1830	77	
	5	02050801	Animal Tissue	N	Grebe	*Aechmophorus clarkii*	ND	ND	398	1110	64	UJ
	5	02050802	Animal Tissue	N	Grebe	*Aechmophorus clarkii*	ND	ND	392	1150	66	UJ

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Data Summary-Clayton Laboratory

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	5	02050803	Animal Tissue	N	Grebe	*Aechmophorus clarkii*	ND	ND	398	1210	67	UJ
	5	02050804	Animal Tissue	N	Coot	*Fulica americana*	ND	ND	400	1110	64	UJ
	5	02050805	Animal Tissue	N	Coot	*Fulica americana*	ND	ND	400	1430	72	UJ
	5	02050806	Animal Tissue	N	Coot	*Fulica americana*	ND	ND	390	1220	68	UJ
	5	02050807	Animal Tissue	N	Coot	*Fulica americana*	ND	ND	402	1060	62	UJ
	5	02050808	Animal Tissue	N	Coot	*Fulica americana*	ND	ND	398	1240	68	UJ
	5	02050901	Soil	N			ND	ND	79.2	110	28	
	5	02050902	Soil	N			210	250	79.8	95.0	16	
	5	02050903	Soil	N			672	830	80.2	99.0	19	
	5	02050904	Soil	N			352	400	80.1	91.0	12	
	5	02050905	Soil	N			648	810	80.0	100	20	J
	5	02051001	Plant Tissue	N	Grass	Family Poaceae	2420	7800	403	1300	69	
	5	02051002	Plant Tissue	N	Grass	Family Poaceae	4800	16000	390	1300	70	
	5	02051003	Plant Tissue	N	Grass	Family Poaceae	2820	9400	390	1300	70	
	5	02051004	Plant Tissue	N	Grass	Family Poaceae	4320	16000	405	1500	73	
	5	02051005	Plant Tissue	N	Grass	Family Poaceae	5720	22000	416	1600	74	
	5	02051006	Plant Tissue	N	Bulrush	*Scirpus sp.*	616	4400	392	2800	86	J
	5	02051007	Plant Tissue	N	Bulrush	*Scirpus sp.*	1950	13000	390	2600	85	
	5	02051008	Plant Tissue	N	Bulrush	*Scirpus sp.*	516	4300	396	3300	88	J
	5	02051009	Plant Tissue	N	Bulrush	*Scirpus sp.*	1950	13000	390	2600	85	
	5	02051101	Animal Tissue	N	Long-tailed pocket mouse	*Perognathus formosus*	ND	ND	412	1150	64	UJ
	5	02051102	Animal Tissue	N	House mouse	*Mus musculus*	443	1390	394	1230	68	J
	5	02051103	Animal Tissue	N	Long-tailed pocket mouse	*Perognathus formosus*	447	1490	394	1310	70	J
	5	02051104	Animal Tissue	N	Long-tailed pocket mouse	*Perognathus formosus*	433	1350	398	1240	68	J
	5	02051105	Animal Tissue	N	Cactus mouse	*Peromyscus eremicus*	ND	ND	417	1190	65	UJ
	5	02051120	Animal Tissue	N	Western pipestrelle bat	*Pipistrellus hesperus*	ND	ND	396	966	59	UJ
	5	02051201	Animal Tissue	N	Gambel's quail	*Callipepla gambelii*	401	1340	385	1280	70	
	5	02051202	Animal Tissue	N	Gambel's quail	*Callipepla gambelii*	ND	ND	392	1230	68	
	5	02051301	Animal Tissue	N	Terrestrial insect (misc.)	Class Insecta	ND	ND	388	1080	64	

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13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
3 ABL												
	1	03010901	Soil	N			22100	24000	80.0	87.0	8	
	1	03010902	Soil	N			371	390	79.8	84.0	5	
	1	03010903	Soil	N			1350	1500	80.1	89.0	10	
	1	03010904	Soil	N			468	550	79.9	94.0	15	
	1	03010905	Soil	N			4230	4600	80.0	87.0	8	J
	1	03010906	Soil	N			1500	1600	79.9	85.0	6	
	1	03010907	Soil	N			1060	1200	80.1	91.0	12	
	1	03010908	Soil	N			117	150	78.0	100	22	
	1	03010909	Soil	N			282	320	80.1	91.0	12	
	1	03010909	Soil	FD			329	370	80.1	90.0	11	
	1	03011001	Plant Tissue	N	Johnson grass	*Sorghum halepense*	126000	350000	19800	55000	64	
	1	03011002	Plant Tissue	N	Johnson grass	*Sorghum halepense*	28400	79000	3960	11000	64	
	1	03011003	Plant Tissue	N	Johnson grass	*Sorghum halepense*	9920	31000	2400	7500	68	
	1	03011004	Plant Tissue	N	Johnson grass	*Sorghum halepense*	48000	160000	3900	13000	70	
	1	03011005	Plant Tissue	N	Johnson grass	*Sorghum halepense*	101000	360000	8120	29000	72	
	1	03011006	Plant Tissue	N	Johnson grass	*Sorghum halepense*	300000	910000	39600	20000	67	
	1	03011007	Plant Tissue	N	Johnson grass	*Sorghum halepense*	46200	140000	3960	12000	67	
	1	03011008	Plant Tissue	N	Johnson grass	*Sorghum halepense*	19800	64000	2020	6500	69	
	1	03011009	Plant Tissue	N	Johnson grass	*Sorghum halepense*	70400	220000	8320	26000	68	
	1	03011101	Animal Tissue	N	White-footed mouse	*Peromyscus leucopus*	ND	ND	390	1220	68	UJ
	1	03011102	Animal Tissue	N	White-footed mouse	*Peromyscus leucopus*	ND	ND	392	1350	71	UJ
	1	03011201	Animal Tissue	N	Starling	*Sturnus vulgaris*	ND	ND	394	1190	67	UJ
	1	03011202	Animal Tissue	N	Starling	*Sturnus vulgaris*	ND	ND	406	1070	62	UJ
	1	03011203	Animal Tissue	N	Starling	*Sturnus vulgaris*	ND	ND	394	1100	64	UJ
	1	03011204	Animal Tissue	N	Mourning dove	*Zenaida macroura*	423	1240	419	1230	66	J
	1	03011205	Animal Tissue	N	American crow	*Corvus brachyrhynchos*	ND	ND	400	1330	70	UJ
	1	03011301	Animal Tissue	N	Terrestrial insect (misc.)	Class Insecta	12600	32300	2040	5230	61	
	1	03011303	Animal Tissue	N	Moths	Order Lepidoptera	ND	ND	419	1680	75	UJ
	2	03020901	Soil	N			ND	ND	80.0	92.0	13	

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13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	2	03020902	Soil	N			ND	ND	83.6	110	24	
	2	03020903	Soil	N			ND	ND	76.8	120	36	
	2	03020904	Soil	N			ND	ND	80.4	98.0	18	
	2	03020905	Soil	N			ND	ND	80.1	89.0	10	
	2	03020906	Soil	N			ND	ND	80.0	92.0	13	
	2	03021001	Plant Tissue	N	Johnson grass	*Sorghum halepense*	ND	ND	408	1200	66	UJ
	2	03021002	Plant Tissue	N	Johnson grass	*Sorghum halepense*	ND	ND	396	1200	67	UJ
	2	03021003	Plant Tissue	N	Johnson grass	*Sorghum halepense*	ND	ND	384	1200	68	UJ
	2	03021004	Plant Tissue	N	Johnson grass	*Sorghum halepense*	290	1000	406	1400	71	UJ
	2	03021005	Plant Tissue	N	Johnson grass	*Sorghum halepense*	ND	ND	396	1200	67	UJ
	2	03021006	Plant Tissue	N	Johnson grass	*Sorghum halepense*	288	800	396	1100	64	UJ
	2	03021301	Animal Tissue	N	Praying mantis	Family Mantidae	ND	ND	421	1170	64	UJ
	2	03021302	Animal Tissue	N	Earthworm	Class Oligochaeta	ND	ND	404	1190	66	UJ
	3	03030901	Soil	N			88.0	110	80.0	100	20	
	3	03030902	Soil	N			990	1100	80.1	89.0	10	
	3	03030903	Soil	N			122	150	80.2	99.0	19	
	3	03031001	Plant Tissue	N	Johnson grass	*Sorghum halepense*	ND	ND	408	1200	66	
	3	03031002	Plant Tissue	N	Johnson grass	*Sorghum halepense*	1160	3400	408	1200	66	
	3	03031003	Plant Tissue	N	Johnson grass	*Sorghum halepense*	1020	3400	390	1300	70	
	3	03031004	Plant Tissue	N	Johnson grass	*Sorghum halepense*	468	1800	390	1500	74	
	3	03031005	Plant Tissue	N	Johnson grass	*Sorghum halepense*	ND	ND	396	1800	78	
	3	03031006	Plant Tissue	N	Johnson grass	*Sorghum halepense*	ND	ND	384	1200	68	
	3	03031301	Animal Tissue	N	Terrestrial insect (misc.)	Class Insecta	ND	ND	398	1330	70	UJ
	3	03031302	Animal Tissue	N	Terrestrial insect (misc.)	Class Insecta	ND	ND	396	1170	66	UJ
	3	03031303	Animal Tissue	N	Terrestrial insect (misc.)	Class Insecta	ND	ND	390	1860	79	UJ
	4	03040101	Surface Water	N				5.75		4.00		
	4	03040102	Surface Water	N				6.15		4.00		
	4	03040103	Surface Water	N				6.60		4.00		
	4	03040301	Sediment	N			104	200	78.0	150	48	
	4	03040302	Sediment	N			ND	ND	79.8	140	43	

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13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	4	03040303	Sediment	N			ND	ND	79.8	140	43	
	4	03040500	Animal Tissue	N	Fish eggs (misc.)	Family Centrarchidae	ND	ND	404	1300	69	
	4	03040501	Animal Tissue	N	White sucker	*Catostomus commersonii*	ND	ND	400	1600	75	
	4	03040502	Animal Tissue	N	White sucker	*Catostomus commersonii*	ND	ND	394	1520	74	
	4	03040503	Animal Tissue	N	White sucker	*Catostomus commersonii*	ND	ND	410	1520	73	
	4	03040504	Animal Tissue	N	White sucker	*Catostomus commersonii*	ND	ND	410	1580	74	
	4	03040505	Animal Tissue	N	Red breast sunfish	Family Centrarchidae	ND	ND	408	1700	76	
	4	03040506	Animal Tissue	N	Pumpkin seed	Family Centrarchidae	ND	ND	390	1450	73	
	4	03040507	Animal Tissue	N	Pumpkin seed	Family Centrarchidae	ND	ND	388	1440	73	
	4	03040508	Animal Tissue	N	Rock bass	Family Centrarchidae	ND	ND	404	1920	79	
	4	03040509	Animal Tissue	N	Rock bass	Family Centrarchidae	ND	ND	402	1680	76	
	4	03041301	Animal Tissue	N	Terrestrial insect (misc.)	Class Insecta	ND	ND	392	1090	64	UJ
	4	03041302	Animal Tissue	N	Terrestrial insect (misc.)	Class Insecta	ND	ND	400	952	58	UJ
	5	03050101	Surface Water	N				240		80.0		
	5	03050102	Surface Water	N				270		80.0		
	5	03050103	Surface Water	N				270		80.0		
	5	03050104	Surface Water	N				250		80.0		
	5	03050105	Surface Water	N				260		80.0		
	5	03050106	Surface Water	N				260		80.0		
	5	03050301	Sediment	N			ND	ND	80.0	250	68	
	5	03050302	Sediment	N			ND	ND	81.6	170	52	
	5	03050303	Sediment	N			119	340	80.5	230	65	
	5	03050401	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	ND	ND	396	3600	89	
	5	03050402	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	ND	ND	400	5000	92	
	5	03050403	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	ND	ND	392	4900	92	
	5	03050404	Plant Tissue	N	Duckweed	*Lemna sp.*	ND	ND	396	9900	96	
	5	03050405	Plant Tissue	N	Duckweed	*Lemna sp.*	ND	ND	405	8100	95	
	5	03050406	Plant Tissue	N	Duckweed	*Lemna sp.*	ND	ND	395	7900	95	
	5	03050407	Plant Tissue	N	Small pondweed	*Potamogeton pusillus*	ND	ND	391	2300	83	
	5	03050408	Plant Tissue	N	Small pondweed	*Potamogeton pusillus*	ND	ND	408	2400	83	

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Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	5	03050409	Plant Tissue	N	Small pondweed	*Potamogeton pusillus*	ND	ND	396	3300	88	
	5	03050501	Animal Tissue	N	Green sunfish	*Lepomis cyanellus*	451	1740	410	1580	74	J
	5	03050502	Animal Tissue	N	Green sunfish	*Lepomis cyanellus*	ND	ND	410	1580	74	UJ
	5	03050701	Animal Tissue	N	Leopard frog	*Rana pipiens*	ND	ND	404	1620	75	UJ
	5	03050702	Animal Tissue	N	Leopard frog	*Rana pipiens*	ND	ND	417	2320	82	UJ
	5	03050901	Soil	N			ND	ND	77.0	100	23	
	5	03050902	Soil	N			ND	ND	80.2	99.0	19	
	5	03050903	Soil	N			ND	ND	81.4	110	26	
	5	03051101	Animal Tissue	N	Meadow vole	*Microtus pennsylvanicus*	ND	ND	417	1230	66	UJ
	5	03051102	Animal Tissue	N	Meadow vole	*Microtus pennsylvanicus*	ND	ND	417	1390	70	UJ
	5	03051103	Animal Tissue	N	Meadow vole	*Microtus pennsylvanicus*	ND	ND	406	1310	69	UJ
	5	03051301	Animal Tissue	N	Grasshopper and cricket	Order Orthoptera	ND	ND	421	1240	66	UJ
	5	03051302	Animal Tissue	N	Praying mantis	Family Mantidae	ND	ND	407	904	55	UJ
	6	03060101	Surface Water	N				ND		80.0		
	6	03060102	Surface Water	N				ND		80.0		
	6	03060103	Surface Water	N				ND		80.0		
	6	03060104	Surface Water	N				ND		80.0		
	6	03060105	Surface Water	N				ND		80.0		
	6	03060106	Surface Water	N				ND		80.0		
	6	03060106	Surface Water	FD				ND		80.0		
	6	03060301 BOTTOM	Sediment	N			ND	ND	81.6	120	32	
	6	03060301 TOP	Pore Water	N				14.0		4.00		
	6	03060301 TOP	Pore Water	N				14.0		4.00		
	6	03060302	Sediment	N			ND	ND	81.6	120	32	
	6	03060303 BOTTOM	Sediment	FD			ND	ND	79.8	140	43	
	6	03060303 BOTTOM	Sediment	N			ND	ND	81.0	150	46	
	6	03060303 TOP	Pore Water	N				23.0		4.00		
	6	03060401	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	ND	ND	390	1300	70	
	6	03060402	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	ND	ND	399	2100	81	
	6	03060403	Plant Tissue	N	Small pondweed	*Potamogeton pusillus*	ND	ND	400	2500	84	

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Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	6	03060901	Soil	N			112	160	77.0	110	30	
	6	03060902	Soil	N			ND	ND	80.0	92.0	13	
	6	03060902	Soil	FD			ND	ND	83.6	110	24	
	6	03060903	Soil	N			ND	ND	80.1	91.0	12	
	7	03070301	Sediment	N			ND	ND	79.2	220	64	
	7	03070302	Sediment	N			ND	ND	79.8	210	62	
	7	03070303	Sediment	N			ND	ND	81.9	210	61	
	7	03071301	Animal Tissue	N	Praying mantis	Family Mantidae	ND	ND	407	1040	61	UJ
	7	03071302	Animal Tissue	N	Earthworm	Class Oligochaeta	ND	ND	392	800	51	UJ
	7	03071303	Animal Tissue	N	Earthworm	Class Oligochaeta	ND	ND	417	926	55	UJ
	8	03080101	Surface Water	N				270		80.0		
	8	03080102	Surface Water	N				280		80.0		
	8	03080103	Surface Water	N				280		80.0		
	8	03080104	Surface Water	N				260		80.0		
	8	03080105	Surface Water	N				270		80.0		
	8	03080106	Surface Water	N				250		80.0		
	8	03080301	Sediment	N			ND	ND	79.2	110	28	
	8	03080302	Sediment	N			ND	ND	77.0	110	30	
	8	03080303	Sediment	FD			ND	ND	79.2	110	28	
	8	03080303	Sediment	N			ND	ND	80.4	120	33	
	8	03080901	Soil	N			ND	ND	80.1	91.0	12	
	8	03080902	Soil	N			ND	ND	81.4	110	26	
	8	03080903	Soil	N			ND	ND	83.6	110	24	
	8	03081301	Animal Tissue	N	Earthworm	Class Oligochaeta	ND	ND	800	1160	31	UJ
	8	03081302	Animal Tissue	N	Bees	Order Hymenoptera	ND	ND	388	996	61	UJ
	8	03081303	Animal Tissue	N	Bees	Order Hymenoptera	ND	ND	412	1030	60	UJ

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13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
4 Holloman AFB												
	0	MW03	Groundwater	N				ND		4.00		
	0	MW03A	Groundwater	N				ND		4.00		
	0	MW04	Groundwater	N				ND		4.00		
	0	MW04A	Groundwater	N				ND		4.00		
	0	MW3801	Groundwater	N				5.70		4.00		J
	0	MW3801A	Groundwater	N				9.50		4.00		
	0	MW3802	Groundwater	N				12.0		4.00		
	0	MW3802A	Groundwater	N				11.0		4.00		
	0	MW3803	Groundwater	N				7.60		4.00		
	0	MW3803A	Groundwater	N				5.70		4.00		
	0	MW3901	Groundwater	N				34.0		4.00		
	0	MW3901A	Groundwater	N				38.0		4.00		
	0	MW3902	Groundwater	N				24.0		4.00		
	0	MW3902A	Groundwater	N				22.0		4.00		
	0	MW4102	Groundwater	N				5.00		4.00		J
	0	MW4102A	Groundwater	N				4.70		4.00		J
	0	MW4102DUP	Groundwater	N				8.00		4.00		J
	0	MW4103	Groundwater	N				8.00		4.00		
	0	MW4103A	Groundwater	N				11.0		4.00		
	1	04010301	Sediment	N			ND	ND	79.2	120	34	
	1	04010302	Sediment	N			ND	ND	81.6	120	32	
	1	04010303	Sediment	N			ND	ND	80.4	120	33	
	1	04010901	Soil	N			ND	ND	79.0	100	21	
	1	04010902	Soil	N			ND	ND	78.0	100	22	
	1	04010903	Soil	N			ND	ND	80.2	99.0	19	
	1	04010904	Soil	N			ND	ND	78.1	110	29	
	1	04010905	Soil	N			83.6	110	83.6	110	24	
	1	04010906	Soil	N			98.8	130	83.6	110	24	
	1	04010907	Soil	N			99.4	140	78.1	110	29	

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Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	1	04010908	Soil	N			118	160	81.4	110	26	
	1	04010909	Soil	N			88.0	110	80.0	100	20	
	1	04010910	Soil	N			2800	3500	800	1000	20	
	1	04010911	Soil	N			328	410	80.0	100	20	
	1	04010912	Soil	N			60.9	70.0	80.0	92.0	13	J
	1	04010913	Soil	N			348	400	80.0	92.0	13	
	1	04011001	Plant Tissue	N	Sea lavender	*Limonium sp.*	ND	ND	391	1700	77	UJ
	1	04011002	Plant Tissue	N	Sea lavender	*Limonium sp.*	ND	ND	378	1400	73	UJ
	1	04011003	Plant Tissue	N	Sea lavender	*Limonium sp.*	ND	ND	400	1600	75	UJ
	1	04011004	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	396	1800	78	UJ
	1	04011005	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	420	2000	79	UJ
	1	04011006	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	400	2000	80	UJ
	1	04011007	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	408	2400	83	UJ
	1	04011008	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	380	1900	80	UJ
	1	04011009	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	420	2000	378	1800	79	J
	1	04011010	Plant Tissue	N	Salt cedar leaf	*Tamarix ramosissima*	ND	ND	385	1100	65	
	1	04011011	Plant Tissue	N	Salt cedar leaf	*Tamarix ramosissima*	ND	ND	396	1100	64	UJ
	1	04011012	Plant Tissue	N	Salt cedar leaf	*Tamarix ramosissima*	ND	ND	370	1000	63	UJ
	1	04011013	Plant Tissue	N	Four-winged saltbush	*Atriplex canescens*	675	2500	378	1400	73	J
	1	04011014	Plant Tissue	N	Four-winged saltbush	*Atriplex canescens*	918	3400	405	1500	73	J
	1	04011015	Plant Tissue	N	Four-winged saltbush	*Atriplex canescens*	1550	4200	407	1100	63	
	1	04011101	Animal Tissue	N	Merriam's kangaroo rat	*Dipodomys merriami*	ND	ND	398	1470	73	UJ
	1	04011102	Animal Tissue	N	Merriam's kangaroo rat	*Dipodomys merriami*	ND	ND	415	1380	70	UJ
	1	04011103	Animal Tissue	N	Merriam's kangaroo rat	*Dipodomys merriami*	ND	ND	389	1340	71	UJ
	1	04011104	Animal Tissue	N	Merriam's kangaroo rat	*Dipodomys merriami*	ND	ND	408	1460	72	UJ
	1	04011104 DUP	Animal Tissue	N	Merriam's kangaroo rat	*Dipodomys merriami*	ND	ND	404	1440	72	UJ
	1	04011105	Animal Tissue	N	Cottontail rabbit	*Sylvilagus audubonii*	ND	ND	396	1650	76	UJ
	1	04011106	Animal Tissue	N	Desert pocket mouse	*Chaetodipus penicillatus*	ND	ND	410	1110	63	UJ
	1	04070301 TOP	Pore Water	N				ND		400		
	2	04020901	Soil	N			ND	ND	79.2	110	28	

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Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	2	04020902	Soil	N			ND	ND	803	1100	27	
	2	04020903	Soil	FD			ND	ND	79.2	110	28	
	2	04020903	Soil	N			ND	ND	814	1100	26	
	2	04021001	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	416	1300	68	UJ
	2	04021002	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	372	1200	69	UJ
	2	04021003	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	392	1400	72	UJ
	2	04021004	Plant Tissue	N	Saltbush	*Atriplex sp.*	ND	ND	418	1900	78	UJ
	2	04021005	Plant Tissue	N	Saltbush	*Atriplex sp.*	ND	ND	378	1800	79	UJ
	2	04021006	Plant Tissue	N	Saltbush	*Atriplex sp.*	ND	ND	400	1600	75	UJ
	2	04021006	Plant Tissue	FD	Saltbush	*Atriplex sp.*	ND	ND	400	2000	80	UJ
	2	04021007	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	396	1800	78	UJ
	2	04021008	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	400	2000	80	UJ
	2	04021009	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	396	1800	78	UJ
	2	04021010	Plant Tissue	N	Salt cedar leaf	*Tamarix ramosissima*	481	1300	407	1100	63	J
	2	04021011	Plant Tissue	N	Salt cedar leaf	*Tamarix ramosissima*	7600	19000	396	990	60	
	2	04021012	Plant Tissue	N	Salt cedar leaf	*Tamarix ramosissima*	ND	ND	377	1300	71	UJ
	2	04021013	Plant Tissue	FD	Saltbush	*Atriplex sp.*	2990	13000	414	1800	77	
	2	04021013	Plant Tissue	N	Saltbush	*Atriplex sp.*	3300	10000	396	1200	67	
	2	04021014	Plant Tissue	N	Saltbush	*Atriplex sp.*	3200	16000	400	2000	80	
	2	04021015	Plant Tissue	N	Saltbush	*Atriplex sp.*	864	3600	408	1700	76	J
	2	04021101	Animal Tissue	N	Desert pocket mouse	*Chaetodipus penicillatus*	ND	ND	400	1210	67	UJ
	2	04021102	Animal Tissue	N	Desert pocket mouse	*Chaetodipus penicillatus*	ND	ND	404	1300	69	UJ
	2	04021103	Animal Tissue	N	Hispid pocket rat	*Sigmodon hispidus*	ND	ND	392	1350	71	UJ
	2	04021104	Animal Tissue	N	Merriam's kangaroo rat	*Dipodomys merriami*	ND	ND	417	1390	70	UJ
	2	04021105	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	406	1560	74	UJ
	2	04021106	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	400	1430	72	UJ
	2	04021107	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	404	1440	67	UJ
	2	04021107	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	404	1440	72	UJ
	2	04021107	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	406	1230	72	UJ
	2	04021107	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	406	1230	67	UJ

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13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	2	04021107	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	406	1230	67	UJ
	2	04021107	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	404	1440	72	UJ
	2	04021107	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	406	1230	72	UJ
	2	04021107	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	404	1440	67	UJ
	2	04021108	Animal Tissue	N	Desert pocket mouse	*Chaetodipus penicillatus*	ND	ND	394	1310	70	UJ
	2	04021109	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	396	1720	77	UJ
	2	04021201	Animal Tissue	N	Mourning dove	*Zenaida macroura*	ND	ND	396	921	57	UJ
	2	04021202	Animal Tissue	N	Mourning dove	*Zenaida macroura*	ND	ND	410	1210	66	UJ
	2	04021203	Animal Tissue	N	Mourning dove	*Zenaida macroura*	ND	ND	414	1120	63	UJ
	2	04021204	Animal Tissue	N	Mourning dove	*Zenaida macroura*	ND	ND	398	1170	66	UJ
	2	04021401	Animal Tissue	N	Prairie rattlesnake	*Crotalus viridis*	ND	ND	408	1200	66	UJ
	4	04040301	Sediment	N			ND	ND	79.3	130	39	
	4	04040302	Sediment	N			ND	ND	819	1300	37	
	4	04040303	Sediment	N			ND	ND	78.0	130	40	
	5	04050101	Surface Water	N				ND		400		
	5	04050102	Surface Water	N				ND		400		
	5	04050103	Surface Water	N				ND		400		
	5	04050104	Surface Water	N				ND		400		
	5	04050105	Surface Water	N				ND		400		
	5	04050106	Surface Water	FD				ND		400		
	5	04050106	Surface Water	N				ND		400		
	5	04050301	Sediment	N			ND	ND	81.0	270	70	
	5	04050302	Sediment	N			ND	ND	80.6	260	69	
	5	04050303	Sediment	N			ND	ND	80.5	230	65	
	5	04050303	Sediment	FD			105	340	80.6	260	69	
	5	04050501	Animal Tissue	N	White Sands pupfish	*Cyprinodon tularosa*	ND	ND	419	1680	75	UJ
	5	04050502	Animal Tissue	N	White Sands pupfish	*Cyprinodon tularosa*	ND	ND	398	1530	74	UJ
	5	04050503	Animal Tissue	N	White Sands pupfish	*Cyprinodon tularosa*	ND	ND	388	1490	74	UJ
	5	04050901	Soil	N			ND	ND	78.0	130	40	
	5	04050902	Soil	N			ND	ND	78.2	170	54	

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13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	5	04050903	Soil	FD			ND	ND	80.6	130	38	
	5	04050903	Soil	N			ND	ND	79.3	130	39	
	5	04051001	Plant Tissue	N	Sea lavender	*Limonium sp.*	ND	ND	384	1600	76	UJ
	5	04051002	Plant Tissue	N	Sea lavender	*Limonium sp.*	ND	ND	399	1900	79	UJ
	5	04051003	Plant Tissue	N	Sea lavender	*Limonium sp.*	ND	ND	420	2100	80	UJ
	5	04051003	Plant Tissue	FD	Sea lavender	*Limonium sp.*	ND	ND	400	2000	80	UJ
	5	04051005	Plant Tissue	N	Grass	Family Poaceae	ND	ND	395	790	50	UJ
	5	04051006	Plant Tissue	N	Grass	Family Poaceae	ND	ND	392	890	56	UJ
	5	04051007	Plant Tissue	N	Grass	Family Poaceae	ND	ND	403	760	47	UJ
	5	04051008	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	380	1900	80	UJ
	5	04051009	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	399	1900	79	UJ
	5	04051010	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	396	2200	82	UJ
	5	04051101	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	410	1640	75	UJ
	5	04051102	Animal Tissue	N	Western harvest mouse	*Reithrodontomys megalotis*	ND	ND	392	1350	71	UJ
	5	04051103	Animal Tissue	N	Western harvest mouse	*Reithrodontomys megalotis*	ND	ND	390	1180	67	UJ
	5	04051104	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	410	1470	72	UJ
	5	04051105	Animal Tissue	N	Western harvest mouse	*Reithrodontomys megalotis*	ND	ND	410	1370	70	UJ
	5	04051201	Animal Tissue	N	Mourning dove	*Zenaida macroura*	ND	ND	385	1040	63	UJ
	6	04060101	Surface Water	N				ND		400		
	6	04060102	Surface Water	N				ND		400		
	6	04060103	Surface Water	N				ND		400		
	6	04060104	Surface Water	N				ND		400		
	6	04060105	Surface Water	N				ND		400		
	6	04060106	Surface Water	N				ND		400		
	6	04060301	Sediment	N			ND	ND	80.5	350	77	
	6	04060302	Sediment	N			ND	ND	80.0	320	75	
	6	04060303	Sediment	N			ND	ND	80.5	350	77	
	6	04060501	Animal Tissue	N	Pupfish	*Cyprinodon tularosa*	ND	ND	392	1450	73	UJ
	6	04060502	Animal Tissue	N	Pupfish	*Cyprinodon tularosa*	ND	ND	398	1530	74	UJ
	6	04060503	Animal Tissue	N	Pupfish	*Cyprinodon tularosa*	ND	ND	404	1620	75	UJ

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Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	6	04060901	Soil	N			ND	ND	78.0	150	48	
	6	04060902	Soil	N			ND	ND	78.4	140	44	
	6	04060903	Soil	N			ND	ND	81.6	160	49	
	6	04061001	Plant Tissue	N	Sea lavender	*Limonium sp.*	ND	ND	380	1900	80	UJ
	6	04061002	Plant Tissue	N	Sea lavender	*Limonium sp.*	ND	ND	396	1800	78	UJ
	6	04061003	Plant Tissue	N	Sea lavender	*Limonium sp.*	ND	ND	396	1800	78	UJ
	6	04061004	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	400	2000	80	UJ
	6	04061005	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	406	2900	86	UJ
	6	04061006	Plant Tissue	FD	Pickleweed	*Allenrolfea occidentalis*	ND	ND	399	1900	79	UJ
	6	04061006	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	408	2400	83	UJ
	6	04061008	Plant Tissue	N	Grass	Family Poaceae	ND	ND	396	1100	64	UJ
	6	04061009	Plant Tissue	N	Grass	Family Poaceae	ND	ND	396	1100	64	UJ
	6	04061010	Plant Tissue	N	Grass	Family Poaceae	ND	ND	400	800	50	UJ
	7	04070101	Surface Water	N				ND		400		
	7	04070102	Surface Water	N				ND		400		
	7	04070103	Surface Water	N				ND		400		
	7	04070104	Surface Water	N				ND		400		
	7	04070105	Surface Water	N				ND		400		
	7	04070106	Surface Water	N				ND		400		
	7	04070106	Surface Water	FD				ND		400		
	7	04070301 BOTTOM	Sediment	N			ND	ND	79.2	330	76	
	7	04070302	Sediment	N			ND	ND	81.6	240	66	
	7	04070303	Sediment	N			ND	ND	81.0	300	73	
	7	04070303	Sediment	FD			ND	ND	81.2	290	72	
	7	04070401	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	ND	ND	416	2600	84	UJ
	7	04070402	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	ND	ND	408	2400	83	UJ
	7	04070403	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	ND	ND	396	2200	82	UJ
	7	04070501	Animal Tissue	N	Pupfish	*Cyprinodon tularosa*	ND	ND	385	1480	74	UJ
	7	04070502	Animal Tissue	N	Pupfish	*Cyprinodon tularosa*	ND	ND	408	1630	75	UJ
	7	04070503	Animal Tissue	N	Pupfish	*Cyprinodon tularosa*	ND	ND	396	1580	75	UJ

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13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	7	04070901	Soil	N			ND	ND	81.6	160	49	
	7	04070902	Soil	N			ND	ND	81.6	170	52	
	7	04070903	Soil	N			ND	ND	78.4	140	44	
	7	04070903	Soil	FD			ND	ND	79.5	150	47	
	7	04071001	Plant Tissue	N	Sea lavender	*Limonium sp.*	ND	ND	416	2600	84	UJ
	7	04071002	Plant Tissue	N	Sea lavender	*Limonium sp.*	ND	ND	396	2200	82	UJ
	7	04071003	Plant Tissue	FD	Sea lavender	*Limonium sp.*	ND	ND	400	2500	84	UJ
	7	04071003	Plant Tissue	N	Sea lavender	*Limonium sp.*	ND	ND	400	2500	84	UJ
	7	04071005	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	384	2400	84	UJ
	7	04071006	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	416	2600	84	UJ
	7	04071007	Plant Tissue	N	Pickleweed	*Allenrolfea occidentalis*	ND	ND	396	2200	82	UJ
	7	04071008	Plant Tissue	N	Indian saltgrass	*Distichlis spicata*	ND	ND	395	940	58	UJ
	7	04071009	Plant Tissue	N	Indian saltgrass	*Distichlis spicata*	ND	ND	390	1000	61	UJ
	7	04071010	Plant Tissue	FD	Indian saltgrass	*Distichlis spicata*	ND	ND	380	1000	62	UJ
	7	04071010	Plant Tissue	N	Indian saltgrass	*Distichlis spicata*	ND	ND	390	1000	61	UJ
	7	04071101	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	406	1770	77	UJ
	7	04071102	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	396	1650	76	UJ
	7	04071103	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	415	1660	75	UJ
	7	04071104	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	406	1850	78	UJ
	7	04071105	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	421	1830	77	UJ
	7	04071106	Animal Tissue	N	Hispid cotton rat	*Sigmodon hispidus*	ND	ND	404	1620	75	UJ
	7	04071201	Animal Tissue	N	Mourning dove	*Zenaida macroura*	ND	ND	414	1430	71	UJ

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13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
5 Indian Head Area												
	3	05030101	Surface Water	N				ND		4.00		
	3	05030102	Surface Water	N				4.00		4.00		
	3	05030103	Surface Water	N				ND		4.00		
	3	05030104	Surface Water	N				ND		4.00		
	3	05030105	Surface Water	N				4.00		4.00		
	3	05030106	Surface Water	N				4.00		4.00		
	3	05030106	Surface Water	FD				5.00		4.00		
	3	05030301	Sediment	N			ND	ND	82.5	110	25	
	3	05030302	Sediment	N			3.80	5.00	83.6	110	24	J
	3	05030303 BOTTOM	Sediment	FD			ND	ND	83.6	110	24	
	3	05030303 BOTTOM	Sediment	N			ND	ND	8040	12000	33	
	3	05030303 TOP	Pore Water	N				ND		4.00		
	3	05030701	Animal Tissue	N	Leopard frog	*Rana pipiens*	ND	ND	408	1410	71	UJ
	3	05030702	Animal Tissue	N	Green frog	*Rana clamitans*	ND	ND	410	1870	78	UJ
	3	05030901	Soil	N			ND	ND	163	240	32	
	3	05030902	Soil	N			129	190	81.6	120	32	
	3	05030903	Soil	N			58.5	90.0	78.0	120	35	J
	3	05030905	Soil	N			132	210	81.9	130	37	
	3	05031001	Plant Tissue	N	Water pepper	*Polygonum hydropiperoides*	ND	ND	405	1500	73	UJ
	3	05031002	Plant Tissue	N	Water pepper	*Polygonum hydropiperoides*	420	2000	399	1900	79	UJ
	3	05031003	Plant Tissue	N	Water pepper	*Polygonum hydropiperoides*	891	3300	405	1500	73	J
	3	05031103	Animal Tissue	N	White-footed mouse	*Peromyscus leucopus*	ND	ND	394	1190	67	UJ
	3	05031104	Animal Tissue	N	White-footed mouse	*Peromyscus leucopus*	ND	ND	396	1130	65	UJ
	3	05031105	Animal Tissue	N	White-footed mouse	*Peromyscus leucopus*	ND	ND	390	1120	65	UJ
	3	05031106	Animal Tissue	N	Marsh rice rat	*Oryzomys palustris*	ND	ND	398	1240	68	UJ
	4	05040101	Surface Water	N				22.0		4.00		
	4	05040102	Surface Water	N				23.0		4.00		
	4	05040103	Surface Water	N				22.0		4.00		
	4	05040104	Surface Water	N				23.0		4.00		

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Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	4	05040105	Surface Water	N				23.0		4.00		
	4	05040106	Surface Water	FD				21.0		4.00		
	4	05040106	Surface Water	N				24.0		4.00		
	4	05040301	Sediment	N			92.3	130	78.1	110	29	
	4	05040302	Sediment	N			318	430	81.4	110	26	
	4	05040303	Sediment	FD			ND	ND	81.0	150	46	
	4	05040303	Sediment	N			ND	ND	79.2	110	28	
	4	05040401	Plant Tissue	N	False loosestrife	*Ludwigia palustris*	ND	ND	387	4300	91	UJ
	4	05040402	Plant Tissue	N	False loosestrife	*Ludwigia palustris*	ND	ND	387	4300	91	UJ
	4	05040403	Plant Tissue	N	False loosestrife	*Ludwigia palustris*	ND	ND	387	4300	91	UJ
	4	05040405	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	420	3500	408	3400	88	J
	4	05040406	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	360	4000	396	4400	91	UJ
	4	05040407	Plant Tissue	N	Filamentous algae	*Oscillatoria sp.*	ND	ND	406	5800	93	UJ
	4	05040501	Animal Tissue	N	Mosquito fish (mostly)	*Gambusia sp.*	ND	ND	408	1460	72	UJ
	4	05040601	Animal Tissue	N	Dragonfly larvae	Suborder Anisoptera	ND	ND	398	1470	73	UJ
	4	05040602	Animal Tissue	N	Crawfish	Order Decapoda	ND	ND	406	2260	82	UJ
	4	05040603	Animal Tissue	N	Bivalves	Class Bivalvia	ND	ND	402	874	54	UJ
	4	05040701	Animal Tissue	N	Frog (tadpole-immature)	*Rana sp.*	465	1660	394	1410	72	UJ
	4	05040702	Animal Tissue	N	Frog (tadpole-immature)	*Rana sp.*	ND	ND	410	1280	68	UJ
	4	05040901	Soil	N			63.0	100	81.9	130	37	J
	4	05040902	Soil	N			3.00	5.00	78.0	130	40	J
	4	05040903	Soil	N			ND	ND	161	230	30	
	4	05040903	Soil	FD			ND	ND	79.3	130	39	
	4	05041001	Plant Tissue	N	Water pepper	*Polygonum hydropiperoides*	ND	ND	418	1100	62	UJ
	4	05041002	Plant Tissue	N	Water pepper	*Polygonum hydropiperoides*	ND	ND	392	1400	72	UJ
	4	05041003	Plant Tissue	N	Water pepper	*Polygonum hydropiperoides*	ND	ND	390	1500	74	UJ
	4	05041101	Animal Tissue	N	House mouse	*Mus musculus*	ND	ND	417	1440	71	UJ
	4	05041301	Animal Tissue	N	Dragonfly (mature)	Suborder Anisoptera	ND	ND	406	902	55	UJ
	5	05050101	Surface Water	N				19.0		4.00		
	5	05050102	Surface Water	N				19.0		4.00		

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13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	5	05050103	Surface Water	N				20.0		4.00		
	5	05050104	Surface Water	N				17.0		4.00		
	5	05050105	Surface Water	N				19.0		4.00		
	5	05050106	Surface Water	N				18.0		4.00		
	5	05050301	Sediment	N			93.6	180	78.0	150	48	
	5	05050302	Sediment	N			90.2	110	80.4	98.0	18	
	5	05050303	Sediment	N			ND	ND	81.7	190	57	
	5	05050501	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	394	1640	76	UJ
	5	05050502	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	402	1490	73	UJ
	5	05050503	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	396	1410	72	UJ
	5	05050504	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	412	1590	74	UJ
	5	05050505	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	406	1500	73	UJ
	5	05050506	Animal Tissue	N	Gizzard shad	*Dorosoma cepedianum*	ND	ND	415	1380	70	UJ
	5	05050507	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	402	1750	77	UJ
	5	05050508	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	394	1640	76	UJ
	5	05050509	Animal Tissue	N	Gizzard shad	*Dorosoma cepedianum*	ND	ND	390	1300	70	UJ
	5	05050510	Animal Tissue	N	Gizzard shad	*Dorosoma cepedianum*	ND	ND	392	1350	71	UJ
	5	05050511	Animal Tissue	N	Gizzard shad	*Dorosoma cepedianum*	ND	ND	414	1480	72	UJ
	5	05050512	Animal Tissue	N	Eel	*Anguilla rostrata*	ND	ND	398	1470	73	UJ
	5	05050514	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	412	1650	75	UJ
	5	05050701	Animal Tissue	N	Eastern painted turtle	*Chrysemys picta*	ND	ND	392	1150	66	UJ
	5	05050702	Animal Tissue	N	Eastern painted turtle	*Chrysemys picta*	ND	ND	388	903	57	UJ
	5	05050703	Animal Tissue	N	Eastern painted turtle	*Chrysemys picta*	ND	ND	396	943	58	UJ
	5	05050704	Animal Tissue	N	Eastern painted turtle	*Chrysemys picta*	ND	ND	389	1050	63	UJ
	5	05050705	Animal Tissue	N	Eastern painted turtle	*Chrysemys picta*	ND	ND	398	865	54	UJ
	5	05050706	Animal Tissue	N	Eastern painted turtle	*Chrysemys picta*	ND	ND	394	1130	65	UJ
	5	05050707	Animal Tissue	N	Frog (tadpole-immature)	*Rana sp.*	ND	ND	396	2080	81	UJ
	5	05050901	Soil	N			ND	ND	76.8	120	36	
	5	05050902	Soil	N			ND	ND	81.6	120	32	
	5	05050903	Soil	N			ND	ND	80.3	110	27	

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13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	5	05051001	Plant Tissue	N	Water pepper	*Polygonum hydropiperoides*	931	4900	399	2100	81	J
	5	05051002	Plant Tissue	N	Water pepper	*Polygonum hydropiperoides*	200	1000	400	2000	80	UJ
	5	05051003	Plant Tissue	N	Water pepper	*Polygonum hydropiperoides*	408	1700	384	1600	76	J
	5	05051101	Animal Tissue	N	Northern short-tailed shrew	*Blarina brevicauda*	ND	ND	388	1080	64	UJ
	5	05051101-A	Animal Tissue	N	White-footed mouse	*Peromyscus leucopus*	ND	ND	419	1230	66	UJ
	5	05051102	Animal Tissue	N	Racoon	*Procyon lotor*	ND	ND	412	1210	66	UJ
	5	05051201	Animal Tissue	N	Starling	*Sturnus vulgaris*	ND	ND	402	1120	64	UJ
	5	05051202	Animal Tissue	N	Starling	*Sturnus vulgaris*	ND	ND	402	1150	65	UJ
	5	05051203	Animal Tissue	N	Dove	*Zenaida macroura*	ND	ND	394	857	54	UJ
	5	05051301	Animal Tissue	N	Moths	Order Lepidoptera	ND	ND	398	1140	65	UJ
	5	05051302	Animal Tissue	N	Dragonfly (mature)	Suborder Anisoptera	ND	ND	400	1050	62	UJ
	6	05060101	Surface Water	N				25.0		4.00		
	6	05060102	Surface Water	N				25.0		4.00		
	6	05060103	Surface Water	N				25.0		4.00		
	6	05060104	Surface Water	N				25.0		4.00		
	6	05060105	Surface Water	N				24.0		4.00		
	6	05060106	Surface Water	N				25.0		4.00		
	6	05060301	Sediment	N			ND	ND	81.2	290	72	
	6	05060302	Sediment	N			ND	ND	82.5	110	25	
	6	05060303	Sediment	N			ND	ND	810	3000	73	
	6	05060501	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	396	1470	73	UJ
	6	05060502	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	366	1530	76	UJ
	6	05060503	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	410	1640	75	UJ
	6	05060504	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	406	1620	75	UJ
	6	05060505	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	396	1800	78	UJ
	6	05060506	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	402	1550	74	UJ
	6	05060507	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	396	1720	77	UJ
	6	05060508	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	404	1620	75	UJ
	6	05060509	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	408	1630	75	UJ
	6	05060510	Animal Tissue	N	Crappie	*Pomoxis nigromaculatus*	ND	ND	392	1510	74	UJ

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Data Summary-Clayton Laboratory

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	6	05060592	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	412	1530	73	
	6	05060701	Animal Tissue	N	Eastern painted turtle	*Chrysemys picta*	ND	ND	412	982	58	UJ
	6	05060901	Soil	N			ND	ND	83.6	110	24	
	6	05060902	Soil	N			546	700	164	210	22	
	6	05060903	Soil	N			ND	ND	158	200	21	
	6	05061101	Animal Tissue	N	White-footed mouse	*Peromyscus leucopus*	ND	ND	392	1230	68	UJ
	6	05061301	Animal Tissue	N	Dragonfly (mature)	Suborder Anisoptera	ND	ND	396	966	59	UJ
	6	05061302	Animal Tissue	N	Earthworm	Class Oligochaeta	ND	ND	412	1330	69	UJ
	6	05061303	Animal Tissue	N	Earthworm	Class Oligochaeta	ND	ND	396	1470	73	UJ
	6	05061304	Animal Tissue	N	Earthworm	Class Oligochaeta	ND	ND	394	1070	63	UJ
	7	05070101	Surface Water	N				12.0		4.00		
	7	05070102	Surface Water	N				12.0		4.00		
	7	05070103	Surface Water	N				12.0		4.00		
	7	05070104	Surface Water	N				12.0		4.00		
	7	05070105	Surface Water	N				12.0		4.00		
	7	05070106	Surface Water	N				12.0		4.00		
	7	05070106	Surface Water	FD				11.0		4.00		
	7	05070301	Sediment	N			ND	ND	81.6	160	49	
	7	05070302 BOTTOM	Sediment	N			ND	ND	81.0	180	55	
	7	05070302 TOP	Pore Water	N				ND		4.00		
	7	05070303	Sediment	N			ND	ND	81.6	160	49	
	7	05070401	Plant Tissue	N	Hydrilla	*Hydrilla verticillata*	ND	ND	384	4800	92	UJ
	7	05070402	Plant Tissue	N	Hydrilla	*Hydrilla verticillata*	180	2000	405	4500	91	UJ
	7	05070403	Plant Tissue	N	Hydrilla	*Hydrilla verticillata*	200	2000	400	4000	90	UJ
	7	05070503	Animal Tissue	N	Frog (tadpole-immature)	*Rana sp.*	ND	ND	402	2230	82	UJ
	7	05070504	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	414	1540	73	UJ
	7	05070505	Animal Tissue	N	Eel	*Anguilla rostrata*	ND	ND	417	1810	77	UJ
	7	05070506	Animal Tissue	N	Carp	*Cyprinus carpio*	ND	ND	385	1600	76	UJ
	7	05070507	Animal Tissue	N	Bluegill sunfish	*Lepomis macrochirus*	ND	ND	388	1620	76	UJ
	7	05070508	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	385	1670	77	UJ

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Data Summary-Clayton Laboratory

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	7	05070509	Animal Tissue	N	Brown bullhead catfish	*Ictalurus nebulosus*	ND	ND	398	2210	82	UJ
	7	05070510	Animal Tissue	N	Shiner	*Notropis sp.*	ND	ND	408	1700	76	UJ
	7	05070601	Animal Tissue	N	Crawfish	Order Decapoda	ND	ND	419	1610	74	UJ
	7	05070602	Animal Tissue	N	Crawfish	Order Decapoda	ND	ND	406	1620	75	UJ
	7	05070603	Animal Tissue	N	Crawfish	Order Decapoda	ND	ND	404	1680	76	UJ
	7	05070901	Soil	N			ND	ND	159	230	31	
	7	05070902	Soil	N			ND	ND	79.0	100	21	
	7	05070903	Soil	N			176	210	79.8	95.0	16	
	7	05071001	Plant Tissue	N	Lizard's tail	*Saururus cernuus*	ND	ND	396	1800	78	UJ
	7	05071002	Plant Tissue	N	Lizard's tail	*Saururus cernuus*	ND	ND	400	2000	80	UJ
	7	05071003	Plant Tissue	N	Lizard's tail	*Saururus cernuus*	220	1000	396	1800	78	UJ
	7	05071004	Plant Tissue	N	Lizard's tail	*Saururus cernuus*	ND	ND	385	940	59	UJ
	7	05071005	Plant Tissue	N	Lizard's tail	*Saururus cernuus*	ND	ND	396	1100	64	UJ
	7	05071006	Plant Tissue	N	Lizard's tail	*Saururus cernuus*	ND	ND	403	1300	69	UJ
	7	05071101	Animal Tissue	N	White-footed mouse	*Peromyscus leucopus*	ND	ND	388	1250	69	UJ
	7	05071102	Animal Tissue	N	Marsh rice rat	*Oryzomys palustris*	ND	ND	398	1330	70	UJ
	7	05071301	Animal Tissue	N	Terrestrial insect (misc.)	Class Insecta	ND	ND	400	952	58	UJ
	7	05071301 Alpha	Animal Tissue	N	Terrestrial insect (misc.)	Class Insecta	ND	ND	417	2080	80	UJ
	7	05071302	Animal Tissue	N	Dragonfly (mature)	Suborder Anisoptera	ND	ND	394	1100	64	UJ
	8	05080101	Surface Water	N				ND		4.00		
	8	05080102	Surface Water	N				ND		4.00		
	8	05080103	Surface Water	N				ND		4.00		
	8	05080104	Surface Water	N				ND		4.00		
	8	05080105	Surface Water	N				ND		4.00		
	8	05080106	Surface Water	N				ND		4.00		
	8	05080301	Sediment	N			ND	ND	82.8	120	31	
	8	05080302	Sediment	N			ND	ND	79.8	140	43	
	8	05080303	Sediment	N			ND	ND	80.2	99.0	19	
	8	05080901	Soil	N			ND	ND	164	210	22	
	8	05080902	Soil	N			ND	ND	158	210	25	

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Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	8	05080903	Soil	N			ND	ND	164	200	18	
	8	05081001	Plant Tissue	N	Lizard's tail	*Saururus cernuus*	ND	ND	392	1400	72	UJ
	8	05081002	Plant Tissue	N	Lizard's tail	*Saururus cernuus*	ND	ND	407	1100	63	UJ
	8	05081003	Plant Tissue	N	Lizard's tail	*Saururus cernuus*	270	1000	405	1500	73	UJ
	8	05081101	Animal Tissue	N	Marsh rice rat	*Oryzomys palustris*	ND	ND	402	1340	70	UJ

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Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
6 LHAAP												
	2	06020101	Surface Water	N				3600		2000		
	2	06020102	Surface Water	N				3800		2000		
	2	06020103	Surface Water	N				3700		2000		
	2	06020104	Surface Water	N				3700		2000		
	2	06020105	Surface Water	N				3500		2000		
	2	06020106	Surface Water	N				3800		2000		
	2	06020301	Sediment	N			286	440	78.0	120	35	
	2	06020302	Sediment	N			ND	ND	77.0	110	30	
	2	06020303	Sediment	N			143	220	78.0	120	35	
	2	06020401	Plant Tissue	N	Sedge	*Dichromena sp.*	11000	22900	1990	4150	52	
	2	06020402	Plant Tissue	N	Sedge	*Dichromena sp.*	15700	28100	2020	3610	44	
	2	06020403	Plant Tissue	N	Sedge	*Dichromena sp.*	9140	24100	402	1060	62	
	2	06020404	Plant Tissue	N	Bullrush	*Scirpus sp.*	2160	30800	404	5770	93	
	2	06020405	Plant Tissue	N	Bulrush	*Scirpus sp.*	1220	15200	404	5050	92	
	2	06020406	Plant Tissue	N	Bullrush	*Scirpus sp.*	1260	14000	406	4510	91	
	2	06020601	Animal Tissue	N	Dragonfly larvae	Anisoptera - larvae	624	4460	396	2830	86	J
	2	06020602	Animal Tissue	N	Dragonfly larvae	Anisoptera - larvae	603	3770	404	2530	84	J
	2	06020603	Animal Tissue	N	Dragonfly larvae	Anisoptera - larvae	996	5860	412	2430	83	J
	2	06020701	Animal Tissue	N	Frog (tadpole-immature)	*Rana catesbeiana*	1050	6180	390	2300	83	
	2	06020702	Animal Tissue	N	Frog (tadpole-immature)	*Rana catesbeiana*	867	5420	412	2580	84	
	2	06020703	Animal Tissue	N	Frog (tadpole-immature)	*Rana catesbeiana*	1660	9760	406	2390	83	J
	2	06020704	Animal Tissue	N	Frog (tadpole-immature)	*Rana catesbeiana*	1230	7240	388	2280	83	J
	2	06020901	Soil	N			ND	ND	79.5	82.0	3	
	2	06020902	Soil	N			ND	ND	79.7	83.0	4	
	2	06020903	Soil	N			ND	ND	79.5	82.0	3	
	2	06020904	Soil	N			ND	ND	79.9	85.0	6	
	2	06021001	Plant Tissue	N	Loblolly pine needles	*Pinus taeda*	1960	5030	406	1040	61	
	2	06021002	Plant Tissue	N	Loblolly pine needles	*Pinus taeda*	1490	4140	398	1110	64	
	2	06021003	Plant Tissue	N	Loblolly pine needles	*Pinus taeda*	2340	6160	390	1030	62	

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Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	2	06021101	Animal Tissue	N	Eastern harvest mouse	*Reithrodontomys fulvescens*	571	1970	402	1390	71	J
	2	06021102	Animal Tissue	N	Fulvous harvest mouse	*Reithrodontomys fulvescens*	ND	ND	410	1420	71	UJ
	2	06021103	Animal Tissue	N	Little brown bat	*Myotis lucifugus*	ND	ND	390	1220	68	UJ
	2	06021201	Animal Tissue	N	Eastern bluebird	*Sialia sialis*	ND	ND	390	1220	68	UJ
	2	06021202	Animal Tissue	N	Eastern phoebe	*Sayornis phoebe*	ND	ND	384	1070	64	UJ
	2	06021203	Animal Tissue	N	Eastern phoebe	*Sayornis phoebe*	ND	ND	406	1160	65	UJ
	2	06021301	Animal Tissue	N	Terrestrial insect (misc.)	Class Insecta	ND	ND	385	2020	81	UJ
	3	06030101	Surface Water	N				ND		4.00		
	3	06030102	Surface Water	N				ND		4.00		
	3	06030103	Surface Water	N				ND		4.00		
	3	06030104	Surface Water	N				ND		4.00		
	3	06030105	Surface Water	N				ND		4.00		
	3	06030106	Surface Water	N				ND		4.00		
	3	06030301	Sediment	N			ND	ND	78.2	170	54	
	3	06030302	Sediment	N			ND	ND	79.5	150	47	
	3	06030303	Sediment	N			ND	ND	81.4	220	63	
	3	06030401	Plant Tissue	N	Water lily	Family Nymphaeaceae	ND	ND	394	4930	92	UJ
	3	06030402	Plant Tissue	N	Water lily	Family Nymphaeaceae	ND	ND	398	3620	89	UJ
	3	06030403	Plant Tissue	N	Water lily	Family Nymphaeaceae	ND	ND	394	4380	91	UJ
	3	06030404	Plant Tissue	N	Pondweed	*Potamogeton sp.*	ND	ND	404	3370	88	UJ
	3	06030405	Plant Tissue	N	Pondweed	*Potamogeton sp.*	ND	ND	381	5440	93	UJ
	3	06030406	Plant Tissue	N	Pondweed	*Potamogeton sp.*	ND	ND	394	3580	89	UJ
	3	06030501	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	387	1760	78	
	3	06030502	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	404	1840	78	
	3	06030503	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	406	1620	75	
	3	06030504	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	398	1660	76	
	3	06030505	Animal Tissue	N	Pickerel	*Esox niger*	ND	ND	388	1690	77	
	3	06030506	Animal Tissue	N	Catfish	*Ictalurus sp.*	ND	ND	412	2170	81	UJ
	3	06030507	Animal Tissue	N	Catfish	*Ictalurus sp.*	ND	ND	402	2010	80	UJ
	3	06030508	Animal Tissue	N	Pickerel	*Esox niger*	ND	ND	417	1670	75	UJ

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Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	3	06030701	Animal Tissue	N	Red ear slider turtle	*Trachemys scripta*	ND	ND	404	1550	74	UJ
	3	06030702	Animal Tissue	N	Red ear slider turtle	*Trachemys scripta*	ND	ND	388	1620	76	UJ
	3	06030703	Animal Tissue	N	Red ear slider turtle	*Trachemys scripta*	ND	ND	400	1600	75	UJ
	3	06030901	Soil	N			ND	ND	77.0	110	30	
	3	06030902	Soil	N			ND	ND	79.2	120	34	
	3	06030903	Soil	N			ND	ND	78.0	100	22	
	3	06031001	Plant Tissue	N	Misc. terrestrial vegetation	---	ND	ND	400	1910	79	UJ
	3	06031002	Plant Tissue	N	Misc. terrestrial vegetation	---	ND	ND	408	2040	80	UJ
	3	06031003	Plant Tissue	N	Misc. terrestrial vegetation	---	ND	ND	398	1810	78	UJ
	3	06031004	Plant Tissue	N	Sedge	*Carex sp.*	ND	ND	404	1300	69	UJ
	3	06031005	Plant Tissue	N	Sedge	*Carex sp.*	ND	ND	392	1450	73	UJ
	3	06031006	Plant Tissue	N	Sedge	*Carex sp.*	ND	ND	402	1490	73	UJ
	3	06031101	Animal Tissue	N	Rice Rat	*Oryzomys palustris*	ND	ND	398	1370	71	UJ
	3	06031101A	Animal Tissue	N	Eastern cottontail	*Pseudopamera rubricata*	ND	ND	394	1520	74	UJ
	3	06031102	Animal Tissue	N	Eastern harvest mouse	*Reithrodontomys fulvescens*	ND	ND	386	1380	72	UJ
	3	06031201	Animal Tissue	N	Mourning dove	*Zenaida macroura*	ND	ND	385	1170	67	UJ
	3	06031202	Animal Tissue	N	Eastern bluebird	*Sialia sialis*	ND	ND	408	1240	67	
	3	06031203	Animal Tissue	N	Eastern bluebird	*Sialia sialis*	ND	ND	402	1150	65	
	3	06031204	Animal Tissue	N	Grosebeak	*Guiraca caerulea*	ND	ND	406	923	56	
	3	06031205	Animal Tissue	N	Mourning dove	*Zenaida macroura*	ND	ND	390	1030	62	UJ
	3	06031206	Animal Tissue	N	Mourning dove	*Zenaida macroura*	ND	ND	412	1290	68	UJ
	3	06031207	Animal Tissue	N	Eastern phoebe	*Sayornis phoebe*	ND	ND	410	1210	66	UJ
	3	06031208	Animal Tissue	N	Eastern bluebird	*Sialia sialis*	ND	ND	410	1580	74	UJ
	3	06031301	Animal Tissue	N	Grasshopper	*Melanoplus sp.*	ND	ND	392	1230	68	
	3	06031302	Animal Tissue	N	Eastern bluebird	*Sialia sialis*	ND	ND	408	2150	81	
	4	06040101	Surface Water	N				ND		4.00		
	4	06040102	Surface Water	N				ND		4.00		
	4	06040103	Surface Water	N				ND		4.00		
	4	06040104	Surface Water	N				ND		4.00		
	4	06040105	Surface Water	N				ND		4.00		

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13-Jun-01

Data Summary-Clayton Laboratory

Site	Location	Sample ID	Matrix	Sample Code	Common Name	Scientific Name	Adjusted Result	Reported Result	Adj RL	RL	Percent Moisture	Flags
	4	06040106	Surface Water	N				ND		4.00		
	4	06040106	Surface Water	FD				ND		4.00		
	4	06040301	Sediment	N			ND	ND	82.8	120	31	
	4	06040302	Sediment	N			ND	ND	82.5	110	25	
	4	06040303	Sediment	N			ND	ND	82.8	120	31	
	4	06040303	Sediment	FD			ND	ND	81.4	110	26	
	4	06040501	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	394	1580	75	
	4	06040502	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	402	1610	75	
	4	06040503	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	385	1600	76	
	4	06040504	Animal Tissue	N	Pickerel	*Esox niger*	ND	ND	392	1570	75	
	4	06040505	Animal Tissue	N	Suckers/gambusia/pirate perch	Family Aphredoderidae	ND	ND	390	1770	78	
	4	06040506	Animal Tissue	N	Yellow bullhead catfish	*Ictalurus natalis*	ND	ND	400	2110	81	
	4	06040507	Animal Tissue	N	Yellow bullhead catfish	*Ictalurus natalis*	ND	ND	388	1940	80	
	4	06040508	Animal Tissue	N	Golden shiner	*Notemigonus crysoleucas*	ND	ND	386	1680	77	
	4	06040509	Animal Tissue	N	Golden shiner	*Notemigonus crysoleucas*	ND	ND	390	1500	74	
	4	06040510	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	390	1560	75	
	4	06040701	Animal Tissue	N	Bullfrog (mature)	*Rana catesbeiana*	ND	ND	390	1770	78	
	4	06040901	Soil	N			ND	ND	80.4	98.0	18	
	4	06040902	Soil	N			ND	ND	79.0	100	21	
	4	06040903	Soil	N			ND	ND	80.0	87.0	8	
	4	06041101	Animal Tissue	N	Racoon	*Procyon lotor*	ND	ND	404	1350	70	UJ
	4	06041401	Animal Tissue	N	Cottonmouth watersnake	*Agkistrodon piscivorus*	ND	ND	400	1910	79	UJ
	4	06041451	Animal Tissue	N	Texas bluestripe garter snake	*Thamnophis sirtalis*	ND	ND	404	1500	73	UJ
	5	06050101	Surface Water	N				ND		4.00		
	5	06050102	Surface Water	N				ND		4.00		
	5	06050103	Surface Water	N				ND		4.00		
	5	06050104	Surface Water	N				ND		4.00		
	5	06050105	Surface Water	N				ND		4.00		
	5	06050106	Surface Water	FD				ND		4.00		
	5	06050106	Surface Water	N				ND		4.00		

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	5	06050107	Surface Water	N				ND		4.00		
	5	06050301	Sediment	N			ND	ND	80.5	350	77	
	5	06050302	Sediment	N			ND	ND	80.5	350	77	
	5	06050303	Sediment	FD			ND	ND	81.0	270	70	
	5	06050303	Sediment	N			ND	ND	80.0	320	75	
	5	06050304 TOP	Pore Water	N				ND		4.00		
	5	06050501	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	386	1610	76	UJ
	5	06050502	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	410	1710	76	UJ
	5	06050503	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	394	1710	77	UJ
	5	06050504	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	417	1740	76	UJ
	5	06050505	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	396	1520	74	UJ
	5	06050506	Animal Tissue	N	Sunfish	*Lepomis sp.*	ND	ND	398	1730	77	UJ
	5	06050507	Animal Tissue	N	Largemouth bass	*Micropterus salmoides*	ND	ND	392	1780	78	UJ
	5	06050508	Animal Tissue	N	Catfish	*Ictalurus sp.*	ND	ND	394	1970	80	UJ
	5	06050509	Animal Tissue	N	Minnow/shiner (misc.)	Family Cyprinidae	ND	ND	408	2040	80	UJ
	5	06050510	Animal Tissue	N	Pickerel	*Esox niger*	ND	ND	396	1980	80	UJ
	5	06050601	Animal Tissue	N	Crawfish	Order Decapoda	ND	ND	390	1700	77	UJ
	5	06050602	Animal Tissue	N	Water measures	Family Hydrometridae	ND	ND	413	1250	67	UJ
	5	06050701	Animal Tissue	N	Green tree frog	*Hyla cinerea*	ND	ND	408	1630	75	
	5	06050901	Soil	N			ND	ND	82.5	110	25	
	5	06050902	Soil	N			ND	ND	79.8	95.0	16	
	5	06050903	Soil	N			ND	ND	80.1	90.0	11	
	5	06050903	Soil	FD			ND	ND	80.1	88.0	9	
	5	06051001	Plant Tissue	N	Misc. terrestrial vegetation	---	ND	ND	406	2540	84	UJ
	5	06051002	Plant Tissue	N	Misc. terrestrial vegetation	---	442	2600	404	2380	83	J
	5	06051003	Plant Tissue	N	Misc. terrestrial vegetation	---	428	2520	404	2380	83	J
	5	06051004	Plant Tissue	N	Misc. terrestrial vegetation	---	905	4310	388	1850	79	J
	5	06051005	Plant Tissue	N	Misc. terrestrial vegetation	---	644	3390	398	2100	81	J
	5	06051006	Plant Tissue	N	Misc. terrestrial vegetation	---	641	3050	396	1890	79	J
	5	06051401	Animal Tissue	N	Broad banded watersnake	*Nerodia fasciata*	ND	ND	400	1820	78	UJ

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	5	06051402	Animal Tissue	N	Cottonmouth watersnake	*Agkistrodon piscivorus*	ND	ND	394	1790	78	UJ
	5	06051451	Animal Tissue	N	Skinks	Family Scincidae	ND	ND	404	1260	68	

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