

# **Ecological Risk Assessment For Range 17 (Trap and Skeet Range)**

Patuxent Research Refuge  
Laurel, MD



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## TABLE OF CONTENTS

LIST OF FIGURES .....	v
LIST OF TABLES .....	vi
ACRONYMS AND ABBREVIATIONS.....	vii
EXECUTIVE SUMMARY .....	viii
1.0 INTRODUCTION.....	1
1.1 Site History .....	1
1.2 Ecological Setting.....	1
1.3 Current Conditions .....	2
2.0 TECHNICAL APPROACH.....	2
2.1 Selection of Contaminants of Potential Concern .....	2
2.2 Exposure Characterization .....	3
2.3 Problem Formulation .....	3
2.4 Assessment Endpoints .....	3
2.5 Measurement Endpoints.....	3
2.6 Conceptual Model.....	4
2.7 Assessment Endpoint No. 1 – Survival and Growth of Terrestrial Invertebrates .....	4
2.8 Assessment Endpoint No. 2 – Reproductive Success of Insectivorous Birds.....	5
2.9 Assessment Endpoint No. 3 – Reproductive Success of Insectivorous Mammals.....	5
2.10 Assessment Endpoint No. 4 – Survival of Gallinaceous Birds.....	5
3.0 METHODS .....	6
3.1 Extent of Contamination .....	6
3.1.1 X-ray Fluorescence Analysis for Metals .....	6
3.1.2 Confirmation Analysis .....	7
3.2 Risk Assessment.....	7
3.2.1 TAL Metals .....	7
3.2.2 Organic Compound Analysis.....	7
3.2.3 Grain Size and Total Organic Carbon .....	7
3.2.4 Earthworm Toxicity Test.....	8
3.2.5 Food-Chain Ingestion Models.....	8
3.2.5.1 Insectivorous Birds.....	9
3.2.5.2 Insectivorous Mammals.....	9
3.2.6 Lead Shot and Grit Counts .....	10
3.2.7 Probability Model for Gallinaceous Birds .....	10
3.3 Wetland Evaluation .....	12
3.4 Sampling Equipment Decontamination.....	12
3.5 Sample Documentation and Shipment.....	12
4.0 RESULTS .....	12
4.1 Extent of Contamination Results.....	13
4.2 Risk Assessment Results.....	13
4.2.1 TAL Metals Results .....	13
4.2.2 Organic Compound Results.....	14
4.2.3 Grain Size and Total Organic Carbon .....	14
4.2.4 Earthworm Toxicity Test Results .....	14
4.2.5 Food-Chain Ingestion Models.....	16
4.2.5.1 Insectivorous Birds.....	16
4.2.5.2 Insectivorous Mammals.....	16

4.2.6	Lead Shot and Grit Counts .....	16
4.2.7	Probability Model for Gallinaceous Birds .....	16
4.3	Wetland Evaluation .....	16
5.0	DISCUSSION .....	17
5.1	Assumptions.....	17
5.2	Risk Characterization.....	18
5.3	Discussion of Hazard Evaluation.....	18
5.3.1	Assessment Endpoint No. 1 - Survival and Growth of Terrestrial Invertebrates .....	18
5.3.2	Assessment Endpoint No. 2 - Reproductive Success of Insectivorous Birds .....	19
5.3.3	Assessment Endpoint No. 3 - Reproductive Success of Insectivorous Mammals.....	19
5.3.4	Assessment Endpoint No. 4 - Survival of Gallinaceous Birds.....	19
5.4	Sources of Uncertainty.....	19
6.0	CONCLUSIONS .....	21
7.0	REFERENCES ..	21
APPENDIX A	Life History and Toxicity Profiles	
APPENDIX B	X-ray Fluorescence Final Report	
APPENDIX C	Final Chemical and Physical Analytical Reports	
APPENDIX D	Toxicity Evaluation Report	

## LIST OF FIGURES

No.	Title
1.	Site Location Map, Range 17, Laurel, MD
2.	Lead Concentrations, Range 17, Laurel, MD

## LIST OF TABLES

Number	Title
1.	Results of Arsenic, Lead, Antimony, and Copper in Surface Soil Analyzed Using X-ray Fluorescence
2.	Confirmation Results of Surface Soil Samples Analyzed Using an ICAP Method
3.	Target Analyte List Metals Analysis of Surface Soil Samples
4.	Comparison of Target Analyte List Metals to Ecological Benchmarks
5.	Results of the 28-day Toxicity Test Using the Earthworm, <i>Eisenia foetida</i>
6.	Life History Parameters Used in the Food Chain and Shot Ingestion Models
7.	Hazard Quotient Calculations for the American Robin
8.	Hazard Quotient Calculations for the Short-tailed Shrew
9.	Results of Lead Shot and Grit Counts
10.	Probability of a Mourning Dove Ingesting Lead Shot
11.	Remedial Goals Based on Soil Lead Concentration
12.	Remedial Goals for Lead Shot

## ACRONYMS AND ABBREVIATIONS

%	percent
°C	degrees Centigrade
AAHSTO	American Association of State Highway and Transportation Officials
As	arsenic
ASTM	American Society for Testing and Materials
AUF	area use factor
BERA	baseline ecological risk assessment
BNA	base, neutral, and acid extractable compound
BSAF	biota-soil/sediment accumulation factor
BW	body weight
cm	centimeters
COPC	contaminant of potential concern
Cu	copper
ERTC	Environmental Response Team Center
ft <sup>2</sup>	square foot
g	grams
GPS	global positioning system
HQ	hazard quotient
ICAP	inductively coupled argon plasma analysis
kg	kilograms
LD <sub>50</sub>	lethal dose that kills 50 percent of test animals
LOAEL	lowest observed adverse effect level
M	meter
MDL	method detection limit
µg/kg	microgram per kilogram
mg	milligrams
mg/kg	milligram per kilogram
mL	milliliter
mm	millimeter
MD	Maryland
NOAEL	no observable adverse affect level
PCB	polychlorinated biphenyl
PRR	Patuxent Research Refuge
PAHs	polycyclic aromatic hydrocarbons
Pb	lead
ppm	parts per million
QA	quality assurance
REAC	Response, Engineering, and Analytical Contract
RG	remedial goal
Sb	antimony
SLERA	screening level ecological risk assessment
SOP	standard operating procedure
TAL	Target Analyte List
TCL	Target Compound List
TOC	total organic carbon
TRV	toxicity reference value
U.S. EPA	United States Environmental Protection Agency
U.S. FWS	United States Fish and Wildlife Service
UXO	unexploded ordnance
XRF	x-ray fluorescence meter

## EXECUTIVE SUMMARY

On 28 and 29 July 2003, soil samples were collected from Trap and Skeet Range 17 located on the Patuxent Research Refuge (PRR) located in Laurel, Maryland (MD). The samples were analyzed for antimony (Sb), arsenic (As), copper (Cu), and lead (Pb) using a field portable x-ray fluorescence (XRF) analyzer. Antimony, As, and Cu were selected for analysis because they are impurities in Pb shot. The results of these analyses were used to determine the extent of contamination at this range. Based on XRF results, 6 of the locations were selected for the following additional analyses: Target Analyte List (TAL) metals; base, neutral, and acid extractable compounds (BNAs); polychlorinated biphenyl (PCB)/pesticides (2 samples); grain size; total organic carbon (TOC); Pb shot count (10 samples); and a 28-day earthworm toxicity test. The results of these analyses were used to determine the risk to biota from the exposure to contaminants in the soil and from the ingestion of Pb shot. In addition, a wetland delineation was conducted to determine the presence of jurisdictional wetlands on this range.

The concentration of Pb in the soil ranged from below the method detection limit (MDL) of 39 milligrams per kilogram (mg/kg) along the periphery of the range to 22,000 mg/kg in the center of the range's fall zone. These results were based on the XRF analysis of soil samples.

Six soil samples were then analyzed for TAL metals and the concentration of these metals were compared to ecological benchmarks. Based on this comparison, 9 metals (in addition to As, Sb, Cu, and Pb) were further evaluated in this risk assessment. Soil samples were analyzed for BNAs to determine the presence of coal tar pitch (a binding agent used in the clay targets). No BNAs were detected above the MDL. Two soil samples were analyzed for pesticides and PCBs to determine if these compounds were present on the range at significant levels. No pesticides or PCBs were detected above the MDL in the samples.

The number of Pb shot was measured in 10 samples. The number of Pb shot in the samples ranged from 10 to 2,946 per square foot (ft<sup>2</sup>). No shot were found at the reference location.

There was a significant reduction in the survival of the earthworms in all on-site samples when compared to the reference soil. There was 97.5 percent (%) earthworm survival in the reference soil sample (which contained 46 mg/kg Pb). The earthworms exposed to soil collected from Location 150R-150D (which contained 260 mg/kg Pb) had 81.5% survival and from Location 150R-50D (which contained 270 mg/kg Pb) had 67.5% survival following a 28-day exposure. No earthworms survived in the remaining 3 samples tested (containing 540 mg/kg, 3,000 mg/kg, and 44,000 mg/kg Pb). The concentrations of the 9 additional metals that required further evaluation were compared to the results of the toxicity tests. This comparison indicated that, with the exception of Pb, metals do not represent a risk to biota.

Food chain accumulation models were used to evaluate the impact of Pb contaminated soil to biota that may feed on the range. The models indicated that there was risk to both insectivorous birds and mammals. In addition, a grit ingestion model was used to determine risk from the ingestion of Pb shot. This model indicated that there was risk to gallinaceous birds.

Based on the results of the wetland delineation, no jurisdictional wetlands were found at this site.

The results of the toxicity test and the food chain accumulation models were used to develop soil remedial goals for Pb in soil. The lowest observed adverse effect level (LOAEL) based clean up goal for Pb was 260 mg/kg and the no observed adverse effect level (NOAEL) based clean up goal for Pb was 46 mg/kg. A grit ingestion model was used to determine a remedial goal for Pb shot. A remedial goal of 3 shot/ft<sup>2</sup>, not to exceed 13 shot/ft<sup>2</sup>, was calculated based on a 10 % probability of a gallinaceous bird ingesting Pb shot.



## 1.0 INTRODUCTION

The first objective of this project was to determine the extent of Pb contamination at Trap and Skeet Range 17, located on the North Tract of the PRR, Laurel, MD (Figure 1). The second objective of the project was to evaluate the ecological effects of Pb shot and Pb-contaminated soil at the range. A soil toxicity test using earthworms, food chain accumulation models, and a Pb shot ingestion probability model were used to evaluate risk to receptor species. Based on the results of the risk assessment, remedial goals were developed for the clean up of Pb shot and Pb-contaminated soil. The third objective of this project was to determine if any portion of the site meets the criteria of a jurisdictional wetland.

### 1.1 Site History

Range 17 was used as a trap and skeet range. A trap range uses 5 shooting positions to fire at clay targets launched from a center trap house. The targets are thrown at different angles away from the trap house. A skeet range uses 8 shooting positions to fire at clay targets launched from both high and low houses. The targets are thrown at the same pattern but the angle of shot varies because the shooter moves to the different positions. These shooting angles tend to create a semicircular pattern of Pb shot as it falls to the ground.

Range 17 opened in the mid 1970s and was originally part of Fort George G. Meade (Vyas et al. 2000). The range was transferred to the U.S. Fish and Wildlife Service (U.S. FWS) in 1991. Since the transfer, the range operated from one to two nights per week until 1999, when the range was closed. The range was closed because data indicated that birds using the site were exposed to Pb (Vyas et al. 2000). Because the mission of the PRR is to conserve and protect wildlife, this risk assessment was completed in order to determine the risk to biota that use this site as well as to develop clean up goals.

Prior to the transfer of the North Tract to PRR, there were 17 active shooting ranges on this property. These ranges were used for rifle, pistol, and larger weapon practice. Currently, there are still 10 active ranges used for target practice by federal, state, and local organizations. All of the ranges are situated so that the impact zone is towards the center of the North Tract. Therefore, although Range 17 was historically used as a recreational range, there is the potential for unexploded ordnance (UXO) from other ranges. For example, during this sampling event, an empty 155-mm shell casing, as well as several impact craters, was noted within the sampling grid. Although it is not expected that the presence of larger ordnance will confound the results of the risk assessment, a subset of the soil samples were analyzed for a variety of organic and inorganic compounds to determine the presence of other contaminants.

In addition, Range 25, which is located north of Range 17, was used for small arms target practice. This range is inactive and is currently used as a wildlife viewing area. However, the safety fan for Range 17 overlaps Range 25, therefore, sampling activities were not restricted to within the actual boundaries of Range 17.

### 1.2 Ecological Setting

The 8,100 acre North Tract of the PRR is located in the coastal plain of central MD and is comprised mostly of a large contiguous forest (approximately 6,400 acres). This forest is connected with the larger, forested acreage of the Central and South Tracts of the PRR and the Beltsville Agricultural Research Center to the south. Together, these lands constitute the largest (more than 13,000 acres) contiguous forest in the coastal plain of MD.

Within the largely upland oak and pine forests lie extensive bottomland hardwood forests along the Little Patuxent and Patuxent Rivers. The North Tract is located in an area known as the Great Fork area, due to the presence of these two rivers. The largely undisturbed bottomland hardwood forest serves to protect the water quality in the Chesapeake Bay. A large portion of the North

Tract, including Range 17, within a 100-year flood plain, and thus this area is part of the Chesapeake Bay Critical Area.

The Critical Area Act was a resource protection program that was passed in 1984 by the MD General Assembly. The act identified Critical Area as land within 1,000 feet of the mean high water line of tidal waters or the landward edge of tidal wetlands and all waters of and lands under the Chesapeake Bay and its tributaries.

Several unique wetland systems occur in the North Tract and are largely associated with the bottomland hardwoods. A cranberry bog, several oxbow marshes along the Little Patuxent River, and mature forested wetlands along the Patuxent River can be found. One wetland near the cranberry bog contains a nematode species known to control mosquitoes. Also, wet sphagnum bogs are scattered throughout the uplands.

Open fields and meadows occur sporadically within the North Tract. Most of these non-forested areas are associated with former gunnery ranges on the eastern half of the tract and within the former Walter Reed Medical Farm in the far western corner (the information in Section 1.2 was provided by Holliday Obrecht, PRR Biologist).

### 1.3 Current Conditions

Since the closing of the range in 1999, the areas around the trap houses and within the fall zone have not been cut and are in the early succession stages between old-field and scrub-shrub habitat. The areas to the north and south of the trap range are forested, while the area along the eastern edge is comprised of a transition area of pine and bramble leading to a forested area. While the range was in use, the large deciduous trees blocked most of the Pb shot from entering the wooded area.

The site slopes slightly towards the east and a shallow ephemeral drainage channel is located along the northeastern edge of the range. This channel collects surface water runoff from the site and carries it to the northeast. This channel, although apparently dry for most of the year, does contain Pb shot and appears to allow movement of shot off the site (Figure 1). The presence of this channel and the associated low-lying area prompted the wetland delineation.

## 2.0 TECHNICAL APPROACH

### 2.1 Selection of Contaminants of Potential Concern

Lead has been identified as a contaminant of potential concern (COPC) at the site based on the historical use of Pb shot at this range. Other metals such as As, Sb, and Cu may also be present because they are impurities in Pb shot. Clay pigeons contain high levels of polycyclic aromatic hydrocarbons (PAHs) because coal tar pitch is used as a binding agent. Therefore, PAHs are also COPCs. A site walk through indicated clay pigeon fragments throughout the range.

### 2.2 Exposure Characterization

The objective of the exposure characterization was to determine the media and the pathways through which assessment endpoints may be affected by site contaminants. The exposure pathways were dependent on the extent and magnitude of contamination, the site habitat, the receptor species present at the site, and the environmental fate and transport of the COPCs.

On-site receptors are potentially exposed to contaminants in soil and sediment through direct contact, intentional ingestion (e.g., consumption of grit-sized particles), and incidental ingestion (e.g., soil particles adhered to or entrained in food items). Transfer of the contaminants to receptors could also occur through the process of bioaccumulation and bioconcentration, whereby upper trophic level receptors are exposed to site contaminants through the ingestion of

contaminated prey items.

### 2.3 Problem Formulation

The ecological risk assessment was designed to evaluate the potential threats to receptors from the direct exposure to Pb contaminated soil as well as from the direct ingestion of Pb shot. The problem formulation process for this risk assessment includes the identification of the COPC, the identification of the exposure pathways for the COPC, a determination of the assessment endpoints for the site, the formulation of testable hypotheses, the development of a conceptual model, the determination of the measurement endpoints for the site, and an analysis of the uncertainties that may be associated with the risk assessment. The problem formulation presented below was developed according to the guidance established in the Ecological Risk Assessment Guidance for Superfund (U.S. EPA 1997).

### 2.4 Assessment Endpoints

Assessment endpoints are explicit expressions of the actual environmental values (e.g., ecological resources) that are to be protected. Valuable ecological resources include those without which ecosystem function would be significantly impaired or those providing critical resources (e.g., habitat). Appropriate selection and definition of assessment endpoints are critical to the utility of a baseline ecological risk assessment (BERA) as they focus risk assessment design and analysis. It is not practical or possible to directly evaluate potential risks to all of the individual components of the ecosystem at the site, so assessment endpoints are used to focus the risk assessment on particular components of the ecosystem that could be adversely affected by the contaminants released from the site. In general, the assessment endpoints selected for the site are aimed at the viability of terrestrial and aquatic populations and organism survivability.

A review of the habitat at Range 17 provided information for the selection of assessment endpoints. A variety of invertebrates, vertebrates, and plants inhabit the site. In addition, many birds and mammals inhabiting this and adjacent areas could prey on the flora and fauna in the study area. Therefore, the assessment endpoints focused on these biological groups.

### 2.5 Measurement Endpoints

Measurement endpoints are measurable ecological characteristics that are related to the assessment endpoints by the mechanisms of toxicity and routes of exposure. Measurement endpoints are used to derive a quantitative estimate of potential effects, and to form a basis for extrapolation to the assessment endpoints.

Measurement endpoints were selected on the basis of potential presence of receptors at the site, and the potential for exposure to COPCs. The availability of the appropriate toxicity information on which risk calculations could be based was also an important consideration. Endpoints selected were determined to be representative of exposure pathways and assessment endpoints identified for the site.

Lower trophic levels were evaluated using site-specific toxicity tests. For example, although the assessment of terrestrial community structure and function was not directly evaluated, the potential impacts to terrestrial invertebrate populations may be assessed via toxicity tests with a surrogate species.

Food chain exposure models and comparison to literature-based toxicity data were used to evaluate risk to avian and mammalian species that use the site for feeding. Appropriate forage species were identified and the dietary exposure of receptors to contaminants was quantified. The results were compared to existing toxicity data for these or other closely related species.

Receptor species were selected from different trophic levels. Organisms that are likely to be exposed to contaminants because of specific behaviors, patterns of habitat use, or feeding habits were selected for evaluation in this BERA. The availability of appropriate toxicity information on which risk calculations could be based was also an important consideration.

## 2.6 Conceptual Model

The conceptual model is based on contaminant and habitat characteristics and was used to identify critical exposure pathways to the selected assessment endpoints. Mammals and birds may be exposed to site contaminants via ingestion of contaminated food, incidental ingestion of soil, and for birds, ingestion of Pb shot.

Although plants make up an important component of this ecosystem, the bioavailability of Pb in soil to plants is limited (although high concentrations of lead in soil may be acutely toxic to plants). Lead is absorbed mainly by root hairs and is stored in the cell walls; translocation of Pb to aboveground tissues does not readily occur (Fleming 1994). Therefore, this assessment endpoint was not evaluated in this BERA.

Lead in the soil may also impact the reptile and amphibian community. However, little information exists with which to conduct an assessment on these species. Therefore, this assessment endpoint was not evaluated in this BERA.

Secondary Pb poisoning occurs when a predator or scavenger consumes animals that have shot embedded in their bodies or consumes the gizzard of a bird that has ingested Pb shot. Secondary Pb poisoning has been documented in several carnivorous bird species (USFWS 1986, Pain and Amiard-Triquet 1993, Pain et al. 1993, and Pain et al. 1994). However, due to the difficulty in linking the secondary exposure to Pb shot to this site, this endpoint was not evaluated in this BERA. An objective of this risk assessment is to determine the probability of a gallinaceous bird ingesting Pb shot. Based on this probability, a clean up goal for Pb shot will be developed to reduce the risk to gallinaceous birds. Therefore, the removal of the primary exposure pathway (direct ingestion of Pb shot) should also protect carnivorous birds from the secondary ingestion of Pb shot from this site.

Based on this conceptual model, and dependent upon the availability of information, the following receptors were evaluated in this risk assessment:

- I. Terrestrial Invertebrates
  - Direct contact with soil
  - Ingestion of soil
- II. Insectivorous Birds
  - Ingestion of soil
  - Ingestion of invertebrates
- III. Insectivorous Mammals
  - Ingestion of soil
  - Ingestion of invertebrates
- IV. Gallinaceous Bird
  - Direct ingestion of Pb shot

## 2.7 Assessment Endpoint No. 1 – Survival and Growth of Terrestrial Invertebrates

Terrestrial invertebrate communities constitute a large portion of the base of the food chain for the entire ecosystem. Impacts to invertebrate communities would have significant direct and indirect

effects (e.g., loss or reduction of forage or transfer of bioaccumulative compounds) on higher trophic-level organisms (e.g., birds and mammals). Terrestrial invertebrates process organic material in the soil and are therefore important in nutrient and energy transfer.

The hypothesis for this assessment endpoint is as follows: The toxicity of COPCs in on-site soil is not significantly greater than at the reference location.

Direct contact and ingestion of contaminated soil are the primary routes of exposure for terrestrial invertebrate communities. An earthworm toxicity test was selected as the measurement endpoint for this assessment endpoint. Risk was evaluated by exposing earthworms (*Eisenia foetida*) to soil collected on the range and from a reference location for 28 days.

#### 2.8 Assessment Endpoint No. 2 – Reproductive Success of Insectivorous Birds

Impacts to insectivorous birds would allow species of potentially harmful insects to obtain higher population levels than would typically occur in a system that was not impacted. Insectivores are important in nutrient processing and energy transfer between the aquatic and terrestrial environment.

The hypothesis for this assessment endpoint is as follows: The concentration of COPCs in the food items of the modeled receptor at on-site locations do not result in HQ values greater than 1.0.

The ingestion of contaminated food and the incidental ingestion of soil are the primary routes of exposure for insectivorous birds. A food chain accumulation model using the American robin, *Turdus migratorius*, was selected as the measurement endpoint for this assessment endpoint. Risk was evaluated by comparing the dose calculated from the food chain models to literature values.

#### 2.9 Assessment Endpoint No. 3 – Reproductive Success of Insectivorous Mammals

Impacts to insectivorous mammals would also allow species of potentially harmful insects to obtain higher population levels than would typically occur in a system that was not impacted. Insectivores are important in nutrient processing and energy transfer in the terrestrial environment, and play an important role in the terrestrial food chain.

The hypothesis for this assessment endpoint is as follows: The concentration of COPCs in the food items of the modeled receptor at on-site locations do not result in HQ values greater than 1.0.

The ingestion of contaminated food and the incidental ingestion of soil are the primary routes of exposure for insectivorous mammals. A food chain accumulation model using the short-tailed shrew, *Blarina brevicauda*, was selected as the measurement endpoint for this assessment endpoint. Risk was evaluated by comparing the dose calculated from the food chain models to literature values.

#### 2.10 Assessment Endpoint No. 4 – Survival of Gallinaceous Birds

Gallinaceous birds were selected for evaluation because of their method of foraging (grazing for seeds) and for their selection of grit. The enzymes within a bird's digestive system are able to dissolve the soft inner portion of a seed. However, tough seed coats can block enzymes from reaching the inner portion of the seed. Therefore, birds ingest small rocks and stones that aid in grinding and wearing away the seed coat. Grit particles are usually defined as small stones or other hard inorganic particles that a bird selectively ingests to aid in digesting seeds.

The size and shape of grit particles that a bird will select are species specific. For example, Best and Giofriddo (1991) found that 80% of the grit particles found in the crop of a mourning dove ranged from 1.0 to 2.6 mm in diameter, with a total range from 0.2 to 4.4 mm in diameter. The

size of Pb shot used at trap and skeet ranges is usually No. 7 ½ (2.41 mm in diameter) or No. 8 (2.29 mm diameter).

Because these Pb shot sizes are within the range of grit particles selected by mourning doves, this species is susceptible to the ingestion of Pb shot. Once Pb shot are ingested, they are stored in the crop where they are used to process food. During this processing of food and combined with the acidic content of the crop, the Pb shot are eroded, which releases Pb. This allows the Pb to be quickly absorbed by the blood, which induces Pb poisoning.

The hypothesis for this assessment endpoint is as follows: The probability of the modeled receptor selecting Pb shot does not exceed 0.10.

The ingestion of Pb shot is the primary route of exposure for gallinaceous birds. An ingestion-based probability model (Peddicord and LaKind 2000) using the mourning dove, *Zenaida macroura*, was selected as the measurement endpoint for this assessment endpoint. This model uses the ratio of grit (natural, non-Pb inorganic particles) to Pb shot to evaluate risk to these species.

### 3.0 METHODS

A Global Positioning System (GPS) was used to establish a grid on the shooting range. Parallel sampling transects were established perpendicular to the alignment of the shooting ranges. Each transect was located approximately 50 meters (m) from the adjacent transect and extended from the trap houses to a maximum distance of approximately 300 m down range. Sampling locations were established every 50 m along each transect. A standard design for a trap range includes a semicircular safety fan with a radius of 300 yards (approximately 274 m) from the trap house. Therefore, the grid was established to place the nodes within the semicircular fall zone (Figure 1).

On July 28 and 29, 2003, representatives of the U.S. FWS Chesapeake Bay Field Office, the U.S. FWS Technical Liaisons, and representatives of the U.S. Environmental Protection Agency (EPA) Environmental Response Team Center's (ERTC) Response, Engineering, and Analytical Contract (REAC) Operations Group collected samples at this site.

#### 3.1 Extent of Contamination

Surface soil samples were collected following ERTC/REAC Standard Operating Procedure (SOP) #2012, *Soil Sampling*. The samples were collected from 67 locations on-site and from a reference area located on the west side of Wildlife Loop. Because of concerns with UXO at this site, each sample location was screened for UXO avoidance using a magnetometer prior to sample collection. At each node, surface debris such as leaves and twigs were removed and then a disposable plastic trowel was used to collect a small amount of surface soil. The soil sample was placed into a labeled plastic bag and returned to the staging area for analysis by XRF.

##### 3.1.1 X-ray Fluorescence Analysis for Metals

Soil samples were prepared and analyzed in accordance with ERTC/REAC SOP #1713, *Spectrace 9000 Field Portable X-ray Fluorescence Operating Procedure*. First, the soil was homogenized in the bag and then a small aliquot of the sample was placed into a labeled aluminum weight boat. The sample was dried at approximately 100 degrees Centigrade (°C) for 15 to 20 minutes. The sample was removed from the oven, sieved using a 0.5-millimeter (mm) mesh sieve and the soil sample placed into a plastic XRF cup. Lead shot were removed during the sieving of the samples as shot number was evaluated separately. The sample was analyzed by XRF for Sb, As, Cu, and Pb.

### 3.1.2 Confirmation Analysis

In order to determine if the analytical results of the XRF meet quality assurance (QA) criteria, 10 soil samples were selected for confirmatory As, Sb, Cu, and Pb analysis. These samples were selected by the XRF operator in order to provide a range of concentrations (based on Pb). The sample cup was provided to the laboratory for analysis using inductively coupled argon plasma (ICAP) method. Following the analysis of the samples, the ICAP results were compared to the XRF results. If the correlation between the XRF results and the ICAP results was greater than 0.7, the data met QA 2 level data objective. In addition, the XRF operator analyzed field duplicates and daily standards (U.S. EPA 1991).

## 3.2 Risk Assessment

Following a review of the XRF analysis, 6 sample locations were chosen that exhibited a range of Pb concentrations. These locations selected were as follows: Reference, 0-150D, 100R-50D, 100R-100D, 150R-50D, and 150R-100D. The purpose of selecting a range of samples was so that a dose response relationship between survival and/or growth and contaminants could be developed. This relationship could then be used to develop a soil remedial goal (RG). The sampling crew returned to each of these 6 locations and collected sufficient soil to fill a 2-gallon plastic bucket. The sample was homogenized in the bucket and then aliquots removed for TAL metals analysis, BNA analysis, TOC, and grain size. The remainder of the sample was used for earthworm toxicity testing.

### 3.2.1 TAL Metals Analysis

The TAL metals (except mercury) analyses were conducted following ERTC/REAC SOP # 1818, *Determination of Metals by Graphite Furnace Atomic Absorption (GFAA) Methods* or ERTC/SOP #1811, *Determination of Metals by Inductively Coupled Plasma (ICP) Methods*. The samples were analyzed for mercury following ERTC/REAC SOP #1832, *Determination of Mercury by Cold-Vapor Atomic Absorption (CVAA) Methods*. Soil samples were analyzed for TAL metals because of the potential contamination from other sources (e.g., UXO and empty shell casings). The maximum metal concentrations were compared to ecological screening criteria for soil (Friday 1998). Metals that exceeded the screening criteria were evaluated further in the risk assessment.

### 3.2.2 Organic Compound Analysis

The BNA analyses were conducted following ERTC/REAC SOP # 1805, *Routine Analysis of Semivolatile Compounds in Soil/Sediment Samples by GC/MS*. Soil samples were analyzed for these compounds because of the potential contamination from the coal tar pitch used as a binder in the clay targets. In addition, 2 of the soil samples (150R-100D and 150R-50D) were analyzed for pesticides and PCBs following ERTC/REAC SOP # 1801, *Routine Analysis of PCBs in Water and Soil/Sediment Samples by GC/ECD* and SOP# 1809, *Routine Analysis of Pesticides in Soil/Sediment Samples by GC/ECD*. Soil samples were selected for these analyses because they corresponded to the samples in which there was a response in the earthworm toxicity test.

### 3.2.3 Grain Size and Total Organic Carbon

The grain size analysis was conducted following the American Society of Testing and Materials (ASTM) Method D-420 and the TOC analysis (defined as loss on ignition) was conducted following the American Association of State Highway and Transportation Officials (AASHTO) Method T-267-86. The results of these analyses were used to evaluate the results of the earthworm toxicity test.

### 3.2.4 Earthworm Toxicity Test

Acute soil toxicity evaluations using *Eisenia foetida* were performed according to ASTM Guide E1676-97, “Standard Guide for Conducting Laboratory Soil Toxicity or Bioaccumulation Tests with the Lumbricid Earthworm *Eisenia foetida*” (ASTM 1997). This testing provided data concerning the availability and toxicity of contaminants present in the soil (USEPA 1989). The earthworm *E. foetida* is widely distributed in soil, is an important component of the terrestrial invertebrate community, and often comprises a significant proportion of the soil biomass. In addition to being in intimate physical contact with the substrate, *E. foetida* feeds on detritus and vegetative debris incorporated into the soil. Therefore, the predominant route of exposure to Pb is from the ingestion of soil. These toxicity tests were used to evaluate the risk to the terrestrial invertebrate communities as well as to provide a site-specific bioaccumulation factor for Pb to invertebrates.

The earthworms were exposed to site soils for 28 days. Survival was also recorded at 14 days. Following a 28-day exposure, the surviving worms from each replicate sample were allowed to depurate for 24 hours, and then the tissue samples were frozen for later analysis. In addition, a dose response relationship was used to establish a NOAEL and LOAEL for Pb in soil.

### 3.2.5 Food-Chain Ingestion Models

To determine the risk associated with the exposure of higher trophic level receptors to site-related contaminants, ingestion-based exposure models were used. Life history information was obtained for each receptor (Appendix A). The hazard quotients (HQs) for higher trophic level species were calculated using food chain models with site-specific risk assumptions. A variety of soil concentrations were used together with literature-based NOAELs and LOAELs (Appendix A). A literature search was conducted to determine levels of exposure to contaminants at which no adverse effects would be expected. If a NOAEL was not available for Pb or receptor species, then a converted LOAEL or Lethal Dose that kills 50% of the test animals (LD<sub>50</sub>) was used. A factor of 10 was used to convert an LD<sub>50</sub> to a LOAEL, and to convert a LOAEL to a NOAEL. All NOAELs and LOAELs were based on the most sensitive endpoint of survival, growth, or reproduction.

Exposure to upper trophic level receptors is expressed using the following food chain model:

$$D_t = \frac{(IR_f + IR_s) \times AUF}{BW}$$

Where:

$D_t$  = total dietary dose (mg/kg/day)  
 $IR_f$  = food ingestion rate (kg/day) x contaminant concentration in prey (mg/kg)  
 $IR_s$  = soil ingestion rate (kg/day) x contaminant concentration in soil (mg/kg)  
 $BW$  = body weight (kg)  
 $AUF$  = area use factor

#### 3.2.5.1 Insectivorous Birds

The American robin was selected as the representative insectivorous bird. Life history parameters were used which provide a reasonable exposure to Pb contaminated food items and soil. The specific life history parameters used in the food chain models are fully described in Appendix A. The concentration of Pb measured at each of the 6



sampling locations (Section 3.2) was used as exposure point concentrations. The dose calculated from these exposure scenarios were compared to both NOAEL and LOAEL toxicity reference value (TRV).

A description of all the literature that was reviewed for NOAEL and LOAEL values is included in Appendix A. Below is a brief description of the study and the TRV derivation that was used for this BERA. Edens et al. (1976) exposed Japanese quail to four dietary concentrations of lead acetate (1, 10, 100 and 1,000 mg/kg) for a period of 12 weeks. Percent hatch of settable eggs was significantly decreased in hens exposed to 100 mg/kg lead. A dietary lead concentration of 1,000 mg/kg almost completely suppressed the quail's egg production. The results from this experiment were used to develop the NOAEL and LOAEL values because of the ecological significance of the endpoints and the method and duration of exposure. An ingestion rate of 18 g/day and adult body weight of 0.12 kg ([feathersite.com/Poultry/Stuff/FeatherFancier/FeathFancQuail.html](http://feathersite.com/Poultry/Stuff/FeatherFancier/FeathFancQuail.html)) were used to convert the exposure concentration to units of mg/kgBW/day. This resulted in a LOAEL of 15 mg/kg BW/day (100 mg/kg) and a NOAEL of 1.5 mg/kg BW/day.

In addition, the soil Pb concentration in the food chain accumulation model was adjusted to calculate a HQ of 1.0. This allowed for the development of a RG based on the exposure of insectivorous birds to contaminated soil.

#### 3.2.5.2 Insectivorous Mammals

The short-tailed shrew was selected as the representative insectivorous mammal. Life history parameters were selected which provide a reasonable exposure to Pb contaminated food items and soil. The specific parameters used in the food chain models are fully described in Appendix A. The concentration of Pb measured at each of the 6 sampling locations (Section 3.2) was used as exposure point concentrations. The dose calculated from these exposure scenarios were compared to both NOAEL and LOAEL TRV.

A description of all the literature that was reviewed for NOAEL and LOAEL values is included in Appendix A. Below is a brief description of the study and the TRV derivation that was used for this BERA. Azar et al. (1973) administered Pb to rats at six dietary levels (1, 10, 50, 100, 1,000 and 2,000 mg/kg) for three generations and measured changes in reproduction and growth. No effects on number of pregnancies, the number of pups born alive, the fertility index, the viability index, or the lactation index were observed at any exposure levels. An exposure concentration of 1,000 mg/kg resulted in reduced offspring weight and kidney damage in the young. An ingestion rate of 0.027 kg/day and adult body weight of 0.35 kg (U.S. EPA 1988) were used to convert the exposure concentration to units of mg/kgBW/day. This resulted in a LOAEL of 77 mg/kg BW/day, and a NOAEL of 7.7 mg/kg BW/day.

In addition, the soil Pb concentration in the food chain accumulation model was adjusted to calculate a HQ of 1.0. This allowed for the development of a RG based on the exposure of insectivorous mammals to contaminated soil.

#### 3.2.6 Lead Shot and Grit Counts

Ten surface soil samples (9 from on the range and 1 from the reference location) were collected to determine the number of lead shot. The sampling locations were selected from within the fall zone as well as from where Pb shot were noted during the collection of samples for XRF analysis. These locations do not overlap with those selected for toxicity testing, as those locations were selected based on a range of Pb concentrations. An area near the grid node was cleared of surface debris and a metal template, measuring

12 inches by 12 inches by 1 inch was placed on the ground surface. The soil within the outline of the template was removed using a trowel or shovel and placed into a labeled plastic bag. Standardized SOPs were not available for counting Pb shot or grit particles therefore the following methods were used to count these particles.

Because these samples were used to determine the number of Pb shot as well as the number of grit-sized particles, sieve sizes were selected that would satisfy both of the requirements. For this BERA, grit was defined as the size particle that would pass through a 2.8-mm mesh sieve but be retained on a 0.5-mm mesh sieve. This size range was selected because it spans the grit sizes that would be selected by a mourning dove (Best and Giofriddo 1991). The fraction of grit-sized particles that are Pb shot are used in a probability model as described in the next section. The ingestion of lead shot by gallinaceous birds was selected as a measurement endpoint for this BERA.

A previous study conducted at this site indicated that lead shot were effectively captured on a 0.5-mm mesh sieve. The size of lead shot that were fired at this site consisted of mostly No. 7 ½ (2.41 mm in diameter) and No. 8 (2.29 mm in diameter) shot. Therefore, it was assumed that this size shot would pass through a 2.8-mm mesh sieve, but that whole shot and smaller broken pieces of lead would be retained on a 0.5-mm mesh sieve.

The following steps were used to separate and count grit particles and Pb shot. The process was iterative because it took several attempts to remove the large amount of organic material in these samples to facilitate counting the grit particles and the shot. First, the soil sample was wet sieved through a 2.8-mm mesh sieve and the material that passed through the sieve was retained on a 0.5-mm mesh sieve. The material that was retained on the 0.5-mm mesh sieve was placed in an oven at 100 °C to dry. The sample was then again sieved through a 0.5-mm mesh sieve to further remove the fine organic material. The contents remaining on the sieve were placed in a white plastic pan and the Pb shot removed. Once the shot were removed, the remaining sample was placed in a porcelain crucible and the crucible placed into a muffle furnace at 450 °C overnight to burn off the remaining organic material. The resulting sample contained uniform, non-organic grit particles as well as small fragments of lead (which turned a light gray, which facilitated the removal and enumeration of them).

A sub-sample of the grit particles was counted into an aluminum weigh boat until the boat held 1.0 g. After determining the number of particles in 1.0 g, this sub-sample was placed back into the original sample, and then the entire sample weighed. Next, the number of grit particles in a 1.0 g sample was multiplied by the total weight of the sample in order to determine the total number of grit particles. In order to reduce operator bias, each sample was counted separately by two individuals and the mean number of particles used in a grit ingestion model.

### 3.2.7 Probability Model for Gallinaceous Birds

Exposure to Pb shot for the mourning dove was calculated by using the model developed by Peddicord and LaKind (2000) for evaluating the probability that a bird will ingest Pb shot in its lifetime. There are 2 parameters that need to be calculated for use in the model. The first parameter is P, which is defined as the probability that a single selected particle will be Pb shot. This parameter is based on an area use factor (S), the fraction of grit-sized particles on site that are Pb shot ( $P_s$ ) and the fraction of grit sized particles that are selected off of the site that are Pb shot ( $P_o$ ). The formula for calculating P is as follows:

$$P = S * P_s + (1 - S)P_o$$

Where,

- P = Probability that a single selected particle will be a Pb shot.
- P<sub>s</sub> = Fraction of grit sized particles onsite that are Pb shot.
- P<sub>o</sub> = Fraction of grit sized particles off site that are Pb shot (we assumed 100% of Pb shot was coming from this site. Therefore, P<sub>o</sub> is 0 for this model).
- S = Fraction of foraging time (AUF) on site.

The next parameter that needs to be calculated for this model is N, which is defined as the number of particles that a bird selects and retains in its gizzard. This factor is based on the bird's lifespan (Y), the number of foraging days per year (D<sub>e</sub>) and the grit retention time (D<sub>p</sub>). The formula for calculating N is as follows:

$$N = Y(D_e/D_p)$$

Where,

- N = Number of particles selected and retained in the gizzard in its lifetime.
- Y = Number of years a bird lives. The value was derived from literature and for the dove it was assumed 1.5 years (McConnell 1967).
- D<sub>e</sub> = Number of days per year that a bird forages in the area (we assumed this species is migratory and would be in the area from March 15 to November 15, thus 245 days).
- D<sub>p</sub> = Retention time for a shot in gizzard (days). Literature base values were chosen for D<sub>p</sub>. For the dove we assumed a retention time of 6 days (McConnell, 1967).

Lastly, to determine the risk to gallinaceous birds, both parameters P and N are used to calculate the probability of selecting Pb shot as grit (P<sub>t</sub>). The formula for calculating P<sub>t</sub> is as follows:

$$P_t = 1 - (1 - P)^N$$

Where,

- P<sub>t</sub> = Probability that a bird will ingest at least one Pb shot in its lifetime.

Appropriate life history information (e.g., life span, foraging days, and shot retention time) was determined from the literature (Table 6). Although mourning doves have a relatively large home range, the following rationale was used to select a value of 1.0 for an AUF (defined as S in the above model). Vyas et al. (2000) measured free-erythrocyte protoporphyrin levels in blood from passerine birds mist netted at this range. Juncos (*Junco hyemalis*) had significantly higher protoporphyrin levels than birds collected from an uncontaminated site. Free erythrocyte protoporphyrin is used as an indicator of Pb poisoning in birds (Beyer et al. 1988) and the levels are positively correlated with blood Pb levels (Pain 1989). Juncos are migratory birds and their primary food source is seeds. Mourning doves are also migratory and primarily eat seeds. Therefore, because of the evidence that indigenous birds are exposed to Pb at this site, an AUF of 1.0 was used in the shot ingestion model.

In order to determine a RG for Pb shot, it was necessary to define an acceptable level of probability (analogous to a TRV in a food chain model) that a bird will select a grit particle that is actually Pb shot.

Suter et al. (2000) indicate that an acceptable exposure level is 20 % based on the level of effect that is considered biologically significant for an ecological community or population. The authors indicate that adverse effects to a community or population that occur at a frequency less than 20 % are indistinguishable from the natural variability inherent in natural biological systems and are considered biologically insignificant.

However, as the mission of this refuge is to conserve and protect the nation's wildlife and habitat through research and wildlife management techniques, a more conservative endpoint was selected for this risk assessment. For this BERA, a probability of 10% was selected to determine risk to gallinaceous birds.

Remedial goals were determined by calculating a number of Pb shot per unit area that would result in an acceptable probability (less than or equal to 10%) that Pb shot would be ingested. In order to determine an acceptable number of Pb shot per unit area of surface soil, calculations were made by manipulating the number of Pb shot found at each of the nine locations used in the probability models, while leaving the number of grit-sized particles unchanged. The number of Pb shot at each location was adjusted until the calculated ingestion probability was dropped to 10%.

### 3.3 Wetland Evaluation

On July 29, 2003, a site reconnaissance was conducted on the skeet range to determine if jurisdictional wetlands were present on the site, and if so, the extent of the wetlands or the waters of the United States. This evaluation was conducted to determine if the area along the drainage swale was a wetland and to determine if there was Pb contamination within this area. Several shallow soil borings (12 to 14 inches deep) were conducted throughout the area. The soil samples were compared to Munsell Soil Charts in order to determine the presence of hydric soils. For the purposes of this wetland evaluation, the three criteria (the presence of wetland vegetation, hydric soils, and flooding) noted in the U.S. Army Corps of Engineers Wetland Delineation Manual (U.S. ACOE 1987) had to be present.

### 3.4 Sampling Equipment Decontamination

All soil samples were collected with disposable trowels, therefore, no equipment decontamination was required.

### 3.5 Sample Documentation and Shipment

Sample documentation was completed following ERTC/REAC SOP #2002, *Sample Documentation* and ERTC/REAC SOP #4005, *Chain of Custody Procedures*. In addition, the sample packaging and shipment were completed following ERTC/REAC SOP #2004, *Sample Packaging and Shipment*.

## 4.0 RESULTS

Soil samples were collected from 67 locations on the range and from the reference location. The analytical results for the metals and TOC are reported in mg/kg and the analytical results for organic compounds are reported in micrograms per kilogram ( $\mu\text{g}/\text{kg}$ ). Results of the grain size analysis are reported as % composition. The analytical results are reported on a dry weight basis.

### 4.1 Extent of Contamination Results

Soil samples were collected from a total of 67 locations on the range and from the reference location and analyzed for Sb, As, Cu, and Pb (Table 1). In addition, a duplicate analysis was conducted on average of one in every 10 samples (7 duplicates). In order to calculate a

statistically derived MDL, a low concentration standard was analyzed in the morning as well as periodically throughout the day.

Lead ranged from below the MDL (39 mg/kg) at the far ends of the range (300R and 300L transects) to 22,000 mg/kg (location 0-150D). Generally, the concentration of Pb in soil at the locations 150m from the trap houses was the highest (e.g., 50L-150D contained 18,000 mg/kg). Based on the concentration of Pb, there was generally a semicircular pattern of Pb contamination that corresponds with the distance that Pb shot will travel when fired from a shotgun (Figure 2). Arsenic was detected above the MDL (33 mg/kg) at 6 out of 67 locations. Lead can interfere with As analysis at Pb:As ratios of 5:1 or greater. Therefore, the As detection limit was calculated as a statistical value (3 times the standard deviation of the analyses of the low concentration instrument standard) or 1/10 the Pb concentration, whichever is greater. This interference may explain the low number of samples in which As was detected. Lastly, Cu was only detected above the MDL (81 mg/kg) in 7 samples and Sb was detected above the MDL (100 mg/kg) in only 1 sample.

In order to confirm the results of the XRF analyses, 10 of the samples were submitted for confirmation analysis using an ICAP method (Table 2). The samples that were submitted were the prepared XRF cup and they were selected across a concentration range. The results from the ICAP method were compared to the XRF results and a correlation analysis conducted on the results. The correlation coefficient was 0.99 (pg. 3, Appendix B – Final X-Ray Fluorescence Report), which was greater than the U.S. EPA (1991) recommended guideline of 0.7 therefore the XRF results met QA2 level criteria (Appendix B).

To present the results graphically, a figure was prepared which shows several contaminant concentration contours (Figure 2). These contours were developed using a linear Kriging of the Pb results from the XRF analysis. This is a mathematical process by which the Pb results are interpolated in order to plot the probability of Pb contamination at all the locations. This particular krigging program is a standard statistical algorithm used as a component of the Environmental Systems Research Institute (ESRI) ArcInfo software package.

## 4.2 Risk Assessment Results

Below are the results of the 6 soil samples analyzed TAL metals, BNAs, TOC and grain size. These results, earthworm toxicity test results and food chain and shot ingestion models, are used to describe the risk to biota.

### 4.2.1 TAL Metals Results

Soil samples were analyzed for TAL metals (Table 3). Of note is the high concentration of lead (44,000 mg/kg) detected at Location 0-150D. This concentration was 2 times the concentration of lead found during the XRF analyses, although the highest concentration of all COPCs was found at the same location.

In order to determine if the concentrations of metals detected at this site pose a risk to biota, the maximum concentration detected on the site was compared to an ecological screening benchmark (Table 4). If the benchmark was greater than the maximum concentration, the metal was not evaluated further in the risk assessment. If the benchmark was less than the maximum concentration detected on the site, the metal was retained as a COPC.

Beryllium (Be), cadmium (Cd), sodium (Na), and thallium (Th) were not detected in any of the samples. Ecological benchmarks were available for Be, Cd, and Th, and the benchmarks were higher than the MDLs. Therefore, these three metals were not evaluated further in the risk assessment. Sodium was not detected and no benchmark was

available, therefore Na was not evaluated further in the risk assessment.

Barium (Ba), cobalt (Co), mercury (Hg), nickel (Ni), silver (Ag), and zinc (Zn) were detected in the soil samples. The maximum concentration detected on the site was compared to an ecological benchmark. The benchmarks were greater than the maximum concentration of the metals therefore these metals were not evaluated further in the risk assessment.

Based on the screening level risk assessment, aluminum (Al), Sb, As, chromium (Cr), iron (Fe), Pb, manganese (Mn), selenium (Se), and vanadium (V) will be retained as COPCs. Benchmarks were not available for calcium (Ca), magnesium (Mg), or potassium (K), therefore, these metals will also be retained in the risk assessment. Although the benchmark for copper (Cu) was greater than the concentration detected in these samples, Cu has already been identified as a COPC because it is an impurity in Pb shot. Therefore, Cu was retained as a COPC (as were As and Sb).

#### 4.2.2 Organic Compound Results

No BNAs were detected in any sample above the MDL. However, a common laboratory contaminant, bis-2(ethylhexyl) phthalate, was recorded at an estimated concentration below the MDL at locations 0-150D, 150R-150D, and 150R-100D of 430, 130, and 130 ug/kg, respectively (Appendix C).

Because of the toxicity noted in the earthworm tests, 2 soil samples were analyzed for pesticides and PCBs. The samples that were selected were those in which there was statistically significant mortality in the earthworms (compared to the reference) at location 150R-100D and 150R-50D. No pesticides or PCBs were detected above the MDLs. However, p,p'-DDE was recorded at an estimated concentration below the MDL at location 150R-100D of 0.9 µg/kg (Appendix C).

#### 4.2.3 Grain Size and Total Organic Carbon

The TOC ranged from 1.8 to 9.1% in samples collected on the site and was 5.2% in the sample collected from the reference location. The grain size analysis for the samples collected on the site indicated that most of the soil was comprised of sand. The samples collected on site contained from 34 to 87% sand. The remaining fractions were comprised of mostly silt and clay (Appendix C).

#### 4.2.4 Earthworm Toxicity Test Results

Soil samples were tested using a 28-day earthworm toxicity test (Table 5). Each soil sample was testing using four replicate exposure chambers with 20 worms added to each chamber. Worms were counted to determine survival at 14 days (only to determine if the test should be continued for the entire 28 days) and at 28 days. At the completion of the test, the surviving worms were weighed to determine the growth compared to the reference and control soils (Appendix D).

In addition, for those chambers with surviving worms at the completion of the test, the worms were allowed to depurate for 24 hours. Then the worms were dried and submitted for total Pb analysis. The samples that were analyzed were the Reference, Location 150R-50D, and Location 150R-100D. In addition, the concentration of Pb was measured in the control worms.

The worms from locations 0-150D (44,000 mg/kg Pb), 100R-50D (3,000 mg/kg Pb) and 100R-100D (540 mg/kg Pb) exhibited 100% mortality at 14 days (Table 5). At 28 days,

the worms from locations 150R-50D (270 mg/kg Pb) exhibited 22.5 % mortality, 150R-100D (250 mg/kg Pb) had 18.5% mortality and, and the Reference location (46 mg/kg Pb) had 2.5% mortality. When compared to the Reference location, there was statistically significant mortality in all the soil samples collected on the range. Therefore, based on the results of this test, the NOAEL is 46 mg/kg Pb and the LOAEL is 260 mg/kg Pb.

Because there was significant earthworm mortality, the accumulation of Pb in tissue cannot be used to develop a bioaccumulation factor. However, in order to determine if Pb was accumulating in the tissue at levels sufficient to cause mortality, a decision was made to analyze the worms for Pb. In order to make analytical comparisons, each of the 4 replicate worm samples from the following test matrix were analyzed: Pre-test worms; the culture control; the ASTM control soil, the reference location; station 150R-100D; and 150R-50D. Lead was not detected in the pretest worms, the culture control, and the ASTM control. Worms exposed to soil containing 260 mg/kg Pb (Location 150R-100D) accumulated Pb in each replicate at 1,200, 1,300, 1,400, and 1,600 mg/kg. The worms exposed to soil containing 270 mg/kg (Location 150R-50D) accumulated Pb in each replicate at 1,300, 1,400, 1,300, and 1,300 mg/kg. The worms exposed to the soil samples collected from the reference location (which contained 46 mg/kg Pb) accumulated Pb in each replicate at 320, 410, 460, and 670 mg/kg (Appendix C).

Because the surviving worms accumulated high concentrations of lead, the test report was further reviewed to determine if there were any factors that could explain the high accumulation. This review indicated that the soils were acidic. For example, the soil from the reference location had a pH of 4.3 standard units and the pH of the soils from the site ranged from 4.5 to 5.4 standard units. In addition, prior to testing, the soils were hydrated with deionized water. Therefore, the combination of a low pH and the addition of deionized water to the soil may have dissolved some of the lead, making it more bioavailable to the worms.

For example, Ma (1982) found that factors such as soil pH or percent organic matter affected metals uptake by earthworms. In addition, Ma et al. (1983) found that soil Pb content, soil pH, and soil organic matter account for almost 70% of the variance in the worm Pb content. This demonstrates that earthworm accumulate more Pb in soils with a low pH and low organic matter. Morgan and Morgan (1988) also found similar results in that soil pH and soil calcium were a major influence on Pb accumulation in worms.

For the remaining TAL metals identified in Section 4.2.1, the analytical results were compared to the survival of the worms to determine if metals posed a risk to biota. For all the metals except Pb and Sb, there was no correlation between % survival and concentration. In addition, the concentration of each metal at the Reference Location exceeded the concentration at a Location that had significant mortality. Based on this information, Pb and Sb are the remaining compounds that pose risk to biota.

The concentration of Pb and Sb were compared to determine if there was a relationship between the concentrations. A correlation analysis was conducted (using ½ the MDL for the non detected values) which indicated that the concentrations were positively correlated ( $r^2 > 0.99$ , Table 3). Although there may be risk from Sb, it may be masked by the high concentration of Pb. Therefore, the risk from Sb will not be further evaluated in this risk assessment.

#### 4.2.5 Food - Chain Ingestion Models

##### 4.2.5.1 Insectivorous Birds

Life history parameters were compiled for the American robin and used in a food chain ingestion model (Table 6). Using average ingestion rates, body weights, and an AUF of 1.0, a dose was calculated to the American robin (Table 7). Based on a NOAEL TRV, the concentration of Pb at all locations posed a risk to the robin (HQ>1).

The soil Pb concentration was manipulated in order to yield an HQ that equaled 1.0. This manipulation resulted in a soil Pb concentration of 35 mg/kg resulted in an HQ of 1.0 using a NOAEL TRV and a soil Pb concentration of 320 mg/kg resulted in an HQ of greater than 1.0 using a LOAEL TRV.

#### 4.2.5.2 Insectivorous Mammals

Life history parameters were compiled for the short-tailed shrew (Table 6). Using average ingestion rates, body weights, and an AUF of 1.0, a dose was calculated to the short-tailed shrew (Table 8). Based on a NOAEL TRV, the concentration of Pb at all locations posed a risk to short-tailed shrew.

The soil Pb concentration was manipulated in order to yield an HQ equal to 1.0. This manipulation indicated a soil Pb concentration greater than 44 mg/kg resulted in an HQ of greater than 1.0 using a NOAEL TRV and a soil Pb concentration greater than 440 mg/kg resulted in an HQ of greater than 1.0 using a LOAEL TRV.

#### 4.2.6 Lead Shot and Grit Counts

Ten soil samples were collected for the determination of Pb shot and grit particles. The number of Pb shot found in the samples ranged from 0 at the reference area to a maximum of 2,946 at location 0-150D (Table 9). The number of Pb shot found in a sample was compared to the total Pb concentration in order to determine if these two factors were correlated. A correlation analysis was conducted and the resulting correlation coefficient was 0.79 (Table 9). This indicates that the concentration of Pb in the soils is related to the number of Pb shot found in a soil sample.

The number of grit-sized particles found in the samples on site ranged from 1,602 at 50R-150D to 7,713 at location 100L-200D (Table 9).

#### 4.2.7 Probability Model for Gallinaceous Birds

Life history parameters were compiled for the mourning dove (Table 6). Then ratio of Pb shot and grit particles were used in a probability model to determine risk to gallinaceous birds (Table 10). For this BERA, a probability of greater than 0.10 indicated risks to birds. The results of the model indicated that the combination of Pb shot and grit particles found on this site posed a risk to birds, except at Location 100L-200D.

### 4.3 Wetland Evaluation

An ephemeral drain was encountered near the center of the site and Pb shot were visible on the soil surface within this drain. Because the flow from this ephemeral drain discharges into an upland-woodland and not an intermittent or perennial stream, the channel is not considered a water of the United States. In addition, no hydric soils were encountered within the potential impact zone of the range. Therefore, no jurisdictional wetlands are located within the boundary of Range 17. However, 2 wetland complexes were encountered in close proximity to the range. If the project proceeds to a clean up phase, these areas should be protected from run-off and heavy equipment.

## 5.0 DISCUSSION



## 5.1 Assumptions

The following conservative assumptions were made to conduct the BERA:

For direct toxicity, total Pb was used as the dose (the benchmarks are expressed in total Pb).

Mean body weight and mean ingestion rates were used when possible to estimate dose in food chain exposure models.

There was an acute response in the earthworm toxicity test and therefore, the calculation of a bioaccumulation factor (BAF) for Pb was not valid. Therefore, a BAF was selected from a risk assessment conducted on another Trap and Skeet Range (Prime Hook National Wildlife Refuge, DE). A BAF of 0.39 was calculated (USFWS 2003) at this site.

Contaminants in food items were assumed to be 100 % bioavailable and not metabolized and/or excreted during the life of the receptor.

Diets for American robin and short-tailed shrew were simplifications of complex diets.

A literature search was conducted to determine the chronic toxicity of the Pb evaluated in the food chain model. Acute toxicity values for Pb were also obtained from the literature. If no toxicity values could be located for the receptor species, values reported for a closely related species were used. Studies were critically reviewed to determine whether study design and methods were appropriate. If values for chronic toxicity were not available, LD<sub>50</sub> (median lethal dose) values were used. For the purposes of this BERA, a factor of 10 was used to convert the reported LD<sub>50</sub> to a LOAEL, and then a factor of 10 was used to convert a reported LOAEL to a NOAEL. If several toxicity values were reported for a receptor species, the most conservative value was used in the risk calculations as long as the study design, exposure route, mechanism, and species tested were deemed appropriate. For the chronic toxicity endpoints, values obtained from long-term feeding studies were used in preference to those obtained from single dose oral studies.

The soil ingestion rates for both the American robin and the short-tailed shrew were based on information for a similar species (as a percentage of the diet). It was assumed that these estimated ingestion rates were representative for the receptor species in question.

For the mourning dove, it was assumed that the ingestion of one Pb shot would be sufficient to cause a response in the bird. The response evaluated was Pb poisoning, which may cause behavioral changes or mortality.

For the TRVs used in the American robin and short-tailed shrew food chain models, the toxicity values were reported as mg/kg contaminant in the diet. These values were converted to daily intake (in milligrams per kilogram body weight per day; [mg/kg BW/day]) by using the following formula:

$$DI = CD \times IR \times 1/BW$$

Where:

DI = Daily Intake (mg/kgBW/day)  
CD = Contaminant Dose (mg/kg diet)  
IR = Ingestion Rate (kg/day)  
BW = Body Weight (kg)

This conversion allowed dietary toxicity levels cited to be converted to a daily dose based on body weight.

## 5.2 Risk Characterization

The BERA was conducted to determine the risk associated with the exposure of biota to site-related contaminants using relevant ecological benchmarks and ingestion-based exposure models. The following steps were completed for this assessment.

- 1) A literature search was conducted to define the life history information for selected indicator species, and assessment and measurement endpoints were chosen;
- 2) Risk exposure scenarios were determined based on site contaminant levels, the extent and magnitude of contamination, and the toxicological effects of the COPCs;
- 3) Indicator species were selected based on species present and or potentially present at the site, the availability of toxicity information from the literature, and the potential for exposure to site contaminants based on habitat use or behavior;
- 4) Exposure pathways were determined for each species;
- 5) Daily dose estimates were calculated using site specific and literature based assumptions such as media concentrations, food, and soil ingestion rates, body weights, and area use factors;
- 6) A risk characterization was conducted by determining the NOAEL and LOAEL using site-specific toxicity tests. For food chain models, an HQ was calculated by dividing several exposure concentrations by a TRV. For the risk from Pb shot ingestion, a probability model was used to determine risk.

For this BERA, it was concluded that there is acceptable ecological risk if the HQ calculated using the assumptions presented and the NOAEL is equal to or less than 1.0. It was concluded that there is an unacceptable ecological risk if the HQ calculated using the assumptions presented and the LOAEL is greater than 1.0. The concentrations of Pb that fall between the NOAEL and the LOAEL have the potential to cause ecological risk.

## 5.3 Discussion of Hazard Evaluation

### 5.3.1 Assessment Endpoint 1: Survival and Growth of Terrestrial Invertebrates

Direct contact with and ingestion of contaminated soil are the primary routes of exposure for terrestrial invertebrates. Risk was evaluated by calculating a NOAEL and a LOAEL from a site-specific toxicity test using the earthworm. These calculations indicate that concentrations of Pb in soils below 46 mg/kg (NOAEL) are unlikely pose a risk to terrestrial invertebrates. This endpoint is based on both survival and growth. However, Pb concentrations in soils above 260 mg/kg (LOAEL) are sufficient to pose a risk to terrestrial invertebrates. It should be noted that this LOAEL is based on a lethal response by the worms (significantly reduced survival when compared to the control animals). This response may be due to the soil Pb concentration as well as the bioavailability of the Pb.

### 5.3.2 Assessment Endpoint 2: Reproductive Success of Insectivorous Birds

The ingestion of contaminated prey and the incidental ingestion of soil are the primary routes of exposure to by insectivorous birds. A food chain accumulation model using the American robin was used to determine a NOAEL and a LOAEL. The results of the HQ calculation in the food chain models indicate that soil Pb concentration below 35 mg/kg are unlikely to pose a risk to insectivorous birds. However, Pb concentrations above 320

mg/kg are sufficient to pose a risk to insectivorous birds.

### 5.3.3 Assessment Endpoint 3: Reproductive Success of Insectivorous Mammals

The ingestion of contaminated prey and the incidental ingestion of soil are the primary routes of exposure to by insectivorous mammals. A food chain accumulation model using the short-tailed shrew was used to determine a NOAEL and a LOAEL. The results of the HQ calculation in the food chain models indicated that soil Pb concentrations below 44 mg/kg are unlikely to pose a risk to insectivorous mammals. However, soil Pb concentrations above 440 mg/kg are sufficient to pose a risk to insectivorous mammals.

### 5.3.4 Assessment Endpoint 4: Survival of Gallinaceous Birds

The mourning dove was selected to represent an upland bird that may directly ingest Pb shot. Risk was evaluated by determining the probability that a mourning dove will ingest Pb shot from this site in its lifetime. The results indicate that the Pb shot in surface soil pose a risk to survival of gallinaceous birds. This risk estimate is based on accepting a 10% probability that a bird will ingest Pb shot.

## 5.4 Sources of Uncertainty

There are factors inherent in the risk assessment process that contribute to uncertainty and must be considered when interpreting results. Major sources of uncertainty include natural variability, error, and insufficient knowledge.

Natural variability is an inherent characteristic of ecological receptors, their stressors, and their combined behavior in the environment. Biotic and abiotic parameters in these systems may vary to such a degree that the exposure to ecological receptors in two identical conceptual models may differ temporally and spatially. Factors that contribute to temporal and spatial variability may be differences in an individual organism's behavior (within the same species), changes in the weather or ambient temperature, unanticipated interference from other stressors, differences between microenvironments, stochasticity, and numerous other factors. Thus, the conservative nature of this BERA assumes that the highly variable environmental conditions and the behavior of organisms and their stressors are interacting in such a manner that allows the contaminants to move freely through the identified exposure pathways, and to produce the same effects identified in the exposure profile.

Uncertainty associated with natural variability also arises from the use of literature toxicity values in which a study has examined a single species/single contaminant system under highly controlled conditions. If conducted in a laboratory, these studies do not take into account the effects of the environmental factors and other stressors that are present in natural systems. These factors may have synergistic, antagonistic, or neutral effects upon the receptor-contaminant interaction. Point estimates of exposure such as a NOAEL, LOAEL, LD50, or mathematical mean (which are presented in the literature) also have an inherent variability that is by default incorporated into the BERA.

In addition, uncertainty associated with natural variability is introduced from the use of literature values for sediment, water, and food ingestion rates, dietary compositions, and body weights. These values reported in the literature are from studies that may have been conducted at a certain time of year or in a certain location that does not necessarily give an accurate representation of the life histories of the species assessed at the site under consideration in the BERA.

Error may be introduced into the BERA through the use of invalid assumptions in the conceptual

model. Conservative assumptions were made in light of the uncertainty associated with the risk assessment process (i.e., natural variability). Conservative assumptions were used to minimize the possibility of concluding that risk is not present when a threat actually does exist (i.e., the elimination of false negatives). While there is uncertainty associated with each conservative assumption used, this consistent selection process assures that the uncertainty associated with this type of error will err on the side of a protective outcome.

Literature values for the toxicity of Pb were not available for all receptor species. An attempt was made to identify studies using closely related species to make risk estimates for the selected receptors. Species respond differently to exposure to toxins; responses to Pb by the indicator species may be different from species for which the toxicity data are reported. Methodological problems were also apparent in several of the studies from which the NOAEL was obtained. Unfortunately, studies more suitable for this BERA were not found for some of the selected receptors.

A literature search was conducted to identify an appropriate NOAEL and LOAEL for this BERA. In many of the studies reviewed, adverse effects were observed at the lowest exposure concentration. This made it impossible to identify an appropriate NOAEL for some receptors. In these cases, a factor of 10 was used to convert the LOAEL to a NOAEL, which adds uncertainty to the NOAEL-based calculations.

Doses in toxicological studies can be reported in units of mg contaminant/kg diet, or in units of mg contaminant/kg body weight/day. All doses reported as mg/kg in diet were converted to units of mg/kg BW/day. If body weights were reported for the test animals in a given study, these values were used for making this conversion. Otherwise, the body weight and ingestion rate for the species reported in other literature sources were used.

Exposure concentrations were calculated (daily intake as described previously) for each target receptor species based on levels of contaminants detected in site media, daily food ingestion rates, incidental soil/sediment ingestion rates, and body weight reported in the literature.

This BERA did not examine the contribution of dermal absorption, transfer across epithelial membranes, or inhalation exposure as part of the exposure pathway. In contrast to the use of conservative assumptions, the error introduced into this BERA by the omission of these routes of exposure may err on the side of a less protective outcome. The relative contribution of this error to alter the outcome of the BERA is unknown at this time.

Data gaps are defined here as the incompleteness of data or information upon which the BERA is based. Specifically, these may be an incomplete contaminant data set, missing pieces of life history information, and the absence of toxicity-based literature for the receptor of concern.

Life history information and literature values for the toxicity of the contaminants of concern are not always available for all of the receptor species. By using closely related species, it is possible to make risk estimates. In reality, however, the information may vary substantially among species, thereby introducing another source of uncertainty.

Soil pH and reduction/oxidation potential are important variables when determining plant uptake of Pb (Swaine 1986). Fixation of soluble Pb by organic matter often occurs in the humus soil layer, reducing the amount of soluble Pb available for uptake by plants (Manninen and Tanskanen 1993). A decision was made prior to conducting the risk assessment not to evaluate the affect of lead to plants. However, the analysis of the soil indicated a low pH (as discussed in Section 4.3 with regards to the toxicity tests) and high sand content (Section 4.2.3). These physical factors increase the availability of lead to plants and therefore the impact plants remains an uncertainty in this risk assessment.

## 6.0 CONCLUSIONS

The results of the samples collected from the range indicate that the site has been contaminated with Pb and Pb shot. Results of the BERA indicate that there is risk to each receptor group.

The LOAELs developed from the toxicity test and the food chain models were tabulated and the lowest LOAEL of 260 mg/kg Pb was selected as an acceptable upper bound RG (Table 11). The NOAELs developed from the toxicity test and food chain models were also tabulated and the highest NOAEL below the lowest LOAEL of 46 mg/kg Pb was selected as an acceptable lower bound RG. These values provide an acceptable range of RGs for this site.

In order to determine the aerial extent of contamination, Pb isopleths were plotted based on a krigging analysis of the data. The results of this analyses indicate that approximately 15 acres of the range exceed the LOAEL based RG.

Since the probability model indicated that gallinaceous birds at Range 17 are at risk due to the ingestion of Pb shot (Table 10), remedial goals were determined by calculating a number of Pb shot per unit area that would result in an acceptable probability (less than or equal to 10%) that Pb shot would be ingested. The range of acceptable numbers was between three and 13 Pb shot/ft<sup>2</sup> (Table 12). Since the number of grit sized particles varied between locations, the most conservative value was chosen for this BERA. The RG for Pb shot in Range 17 soil was determined to be three Pb shot/ft<sup>2</sup> (Table 12).

## 7.0 REFERENCES

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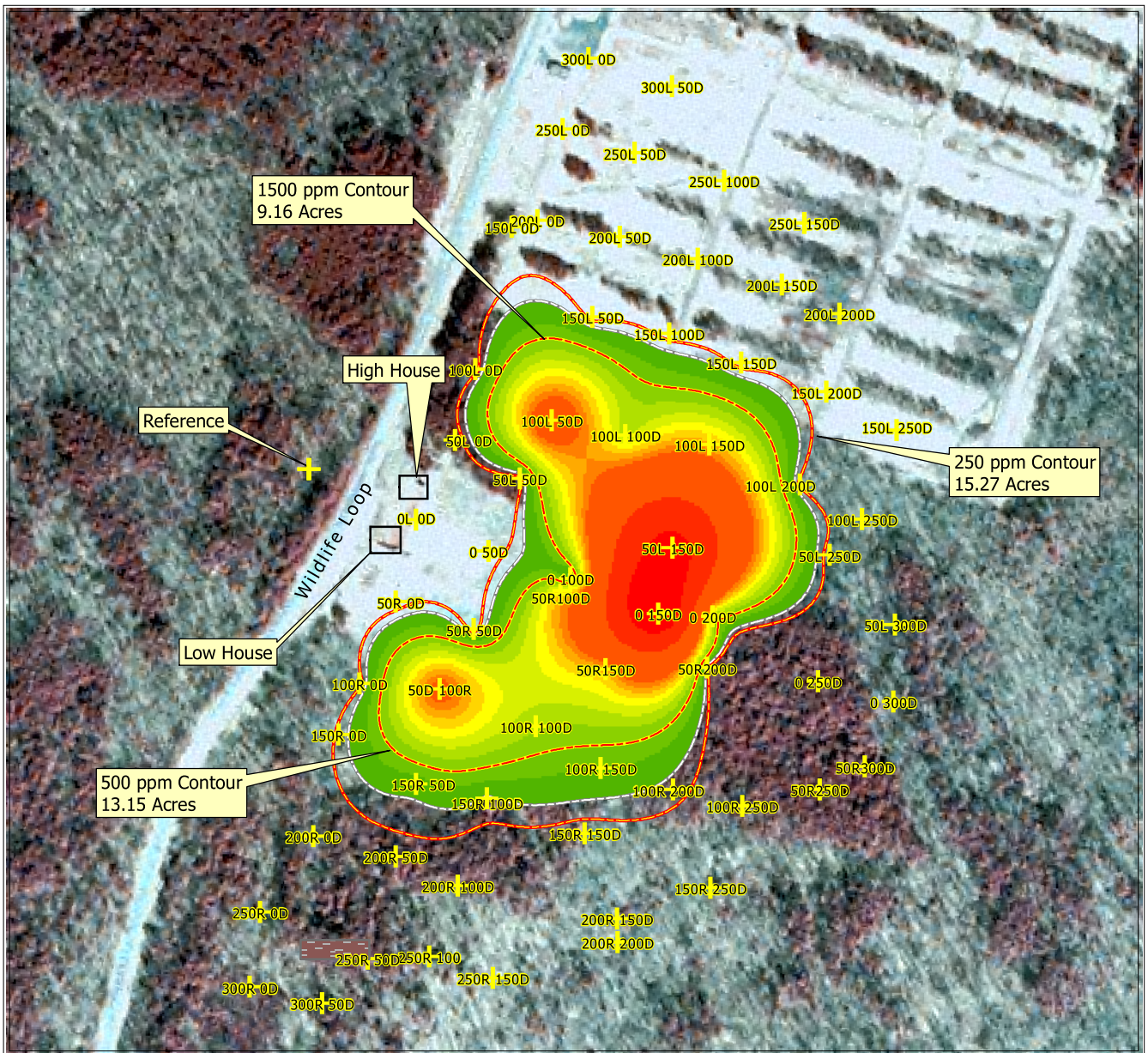
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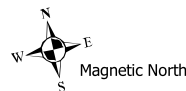
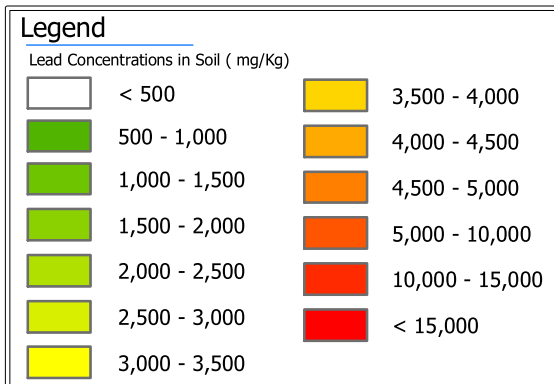


Coordinates Not Available for Locations 150R 200D  
50L 200D  
50L 100D

Map created using site survey GPS data and a reprojected USGS DOQQ.

Map creation date: 31March2004

Projection: Maryland State Plane  
Datum: NAD83  
Units: Feet  
FIPS: 1900



U.S. EPA Environmental Response Team Center  
Response Engineering and Analytical Contract  
68-C99-223  
W.A. #R1A00318

**Figure 2**  
**Lead Concentrations**  
**Range 17**  
**Laurel, MD**

Table 1. Results of Arsenic, Lead, Antimony, and Copper  
in Surface Soil Analyzed Using X-ray Fluorescence  
Range 17 - Patuxent Research Refuge  
Laurel, MD  
March 2004

Location	Date Analyzed	As	Pb	Cu	Sb
0L-0D	28-Jul-03	U	150	U	U
0L-0D (DUP)	28-Jul-03	U	150	U	U
0-50D	28-Jul-03	U	110	U	U
0-100D	28-Jul-03	U	230	U	U
0-150D	28-Jul-03	U	22,000	U	160
0-200D	28-Jul-03	U	2,000	110	U
0-250D	28-Jul-03	U	51	U	U
0-300D	28-Jul-03	U	U	U	U
50L-0D	28-Jul-03	U	43	U	U
50L-50D	28-Jul-03	U	120	U	U
50L-100D	28-Jul-03	180	1,800	U	U
50L-150D	28-Jul-03	U	18,000	U	U
50L-150D (DUP)	28-Jul-03	U	17,000	U	U
50L-200D	29-Jul-03	U	3,200	110	U
50L-250D	28-Jul-03	U	57	U	U
50L-300D	29-Jul-03	U	51	U	U
50L-300D (DUP)	29-Jul-03	U	U	U	U
50R-0D	28-Jul-03	U	48	U	U
50R-50D	28-Jul-03	U	42	U	U
50R-50D (DUP)	28-Jul-03	U	100	U	U
50R-100D	28-Jul-03	U	4,300	U	U
50R-150D	28-Jul-03	U	4,200	U	U
50R-200D	28-Jul-03	U	280	U	U
50R-250D	28-Jul-03	U	39	U	U
50R-300D	28-Jul-03	U	40	U	U

Results reported in mg/kg, dry weight

Method detection limits (MDLs):

Arsenic	33
Lead	39
Copper	81
Antimony	100

U denotes not detected at a concentration greater than the MDL

Dup denotes duplicate analyses

Table 1 (cont'd). Results of Arsenic, Lead, Antimony, and Copper  
in Soil Surface Analyzed Using X-ray Fluorescence  
Range 17 - Patuxent Research Refuge  
Laurel, MD  
March 2004

Location	Date Analyzed	As	Pb	Cu	Sb
100L-0D	28-Jul-03	U	100	U	U
100L-0D (DUP)	28-Jul-03	33	110	U	U
100L-50D	29-Jul-03	U	7,200	U	U
100L-100D	29-Jul-03	U	2,200	U	U
100L-150D	29-Jul-03	U	4,200	U	U
100L-200D	28-Jul-03	U	140	100	U
100L-250D	29-Jul-03	U	75	95	U
100R-0D	28-Jul-03	U	120	U	U
100R-50D	28-Jul-03	U	5,900	U	U
100R-100D	28-Jul-03	U	2,100	U	U
100R-150D	28-Jul-03	U	1,000	U	U
100R-200D	28-Jul-03	U	370	U	U
100R-250D	29-Jul-03	U	81	120	U
150L-0D	28-Jul-03	U	170	U	U
150L-50D	28-Jul-03	U	91	U	U
150L-100D	29-Jul-03	U	97	U	U
150L-150D	28-Jul-03	40	85	88	U
150L-200D	29-Jul-03	U	58	U	U
150L-250D	28-Jul-03	U	64	U	U
150R-0D	28-Jul-03	U	U	U	U
150R-50D	28-Jul-03	U	700	U	U
150R-100D	28-Jul-03	U	440	U	U
150R-150D	28-Jul-03	U	89	U	U
150R-200D	28-Jul-03	40	56	U	U
150R-250D	28-Jul-03	U	54	U	U
200L-0D	28-Jul-03	U	U	U	U

Results reported in mg/kg, dry weight

Method detection limits (MDLs):

Arsenic            33

Lead                39

Copper            81

Antimony        100

U denotes not detected at a concentration greater than the MDL

Dup denotes duplicate analyses

Table 1 (cont'd). Results of Arsenic, Lead, Antimony, and Copper  
in Surface Soil Analyzed Using X-ray Fluorescence  
Range 17 - Patuxent Research Refuge  
Laurel, MD  
March 2004

Location	Date Analyzed	As	Pb	Cu	Sb
200L-0D (DUP)	28-Jul-03	U	U	U	U
200L-50D	28-Jul-03	U	43	U	U
200L-100D	28-Jul-03	U	U	U	U
200L-150D	28-Jul-03	U	59	U	U
200L-200D	29-Jul-03	U	79	U	U
200R-0D	29-Jul-03	U	U	U	U
200R-50D	29-Jul-03	U	U	U	U
200R-50D (DUP)	29-Jul-03	U	57	U	U
200R-100D	29-Jul-03	U	190	U	U
200R-150D	29-Jul-03	U	U	110	U
200R-200D	29-Jul-03	U	84	U	U
250L-0D	29-Jul-03	U	U	U	U
250L-50D	28-Jul-03	U	U	U	U
250L-100D	28-Jul-03	U	96	U	U
250L-150D	28-Jul-03	U	40	U	U
250R-0D	29-Jul-03	U	55	U	U
250R-50D	29-Jul-03	U	85	U	U
250R-100D	29-Jul-03	37	81	U	U
250R-150D	29-Jul-03	U	59	U	U
300L-0D	28-Jul-03	U	U	U	U
300L-50D	28-Jul-03	U	U	U	U
300R-0D	29-Jul-03	42	U	U	U
300R-50D	29-Jul-03	U	U	U	U

Results reported in mg/kg, dry weight

Method detection limits (MDLs):

Arsenic	33
Lead	39
Copper	81
Antimony	100

U denotes not detected at a concentration greater than the MDL

Dup denotes duplicate analyses

Table 2. Confirmation Results of Surface Soil Samples Analyzed Using an ICAP Method  
 Range 17 - Patuxent Research Refuge  
 Laurel, MD  
 March 2004

Location Parameter	Mean MDL	0L-0D	0-150D	50L-200D	100L-50D	100L-100D	100R-150D	150R-50D	150R-100D	200R-0D	250R-50D
Antimony	1.0	U	190	5	23	2.3	1.3	U	U	U	U
Arsenic	1.0	2.3	130	11	19	7.5	5.9	1.9	2.7	2.6	2.6
Copper	0.5	17	22	51	8.9	7.4	20	3.6	5.0	4.5	12
Lead	1.0	120	18,000	2,300	5,800	2,100	720	510	300	19	54

Results report in mg/kg, dry weight

MDL denotes method detection limit

U denotes not detected at a concentration greater than the MDL

ICAP denotes inductively coupled argon plasma

Table 3. Target Analyte List Metals Analysis of Surface Soil Samples  
 Range 17 - Patuxent Research Refuge  
 Laurel, MD  
 March 2004

Location Parameter	0-150D		100R-100D		100R-50D		150R-100D		150R-50D		Reference	
	Conc	MDL	Conc	MDL	Conc	MDL	Conc	MDL	Conc	MDL	Conc	MDL
Aluminum	5,800	10	1,700	8.6	8,300	11	2200	8.7	1,800	9.4	3,100	9.7
Antimony	340	1	0.97	0.86	5.2	1.1	U	0.87	U	0.94	U	0.97
Arsenic	220	1	2.9	0.86	12	1.1	2.8	0.87	2.1	0.94	3	0.97
Barium	22	0.51	6.2	0.43	35	0.56	7.3	0.44	7.3	0.47	15	0.48
Beryllium	U	0.51	U	0.43	U	0.56	U	0.44	U	0.47	U	0.48
Cadmium	U	0.51	U	0.43	U	0.56	U	0.44	U	0.47	U	0.48
Calcium	200	10	47	8.6	150	11	95	8.7	130	9.4	38	9.7
Chromium	13	0.51	3.4	0.43	11	0.56	4.9	0.44	3.9	0.47	6.4	0.48
Cobalt	3.7	0.51	U	0.43	6.4	0.56	0.5	0.44	0.52	0.47	1.3	0.48
Copper	25	0.51	5.9	0.43	15	0.56	5.9	0.44	3.4	0.47	9.2	0.48
Iron	13,000	4	2,500	3.4	10,000	4.5	3,700	3.5	2,800	3.8	5,700	3.9
Lead	44,000	5.1	540	0.86	3,000	1.1	260	0.87	270	0.94	46	0.97
Magnesium	270	51	89	43	450	56	140	44	120	47	130	48
Manganese	110	0.51	11	0.43	430	0.56	17	0.44	16	0.47	37	0.48
Mercury	0.072	0.038	0.099	0.032	0.098	0.04	0.037	0.033	U	0.033	0.056	0.036
Nickel	4.7	0.51	1.1	0.43	5.9	0.56	1.8	0.44	1.5	0.47	1.8	0.48
Potassium	270	51	92	43	360	56	100	44	84	47	230	48
Selenium	1.9	0.51	0.8	0.43	1.9	0.56	0.89	0.44	0.68	0.47	0.94	0.48
Silver	1.5	0.51	U	0.43	U	0.56	U	0.44	U	0.47	U	0.48
Sodium	U	100	U	86	U	110	U	87	U	94	U	97
Thallium	U	1	U	0.86	U	1.1	U	0.87	U	0.94	U	0.97
Vanadium	27	0.51	5.7	0.43	22	0.56	9.6	0.44	7	0.47	15	0.48
Zinc	28	1	8.6	0.86	35	1.1	11	0.87	12	0.94	15	0.97

MDL denotes method detection limit

U denotes not detected

mg/kg denotes milligrams per kilogram

Conc. denotes concentration

Pb vs. Sb are correlated (0.99, using 1/2 the MDL for non detect values)

Table 4. Comparison of Target Analyte List Metals to Ecological Benchmarks  
 Range 17 - Patuxent Research Refuge  
 Laurel, MD  
 March 2004

Parameter	Mean Concentration	Maximum Concentration	Screening Benchmark <sup>(1)</sup>	Hazard Quotient	COPC	Explanation
Aluminum	3,960	8,300	50	166	Y	Retained - Hazard Quotient > 1
Antimony	115	340	3.5	97	Y	Retained - Previously selected as a COPC
Arsenic	48	220	10	22	Y	Retained - Previously selected as a COPC
Barium	16	35	165	0.2	N	Eliminated - Hazard Quotient < 1
Beryllium	U	U	1.1	na	N	Eliminated - Not detected and the MDL < Benchmark
Cadmium	U	U	1.6	na	N	Eliminated - Not detected and the MDL < Benchmark
Calcium	124	200	na	na	Y	Retained - No benchmark available
Chromium	7	13	0.4	32.5	Y	Retained - Hazard Quotient > 1
Cobalt	3	6.4	20	0.3	N	Eliminated - Hazard Quotient < 1
Copper	11	25	40	0.6	Y	Retained - Previously selected as a COPC
Iron	6,400	13,000	200	65	Y	Retained - Hazard Quotient > 1
Lead	9,614	44,000	50	880	Y	Retained - Previously selected as a COPC
Magnesium	214	450	na	na	Y	Retained - No benchmark available
Manganese	117	430	100	4	Y	Retained - Hazard Quotient > 1
Mercury	0.077	0.099	0.1	0.99	N	Eliminated - Hazard Quotient < 1
Nickel	3	5.9	30	0.2	N	Eliminated - Hazard Quotient < 1
Potassium	181	360	na	na	Y	Retained - No benchmark available
Selenium	1	1.9	0.81	2	Y	Retained - Hazard Quotient > 1
Silver	na	1.5	2	0.8	N	Eliminated - Hazard Quotient < 1
Sodium	U	U	na	na	N	Eliminated - Not detected and no benchmark available
Thallium	U	U	1	na	N	Eliminated - Not detected and the MDL < Benchmark
Vanadium	14	27	2	13	Y	Retained - Hazard Quotient > 1
Zinc	19	35	50	0.7	N	Eliminated - Hazard Quotient < 1

Concentrations reported in mg/kg

U denotes not detected at a concentration greater than the MDL

na denotes not applicable

COPC denotes contaminant of potential concern

MDL denotes method detection limit

Hazard Quotient = Maximum Concentration/Ecological Benchmark

(1) Friday, G.P. 1998. Ecological Screening Values for Surface Water, Sediment, and Soil.

Westinghouse Savannah River Co. Report TR-98-00110. Aiken, SC

Table 5. Results of the 28-day Toxicity Test  
 Using the Earthworm, *Eisenia foetida*  
 Range 17 - Patuxent Research Refuge  
 Laurel, MD  
 March 2004

Location	Soil Lead Conc. (mg/kg)	14-day Survival * (%)	28-day Survival * (%)
Reference	46	97.5	97.5
150R-100D	260	100	81.5 <sup>(a)</sup>
150R-50D	270	82.5	67.5 <sup>(a)</sup>
100R-100D	540	0	0 <sup>(a)</sup>
100R-50D	3,000	0	0 <sup>(a)</sup>
0-150D	44,000	0	0 <sup>(a)</sup>

Conc. denotes concentration

mg/kg denotes milligrams per kilogram

% denotes percent

\* Survival is reported as the average of the 4 replicates tested

(a) Indicates statistically different than the Reference (p<0.05)



Table 6. Life History Parameters Used in the Food Chain and Pellet Ingestion Models  
 Range 17 - Patuxent Research Refuge  
 Laurel, MD  
 March 2004

	Value	Units	Reference
<b>Short-tailed shrew</b>			
<i>Blarina brevicauda</i>			
Home Range	≤ 1	acre	Merritt 1987
Area Use Factor	1.0	unitless	
Food Ingestion Rate	0.00795	kg/day	U.S. EPA 1993
Soil Ingestion Rate	0.00075 <sup>(a)</sup>	kg/day	Beyer et al. 1994
Body Weight	0.021	kg	Jones and Birney 1988
<b>American robin</b>			
<i>Turdus migratorius</i>			
Home Range	≤ 2	acres	U.S. EPA 1993
Area Use Factor	1.0	unitless	
Food Ingestion Rate	0.00696	kg/day	Various authors, See App. A
Soil Ingestion Rate	0.000724 <sup>(b)</sup>	kg/day	Beyer et al. 1994
Body Weight	0.0771	kg	Various authors, See App. A
<b>Mourning dove</b>			
<i>Zenaida macroura carolinensis</i>			
Home Range	218	hectares	Mirarchi and Baskett, 1994
Average Lifespan	1.5	years	Mirarchi and Baskett, 1994
No. of Foraging Days	245	days (Mar15-Nov15)	
Grit Retention Time	6	days	McConnell, 1967

- (a) - Soil ingestion rate based on a opossum (9.4% of the diet)  
 (b) - Soil ingestion rate based on a woodcock (10% of the diet)

References:

- (1) Beyer, W. N., E. E. Connor, and S. Gerould. 1994. Estimates of soil ingestion by wildlife. *J. Wildl. Manage.* 58(2):375-382.
- (2) Jones, Jr., J. K. J. and E.C. Birney. 1988. *Handbook of Mammals of the North Central States*. Minneapolis, MN: University of Minnesota Press, 346 pp.
- (3) McConnell, C.A. 1967. Experimental lead poisoning of bobwhite quail and mourning doves. *Proc. Ann. Conf. Southeast Assoc. Game Fish Comm.* 21:208-219.
- (4) Merritt, J. F. 1987. *Guide to the Mammals of Pennsylvania*. Pittsburgh, PA: University of Pittsburgh Press, 408 pp.
- (5) Mirarchi, R.E. and T.S. Baskett. 1994. Mourning Dove. In *The Birds of North America*, A. Poole, P. Stettenheim, F.B. Gill (eds.). Amer. Ornithol. Union & Acad. of Nat'l Sci., Phila., PA No. 117.
- (6) U.S. EPA. 1993. *Wildlife Exposure Factors Handbook, Volume I of II*. U. S. EPA, Office of Research and Development, Washington, D.C. EPA/600/R-93/187a

Table 7. Hazard Quotient Calculations for the American Robin  
 Range 17 - Patuxent Research Refuge  
 Laurel, MD  
 March 2004

Location	Soil Lead Conc. (mg/kg)	Soil Ingestion Rate (kg/day)	BAF	Worm Lead Conc. (mg/kg)	Food Ingestion Rate (kg/day)	Area Use Factor	Body Weight (kg)	Dose (mg/kg BW/day)	LOAEL TRV (mg/kg BW/day)	LOAEL HQ	NOAEL TRV (mg/kg BW/day)	NOAEL HQ
0-150D	44,000	0.000724	0.39	17,160	0.00696	1.00	0.0771	1962.3	15.0	130.8	1.5	1308.2
100R-50D	3,000	0.000724	0.39	1,170	0.00696	1.00	0.0771	133.8	15.0	8.9	1.5	89.2
100R-100D	540	0.000724	0.39	211	0.00696	1.00	0.0771	24.1	15.0	1.6	1.5	16.1
150R-50D	270	0.000724	0.39	105	0.00696	1.00	0.0771	12.0	15.0	0.8	1.5	8.0
150R-100D	260	0.000724	0.39	101	0.00696	1.00	0.0771	11.6	15.0	0.8	1.5	7.7
Reference	46	0.000724	0.39	18	0.00696	1.00	0.0771	2.1	15.0	0.1	1.5	1.4

BAF denotes bioaccumulation factor (Section 5.1)  
 LOAEL denotes lowest observed apparent effect level  
 TRV denotes toxicity reference value  
 HQ denotes hazard quotient  
 NOAEL denotes no observed apparent effect level

Table 8. Hazard Quotient Calculations for the Short-tailed Shrew  
 Range 17 - Patuxent Research Refuge  
 Laurel, MD  
 March 2004

Location	Soil Lead Conc. (mg/kg)	Soil Ingestion Rate (kg/day)	BAF	Worm Lead Conc. (mg/kg)	Food Ingestion Rate (kg/day)	Area Use Factor	Body Weight (kg)	Dose (mg/kg BW/day)	LOAEL TRV (mg/kg BW/day)	LOAEL HQ	NOAEL TRV (mg/kg BW/day)	NOAEL HQ
0-150D	44,000	0.00075	0.39	17,160	0.00795	1.00	0.021	8067.7	77.0	104.8	7.7	1047.8
100R-50D	3,000	0.00075	0.39	1,170	0.00795	1.00	0.021	550.1	77.0	7.1	7.7	71.4
100R-100D	540	0.00075	0.39	211	0.00795	1.00	0.021	99.0	77.0	1.3	7.7	12.9
150R-50D	270	0.00075	0.39	105	0.00795	1.00	0.021	49.5	77.0	0.6	7.7	6.4
150R-100D	260	0.00075	0.39	101	0.00795	1.00	0.021	47.7	77.0	0.6	7.7	6.2
Reference	46	0.00075	0.39	18	0.00795	1.00	0.021	8.4	77.0	0.1	7.7	1.1

BAF denotes bioaccumulation factor (Section 5.1)  
 LOAEL denotes lowest observed apparent effect level  
 TRV denotes toxicity reference value  
 HQ denotes hazard quotient  
 NOAEL denotes no observed apparent effect level

Table 9. Results of Lead Shot and Grit Counts  
 Range 17 - Patuxent Research Refuge  
 Laurel, MD  
 March 2004

Location	Lead Concentration (mg/kg)	Number of Lead Shot	Number of Grit Particles*
Reference	46	0	844
50L-200D	3,200	10	2,272
100L-200D	140	12	7,713
0-100D	230	41	4,803
0-200D	2,000	60	5,742
50R-100D	4,300	136	3,767
50L-100D	1,800	262	2,871
50L-150D	18,000	460	5,532
50R-150D	4,200	992	1,602
0-150D	22,000	2,946	4,722

\* - Defined as passing through a 2.8 mm-mesh sieve  
 and retained on a 0.5 mm-mesh sieve  
 mg/kg denotes milligrams per kilogram

Lead concentration and lead shot are correlated ( $r^2=0.79$ )

Table 10. Probability of a Mourning Dove Ingesting Lead Shot  
 Range 17 - Patuxent Research Refuge  
 Laurel, MD  
 March 2004

Location	Number of Lead Shot	Number of Grit Particles	Percent Grit that is Lead Shot (Ps)	Area Use Factor (S)	Percent Grit * AUF (P)	Lifespan (Y)	Foraging days per Year (De)	Grit Retention Time (Dp)	Total Number of Particles Selected (N)	Probability (Pt)
Reference	0	844	0.0000	1.0	0.0000	1.5	245	6.0	61.25	0.00
50L-200D	10	2,272	0.0044	1.0	0.0044	1.5	245	6.0	61.25	0.24
100L-200D	12	7,713	0.0016	1.0	0.0016	1.5	245	6.0	61.25	0.09
0-100D	41	4,803	0.0085	1.0	0.0085	1.5	245	6.0	61.25	0.41
0-200D	60	5,742	0.0103	1.0	0.0103	1.5	245	6.0	61.25	0.47
50R-100D	136	3,767	0.0348	1.0	0.0348	1.5	245	6.0	61.25	0.89
50-100D	262	2,871	0.0836	1.0	0.0836	1.5	245	6.0	61.25	1.00
50L-150D	460	5,532	0.0768	1.0	0.0768	1.5	245	6.0	61.25	0.99
150R-150D	992	1,602	0.3824	1.0	0.3824	1.5	245	6.0	61.25	1.00
0-150D	2,946	4,722	0.3842	1.0	0.3842	1.5	245	6.0	61.25	1.00

Formula

$$P = S(Ps) + (1-S)Po$$

$$N = Y(De/Dp)$$

$$Pt = 1 - (1 - P)^N$$

Note: For this risk assessment, we assumed that  $Po = 0$ , therefore, this term is not included in the calculation

Table 11. Remedial Goals Based on Soil Lead Concentration  
 Range 17 - Patuxent Research Refuge  
 Laurel, MD  
 March 2004

Assessment Endpoint	Measurement Endpoint	NOAEL (mg/kg)	LOAEL (mg/kg)
Terrestrial invertebrates	Earthworm	<b>46</b>	<b>260</b>
Avian insectivore	American robin	35	320
Terrestrial insectivore	Short-tailed shrew	44	440

**Remedial Goals** - The range from the highest NOAEL below the lowest LOAEL to the lowest LOAEL of the data set.

mg/kg denotes milligrams per kilogram

NOAEL denotes no observed adverse effect level

LOAEL denotes lowest observed adverse effect level

Table 12. Remedial Goals for Lead Shot  
 Range 17 - Patuxent Research Refuge  
 Laurel, MD  
 March 2004

Location	Number of Lead Shot	Number of Grit Particles	Percent Grit that is Lead Shot (Ps)	Area Use Factor (S)	Percent Grit * AUF (P)	Lifespan (Y)	Foraging days per Year (De)	Grit Retention Time (Dp)	Total Number of Particles Selected (N)	Probability (Pt)
Reference	0	844	0.0000	1.0	0.0000	1.5	245	6.0	61.25	0.00
50L-200D	4	2,272	0.0018	1.0	0.0018	1.5	245	6.0	61.25	0.10
100L-200D	13	7,713	0.0017	1.0	0.0017	1.5	245	6.0	61.25	0.10
0-100D	8	4,803	0.0017	1.0	0.0017	1.5	245	6.0	61.25	0.10
0-200D	10	5,742	0.0017	1.0	0.0017	1.5	245	6.0	61.25	0.10
50R-100D	7	3,767	0.0019	1.0	0.0019	1.5	245	6.0	61.25	0.11
50L-100D	5	2,871	0.0017	1.0	0.0017	1.5	245	6.0	61.25	0.10
50L-150D	10	5,532	0.0018	1.0	0.0018	1.5	245	6.0	61.25	0.10
150R-150D	3	1,602	0.0019	1.0	0.0019	1.5	245	6.0	61.25	0.11
0-150D	8	4,722	0.0017	1.0	0.0017	1.5	245	6.0	61.25	0.10

Formula

$$P = S(Ps) + (1-S)Po$$

$$N = Y(De/Dp)$$

$$Pt = 1-(1-P)N$$

Note: For this risk assessment, we assumed that  $Po = 0$ , therefore, this term is not included in the calculation

Appendix A  
Life History and Exposure Profiles  
Range 17 - Patuxent Research Refuge  
Laurel, MD  
March 2004



## Life History/Exposure Profile Information

Listed below is review of the general life history of each of the selected receptor species to evaluate the measurement endpoints. This information indicates that the selected species may use or inhabit the site areas, and thus supports their use as valid measurement endpoints.

### **Terrestrial Invertebrates**

#### Justification

Earthworms are in direct contact with soil, and may comprise as much as three fourths of the soil animal biomass in many terrestrial ecosystems (Cocking et al. 1994). They benefit the soil structure by increasing aggregate formation, aerating, and increasing moisture-holding capacity. Earthworms are an important food source for many terrestrial mammals and birds.

#### Life History

The oligochaetes include earthworms and a group of related, mostly freshwater, species of annelids, and over 3,000 species are known (Hickman and Roberts 1994). Earthworms are segmented, and segments each contain elements of such body systems as circulatory, nervous, and excretory tracts (Brusca and Brusca 1990). Segmentation increases the efficiency of body movement by allowing the effect of muscle contraction to be extremely localized, and it enables the development of greater complexity in general body organization (Brusca and Brusca 1990).

Besides being segmented, the body wall of earthworms is characterized by circular and longitudinal muscle fibers surrounded by a moist, acellular cuticle that is secreted by an epidermal epithelium. Earthworms are schizocoelous, with a large and well-developed true coelom that is lined with mesoderm. The coelom is partially subdivided by septa. Hydrostatic pressure is maintained across segments and helps maintain body rigidity, allowing muscle contractions to bend the body without collapsing it (Brusca and Brusca 1990).

The internal organs of earthworms are well developed. They include a closed, segmentally-arranged circulatory system. The digestive system is a complete tube with mouth and anus. Gases are exchanged through the skin, or sometimes through specialized gills or modified parapodia. Each segment typically contains a pair of nephridia. The nervous system includes a pair of cephalic ganglia attached to double nerve cords that run the length of the animal along the ventral body wall, with ganglia and branches in each segment. Earthworms have some combination of tactile organs, chemoreceptors, balance receptors, and photoreceptors; and some species have fairly well developed eyes, including lenses (Brusca and Brusca 1990). Oligochaetes possess permanent sex organs. Most are hermaphroditic, and development is direct, resulting in young that resemble tiny adults (Hickman and Roberts 1994).

Ecologically, earthworms range from passive filter feeders to voracious and active predators, and feed primarily on detritus and algae. Earthworms cycle large quantities of soil through their guts, a process that speeds the turnover of nutrients in soil and increases productivity.

Earthworms pass a mixture of both organic and inorganic materials through their guts when feeding (Cocking et al. 1994). Earthworms are sometimes classified into two groups depending on depth of activity. The first group, the deep-working group, move through the full depth of available surface and subsurface soil; whereas the second group, the shallow-working group, confine their activities to the upper 15 centimeters (Cocking et al. 1994). Larger earthworms, that feed on organic matter by drawing leaves and other materials into their mouth, ingest larger quantities of soil, compared to smaller worms that consume fragmented litter (Cocking et al. 1994).

#### Exposure Profile

Earthworms cycle large quantities of soil through their guts, as they feed. Since direct contact with and ingestion of contaminated soil are the primary routes of exposure for *Eisenia foetida*.

## **Upland birds exposed directly to lead shot**

### Justification

Large numbers of upland birds use areas within the former skeet and trap range either seasonally or year-round to hunt for food. Exposure to lead shot creates a potential for direct toxicity to these birds. The mourning dove (*Zenaida macroura carolinensis*) was selected as a receptor for the lead shot ingestion pathway, because it is a common bird species that uses grit. Also, several publications have documented the potential for toxicity to this species following ingestion of lead shot (Lewis and Legler 1968; Kendall et al. 1996; Buerger et al. 1986; McConnell 1967; Marn et al. 1988).

### Life History

Mourning doves are one of the most abundant game birds in North America. These birds are medium sized, brownish, with a rounded or pointed white-tipped tail. The males are larger (130.4 g) than the females (124.7 g), and are typically brighter colored (Basket et al. 1993).

Mourning doves are very common throughout North America. This species breeds throughout south Canada, and all of the continental United States into Baja California and Mexico south to Puebla. As migratory birds, they winter throughout most of their breeding range, except central Canada and north-central United States south to Central America (Mirarchi and Baskett 1994).

Doves mate for life, with a breeding season ranging from April to August. Doves typically nest in trees along the edges of fields, pastures, or clearings. Flimsy nests, in trees and shrubs, are made using grass and twigs. The clutch size ranges from one to four, with a mean of two eggs, and egg color is pure white. Incubation is performed by both sexes, male by day, female by night, and generally lasts between 13 and 14 days (Ehrlich et al. 1988). The pair may raise 2-5 broods/season, with fledging occurring within 12 to 14 days.

Mourning doves are predominately seed eaters, and consume a wide variety of seeds, including buckwheat, millet, corn, wheat, rye, and peanuts, (Ehrlich et al. 1988). Favorite non-agricultural seeds include a variety of grasses, supurges, goosefoots and saltbushes, ragweed, pokeweed, and poppies. Grit is an essential component of diet, but function appears mechanical rather than nutritive (Mirarchi and Baskett 1994). Doves prefer seeds that lie on the ground, and pick up grit to help grind the seeds. It has been estimated that 20 percent of each day is spent feeding, including searching procuring and handling food and grit, drinking, and pecking at bark (Mirarchi and Baskett 1994).

### Exposure Profile

Mourning doves average 22.5 to 34 cm, and have an average wingspread of 43 to 48 cm (Mirarchi and Baskett 1994). Daily home ranges vary from 50 to 1,200 hectare (ha), with an average of 218 ha (Mirarchi and Baskett 1994) and the average number of years a bird lives is 1.5 years (Mirarchi and Baskett, 1994). Given the results of the blood results for the juncos collected from the site (elevated indicating exposure), we felt it appropriate to use 1 as an area use factor in this model. The number of days per year that a bird forages was assumed to be 245. This assumption was based on a bird migrating from November 15 to March 15. The retention time for shot in the gizzard (6 days) is based on literature (McConnell, 1967).

## **Avian Insectivore**

### Justification

The American robin (*Turdus migratorius*) was selected as an appropriate omnivorous bird species to evaluate effects of accumulation of lead within the food web. The diet of the American robin consists of seasonally variable proportions of invertebrates (earthworms, snails, beetles, caterpillars, spiders, etc.) and fruit (dogwood, cherry, sumac, hackberries, raspberries, etc.)(U.S. EPA 1993 and Ehrlich et al. 1988). They are common in the area and are

likely summer residents at the site.

### Life History

The American robin (*Turdus migratorius*) occurs throughout most of the continental United States and Canada, wintering in the southern half of North America and into Central America (Bull and Farrand 1977; Peterson 1990). Given the increase in open habitat and lawns, the robin's breeding range has expanded in recent times (Collins and Boyaijan 1965; Ehrlich *et al.* 1988). Habitat requirements for breeding robins include access to fresh water, protected nesting sites, and productive foraging areas (Howell 1942; Ehrlich *et al.* 1988). These requirements are commonly met in moist forests, swamps, open woodlands, and other open areas (Bull and Farrand 1977). Non-breeding robins occupy similar habitats, although proximity to fruit bearing trees is of more importance.

Male robins are characterized by a dark grey to black head and back with a bright red to orange breast. Females and juveniles are similar to males in appearance but much duller in coloring, and juveniles have black spots on their breasts. The largest of the North American thrushes, both males and female robins grow to 9 to 11 inches long. Robin legs are classified as booted tarsi, a long leg with few scales (Collins and Boyaijan 1965).

The primary foraging technique for robins is to hop along the ground in search of ground-dwelling invertebrates, although they commonly search for insects and fruit in tree branches as well. The robin's diet during the breeding season consists mainly of invertebrates and some fruit, but fruit is the primary food consumed outside of the breeding season. Robins exhibit a low digestive efficiency for fruit, and they often consume more than their own body weight (BW) in fruit to meet their metabolic needs (Hazelton *et al.* 1984).

The diet of the American robin consists of seasonally variable proportions of invertebrates (e.g., earthworms, snails, beetles, caterpillars, spiders) and fruit (e.g., dogwood, cherry, sumac, holly, hackberries, and juneberries) (Martin *et al.* 1951; Paszkowski 1982; Wheelwright 1986; Ehrlich *et al.* 1988). The ratio of percent (%) invertebrates to % fruit in the diet is reported to change from 94:6 in spring (nesting season) to 34:66 in summer to 4:96 in fall (migratory season) to 7:93 in winter (Wheelwright 1986). Year round, the diet of the robin averages 63% fruits and 37% invertebrates (Martin *et al.* 1951; Eiserer 1976; Wheelwright 1988). Robin diets are diverse; analysis of the stomach contents of 1900 robins showed that the birds consumed fruit from more than 50 plant genera and invertebrates from over 100 families (Wheelwright 1986).

Robins typically use the same foraging site for many weeks at a time but join a variety of roosts, usually within 2 kilometers (km) of the foraging area (Morrison and Caccamise 1990). During the breeding periods, male robins establish territories, the size of which is determined by population density: smaller territories are found where robin densities are high. Most foraging occurs within these territories; however, if food resources are limited, adult robins will leave temporarily to forage elsewhere. Breeding territories are vigorously defended; robins will attack man, snake or other enemies to defend its territory or nest, except in more remote locations (Howell 1942; Collins and Boyaijan 1965). Females lay eggs in nests made of mud, grass, and twigs, built 0.9 to 7.5 meters above the ground in trees, buildings, or shrubs. Eggs are characteristically bright blue, number from 3 to 6, and hatch in 12 to 14 days. Young juveniles leave two weeks after hatching and can live up to ten years in the wild, though most rarely survive past 14 months (Collins and Boyaijan 1965; Cassidy 1990).

Predators that feed on adult robins include cats, dogs, owls, and hawks. Crows, jays, grackles, snakes and squirrels are nest predators, attacking both eggs and nestlings. A robin that survives to adulthood has a life expectancy of 10 years (Eiserer 1976; Wauer 1999).

### Exposure Profile

Adult American robins weigh from 0.055 to 0.103 kg (Eiserer 1976; Clench and Leberman 1978; Hazelton *et al.* 1984; Skorupa and Hothem 1985; Wheelwright 1986; Wheelwright 1988; Wauer 1999). The average value (0.0771 kg) was used as a representative measure of BW.

The diet of the American robin primarily consists of fruit and invertebrates. Diet varies seasonally, and depends on habitat and time of day (Wheelwright 1988). The year-round diet is comprised of 37% invertebrates and 63% fruits

(Martin *et al.* 1951; Eiserer 1976; Wheelwright 1988). For this risk assessment, however, the diet of the American robin was assumed to consist solely of invertebrates.

Food ingestion rates (FIR) for adult robins are highly dependant on whether fruits or invertebrate prey are consumed. Several studies were located that reported daily fruit ingestion rates ranging from 0.0571 to 0.1078 kilograms per day (kg/d), wet weight (Hazelton *et al.* 1984; Tobin 1984; Skorupa and Hothem 1985; Levey and Karasov 1989), with the average being 0.753 kg/d. One study was located that reported a food ingestion rate for robins feeding on invertebrates; Levey and Karasov (1989) reported an ingestion rate of 0.024 kg/d wet weight for robins consuming crickets. These values were converted to dry weights using the water content of the dietary components (U.S. EPA 1993). In this risk assessment, assuming a diet consisting of 100% invertebrates, robins consume 0.00696 kg/d dry weight under both conservative and representative scenarios.

Soil ingestion for the American robin was derived from calculated values determined by (Beyer *et al.* 1994) for the American woodcock. Given that the diets of the woodcock and robin are similar, soil ingestion by the robin can be expected to be 10.4% of the diet. For robins eating only invertebrates, the soil ingestion rate of 10.4% was multiplied by the food ingestion rate of 0.00696 kg/d to yield soil ingestion estimates of 0.000724 kg/d for both the conservative and representative scenarios.

The reported HR size of the American robin ranges from 0.11 hectares (ha) to 0.42 ha (Howell 1942; Eiserer 1976; Stokes 1979; Pitts 1984; Wauer 1999). For this risk assessment, an AUF of 1 was selected because of the data that indicates that species that use this site are exposed to lead.

## **Insectivorous Mammals**

### Justification

The short-tailed shrew (*Blarina brevicauda*) was selected as representative of insectivorous mammals, because of its dietary composition, relative abundant distribution in both moist and dry habitats, and likelihood of occurrence at the site. Although their diets may consist of plants and insects, they tend to favor soil invertebrates when they are in abundance. Hence, by assuming that their dietary composition comprises solely invertebrates in this risk assessment, this species may represent an insectivorous mammal.

### Life History

The short-tailed shrew is an extremely active, large, and heavy-bodied shrew common within its range (Jones and Birney 1988). It occupies a variety of moist and dry habitats such as marshes, bogs, moist forest floors with ample decaying matter, brushland, fencerows, weedfields, and pastures (Barbour and Davis 1974; Jones and Birney 1988). Short-tailed shrews are active both day and night throughout the year, although most of this activity is subnivian (Merritt 1987). During harsh winters, this species may undergo a period of torpor (Hoffmeister 1989).

The home range of this species varies with their dramatic population cycles. In peak years, animal density may be greater than 25 individuals per acre (Schwartz and Schwartz 1981). In other years, this species may have an animal density of one individual per acre (Merritt 1987).

Although short-tailed shrews strongly prefer animal matter, they are opportunistic omnivores and voraciously consume whatever food items are in ample supply (Barbour and Davis 1974). These food items include earthworms, slugs, snails, insects, arthropods, fungi, vegetable matter, seeds, snakes, salamanders, small mammals, and young birds (Barbour and Davis 1974; Jones and Birney 1988; Schwartz and Schwartz 1981). Prey items that are not consumed immediately are stored in a cache (Merritt 1987). Plant matter is generally consumed to a greater extent in winter (Schwartz and Schwartz 1981). In some regions, plant matter may constitute up to 20 percent of the shrew's diet (Barbour and Davis 1974). Submaxillary glands produce a venom that quickly immobilizes their prey (Merritt 1987).

Using echolocation and scent-marking, short-tailed shrews rely heavily on their hearing and sense of smell to locate food and to move about (Hoffmeister 1989). An elaborate system of runways and tunnels are constructed, usually

just a few inches below the ground surface (Schwartz and Schwartz 1981). Two types of nests are built by this species, a breeding nest and a resting nest. Both nests are built underground beneath a log, rock, or other cover, and have multiple entrances. The breeding nest is typically larger than the resting nest (Merritt 1987).

Breeding appears to commence in early spring and extends into the fall, although in some regions, breeding may subside in early and midsummer, but peak again in early fall (Hoffmeister 1989; Jones and Birney 1988). Gestation periods are approximately 21 to 22 days with litter sizes of approximately 4 to 10 young (Jones and Birney 1988; Schwartz and Schwartz 1981). The young are fully mature from one to three months of age (Barbour and Davis 1974; Schwartz and Schwartz 1981). Both sexes may breed their first spring (Schwartz and Schwartz 1981).

Natural predators of the short-tailed shrew include fish, snakes, owls, hawks, shrikes, opossums, raccoons, foxes, weasels, bobcats, skunks, and feral cats, although most of these predators do not consume the shrew (or at least all of the shrew), because of their distasteful musk glands (Barbour and Davis 1974; Jones and Birney 1988; Merritt 1987; Schwartz and Schwartz 1981). The life expectancy of a short-tailed shrew in the wild is approximately one year (Schwartz and Schwartz 1981).

### Exposure Profile

Adult short-tailed shrews weigh from 12 to 30 g (Jones and Birney 1988; Merritt 1987). The mean body weight of 21 g, and an area use factor of 1 were used for this risk assessment.

The short-tailed shrew is primarily carnivorous. Its diet includes invertebrates (insects, earthworms, snails, spiders), but it also feeds on vertebrates, such as voles, amphibians, and birds (Merritt 1987, U.S. EPA 1993). Plant roots, nuts, fruits, and fungi are also part of the shrew's diet (Merritt 1987). Food ingestion rates ranging from 0.49 to 0.62 g/g of BW per day (g/g BW/day) have been reported (U.S. EPA 1993). An average food ingestion rate of 11.66 g/day was used for this risk assessment.

A soil ingestion rate for the short-tailed shrew was not available from the literature therefore the soil ingestion rate of the opossum was used. The opossum's diet is similar to that of the short-tailed shrew, since they are both opportunistic omnivores with a strong preference for animal matter (Schwartz and Schwartz 1981). A soil ingestion rate of 9.4 percent of the diet was reported for the opossum (Beyer et al. 1994). This value was multiplied by the highest food ingestion rate of the short-tailed shrew (11.66 g/day) to yield a soil ingestion rate of 1.1 g/day. For the food chain model in this risk assessment, it was assumed that 100 percent of the diet of the short-tailed shrew was comprised of invertebrates.

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## Toxicity Profile

### Lead Shot

Birds ingest lead shot while foraging in the wetland substrate and on adjacent soils in search of food and grit. Grit refers to the small stones or other hard material retained in the muscular stomach of some birds that is used to help grind up food items. The size of the lead shot used for trap and skeet is the preferred size of grit and plant seeds for ducks and geese. Transfer of lead shot through the food chain may also occur in animals that prey on birds that have ingested lead shot. For example, a mallard may ingest two pellets of #4 lead shot in its gizzard, and as the bird becomes weak and disoriented from lead poisoning, it becomes an easy food source for all predators, including raptors. Once the bird is eaten by a hawk, owl, or bald eagle, the lead enters the raptor's body and may cause its death. Bald eagles appear especially susceptible, since they utilize dead or crippled waterfowl extensively, and appear to be poor at regurgitating the shot once they ingest it. Heavy predation at wetlands helps prevent the accumulation of dead animals that might cause a noticeable die-off.

The toxicity of ingested lead shot is dependent on many factors, such as temperature, diet, age, sex, and species. The toxic action of lead is that it blocks the sulfur-hydrogen linkages in enzymes, resulting in a reduction of oxygen consumption by all tissues, a reduction in glycolysis, and an almost complete cessation of hydrogen transfer reactions in nerve tissues. It also interferes with the production of hemoglobin, resulting in severe anemia (Pain 1996), and may impair reproduction and immune system functions. Clinical signs of lead poisoning often include muscular weakness. A progressive illness results in a few weeks and may terminate in death with the ingestion of only a single lead shot (Buerger et al. 1986).

Waterfowl are believed to be at the greatest risk from deposited lead shot because of their food habits, grit use, and attraction to wetlands. The waterfowl-lead shot exposure pathway may be incomplete in deep water and dense emergent vegetation. The mourning dove may be at the greatest risk from lead shot contamination in uplands, due to their food habits and grit use. The shot remains available on the ground or in the sediment until it is turned under or settles deep enough to no longer pose a threat. This can be a period of extended time, since studies have shown no significant difference in the settling rates between large (#2) and small (#6) shot. One study in Utah showed that 75% of the #4 shot deposited were still in the top inch of the marsh sediment after 1 year (Low and Studinski 1967).

### Lead

#### Birds

The gastric motility of adult male and female red-tailed hawks (*Buteo jamaicensis*) fed 0.82 and 1.64 mg/kg BW/day (mg/kgBW/day concentration reported by authors) for 3 weeks was evaluated through the use of surgically implanted transducers. Neither concentration had any effect on gastric contractions or egestion of undigested material pellets (Lawler et al. 1991). Adult male and female red-tailed hawks were administered lead acetate by gavage at a concentration of 0.82 mg Pb/kg BW/day for 3 weeks (Redig et al. 1991). Compared to control birds, there was an 83 percent decrease in delta-aminolevulinic acid dehydratase activity and a 74 percent increase in the levels of free porphyrins circulating in the blood of experimental birds. Immune function (as measured by antibody titers to foreign red blood cells or mitogenic stimulation of T-lymphocytes) was not significantly affected at this exposure level.

Beyer et al. (1988) fed red-winged blackbirds, brown-headed cowbirds, common grackles, northern bobwhites and eastern screech owls diets containing lead acetate. The dietary concentration was increased by 60 percent weekly until half of the birds in each treatment group died. Because the exposure concentrations changed throughout the experiment, this study was not used to derive TRVs for this risk assessment.

One-day old American kestrel chicks were dosed orally with metallic lead at concentrations of 0, 25, 125 or 625 mg/kgBW/day for 10 days (Hoffman et al. 1985a and 1985b). Forty percent of the birds in the highest dose group died after 6 days of exposure. Growth rates of birds which received lead at concentrations of 125 or 625 mg/kgBW/day were significantly lower than the growth rates of control birds. The effect of lead on survival of



American kestrels was evaluated by feeding the birds either a control diet, or a diet containing mallard ducks which had died of lead poisoning (mean lead concentration was 29.3 mg/kg) for 60 days (Stendell 1980). No kestrels died or exhibited visible signs of lead poisoning during the 60-day exposure period. An ingestion rate of 0.0307 kg/day (Barrett and Mackey 1975) and a body weight of 0.111 kg (Dunning 1993) were used to convert the exposure concentration to units of mg/kgBW/day. A NOAEL of 8.1 mg/kgBW/day was calculated based on the results of this experiment.

Ringed turtle doves received 0 or 100  $\mu\text{g}/\text{ml}$  lead in their drinking water from two weeks prior to breeding throughout a breeding cycle (Kendall and Scanlon 1981). Exposure to lead did not increase the time required to produce eggs, and no adverse effects on egg production or fertility were observed. Bone lead concentrations in adult birds and bone and liver lead concentrations in juveniles were higher than in control birds or progeny of control birds. A water ingestion rate of 0.017 L/day (calculated using an allometric equation from Calder and Braun 1983) and a body weight of 0.16 kg (Schwarzbach et al. 1991) were used to convert the exposure concentration to units of mg/kgBW/day. A NOAEL of 0.01 mg/kgBW/day was calculated based on the results of this experiment.

Bobwhite quail were fed diets supplemented with lead (as lead acetate) at concentrations of 0, 500, 1000, 1500, 2000 and 3000 mg/kg for 6 weeks (Damron and Wilson 1975). Weight gain and food consumption were significantly decreased in birds receiving the two highest exposure concentrations. Mortality of birds receiving 3000 mg/kg lead was 46.7 percent, much greater than any other exposure group; however it was not statistically significant due to large variability among replicate pens. In another experiment, male bobwhite were fed diets containing 0, 500, 1000 or 1500 mg/kg lead (as lead acetate) for 8 weeks. Mortality, food consumption, sperm concentration and sperm viability were measured; no effects were observed at any exposure concentration. A food ingestion rate of 0.0143 kg/day and adult body weight of 0.169 kg were used to convert the exposure concentrations to units of mg/kgBW/day; 2000 mg/kg was selected as the NOAEL level. A NOAEL of 127 (exposure concentration of 1500 mg/kg, endpoint measured sperm concentration and viability) and an estimated LOAEL of 1270 mg/kgBW/day were calculated based on the results of this experiment.

Day-old Canada geese were fed diets supplemented with lead-contaminated sediment at lead concentrations of 1.9 (control diet), 414, 828 and 1656  $\mu\text{g}/\text{g}$  lead for 6 weeks (Hoffman et al. 2000). Mortality was observed only in the highest exposure group (22 percent), but it was not significantly different from the control group. Hematocrit, hemoglobin, and ALAD activity were significantly lower and protoporphyrin levels were higher in the two highest exposure groups. Renal tubular degeneration was observed in one gosling from the 1656  $\mu\text{g}/\text{g}$  group, but histopathologic lesions most commonly associated with lead poisoning in waterfowl were not observed in other geese. Growth was decreased in goslings from the highest exposure group. Because none of the effects measured in this experiment are considered ecologically relevant, results of this experiment were not used to derive TRVs for exposure of birds to lead.

Day-old mallard ducklings were fed diets supplemented with lead-contaminated sediment at lead concentrations of 1.9 (control diet), 414 and 828  $\mu\text{g}/\text{g}$  lead for 6 weeks (Hoffman et al. 2000b). A clean sediment-supplemented control (24 percent sediment) and a positive control diet containing lead acetate at a concentration equivalent to the 828  $\mu\text{g}/\text{g}$  lead-contaminated sediment diet were included in the experimental design. Mortality was observed only in the lead acetate group (7 percent), but was not significantly different from the control group. Hematocrit and hemoglobin were significantly lower in ducklings, which received lead acetate. Blood ALAD activity levels were significantly lower and protoporphyrin levels were higher in both groups, which received lead-contaminated sediment and the ducklings which received lead acetate. Acid-fast renal tubular inclusion bodies and nephrosis are abnormalities associated with lead poisoning; inclusion bodies were observed in 50 percent and tubular nephrosis was observed in 75 percent of ducklings fed lead acetate. Renal inclusion bodies were observed in 2 of 9 ducklings from the 414  $\mu\text{g}/\text{g}$  group, and in 4 of 9 ducklings from the 828  $\mu\text{g}/\text{g}$  group. Growth was affected only in ducklings fed lead acetate. Because none of the effects measured in this experiment are considered ecologically relevant, results of this experiment were not used to derive TRVs for exposure of birds to lead.

Heinz et al (1999) studied the bioavailability and toxicity of lead-contaminated sediment to adult mallards. In the first experiment, ducks were fed a pelleted commercial duck diet containing 0, 3, 6, 12 or 24 percent lead-contaminated sediment (103, 207, 414 and 828  $\mu\text{g}/\text{g}$  lead, respectively) for 5 weeks. Ducks fed the 24 percent lead-contaminated sediment exhibited atrophy of the breast muscles, green staining of the feathers around the vent,

viscous bile, green staining of the gizzard lining, and renal tubular intranuclear inclusion bodies; 1 of 10 birds died. In the second experiment, the dietary concentration of the lead-contaminated sediment was increased to 48 percent, but only about 20 percent was actually ingested due to food washing by the birds. Duration of this experiment was also 5 weeks. Protophyrin levels were elevated, and all of the lead-exposed birds had renal tubular intranuclear inclusion bodies. A third experiment was conducted to determine if the effects of lead were greater when birds were fed a nutritionally deficient diet. Ducks were fed a control diet, a commercial duck mash with 24 percent lead-contaminated sediment, of a ground corn diet with 24 percent lead-contaminated sediment for 15 weeks. Food washing was again observed; actual ingestion rates were 17 and 14 percent for the lead-contaminated duck mash and ground corn diets, respectively. Mortality occurred in 4 of 5 birds fed the lead-contaminated ground corn diet. At necropsy, all birds fed the lead-contaminated ground corn diet were emaciated, had renal tubular intranuclear inclusion bodies, and blackish-green bile. Based on the clinical signs of lead poisoning observed in the first experiment, an exposure concentration of 828  $\mu\text{g/g}$  lead was selected as the LOAEL from this experiment. An ingestion rate of 0.139 kg/day and body weight of 1.25 kg (Piccirillo and Quesenberry 1980) were used to convert the exposure concentrations to units of mg/kgBW/day. A LOAEL of 92 mg/kgBW/day and a NOAEL of 46 mg/kgBW/day were calculated based on the results of this experiment.

Day-old Japanese quail were fed diets containing lead (as lead acetate) at concentrations of 0, 1, 10, 100, 500 or 1000 mg/kg for 5 weeks (Morgan et al. 1975). Body weight, packed cell volume, and hemoglobin were significantly reduced in birds that received 1000 mg/kg lead. At five weeks of age, testes size was also significantly reduced in the highest exposure group. Mean body weights of the 500 and 1000 mg/kg exposure groups at three weeks were 65 and 55 g. Ingestion rates were calculated as a percent of the adult ingestion rate of 18 g/day (body weight of 0.12 kg; <[www.feathersite.com/Poultry/Stuff/FeatherFancier/FeathFancQuail.html](http://www.feathersite.com/Poultry/Stuff/FeatherFancier/FeathFancQuail.html)>), resulting in ingestion rates of 9.8 and 8.3 g/day, respectively. A LOAEL of 151 mg/kgBW/day and a NOAEL of 75.4 mg/kgBW/day were calculated based on the results of this experiment.

Nine raptors (5 red-tailed hawks, 3 rough-legged hawks and 1 golden eagle) were administered 3 mg/kgBW lead daily in the form of a lead acetate trihydrate solution by mouth for 30 weeks. Control birds (6 red-tailed hawks, 1 Swainsons hawk) were dosed with a sodium acetate solution by mouth. Clinical signs of lead toxicosis (anorexia, green bile-stained feces and anemia) were observed in 8 of the 9 experimental birds. Three birds died 3 to 4 weeks following the onset of clinical symptoms. This study was not used to derive the TRVs for this risk assessment because dosing was via solution rather than dietary, and because different species were included within the experimental group.

Edens et al. (1976) exposed Japanese quail to four dietary concentrations of lead acetate (1,10, 100 and 1000 mg/kg) for a period of 12 weeks. Percent hatch of settable eggs was significantly decreased in hens exposed to 100 mg/kg lead. Dietary lead at a concentration of 1000 mg/kg almost completely suppressed egg production. The results from this experiment will be used to develop the NOAEL and LOAEL values because of the ecological significance of the endpoints and the method and duration of exposure. An ingestion rate of 18 g/day and adult body weight of 0.12 kg (<[www.feathersite.com/Poultry/Stuff/FeatherFancier/FeathFancQuail.html](http://www.feathersite.com/Poultry/Stuff/FeatherFancier/FeathFancQuail.html)>) were used to convert the exposure concentration to units of mg/kgBW/day. A LOAEL of 15 mg/kg BW/day (100 mg/kg) and a NOAEL of 1.5 mg/kg BW/day will be used to evaluate the risk posed by Pb to avian receptors.

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## **Mammals**

Mason and MacDonald (1986) evaluated the effect of Pb and Cd on otter (*Lutra lutra*). Daily Pb intake was estimated on the basis of measured fecal Pb levels, the known ingestion rate for otter, and gastrointestinal Pb absorption rates for mammals. Estimated Pb intake correlated well with levels measured in major fish prey species. No apparent impact on population levels was found when Pb intake was less than 0.15 mg/kg BW/day whereas otter populations were reduced in sites where the estimated Pb intake exceeded 2 mg/kg BW/day.

Adult pregnant mice (C57Bl strain) were fed a diet containing Pb concentrations of 0, 0.125, 0.25, 0.5, and 1 percent for 48 hours following observation of the presence of a vaginal plug (Jacquet et al. 1976). Dietary Pb concentrations of 0.125 percent, 0.25 percent, and 0.5 percent resulted in an increase in the number of embryos in the 4-cell stage versus the 8-cell stage. At a dietary exposure level of 1 percent, an increase in the number of undivided embryos was observed. In normal mouse embryo development, after 48 hours the embryo is in the 8-cell stage and is placed near the end of the oviduct ready to be discharged to the uterus. Effects of delayed cleavage on embryo loss prior to implantation is not known. An ingestion rate of 0.0058 kg/day and adult body weight of 0.033 kg (U.S. EPA 1988) were used to convert the exposure concentration to units of mg/kgBW/day. A LOAEL of 220 mg/kg BW/day, and an estimated NOAEL of 80 mg/kg BW/day were calculated based on the results of this experiment.

Pregnant female mice were given lead acetate in their drinking water at concentrations of 0, 500, 750 and 1000 mg/L starting on gestation day 12 and continuing to 4 weeks postpartum (Waalkes et al. 1995). Offspring were weaned and received lead in their drinking water after weaning for 112 weeks. Renal lesions (atypical tubular hyperplasia or tumors) occurred rarely in control male mice (4 percent) and increased in dose related fashion for lead exposed male offspring: 500 ppm, 16 percent; 750 ppm, 24 percent; and 1000 ppm, 48 percent. The number of lesions in the 1000 mg/L group was significantly higher than for the control group. Lead-treated females also developed renal lesions, but at much lower rates. An ingestion rate of 0.0058 kg/day and adult body weight of 0.033 kg (U.S. EPA 1988) were used to convert the exposure concentration to units of mg/kgBW/day. A LOAEL of 176 mg/kg BW/day, and a NOAEL of 132 mg/kg BW/day were calculated based on results of this study.

Azar et al. (1973) administered Pb to rats at six dietary levels (1, 10, 50, 100, 1000 and 2000) for three generations and measured changes in reproduction and growth. No effects on number of pregnancies, number of pups born alive, fertility index, viability index or lactation index were observed at any exposure levels. An exposure concentration of 1000 mg/kg resulted in reduced offspring weight and kidney damage in the young. An ingestion rate of 0.027 kg/day and adult body weight of 0.35 kg (U.S. EPA 1988) were used to convert the exposure concentration to units of mg/kgBW/day. A LOAEL of 77 mg/kg BW/day, and a NOAEL of 7.7 mg/kg BW/day will be used to evaluate the risk posed by Pb to mammalian receptors

## References

Azar, A., H.J. Trochimowicz, and M.E. Maxwell. 1973. Review of Lead Studies in Animals Carried Out at Haskell Laboratory: Two-Year Feeding Study and Response to Hemorrhage Study. In: Environmental Health Aspects of Lead: International Symposium (eds.). D. Barth et al. Commission of European Communities. p. 199-210.

Jacquet, P., A. Leonard and G. B. Gerber. 1976. "Action of Lead on Early Divisions of the Mouse Embryo." *Toxicology*, 6:129-132.

Mason, C.F. and S.M. MacDonald. 1986. "Levels of Cadmium, Mercury and Lead in Otter and Mink Feces from the United Kingdom." *Sci. Total Environ.*, 53:139-146.

U.S. EPA. 1988. Recommendations for and documentation of biological values for use in risk assessment. EPA/600/6-87/008.

Waalkes, M.P., B.A. Diwan, J.M. Ward, D.E. Devor and R.A. Goyer. 1995. Renal tubular tumors and atypical hyperplasias in B6C3F1 mice exposed to lead acetate during gestation and lactation occur with minimal chronic nephropathy. *Cancer Research*. 55:5256-5271.

Appendix B  
X-ray Fluorescence Final Report  
Range 17 – Patuxent Research Refuge  
Laurel, MD  
March 2004



DATE: 13 August 2003  
TO: Jennifer Badner, REAC Task Leader  
THROUGH: Jay Patel, REAC Inorganic Group Leader  
FROM: Dennis Kalnicky, REAC Senior Chemist  
SUBJECT: FPXRF ANALYSES, RANGE 17 SITE, LAUREL, MD  
WORK ASSIGNMENT #R1A00318 - FPXRF ACTIVITIES REPORT

*J. Patel*  
*Dennis Kalnicky*

#### BACKGROUND

A Spectrace 9000 Field-Portable X-ray Fluorescence (FPXRF) analyzer, maintained and operated by Response Engineering and Analytical Contract (REAC) personnel, was used to support United States Environmental Protection Agency/Environmental Response Team Center (U.S. EPA/ERTC) activities at the Range 17 site. REAC personnel analyzed site soil samples for primary target element: lead (Pb), and secondary target elements: copper (Cu), arsenic (As), and antimony (Sb).

#### OBSERVATIONS AND ACTIVITIES

##### Spectrace 9000 FPXRF Analyses

REAC personnel were at the site from 27 to 29 July 2003, to determine the extent of target element contamination in site soil samples utilizing a Spectrace 9000 FPXRF analyzer (S/N Q-114). A total of 67 samples and 7 duplicates were analyzed on site. The Spectrace 9000 FPXRF measurement times (instrument live-time) were 60 seconds for each source: cadmium-109 (Cd-109), iron-55 (Fe-55), and americium-241 (Am-241).

Sample preparation, analysis, and quality assurance/quality control (QA/QC) procedures used in this study conform to those described in the U.S. EPA/ERTC REAC Standard Operating Procedure (SOP) #1713, *Spectrace 9000 Field Portable X-ray Fluorescence Operating Procedure*.

Preliminary results for target elements were reported on 30 July 03.

---

cc: Central File-WA # R1A00318  
D. Charters, U.S. EPA/ERTC Work Assignment Manager  
Rajeshmal Singhvi, U.S. EPA/ERTC

### Sample Preparation

Soil samples were received in labeled plastic bags. Each sample was mixed with a disposable plastic spoon. Stones and debris were removed prior to placing 10-20 grams of the sample into a labeled aluminum weighing dish. Samples were dried in an oven for 1-2 hours as needed. After drying, the sample was passed through a 10-mesh stainless steel sieve to remove rocks and large organic matter. The sample was then placed in a labeled 31-millimeter (mm) polyethylene X-ray sample cup and sealed with 0.2-mil (5 micrometer) thick polypropylene X-ray window film. Duplicates were prepared for every 10 samples, and the suffix "DUP" was added to the sample ID for the duplicate sample. Prior to XRF analysis, each sample cup was tapped against the tabletop to pack the sample evenly against the film window. The sample cup was placed directly on the probe aperture window of the Spectrace 9000 FPXRF analyzer, the safety shield was closed, and analysis was initiated with the measurement times previously noted.

### FPXRF Analysis Results

XRF analysis results for each sample were saved in the Spectrace 9000 internal data logger memory. The data were downloaded and archived on computer disks on a daily basis. Target element results for each analyzed sample and standard were logged into the Spectrace 9000 field logbook (REACII-L-00175). Target element results were qualified using the field method detection and quantitation limits discussed in this report.

### QA/QC Procedures

The reliability of the Spectrace 9000 FPXRF unit and application model was evaluated daily during the site visit. The energy calibration check and detector resolution check were performed at the beginning of each day to ensure that proper instrument calibration was maintained and that the detector resolution was adequate for producing reliable X-ray intensity measurements. The Spectrace 9000 soil application model was verified at the beginning of each day for the target elements. This was accomplished by analyzing a blank sample and National Institute of Standards and Technology (NIST) Standard Reference Materials (SRMs) #2709, #2710, and #2711. Energy calibration checks, detector resolution checks, and application verification results were recorded in the Spectrace 9000 field logbook (REACII-L-00175).

### Method Detection and Quantitation Limits

A low concentration standard, NIST SRM #2709, was analyzed at the beginning of each day and periodically during sample analysis to establish statistically derived method detection and quantitation limits for the target elements. The standard deviation [STD (n-1)] for these analyses was used to calculate the Spectrace 9000 method detection limit (MDL) and method quantitation limit (MQL) for each target element. The MDL was calculated as three times the standard deviation ( $MDL = 3 \times STD$ ) and the MQL was defined as ten times the standard deviation ( $MQL = 10 \times STD$ ) for repeat measurements.

Lead can interfere with the FPXRF analysis of As when the Pb:As ratio is 5:1 or greater. Therefore, the final As MDL was the larger of the statistical value or 1/10 the Pb concentration. The final MDL for Sb was set higher than the statistical value due to potential background effects in the Am-241 XRF spectrum.

Spectrace 9000 results were qualified by a "U" for values less than the MDL (not detected).

### Measurement Precision

Spectrace 9000 FPXRF analysis precision for Pb and As was determined using a synthetic standard, R33. The spiked values in R33 were: Pb=52 and As=273 milligrams per kilogram (mg/kg). Precision for Cu was determined using SRM #2710. Precision for Sb could not be determined because the concentration was too low in the reference standards. The coefficient of variation (COV) values for Pb, As, and Cu were within the specification of 20 percent (U.S. EPA/ERT 1991).

### FPXRF Confirmation Samples

In order to obtain Quality Assurance level 2 (QA2) data, a minimum of 10 percent of the samples must be confirmed by a laboratory method such as Inductively-Coupled Plasma (ICP) emission spectroscopy or Atomic Absorption (AA) analysis. A regression analysis between the Spectrace 9000 data (independent) and the confirmatory data (dependent) must yield a coefficient of determination ( $r^2$ ) greater than 0.7 (U.S. EPA/ERT 1991). The model obtained by the regression may be used to validate or adjust the Spectrace 9000 data.

Approximately 15 percent of the soil samples (10 samples) analyzed by FPXRF methods were submitted for confirmatory laboratory analysis. To minimize potential sample homogeneity problems, the original XRF sample cups were submitted for confirmatory analysis.

### Results

Appendix A contains MDL qualified FPXRF results for the target elements. Appendix B contains MDL and QA/QC data. Preliminary FPXRF field reports are in Appendix C. Appendix D contains FPXRF and laboratory data for confirmation samples. Photocopies of field logbook pages and disks with field FPXRF data are in the REAC Central File.

### FPXRF Confirmation Sample Results

FPXRF and laboratory results less than the MDL (U) were set equal to zero for regression analysis purposes. Comparisons are based on final FPXRF data and preliminary laboratory data. No QA/QC evaluation was performed for the preliminary laboratory data, and it should be used with caution (Appendix D). Regression analysis results obtained for Pb are summarized below:

Element	Number of Observations	$r^2$	Slope	Intercept	Standard Error of Y Estimate
Pb (all data)	10	0.9993	0.818	-33	161

Regression analysis results using all confirmation data indicated that QA2 data quality objectives were met ( $r^2 > 0.70$ ) for FPXRF analysis of Pb.

Regression analysis could not be performed for Cu, As, and Sb because most results were less than the XRF MDL. FPXRF and laboratory data were compared based on XRF MDLs and MQLs. The results of these comparisons are summarized below:

Element	Total number of Confirmation Samples	FPXRF Results	Laboratory Results	FPXRF Confirmed by Laboratory
Cu	10	9 samples < XRF MDL 1 sample = 110	< XRF MDL lab = 51	yes no
As	10	10 samples < XRF MDL	< XRF MDL	yes
Sb	10	9 samples < XRF MDL 1 sample = 160	< XRF MDL lab = 190	yes yes

These comparisons support QA2 data objectives for FPXRF analysis of Cu, As, and Sb.



## REFERENCES

U.S. EPA/ERT. 1991. Quality Assurance Technical Information Bulletin, "Field-Portable X-Ray Fluorescence", Volume 1, Number 4.

**MDL Qualified FPXRF Analysis Results  
FPXRF Activities Report  
Range 17 Site  
August 2003**

WA #R1A00318 Range 17 site  
 Final FPXRF data; MDL Qualified; 2 Significant Figures  
 Spectrace 9000; S/N Q-114  
 Cd109-60; Fe55-60; Am241-60 sec  
 Soil

XRF ID	LOCATION	DATE ANALYZED	Statistical MDL				
			As (mg/kg)	As MDL	Pb (mg/kg)	Cu (mg/kg)	Sb (mg/kg)
0-0-0	000	28-Jul-2003	U	33	150	U	U
0-0-0DUP	000	28-Jul-2003	U	33	150	U	U
0-50D	0-050D	28-Jul-2003	U	33	110	U	U
0-100D	0-100D	28-Jul-2003	U	33	230	U	U
0-150D	0-150D	28-Jul-2003	U	2200	22000	U	160
0-200D	0-200D	28-Jul-2003	U	200	2000	110	U
0-250D	0-250D	28-Jul-2003	U	33	51	U	U
0-300D	0-300D	28-Jul-2003	U	33	U	U	U
50L0D	050L000D	28-Jul-2003	U	33	43	U	U
50L50D	050L050D	28-Jul-2003	U	33	120	U	U
50L100D	050L100D	28-Jul-2003	U	180	1800	U	U
50L150D	050L150D	28-Jul-2003	U	1800	18000	U	U
50L150D-DUP	050L150D	28-Jul-2003	U	1700	17000	U	U
50L200D	050L200D	29-Jul-2003	U	320	3200	110	U
50L250D	050L250D	28-Jul-2003	U	33	57	U	U
50L300D	050L300D	29-Jul-2003	U	33	51	U	U
50L300D-DUP	050L300D	29-Jul-2003	U	33	U	U	U
50R0D	050R000D	28-Jul-2003	U	33	48	U	U
50R50D	050R050D	28-Jul-2003	U	33	42	U	U
50R50D-DUP	050R050D	28-Jul-2003	U	33	100	U	U
50R100D	050R100D	28-Jul-2003	U	430	4300	U	U
50R150D	050R150D	28-Jul-2003	U	420	4200	U	U
50R200D	050R200D	28-Jul-2003	U	33	280	U	U
50R250D	050R250D	28-Jul-2003	U	33	39	U	U
50R300D	050R300D	28-Jul-2003	U	33	40	U	U
100L0D	100L000D	28-Jul-2003	U	33	100	U	U
100L0D-DUP	100L000D	28-Jul-2003	33	33	110	U	U
100L50D	100L050D	29-Jul-2003	U	720	7200	U	U
100L100D	100L100D	29-Jul-2003	U	220	2200	U	U
100L150D	100L150D	29-Jul-2003	U	420	4200	U	U
100L200D	100L200D	28-Jul-2003	U	33	140	100	U
100L250D	100L250D	29-Jul-2003	U	33	75	95	U
100R0D	100R000D	28-Jul-2003	U	33	120	U	U
100R50D	100R050D	28-Jul-2003	U	590	5900	U	U
100R100D	100R100D	28-Jul-2003	U	210	2100	U	U
100R150D	100R150D	28-Jul-2003	U	100	1000	U	U
100R200D	100R200D	28-Jul-2003	U	37	370	U	U
100R250D	100R250D	29-Jul-2003	U	33	81	120	U
150L0D	150L000D	28-Jul-2003	U	33	170	U	U
150L50D	150L050D	28-Jul-2003	U	33	91	U	U
150L100D	150L100D	29-Jul-2003	U	33	97	U	U
150L150D	150L150D	28-Jul-2003	40	33	85	88	U
150L200D	150L200D	29-Jul-2003	U	33	58	U	U
150L250D	150L250D	28-Jul-2003	U	33	64	U	U
150R0D	150R000D	28-Jul-2003	U	33	U	U	U
150R50D	150R050D	28-Jul-2003	U	70	700	U	U
150R100D	150R100D	28-Jul-2003	U	44	440	U	U
150R150D	150R150D	28-Jul-2003	U	33	89	U	U
150R200D	150R200D	28-Jul-2003	40	33	56	U	U
150R250D	150R250D	28-Jul-2003	U	33	54	U	U
200L0D	200L000D	28-Jul-2003	U	33	U	U	U
200L0D-DUP	200L000D	28-Jul-2003	U	33	U	U	U
200L50D	200L050D	28-Jul-2003	U	33	43	U	U
200L100D	200L100D	28-Jul-2003	U	33	U	U	U
200L150D	200L150D	28-Jul-2003	U	33	59	U	U
200L200D	200L200D	29-Jul-2003	U	33	79	U	U

**WA #R1A00318 Range 17 site**  
**Final FPXRF data; MDL Qualified; 2 Significant Figures**  
**Spectrace 9000; S/N Q-114**  
**Cd109-60; Fe55-60; Am241-60 sec**  
**Soil**

XRF ID	LOCATION	DATE ANALYZED	Statistical MDL				
			As (mg/kg)	As MDL	Pb (mg/kg)	Cu (mg/kg)	Sb (mg/kg)
200R0D	200R0000D	29-Jul-2003	U	33	U	U	U
200R50D	200R050D	29-Jul-2003	U	33	U	U	U
200R50D-DUP	200R050D	29-Jul-2003	U	33	57	U	U
200R100D	200R100D	29-Jul-2003	U	33	190	U	U
200R150D	200R150D	29-Jul-2003	U	33	U	110	U
200R200D	200R200D	29-Jul-2003	U	33	84	U	U
250L0D	250L000D	29-Jul-2003	U	33	U	U	U
250L50D	250L050D	28-Jul-2003	U	33	U	U	U
250L100D	250L100D	28-Jul-2003	U	33	96	U	U
250L150D	250L150D	28-Jul-2003	U	33	40	U	U
250R0D	250R000D	29-Jul-2003	U	33	55	U	U
250R50D	250R050D	29-Jul-2003	U	33	85	U	U
250R100D	250R100D	29-Jul-2003	37	33	81	U	U
250R150D	250R150D	29-Jul-2003	U	33	59	U	U
300L0D	300L000D	28-Jul-2003	U	33	U	U	U
300L50D	300L050D	28-Jul-2003	U	33	U	U	U
300R0D	300R000D	29-Jul-2003	42	33	U	U	U
300R50D	300R050D	29-Jul-2003	U	33	U	U	U

MDL - Method Detection Limit; U - Not Detected (less than the MDL); DUP - duplicate sample

Pb can interfere with the As analysis at Pb:As ratios of 5:1 or greater.

The As detection limit is the statistical value or 1/10 the Pb concentration, whichever is greater.

The Sb detection limit (100) is higher than the statistical value (36) due to potential background effects in the Am-241 XRF spectrum.

**MDL and QA/QC Data  
FPXRF Activities Report  
Range 17 Site  
August 2003**

**WA #R1A00318 Range 17 site**  
**MDL and QA/QC Data**  
**Spectrace 9000; S/N Q-114**  
**Cd109-60; Fe55-60; Am241-60 sec**  
**Soil**

ID	DATE ANALYZED	As (mg/kg)	Pb (mg/kg)	Cu (mg/kg)	Sb (mg/kg)
----	---------------	---------------	---------------	---------------	---------------

MDL SAMPLE

#2709	28-Jul-2003	34.39	0.81	56.52	-8.81
#2709	28-Jul-2003	39.74	-3.44	84.02	7.29
#2709	28-Jul-2003	21.67	16.52	90.25	-10.19
#2709	28-Jul-2003	20.99	27.03	81.38	23.9
#2709	28-Jul-2003	26.25	24.01	15.91	13.66
#2709	28-Jul-2003	9.23	27.87	19.49	8.36
#2709	28-Jul-2003	18.22	1.86	79.26	-0.59
#2709	28-Jul-2003	30.96	15.46	88.61	-4.43
#2709	29-Jul-2003	20.11	31.62	35.94	14.74
#2709	29-Jul-2003	32.79	13.48	47.84	-18.95
#2709	29-Jul-2003	28.06	-3.64	77.99	-9.25
#2709	29-Jul-2003	-2.93	29.57	84.65	1.14

AVG	23	15	63	1
STDS	11	13	27	12
MDL	33	39	81	36
MQL	110	130	270	120

Number of Obs	12	12	12	12
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AVG - Average  
 STDS - Standard Deviation (n-1 method)  
 MDL - Method Detection Limit  
 MQL - Method Quantitation Limit

**WA #R1A00318 Range 17 site**  
**MDL and QA/QC Data**  
**Spectrace 9000; S/N Q-114**  
**Cd109-60; Fe55-60; Am241-60 sec**  
**Soil**

ID	DATE ANALYZED	As (mg/kg)	Pb (mg/kg)	Cu (mg/kg)	Sb (mg/kg)
----	---------------	---------------	---------------	---------------	---------------

PRECISION (COV) SAMPLE

#R33	28-Jul-2003	207.91	353.75	8.95	-2.83
#R33	28-Jul-2003	194.22	343.93	-23.58	-40.32
#R33	28-Jul-2003	225.68	271.63	0.11	-23.04
#R33	28-Jul-2003	220.95	302.89	72.6	-24.39
#R33	28-Jul-2003	200.68	370.59	21.91	-24.54
#R33	28-Jul-2003	188.74	368.53	50.38	-28.08
#R33	29-Jul-2003	215.79	317.05	7.34	11.02
#R33	29-Jul-2003	204.48	313.62	-15.9	-42.72
#R33	29-Jul-2003	228.44	307.78	-14.02	-43.93
#R33	29-Jul-2003	222.08	309.03	17.84	-30.46

AVG	211	326	13	-25
STDS	14	32	30	17
COV(%)	6.5	9.8	NA	NA

Number of Obs	10	10	10	10
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Spiked Value (Synthetic Std.)	273	528	NA	NA
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AVG - Average  
 STDS - Standard Deviation (n-1 method)  
 COV(%) - Coefficient of Variation in percent

**WA #R1A00318 Range 17 site**  
**MDL and QA/QC Data**  
**Spectrace 9000; S/N Q-114**  
**Cd109-60; Fe55-60; Am241-60 sec**  
**Soil**

ID	DATE ANALYZED	As (mg/kg)	Pb (mg/kg)	Cu (mg/kg)	Sb (mg/kg)
----	---------------	---------------	---------------	---------------	---------------

QC SAMPLE

#2710	28-Jul-2003	713.83	5489.22	2705.49	-3.58
#2710	28-Jul-2003	702.18	5327.96	2909.66	27.68
#2710	28-Jul-2003	417.58	5418.76	2541.31	38.22
#2710	28-Jul-2003	529.51	5377.95	2594.92	47.37
#2710	28-Jul-2003	438.71	5501.79	2863.8	41.26
#2710	28-Jul-2003	385.39	5636.14	2650.37	5.09
#2710	29-Jul-2003	382.9	5548.23	2866.78	-2.06
#2710	29-Jul-2003	627.61	5744.02	2902.78	26.71
#2710	29-Jul-2003	583.62	5694.68	3062.08	36.64
#2710	29-Jul-2003	464.73	5561.71	2771.82	28.32

AVG	525	5530	2787	25
STDS	126	135	163	18
COV(%)	24.1	2.4	5.8	NA

Number of Obs	10	10	10	10
---------------	----	----	----	----

Certified value	626	5532	2950	38
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AVG - Average

STDS - Standard Deviation (n-1 method)

COV(%) - Coefficient of Variation in percent



**WA #R1A00318 Range 17 site**  
**MDL and QA/QC Data**  
**Spectrace 9000; S/N Q-114**  
**Cd109-60; Fe55-60; Am241-60 sec**  
**Soil**

ID	DATE ANALYZED	As (mg/kg)	Pb (mg/kg)	Cu (mg/kg)	Sb (mg/kg)
----	---------------	---------------	---------------	---------------	---------------

PRECISION (COV) SAMPLE

#2711	28-Jul-2003	154.83	1109.37	176.02	3.57
#2711	29-Jul-2003	55.42	1185.31	131.39	-0.04

AVG	105	1147	154	2
STDS	70	54	32	3
COV(%)	66.9	4.7	20.5	144.6
Number of Obs	2	2	2	2
Certified value	105	1162	114	19

AVG - Average  
 STDS - Standard Deviation (n-1 method)  
 COV(%) - Coefficient of Variation in percent

**WA #R1A00318 Range 17 site**  
**MDL and QA/QC Data**  
**Spectrace 9000; S/N Q-114**  
**Cd109-60; Fe55-60; Am241-60 sec**  
**Soil**

ID	DATE ANALYZED	As (mg/kg)	Pb (mg/kg)	Cu (mg/kg)	Sb (mg/kg)
=====					
ZERO CHECK SAMPLE: 60 SECONDS PER SOURCE					
#SAND	28-Jul-2003	-0.2	-2.59	2.11	-7.83
#SAND	29-Jul-2003	-9.66	9.96	14.58	-26.1
=====					
	AVG	-5	4	8	-17
	Number of Obs	2	2	2	2
	AVG - Average				

**Preliminary FPXRF Field Reports  
FPXRF Activities Report  
Range 17 Site  
August 2003**



WA #R1A00318 Range 17 site  
Preliminary FPXRF data; MDL Qualified; 2 Significant Figures  
Spectrace 9000; S/N Q-114  
Cd109-60; Fe55-60; Am241-60 sec  
Soil

\*\*\*\*\* NO QC EVALUATION HAS BEEN PERFORMED; DATA SHOULD BE USED WITH DISCRETION \*\*\*\*\*

XRF ID	LOCATION	DATE ANALYZED	MDL				
			As (mg/kg)	As MDL	Pb (mg/kg)	Cu (mg/kg)	Sb (mg/kg)
0-0-0	000	28-Jul-2003	U	75	150	U	U
0-0-0DUP	000	28-Jul-2003	U	75	150	U	U
0-50D	0-050D	28-Jul-2003	U	75	110	U	U
0-100D	0-100D	28-Jul-2003	U	75	230	U	U
0-150D	0-150D	28-Jul-2003	U	2200	22000	U	160
0-200D	0-200D	28-Jul-2003	U	200	2000	110	U
0-250D	0-250D	28-Jul-2003	U	75	51	U	U
0-300D	0-300D	28-Jul-2003	U	75	U	U	U
50L0D	050L000D	28-Jul-2003	U	75	U	U	U
50L50D	050L050D	28-Jul-2003	U	75	120	U	U
50L100D	050L100D	28-Jul-2003	U	180	1800	U	U
50L150D	050L150D	28-Jul-2003	U	1800	18000	U	U
50L150D-DUP	050L150D	28-Jul-2003	U	1700	17000	U	U
50L200D	050L200D	29-Jul-2003	U	320	3200	110	U
50L250D	050L250D	28-Jul-2003	U	75	57	U	U
50L300D	050L300D	29-Jul-2003	U	75	51	U	U
50L300D-DUP	050L300D	29-Jul-2003	U	75	U	U	U
50R0D	050R000D	28-Jul-2003	U	75	U	U	U
50R50D	050R050D	28-Jul-2003	U	75	U	U	U
50R50D-DUP	050R050D	28-Jul-2003	U	75	100	U	U
50R100D	050R100D	28-Jul-2003	U	430	4300	U	U
50R150D	050R150D	28-Jul-2003	U	420	4200	U	U
50R200D	050R200D	28-Jul-2003	U	75	280	U	U
50R250D	050R250D	28-Jul-2003	U	75	U	U	U
50R300D	050R300D	28-Jul-2003	U	75	U	U	U
100L0D	100L000D	28-Jul-2003	U	75	100	U	U
100L0D-DUP	100L000D	28-Jul-2003	U	75	110	U	U
100L50D	100L050D	29-Jul-2003	U	720	7200	U	U
100L100D	100L100D	29-Jul-2003	U	220	2200	U	U
100L150D	100L150D	29-Jul-2003	U	420	4200	U	U
100L200D	100L200D	28-Jul-2003	U	75	140	100	U
100L250D	100L250D	29-Jul-2003	U	75	75	U	U
100R0D	100R000D	28-Jul-2003	U	75	120	U	U
100R50D	100R050D	28-Jul-2003	U	590	5900	U	U
100R100D	100R100D	28-Jul-2003	U	210	2100	U	U
100R150D	100R150D	28-Jul-2003	U	100	1000	U	U
100R200D	100R200D	28-Jul-2003	U	75	370	U	U
100R250D	100R250D	29-Jul-2003	U	75	81	120	U
150L0D	150L000D	28-Jul-2003	U	75	170	U	U
150L50D	150L050D	28-Jul-2003	U	75	91	U	U
150L100D	150L100D	29-Jul-2003	U	75	97	U	U
150L150D	150L150D	28-Jul-2003	U	75	85	U	U
150L200D	150L200D	29-Jul-2003	U	75	58	U	U
150L250D	150L250D	28-Jul-2003	U	75	64	U	U
150R0D	150R000D	28-Jul-2003	U	75	U	U	U
150R50D	150R050D	28-Jul-2003	U	75	700	U	U
150R100D	150R100D	28-Jul-2003	U	75	440	U	U
150R150D	150R150D	28-Jul-2003	U	75	89	U	U
150R200D	150R200D	28-Jul-2003	U	75	56	U	U
150R250D	150R250D	28-Jul-2003	U	75	54	U	U
200L0D	200L000D	28-Jul-2003	U	75	U	U	U

WA #R1A00318 Range 17 site  
Preliminary FPXRF data; MDL Qualified; 2 Significant Figures  
Spectrace 9000; S/N Q-114  
Cd109-60; Fe55-60; Am241-60 sec  
Soil

\*\*\*\*\* NO QC EVALUATION HAS BEEN PERFORMED; DATA SHOULD BE USED WITH DISCRETION \*\*\*\*\*

XRF ID	LOCATION	DATE ANALYZED	MDL	75	50	100	100
				As (mg/kg)	Pb (mg/kg)	Cu (mg/kg)	Sb (mg/kg)
200L0D-DUP	200L000D	28-Jul-2003	U	75	U	U	U
200L50D	200L050D	28-Jul-2003	U	75	U	U	U
200L100D	200L100D	28-Jul-2003	U	75	U	U	U
200L150D	200L150D	28-Jul-2003	U	75	59	U	U
200L200D	200L200D	29-Jul-2003	U	75	79	U	U
200R0D	200R0000D	29-Jul-2003	U	75	U	U	U
200R50D	200R050D	29-Jul-2003	U	75	U	U	U
200R50D-DUP	200R050D	29-Jul-2003	U	75	57	U	U
200R100D	200R100D	29-Jul-2003	U	75	190	U	U
200R150D	200R150D	29-Jul-2003	U	75	U	110	U
200R200D	200R200D	29-Jul-2003	U	75	84	U	U
250L0D	250L000D	29-Jul-2003	U	75	U	U	U
250L50D	250L050D	28-Jul-2003	U	75	U	U	U
250L100D	250L100D	28-Jul-2003	U	75	96	U	U
250L150D	250L150D	28-Jul-2003	U	75	U	U	U
250R0D	250R000D	29-Jul-2003	U	75	55	U	U
250R50D	250R050D	29-Jul-2003	U	75	85	U	U
250R100D	250R100D	29-Jul-2003	U	75	81	U	U
250R150D	250R150D	29-Jul-2003	U	75	59	U	U
300L0D	300L000D	28-Jul-2003	U	75	U	U	U
300L50D	300L050D	28-Jul-2003	U	75	U	U	U
300R0D	300R000D	29-Jul-2003	U	75	U	U	U
300R50D	300R050D	29-Jul-2003	U	75	U	U	U

MDL - Method Detection Limit; U - Not Detected (less than the MDL); DUP - duplicate sample

Pb can interfere with the As analysis at Pb:As ratios of 5:1 or greater.

Therefore the As detection limit is the statistical value or 1/10 the Pb concentration, whichever is greater.

**FPXRF Confirmation Sample Data  
FPXRF Activities Report  
Range 17 Site  
August 2003**





Table 1.x (cont.) Results of the Analysis for Metals in Soil  
 WA # 0-318 Range 17  
 Results Based on Dry Weight  
 NO QC EVALUATION HAS BEEN PERFORMED

Client ID	Method	Blank		0-0-0		0-150D		50L200D		100L50D		100L100D	
Location	Lab			000		0-150D		050L200D		100L050D		100L100D	
% Solids	NA	100.00		100.00		100.00		100.00		100.00		100.00	
Parameter	Analysis Method	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg
Antimony	ICAP	U	1.0	U	1.0	190	1.0	5.0	1.0	23	1.0	2.3	1.0
Arsenic	ICAP	U	1.0	2.3	1.0	130	1.0	11	1.0	19	1.0	7.5	1.0
Copper	ICAP	U	0.50	17	0.50	22	0.50	51	0.50	8.9	0.50	7.4	0.50
Lead	ICAP	U	1.0	120	1.0	18000	1.0	2300	1.0	5800	1.0	2100	1.0

Table 1.x (cont.) Results of the Analysis for Metals in Soil  
 WA # 0-318 Range 17  
 Results Based on Dry Weight  
 NO QC EVALUATION HAS BEEN PERFORMED

Client ID	Method	100R150D		150R50D		150R100D		200R0D		250R50D	
Location	100R150D	100R150D		150R050D		150R100D		200R000D		250R050D	
% Solids	100.00	100.00		100.00		100.00		100.00		100.00	
Parameter	Analysis Method	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg
Antimony	ICAP	1.3	0.99	U	0.98	U	1.0	U	0.97	U	0.98
Arsenic	ICAP	5.9	0.99	1.9	0.98	2.7	1.0	2.8	0.97	2.6	0.98
Copper	ICAP	20	0.5	3.6	0.49	5.0	0.50	4.5	0.49	12	0.49
Lead	ICAP	720	0.99	510	0.98	300	1.0	19	0.97	54	0.98

**NO QC EVALUATION HAS BEEN PERFORMED.  
 DATA VALIDITY IS UNSUBSTANTIATED  
 AND THE DATA SHOULD BE USED  
 WITH DISCRETION.**

**CHAIN OF CUSTODY RECORD**

Project Name: Range 17  
 Project Number: RI000318  
 LM Contact: J. Badner Phone: 732-494-4004

No: 15364  
 Sheet 01 of 01 (Do not copy)  
 (for addnl. samples use new form)

8/4/03

**Sample Identification**

**Analyses Requested**

REAC#	Sample No	Sampling Location	Matrix	Date Collected	# of Bottles	Container/Preservative	Pb	As	Cu	Sb
3080	0-0-0	000	Soil	28-Jul-03	1	XRF amp/mar	✓	✓	✓	✓
3081	0-1500	0-1500		↓						
3082	50L200D	050L200D		29-Jul-03						
3083	100L50D	100L050D		↓						
3084	100L100D	100L100D		↓						
3085	100R150D	100R150D		28-Jul-03						
3086	150R50D	150R050D		↓						
3087	150R100D	150R100D		↓						
3088	200R0D	200R000D		29-Jul-03						
3089	250R50D	250R050D		↓						

**Matrix:**

- A- Air
- AT- Animal Tissue
- DL- Drum Liquids
- DS- Drum Solids
- GW- Groundwater
- O- Oil
- PR- Product
- PT- Plant Tissue
- PW- Potable Water
- S- Soil
- SD- Sediment
- SL- Sludge
- SW- Surface Water
- TX- TCLP Extract
- W- Water
- X- Other

**Special Instructions:**

**SAMPLES TRANSFERRED FROM**

**CHAIN OF CUSTODY #:**

Items/Reason	Relinquished by	Date	Received by	Date	Time	Items/Reason	Relinquished by	Date	Received by	Date	Time
10/analyses	D. Kaludny	8/4/03	J. Badner	8/4/03	10:45	611/104	J. Badner	8/4/03	Chaves	8/4/03	11:05

**WA #0-318 range 17 site**  
**Spectrace 9000; S/N Q-114**  
**Cd109-60; Fe55-60; Am241-60 sec**  
**Confirmation Samples**  
**Final FPXRF and Preliminary Laboratory Results; MDL Qualified; 2 Significant Figures**

STATISTICAL MDL	33	1	39	1	81	0.5	100	1
MQL	110		130		270		330	

ID	LOCATION	DATE	As (mg/kg)		Pb (mg/kg)		Cu (mg/kg)		Sb (mg/kg)		
			XRF	XRF MDL	Lab	XRF	Lab	XRF	Lab	XRF	Lab
0-0-0	000	28-Jul-2003	U	33	2.3	150	120	U	17	U	U
0-150D	0-150D	28-Jul-2003	U	2200	130	22000	18000	U	22	160	190
50L200D	050L200D	29-Jul-2003	U	320	11	3200	2300	110	51	U	5
100L50D	100L050D	29-Jul-2003	U	720	19	7200	5800	U	8.9	U	23
100L100D	100L100D	29-Jul-2003	U	220	7.5	2200	2100	U	7.4	U	2.3
100R150D	100R150D	28-Jul-2003	U	100	5.9	1000	720	U	20	U	1.3
150R50D	150R050D	28-Jul-2003	U	70	1.9	700	510	U	3.6	U	U
150R100D	150R100D	28-Jul-2003	U	44	2.7	440	300	U	5	U	U
200R0D	200R000D	29-Jul-2003	U	33	2.6	U	19	U	4.5	U	U
250R50D	250R050D	29-Jul-2003	U	33	2.6	85	54	U	12	U	U

MDL - Method Detection Limit  
 MQL - Method Quantitation Limit  
 U - Not Detected (less than the MDL)

**WA #0-318 range 17 site**  
**Spectrace 9000; S/N Q-114**  
**Cd109-60; Fe55-60; Am241-60 sec**  
**Confirmation Samples**  
**Final FPXRF and Preliminary Laboratory Results; MDL Qualified; 2 Significant Figures**

**REGRESSION ANALYSIS DATA**

ID	LOCATION	DATE	Pb (mg/kg)		
			XRF	Lab	
0-0-0	000	28-Jul-2003	150	120	
0-150D	0-150D	28-Jul-2003	22000	18000	
50L200D	050L200D	29-Jul-2003	3200	2300	
100L50D	100L050D	29-Jul-2003	7200	5800	
100L100D	100L100D	29-Jul-2003	2200	2100	
100R150D	100R150D	28-Jul-2003	1000	720	
150R50D	150R050D	28-Jul-2003	700	510	
150R100D	150R100D	28-Jul-2003	440	300	
200R0D	200R000D	29-Jul-2003	U	19	
250R50D	250R050D	29-Jul-2003	85	54	
			MDL	50	1
			MQL	170	

REGRESSION ANALYSIS: LAB (DEPENDENT) VS. XRF (INDEPENDENT)

Lead (Pb):

XRF ID	FPXRF*	Lab	Lead: all data		
			Pred	Res	StdRes
200R0D	0	19	-33	-52	-0.3
250R50D	85	54	36	-18	-0.1
0-0-0	150	120	90	-30	-0.2
150R100D	440	300	327	27	0.2
150R50D	700	510	540	30	0.2
100R150D	1000	720	785	65	0.4
100L100D	2200	2100	1767	-333	-2.1
50L200D	3200	2300	2585	285	1.8
100L50D	7200	5800	5858	58	0.4
0-150D	22000	18000	17969	-31	-0.2

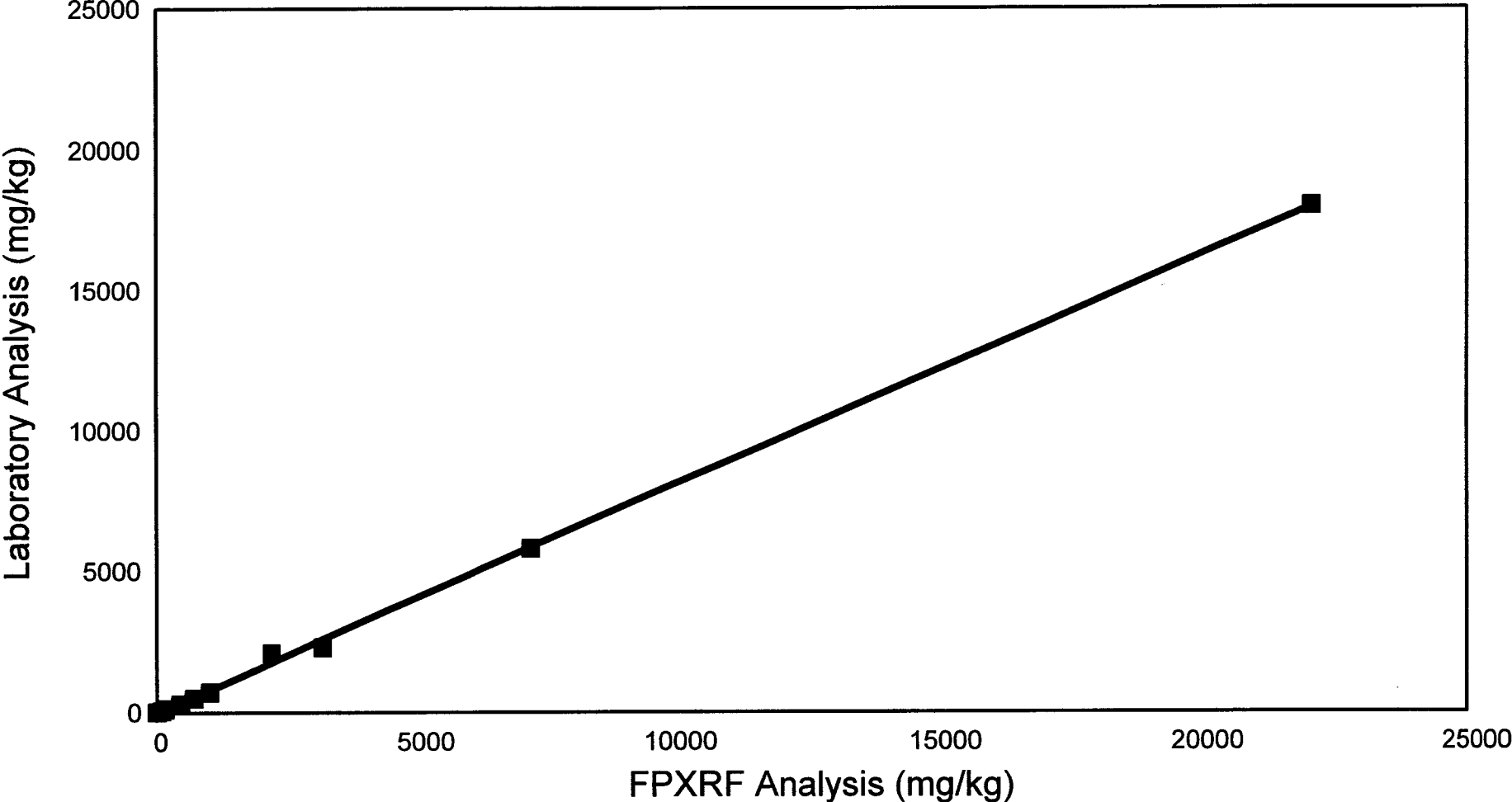
Pb: all data

Regression Output:

Constant	-33.234
Std Err of Y Est	160.641
R Squared	0.99926
No. of Observations	10
Degrees of Freedom	8
X Coefficient(s)	0.81826
Std Err of Coef.	0.00788
t-value	103.882

\* Non detects set to zero (0)

# Laboratory vs FPXRF: Lead



7-28-03 Q-114 Postcard checkout Range 17  
Unit on at 0826 AM

*Wh*

*Wh*

**SPECTRACE 9000 FPXRF DAILY INSTRUMENT CHECKOUT**

DATE 7-28-03

SPECTRACE SERIAL NO Q-114

SITE Range 17

WA# 0318

**ENERGY CALIBRATION CHECK (SAFETY SHIELD IN PLACE)**

*#ECHK*

Source: Cd 109 Range  
Pb La 10.541 KeV(10.50 - 10.58)  
Pb Lβ 12.631 KeV(12.57 - 12.65)  
Source Line 22.100 KeV(22.06 - 22.14)

Source: Fe55 Range  
S Kα 2.303 KeV(2.29 - 2.33)  
Source Line 5.890 KeV(5.87 - 5.91)

Source: Am 241 Range  
Pb La 10.547 KeV(10.49 - 10.59)  
Pb Lβ 12.610 KeV(12.56 - 12.66)  
Source Line 59.49 KeV(59.3 - 59.7)

**IRON Kα RESOLUTION/INTENSITY CHECK (Cd 109, IRON PURE)**

*#MESCK*

Iron at maximum peak height (MPH) = 2484 counts (MPH ≥ 1000 at 6.40 ± 0.02 KeV)  
1/2 MPH = 1242 counts

left (low energy) side, 1/2 MPH  
1767 counts at 6.262 KeV  
1506 counts at 6.246 KeV

6.535  
6.244  
.289

right (high energy) side, 1/2 MPH  
1978 counts at 6.519 KeV  
1719 counts at 6.535 KeV

Calculated FWHM = 0.287 KeV (≤ 0.300)

- Pass  Fail: Counts ≤ 1/2 MPH at 6.25 KeV
- Pass  Fail: Counts ≤ 1/2 MPH at 6.55 KeV

Cd 109 Intensity Check Criteria  
Fe 0.983650 (≥0.95 and ≤1.05)  
Mn 0.00157 (≤ ± 0.006)  
Co 0.00158 (≤ ± 0.006)

**BLANK SAMPLE CHECK**

*#SAND*

Check One:  Quartz  Teflon  Sand  Other (Specify)     

- Pass  Fail: All target elements Cr (z=24) and higher are within ± 3 std. deviations of zero
- Pass  Fail: All non-target elements Cr (z=24) and higher are within ± 5 std. deviations of zero

Comments IR = 60% 34 OK

NOTE: All acquisition times ≥ 60 seconds each source, All checks with standard Soil Application

*Wh*

Rev - 03/14/01

Initials Wh AK

Continued on Page

Read and Understood By

Dominic Kalin  
Signed

7-28-03  
Date

\_\_\_\_\_  
Signed

\_\_\_\_\_  
Date

#	ID	Time	As	Pb	Cu	Sb	Comment
04	#2709	0913	21	27	81	24	SRM
05	#2710	0917	702	5330	2910	28	"
06	#2711	0922	155	1109	176	4	"
07	#R33	0927	194	344	-24	-40	QC
08	#2709	0951	9	28	19	8	SRM
09	#2709	1005	31	15	89	4	"
10	φ-φ-φ	1031	5	150	31	-8	Preference
11	φ-φ-φ-DUP	1036	19	145	18	-19	Duplicate
12	φ-5φD	1040	7	108	φ	-21	Soil
13	φ-1φD	1046	-9	229	16	-9	Soil
14	φ-15φD	1054	1270	22140	-20	163	Soil
15	φ-20φD	1101	151	1983	107	-19	Soil
16	φ-25φD	1107	-9	51	79	-21	Soil
17	φ-30φD	1115	13	39	66	6	Soil
18	5φ4φD	—	9	43	78	-10	Soil
19	5φL5φD	1125	33	121	31	2	Soil
20	#2709	—	26	24	16	14	SRM
21	#2710	1134	439	5500	2860	41	"
22	#R33	1139	221	303	73	-24	QC
23	5φR5φD	1144	-12	42	18	-12	Soil
24	5φR5φD-DUP	1149	12	103	33	-12	Duplicate
25	5φR2φD	1154	28	283	31	-19	Soil
26	5φRφD	1159	22	48	-34	-20	Soil
27	5φR15φD	1204	205	4194	-9	4	Soil
28	100R100D	1209	147	2054	33	-11	Soil
29	50L100D	1214	85	1810	56	-3	Soil
30	100R200D	1219	16	372	19	-9	Soil
31	100RφD	1224	-32	118	47	-14	Soil
32	50R300D	1229	18	40	-27	-19	Soil
33	#2709	1234	18	2	79	-1	SRM
34	#2710	1238	385	5610	2650	5	SRM
35	#R33	—	189	369	50	-28	QC

FILES: ~~PH 17-0728A.BE~~ R170728A.BE ✓  
 R170728A.SPT ✓

Continued on Page

Read and Understood By

Daniel Kalinsky

7/28/03

Signed

Date

Signed

Date



7-28-03		Q114		60 sec / source			COMMENT
#	ID	Time	As	Pb	Cu	Sb	
01	200L OD	1305	13	6	-14	12	Soil
02	200L OD-DUP	1309	27	2	41	-13	Duplicate
03	100R50D	1314	107	5910	56	12	Soil
04	50L150D	1319	610	17630	-48	85	Soil
05	50L150D-DUP	1324	680	16840	20	85	Duplicate
06	50R250D	—	15	39	46	-14	Soil
07	100R150D	1333	96	1034	73	-9	Soil
08	50L250D	1338	-12	57	54	9	Soil
09	150L250D	1343	11	64	46	10	Soil
10	200L50D	1348	1	43	16	22	Soil
11	#2709	1352	22	17	90	-10	SRM (QC)
12	#2710	1357	714	5490	2710	-4	" "
13	#R33	1401	208	354	9	-3	QC
14	150L OD	1406	6	166	10	32	Soil
15	250L50D	1411	26	32	40	2	Soil
16	150R100D	1416	39	443	-41	-1	Soil
17	150R250D	1420	-9	54	-1	-11	Soil
18	200L100D	1430	15	26	61	-9	Soil
19	150R OD	1434	33	37	20	-5	Soil
20	100L200D	1439	20	136	100	-2	Soil
21	50R100D	1444	59	4252	27	56	Soil
22	200L150D	1449	11	59	17	-26	Soil
23	250L150D	1453	7	40	69	-15	Soil
24	#2709	1458	40	-3	84	7	SRM (QC)
25	#2710	1503	530	5380	2590	47	" "
26	#R33	1508	201	371	22	-25	QC
27	150R200D	1513	40	56	51	-11	Soil
28	250L100D	1518	-28	96	22	-1	Soil
29	150L50D	1522	3	91	61	-8	Soil
30	300L OD	1527	-1	2	70	8	Soil
31	150R50D	1532	61	704	2	-24	Soil
32	300L50D	1537	25	16	32	-14	Soil
33	150L150D	1542	40	85	88	-12	Soil

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Dennis Kalinsky  
Signed

7-28-03  
Date

Signed

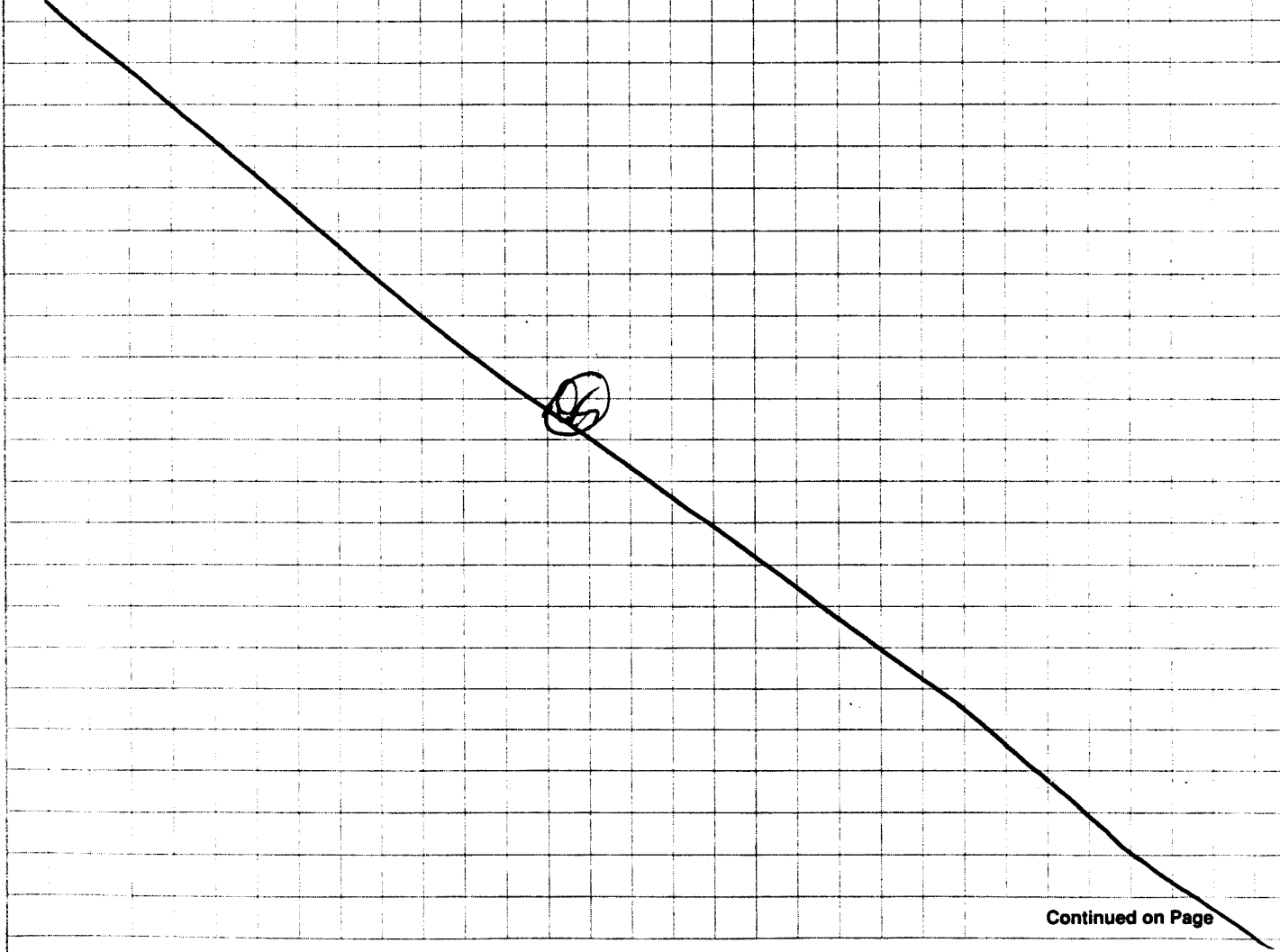
Date

7-28-03

#	ID	Time	As	Pb	Cu	Sb	COMMENT
34	150R1500	1547	5	89	1	-11	Soil
35	100L ØD	1550	30	102	9	-18	Soil
36	100L ØD-DWP	1556	33	112	71	-5	Soil
37	#2709	1601	34	1	57	-9	SRM (QC)
38	#2710	1606	418	5420	2540	38	" "
39	#R33	1611	226	272	Ø	-23	QC

FILES: ~~R170720A~~ R170720B, RES ✓  
R170720B.SAT ✓

*AKA*



Continued on Page

Read and Understood By

*Dennis Kaluichy* 7-28-03  
Signed Date

Signed

Date

7-29-03 Q114 Checkout - unit on at 0736

OK

SPECTRACE 9000 FPXRF DAILY INSTRUMENT CHECKOUT

OK

DATE 7-29-03

SPECTRACE SERIAL NO. Q-114

SITE Rangolf

WA# 0318

ENERGY CALIBRATION CHECK (SAFETY SHIELD IN PLACE)

#EORACK

Source: Cd 109	Range
Pb L $\alpha$ <u>10.547</u>	KeV(10.50 - 10.58)
Pb L $\beta$ <u>12.619</u>	KeV(12.57 - 12.65)
Source Line <u>22.096</u>	KeV(22.06 - 22.14)

Source: Fe55	Range
S K $\alpha$ <u>2.309</u>	KeV(2.29 - 2.33)
Source Line <u>5.885</u>	KeV(5.87 - 5.91)

Source: Am 241	Range
Pb L $\alpha$ <u>10.538</u>	KeV(10.49 - 10.59)
Pb L $\beta$ <u>12.628</u>	KeV(12.56 - 12.66)
Source Line <u>59.472</u>	KeV(59.3 - 59.7)

IRON K $\alpha$  RESOLUTION/INTENSITY CHECK (Cd 109, IRON PURE)

#RESCK

Iron at maximum peak height (MPH) = 3421 counts (MPH  $\geq$  1000 at 6.40  $\pm$  0.02 KeV)  
 1/2 MPH = 1711 counts

left (low energy) side, 1/2 MPH  
1749 counts at 6.260 KeV  
1417 counts at 6.244 KeV

6.549  
6.260  
.289

right (high energy) side, 1/2 MPH  
1740 counts at 6.533 KeV  
1460 counts at 6.549 KeV

Calculated FWHM = 0.289 KeV ( $\leq$  0.300)

- Pass  Fail: Counts  $\leq$  1/2 MPH at 6.25 KeV
- Pass  Fail: Counts  $\leq$  1/2 MPH at 6.55 KeV

Cd 109 Intensity Check Criteria  
 Fe 0.987289 ( $\geq$  0.95 and  $\leq$  1.05)  
 Mn -0.000017 ( $\leq$   $\pm$  0.006)  
 Co 0.00275 ( $\leq$   $\pm$  0.006)

BLANK SAMPLE CHECK

#SAND

Check One:  Quartz  Teflon  Sand  Other (Specify) \_\_\_\_\_

- Pass  Fail: All target elements Cr (z=24) and higher are within  $\pm$  3 std. deviations of zero
- Pass  Fail: All non-target elements Cr (z=24) and higher are within  $\pm$  5 std. deviations of zero

Comments Zr = 40 +/- 2.9 OK

NOTE: All acquisition times  $\geq$  60 seconds each source, All checks with standard Soil Application

OK

Rev - 03/14/01

Initials \_\_\_\_\_ OK

Continued on Page

Read and Understood By

Dominic Kalinsky  
Signed

7/29/03  
Date

Signed

Date

#	ID	Time	As	Pb	Cu	Sb	COMMENT
7-29-03	Q114						60 performance
04	#2709	0817	-3	30	85	1	SPM (QC)
05	#2710	0821	628	5740	2900	27	" "
06	#2711	0826	55	1185	131	0	" "
07	#R33	0830	216	317	7	11	QC
08	50L200D	0835	218	3180	114	-2	Soil
09	100L50D	0839	319	7210	45	25	Soil
10	100L150D	0844	261	4217	35	16	Soil
11	100L250D	0849	-4	75	95	10	Soil
12	250L0D	0854	-5	17	53	-11	Soil
13	100L100D	0858	38	2236	44	-10	Soil
14	250A0D	0903	13	55	33	10	Soil
15	300R50D	0908	14	28	32	-6	Soil
16	50L300D	0913	12	51	2	-40	Soil
17	50L300D-DWP	0918	9	38	29	-10	Soil
18	#2709	0922	33	13	88	-19	SPM (QC)
19	#2710	0927	584	5690	3060	37	" "
20	#R33	0931	204	314	-16	-43	QC
21	250R50D	0936	4	85	-7	6	Soil
22	250R150D	0940	16	59	-22	-3	Soil
23	250R100D	0945	37	81	28	-17	Soil
24	300R0D	0950	42	25	54	-41	Soil
25	150L200D	0955	15	58	62	-4	Soil
26	150L100D	0959	20	97	22	-7	Soil
27	100R250D	1004	2	81	120	1	Soil
28	200R100D	1009	2	193	58	-20	Soil
29	200R50D	1013	9	37	35	-32	Soil
30	200R50D-DWP	1018	4	57	32	-29	Soil
31	#2709	1023	20	32	36	15	SPM (QC)
32	#2710	1027	383	5550	2870	-2	" "
33	#R33	1031	228	308	-14	-44	QC

FILES: R170729 A. RES ✓  
 R170729 A. SPT ✓

Continued on Page

Read and Understood By

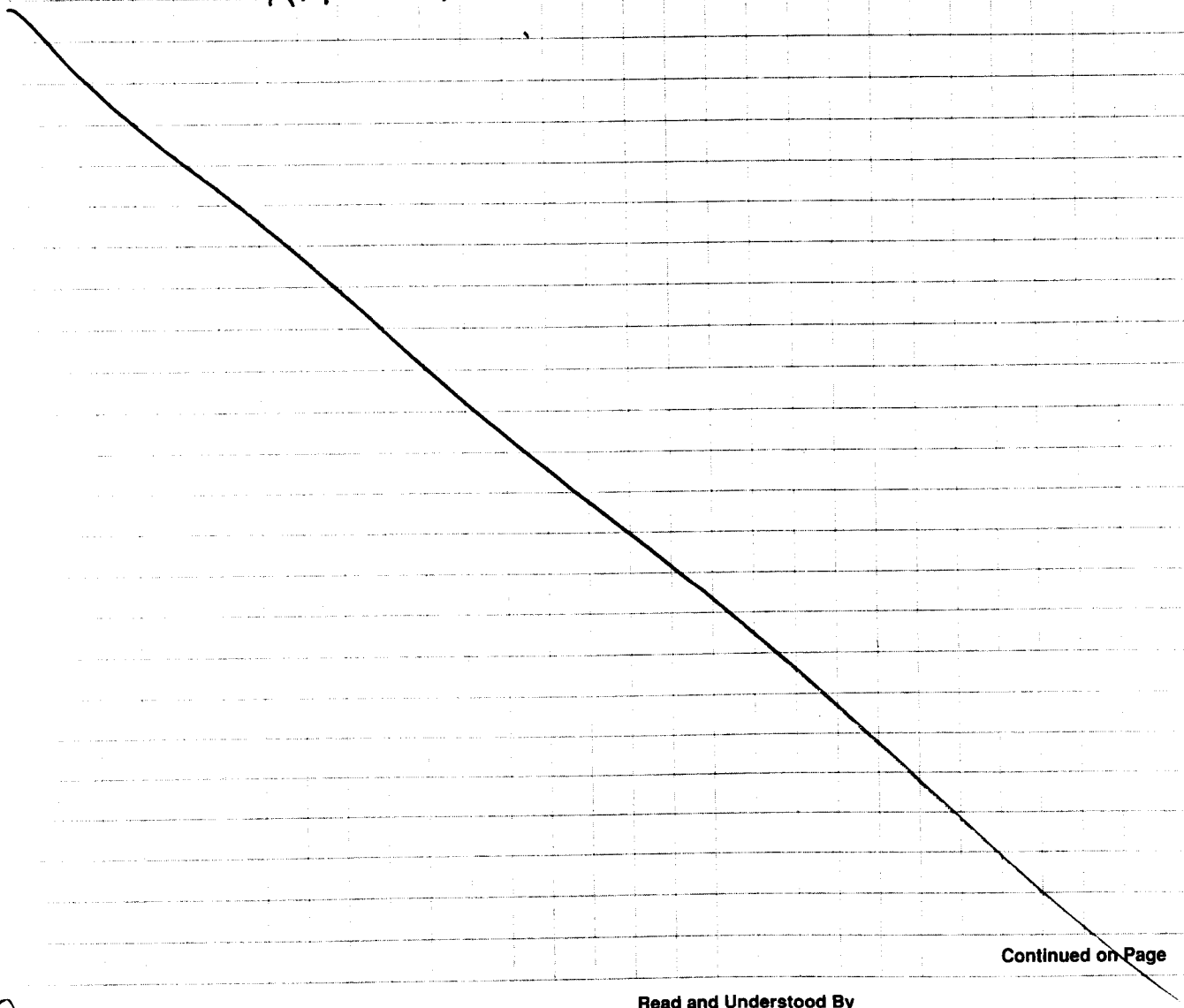
*Dennis Kalinsky* 7/29/03  
 Signed Date

Signed

Date

#	ID	Time	As	Pb	Cu	① Sb	COMMENT
01	200L200D	1051	-10	79	76	-10	Soil
02	200R200D	1056	11	84	59	-8	Soil
03	200 ROD	1100	12	25	22	-4	Soil
04	200R150D	1105	6	37	114	0	Soil
05	#2709	1100	28	97	78	-9	SRM(QC)
06	#2710	1114	465	5560	2770	28	" "
07	#R33	1118	222	309	18	-30	QC

FILES: R170729B.RES ✓  
R170729B.SPT ✓



Continued on Page

Domin Kalinsky 7/29/03  
Signed Date

Read and Understood By \_\_\_\_\_  
Signed Date

Appendix C  
Final Chemical and Physical Analytical Reports  
Range 17 –Patuxent Research Refuge  
Laurel, MD  
March 2004

ANALYTICAL REPORT

Prepared by  
LOCKHEED MARTIN

Range 17 (PNRR) Site  
Fort Meade, Maryland

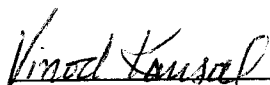
September 2003

EPA Work Assignment No. 0-318  
LOCKHEED MARTIN Work Order No.R1A0318  
EPA Contract No. 68-C99-223

Submitted to  
D. Charters  
EPA-ERTC

 9/5/03  
J. Badner Date  
Task Leader

Analysis by:  
REAC

 9/8/03  
V. Kansal Date  
Analytical Section Leader

Prepared by:  
R. Varsolona

 9/9/03  
D. Miller Date  
Program Manager

Reviewed by:  
J. Soroka

## Table of Contents

<u>Topic</u>	<u>Page Number</u>
Introduction	Page 1
Case Narrative	Page 1
Summary of Abbreviations	Page 2
 Section I	
Analytical Procedure for BNA in Soil	Page 3
Analytical Procedure for Metals in Soil	Page 5
Results of the Analysis for BNA in Soil	Table 1.1 Page 6
Results of TIC for BNA in Soil	Table 1.2 Page 8
Results of the Analysis for Metals in Soil	Table 1.3 Page 15
 Section II	
<b>QA/QC for BNA</b>	Page 18
Results of Internal Standard Areas for BNA in Soil	Table 2.1 Page 19
Results of the Surrogate Percent Recoveries for BNA in Soil	Table 2.2 Page 20
Results of MS/MSD Analysis for BNA in Soil	Table 2.3 Page 21
<b>QA/QC for Metals</b>	Page 22
Results of MS/MSD Analysis for Metals in Soil	Table 2.4 Page 23
Results of the Blank Spike Analysis for Metals in Soil	Table 2.5 Page 25
Results of the LCS Analysis for Metals in Soil	Table 2.6 Page 26
 Section III	
Chains of Custody	Page 27
Appendix A      Data for BNA-Soil	Page M136001
Appendix B      Data for Metals-Soil	Page M133001

Appendices will be furnished on request.



## Introduction

REAC in response to WA #0-318, provided analytical support for environmental samples collected from the Range 17 (PNRR) Site, in Fort Meade, Maryland as described in the following table. The support also included QA/QC, data review, and preparation of an analytical report containing a summary of the analytical methods, the results, and the QA/QC results.

The samples were treated with procedures consistent with those described in SOP #1008.

COC #	Number of Samples	Sampling Date	Date Received	Matrix	Analysis	Laboratory	Data Package
318-0001	6	7/29/03	7/30/03	Soil	BNA	REAC	M136
318-0001	6				Metals		M133
15364	5	7/28/03	8/4/03		Pb, As, Cu, Sb		
	5	7/29/03	8/4/03				

## Case Narrative

The data contained in this report has been validated to two significant figures. Any other interpretation of the data is the responsibility of the user.

### Data Package M136 BNA in Soil

The data was examined and found to be acceptable.

### Data Package M133 Metals in Soil

The method blank contained 1.1 mg/Kg selenium and 1.7 mg/Kg zinc. Samples 318-0001, 318-0002, 318-0003, 318-0004, 318-0005 and 318-0006 were < 5 times the blank value for selenium, their results are considered non-detect. The associated samples for zinc were >5 times the method blank. The data are not affected.

The acceptable QC limits for the percent recoveries were exceeded for antimony in 318-0005 MS (39%), MSD (41%) and manganese in 318-0005 MS (192%). The concentration of antimony and manganese should be considered estimated in samples 318-0001, 318-0002, 318-0003, 318-0004, 318-0005 and 318-0006.

## Summary of Abbreviations

AA	Atomic Absorption				
B	The analyte was found in the blank				
BFB	Bromofluorobenzene				
C	Centigrade				
cont.	Continued				
D	(Surrogate Table) this value is from a diluted sample and was not calculated				
	(Result Table) this result was obtained from a diluted sample				
Dioxin	denotes Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans and/or PCDD and PCDF				
CLP	Contract Laboratory Protocol				
COC	Chain of Custody				
CONC	Concentration				
CRDL	Contract Required Detection Limit				
CRQL	Contract Required Quantitation Limit				
DFTPP	Decafluorotriphenylphosphine				
DL	Detection Limit				
E	The value is greater than the highest linear standard and is estimated				
EMPC	Estimated maximum possible concentration				
ICAP	Inductively Coupled Argon Plasma				
ISTD	Internal Standard				
J	The value is below the method detection limit and is estimated				
LCS	Laboratory Control Sample				
LCSD	Laboratory Control Sample Duplicate				
MDL	Method Detection Limit				
MI	Matrix Interference				
MS (BS)	Matrix Spike (Blank Spike)				
MSD (BSD)	Matrix Spike Duplicate (Blank Spike Duplicate)				
MW	Molecular Weight				
NA	Either Not Applicable or Not Available				
NC	Not Calculated				
NR	Not Requested				
NS	Not Spiked				
% D	Percent Difference				
% REC	Percent Recovery				
PPB	Parts per billion				
PPBV	Parts per billion by volume				
PPMV	Parts per million by volume				
PQL	Practical Quantitation Limit				
QA/QC	Quality Assurance/Quality Control				
QL	Quantitation Limit				
RPD	Relative Percent Difference				
RSD	Relative Standard Deviation				
SIM	Selected Ion Monitoring				
TCLP	Toxic Characteristics Leaching Procedure				
U	Denotes not detected				
W	Weathered analyte; the results should be regarded as estimated				
m <sup>3</sup>	cubic meter	kg	kilogram	µg	microgram
L	liter	g	gram	pg	picogram
mL	milliliter	mg	milligram	ng	nanogram
µL	microliter				
*	denotes a value that exceeds the acceptable QC limit				
	Abbreviations that are specific to a particular table are noted in footnotes on that table				

Revision 2/15/00

## Analytical Procedure for BNA in Soil

### Extraction Procedure

Prior to extraction each sample was spiked with a six component surrogate mixture consisting of nitrobenzene-d<sub>5</sub>, 2-fluorobiphenyl, terphenyl-d<sub>14</sub>, phenol-d<sub>5</sub>, 2-fluorophenol, and 2,4,6-tribromophenol. Thirty grams of sample was mixed with 30 g anhydrous sodium sulfate, and Soxhlet extracted for 16 hours with 300 mL of methylene chloride. The extract was concentrated to 1.0 mL, an internal standard mixture consisting of 1,4-dichlorobenzene-d<sub>4</sub>, naphthalene-d<sub>8</sub>, acenaphthene-d<sub>10</sub>, phenanthrene-d<sub>10</sub>, chrysene-d<sub>12</sub>, and perylene-d<sub>12</sub> was added, and analyzed.

### Analysis Procedure

An Agilent 6890/5973 gas chromatograph/mass spectrometer (GC/MSD), equipped with a 7683 autosampler and controlled by a PC computer equipped with Enviroquant software was used for sample analysis.

The instrument conditions were:

Column:	Restek Rtx-5, 30 meter x 0.25 mm ID, 0.50 µm film thickness (or equivalent)
Injection Temperature:	250°C
Transfer Temperature:	280°C
Source Temperature and Analyzer Temperature:	230°C
Temperature Program:	50°C for 0.5 minutes 20°C/min to 295°C; hold for 5 minutes 35°C/min to 310°C; hold for 8.5 minutes
Pulsed Split Injection:	Pressure Pulse = 16psi for 0.25 min, then normal Splitless at purge flow of 5 mL/min for 0.5 min
Injection Volume:	1 µL (Must use 4 mm ID single gooseneck liners packed with 10 mm plug of silanized and conditioned glass wool).

The GC/MS system was calibrated using 5 BNA standard mixtures at 20, 50, 80, 120, and 160 µg/mL. Before each analysis day, the system was tuned with 50 ng decafluorotriphenylphosphine (DFTPP) and passed a continuing calibration check when analyzing a 50 µg/mL standard mixture in which the responses were evaluated by comparison to the average response of the calibration curve.

The BNA results, based on dry weight, are listed in Table 1.1; the tentatively identified compounds (TICs) are listed in Table 1.2. The concentration of the detected compounds was calculated using the following equation:

$$C_u = \frac{(A_u)(I_{is})(V_i)(DF)}{(A_{is})(RF \text{ or } RF_{ave})(V_i)(W)(D)}$$

where;

$C_u$	=	Concentration of target analyte ( $\mu\text{g}/\text{Kg}$ )
$A_u$	=	Area of target analyte
$I_{is}$	=	Mass of specific internal standard (ng)
$V_i$	=	Volume of extract ( $\mu\text{L}$ )
DF	=	Dilution Factor
$A_{is}$	=	Area of specific internal standard
RF	=	Response Factor (unitless)
$RF_{ave}$	=	Average Response Factor (unitless)
$V_i$	=	Volume of extract injected ( $\mu\text{L}$ )
W	=	Weight of sample (g)
D	=	Decimal per cent solids

The  $RF_{ave}$  is used when a sample is associated with an initial calibration curve. The RF is used when a sample is associated with a continuing calibration.

Response Factor calculation:

The RF for each specific analyte is quantitated based on the area response from the continuing calibration check as follows:

$$RF = \frac{(A_c)(I_{is})}{(A_{is})(I_c)}$$

where;

RF	=	Response factor for a specific analyte
$A_c$	=	Area of the analyte in the standard
$I_{is}$	=	Mass of the specific internal standard
$A_{is}$	=	Area of the specific internal standard
$I_c$	=	Mass of the analyte in the standard

$$RF_{AVE} = \frac{RF_1 + \dots + RF_n}{n}$$

and

n = number of standards used in initial calibration (e.g., n = 5)

Revision of 8/04/03

## Analytical Procedure for Metals in Soil

### Sample Preparation

A representative 1-2 g (wet weight) sample, weighed to 0.01 g accuracy, was mixed with 10-mL 1:1 nitric acid, placed in a 50-mL polypropylene digestion cup and digested in nitric acid and hydrogen peroxide according to SW-846, Method 3050 B on a Hot Block digestion system. The final reflux was either nitric acid or hydrochloric acid depending on the metals to be determined. After digestion, the samples were allowed to cool to room temperature, transferred to 100 mL volumetric flasks and diluted to volume with ASTM Type I water. The samples were analyzed for all metals, except mercury, according to ERTC/REAC SOP #1818 *Determination of Metals by Graphite Furnace Atomic Absorption (GFAA) Methods* or SOP #1811 *Determination of Metals by Inductively Coupled Plasma (ICP) Methods*.

A representative 0.25-0.8 g (wet weight) sample, weighed to 0.01 g accuracy, was transferred to a 300-mL BOD bottle and prepared according to SW-846, Method 7471B. The sample was heated for 1/2 hour on a hot plate at 95° C, cooled to room temperature, and reduced with sodium chloride hydroxylamine hydrochloride solution (NH<sub>2</sub>OH:HCl). Mercury was then analyzed separately on a Leeman Labs PS200II AA Spectrometer according to ERTC/REAC SOP #1832, *Determination of Mercury by Cold-Vapor Atomic Absorption (CVAA) Methods*.

A separate sample was used to determine total solids.

A reagent blank and a blank spike sample were processed for each batch of samples. One matrix spike (MS) and one matrix spike duplicate (MSD) were also processed for each batch or for every ten samples.

### Analysis and Calculations

The AA, ICP and Leeman Labs PS200II instruments were calibrated and operated according to SOP #1818/1811/1832 and the manufacturers operating instructions. After calibration, initial calibration verification (ICV), initial calibration blank (ICB), and quality control check standards were run to verify proper calibration. The continuing calibration verification (CCV) and continuing calibration blank (CCB) standards were run after every ten sample analyses to assure proper operation during sample analysis.

The metal concentration in solution, in micrograms per liter (µg/L) or milligrams per liter (mg/L) was read directly from the read-out system of the AA or ICP instrument. The results were converted to milligrams per kilogram (mg/kg) by correcting the instrument reading for the sample weight and percent solids.

Final concentrations, based on wet weight are given by:

$$\text{mg metal/kg sample} = [(A \times V) / W] \times \text{DF} \times \text{CF}$$

where:

A = Instrument read-out (µg/L for AA; mg/L or µg/L for ICP)  
V = final volume of processed sample (mL)  
W = weight of sample (g)  
DF = Dilution Factor (1.00 for no dilution)  
CF = conversion factor (0.001 for µg/L; 1.00 for mg/L)

For samples that required dilution to be within the instrument calibration range, DF is given by:

$$\text{DF} = (C + B) / C$$

where:

B = amount of acid blank used for dilution (mL)  
C = sample aliquot (mL)

Final concentrations, based on dry weight, are given by:

$$\text{mg/kg(dry)} = [\text{mg/kg (wet)} \times 100] / S$$

where

S = percent solids

The results are listed in Table 1.3

Revision date: 04/24/2003

Table 1.1 Results of the Analysis for BNA in Soil  
 WA # 0-318 Range 17 (PNRR) Site  
 Results Based On Dry Weight

Sample No.	SBLK073103		318-0001		318-0005		318-0002		318-0003	
Sample Location	Lab Blank		0150D		150R50D		100R100D		100R50D	
Dilution Factor	1		1		1		1		1	
% Solids	100		69		87		86		65	
Compound Name	Conc. µg/kg	MDL µg/kg	Conc. µg/kg	MDL µg/kg	Conc. µg/kg	MDL µg/kg	Conc. µg/kg	MDL µg/kg	Conc. µg/kg	MDL µg/kg
Phenol	U	330	U	480	U	380	U	380	U	510
bis(-2-Chloroethyl)Ether	U	330	U	480	U	380	U	380	U	510
2-Chlorophenol	U	330	U	480	U	380	U	380	U	510
1,3-Dichlorobenzene	U	330	U	480	U	380	U	380	U	510
1,4-Dichlorobenzene	U	330	U	480	U	380	U	380	U	510
Benzyl alcohol	U	330	U	480	U	380	U	380	U	510
1,2-Dichlorobenzene	U	330	U	480	U	380	U	380	U	510
2-Methylphenol	U	330	U	480	U	380	U	380	U	510
bis(2-Chloroisopropyl)ether	U	330	U	480	U	380	U	380	U	510
4-Methylphenol	U	330	U	480	U	380	U	380	U	510
N-Nitroso-Di-n-propylamine	U	330	U	480	U	380	U	380	U	510
Hexachloroethane	U	330	U	480	U	380	U	380	U	510
Nitrobenzene	U	330	U	480	U	380	U	380	U	510
Isophorone	U	330	U	480	U	380	U	380	U	510
2-Nitrophenol	U	330	U	480	U	380	U	380	U	510
2,4-Dimethylphenol	U	330	U	480	U	380	U	380	U	510
bis(2-Chloroethoxy)methane	U	330	U	480	U	380	U	380	U	510
2,4-Dichlorophenol	U	330	U	480	U	380	U	380	U	510
1,2,4-Trichlorobenzene	U	330	U	480	U	380	U	380	U	510
Naphthalene	U	330	U	480	U	380	U	380	U	510
4-Chloroaniline	U	330	U	480	U	380	U	380	U	510
Hexachlorobutadiene	U	330	U	480	U	380	U	380	U	510
4-Chloro-3-methylphenol	U	330	U	480	U	380	U	380	U	510
2-Methylnaphthalene	U	330	U	480	U	380	U	380	U	510
Hexachlorocyclopentadiene	U	330	U	480	U	380	U	380	U	510
2,4,6-Trichlorophenol	U	330	U	480	U	380	U	380	U	510
2,4,5-Trichlorophenol	U	330	U	480	U	380	U	380	U	510
2-Chloronaphthalene	U	330	U	480	U	380	U	380	U	510
2-Nitroaniline	U	330	U	480	U	380	U	380	U	510
Dimethylphthalate	U	330	U	480	U	380	U	380	U	510
Acenaphthylene	U	330	U	480	U	380	U	380	U	510
2,6-Dinitrotoluene	U	330	U	480	U	380	U	380	U	510
3-Nitroaniline	U	330	U	480	U	380	U	380	U	510
Acenaphthene	U	330	U	480	U	380	U	380	U	510
2,4-Dinitrophenol	U	330	U	480	U	380	U	380	U	510
4-Nitrophenol	U	330	U	480	U	380	U	380	U	510
Dibenzofuran	U	330	U	480	U	380	U	380	U	510
2,4-Dinitrotoluene	U	330	U	480	U	380	U	380	U	510
Diethylphthalate	U	330	U	480	U	380	U	380	U	510
4-Chlorophenyl-phenylether	U	330	U	480	U	380	U	380	U	510
Fluorene	U	330	U	480	U	380	U	380	U	510
4-Nitroaniline	U	330	U	480	U	380	U	380	U	510
4,6-Dinitro-2-methylphenol	U	330	U	480	U	380	U	380	U	510
N-Nitrosodiphenylamine	U	330	U	480	U	380	U	380	U	510
4-Bromophenyl-phenylether	U	330	U	480	U	380	U	380	U	510
Hexachlorobenzene	U	330	U	480	U	380	U	380	U	510
Pentachlorophenol	U	330	U	480	U	380	U	380	U	510
Phenanthrene	U	330	U	480	U	380	U	380	U	510
Anthracene	U	330	U	480	U	380	U	380	U	510
Carbazole	U	330	U	480	U	380	U	380	U	510
Di-n-butylphthalate	U	330	U	480	U	380	U	380	U	510
Fluoranthene	U	330	U	480	U	380	U	380	U	510
Pyrene	U	330	U	480	U	380	U	380	U	510
Butylbenzylphthalate	U	330	U	480	U	380	U	380	U	510
Benzo(a)anthracene	U	330	U	480	U	380	U	380	U	510
3,3'-Dichlorobenzidine	U	330	U	480	U	380	U	380	U	510
Chrysene	U	330	U	480	U	380	U	380	U	510
Bis(2-Ethylhexyl)phthalate	U	330	430	J	480	130	J	380	U	510
Di-n-octylphthalate	U	330	U	480	U	380	U	380	U	510
Benzo(b)fluoranthene	U	330	U	480	U	380	U	380	U	510
Benzo(k)fluoranthene	U	330	U	480	U	380	U	380	U	510
Benzo(a)pyrene	U	330	U	480	U	380	U	380	U	510
Indeno(1,2,3-cd)pyrene	U	330	U	480	U	380	U	380	U	510
Dibenzo(a,h)anthracene	U	330	U	480	U	380	U	380	U	510
Benzo(g,h,i)perylene	U	330	U	480	U	380	U	380	U	510

Table 1.1 (Cont.) Results of the Analysis for BNA in Soil  
 WA # 0-318 Range 17 (PNRR) Site  
 Based On Dry Weight

Sample No.	318-0004		318-0006	
Sample Location	150R100D		Reference	
Dilution Factor	1		1	
% Solid	87		77	
Compound Name	Conc. µg/kg	MDL µg/kg	Conc. µg/kg	MDL µg/kg
Phenol	U	380	U	430
bis(-2-Chloroethyl)Ether	U	380	U	430
2-Chlorophenol	U	380	U	430
1,3-Dichlorobenzene	U	380	U	430
1,4-Dichlorobenzene	U	380	U	430
Benzyl alcohol	U	380	U	430
1,2-Dichlorobenzene	U	380	U	430
2-Methylphenol	U	380	U	430
bis(2-Chloroisopropyl)ether	U	380	U	430
4-Methylphenol	U	380	U	430
N-Nitroso-Di-n-propylamine	U	380	U	430
Hexachloroethane	U	380	U	430
Nitrobenzene	U	380	U	430
Isophorone	U	380	U	430
2-Nitrophenol	U	380	U	430
2,4-Dimethylphenol	U	380	U	430
bis(2-Chloroethoxy)methane	U	380	U	430
2,4-Dichlorophenol	U	380	U	430
1,2,4-Trichlorobenzene	U	380	U	430
Naphthalene	U	380	U	430
4-Chloroaniline	U	380	U	430
Hexachlorobutadiene	U	380	U	430
4-Chloro-3-methylphenol	U	380	U	430
2-Methylnaphthalene	U	380	U	430
Hexachlorocyclopentadiene	U	380	U	430
2,4,6-Trichlorophenol	U	380	U	430
2,4,5-Trichlorophenol	U	380	U	430
2-Chloronaphthalene	U	380	U	430
2-Nitroaniline	U	380	U	430
Dimethylphthalate	U	380	U	430
Acenaphthylene	U	380	U	430
2,6-Dinitrotoluene	U	380	U	430
3-Nitroaniline	U	380	U	430
Acenaphthene	U	380	U	430
2,4-Dinitrophenol	U	380	U	430
4-Nitrophenol	U	380	U	430
Dibenzofuran	U	380	U	430
2,4-Dinitrotoluene	U	380	U	430
Diethylphthalate	U	380	U	430
4-Chlorophenyl-phenylether	U	380	U	430
Fluorene	U	380	U	430
4-Nitroaniline	U	380	U	430
4,6-Dinitro-2-methylphenol	U	380	U	430
N-Nitrosodiphenylamine	U	380	U	430
4-Bromophenyl-phenylether	U	380	U	430
Hexachlorobenzene	U	380	U	430
Pentachlorophenol	U	380	U	430
Phenanthrene	U	380	U	430
Anthracene	U	380	U	430
Carbazole	U	380	U	430
Di-n-butylphthalate	U	380	U	430
Fluoranthene	U	380	U	430
Pyrene	U	380	U	430
Butylbenzylphthalate	U	380	U	430
Benzo(a)anthracene	U	380	U	430
3,3'-Dichlorobenzidine	U	380	U	430
Chrysene	U	380	U	430
Bis(2-Ethylhexyl)phthalate	U	380	130	J 430
Di-n-octylphthalate	U	380	U	430
Benzo(b)fluoranthene	U	380	U	430
Benzo(k)fluoranthene	U	380	U	430
Benzo(a)pyrene	U	380	U	430
Indeno(1,2,3-cd)pyrene	U	380	U	430
Dibenzo(a,h)anthracene	U	380	U	430
Benzo(g,h,i)perylene	U	380	U	430

Table 1.2 Results of TIC for BNA in Soil  
WA# 0-234 Range 17 (PNRR) Site

Sample #	Compound
SBLK073103	No TIC Found



Table 1.2 (Cont) Results of TIC for BNA in Soil  
 WA # 0-318 Range 17 (PNRR) Site

Sample #	318-0001	Con. Factor	48.24		
LabFile#	PNR2013	Conc.*	µg/kg		
	CAS#	Compound	Q	RT	Conc.*
1		Unknown		4.19	270
2		Unknown alkane		13.36	390
3		Unknown aldehyde		14.12	370
4		Unknown alcohol		14.49	2000
5		C27 Alkane		15.95	390
6		Unknown alcohol		16.02	1700
7		Unknown alkane		16.89	320
8		Unknown aldehyde		17.45	380
9		C29 Alkane		18.06	6900
10		Unknown alcohol		18.18	380
11		Unknown alkane		19.06	380
12		Unknown aldehyde		19.64	530
13		C31 Alkane		20.19	4300
14		Unknown		20.35	1100
15		Unknown ketone		20.57	430
16		Unknown		21.07	670
17		Unknown		22.31	2000
18		Unknown alkane		23.01	550
19		Unknown alcohol		23.31	1600
20		Unknown		24.97	2900

\* Estimated Concentration ( Response Factor = 1 )

Table 1.2 (Cont) Results of TIC for BNA in Soil  
 WA # 0-318 Range 17 (PNRR) Site

Sample #	318-0005				38.54
LabFile#	PNR2014		Con. Factor		
	CAS#	Compound	Q	RT	Conc.* µg/kg
1		Unknown		4.19	100
2	79345	Ethane, 1,1,2,2-tetrachloro-	96	4.81	230
3		Unknown		5.14	85
4		Unknown alcohol		13.36	150
5		Unknown aldehyde		14.12	150
6		Unknown alcohol		14.49	320
7		Unknown aldehyde		15.50	81
8		Unknown alkane		15.94	96
9		Unknown alcohol		16.02	310
10		Unknown aldehyde		17.45	250
11		C29 Alkane		18.05	1000
12		Unknown alcohol		18.18	260
13		Unknown alkane		19.06	110
14		Unknown aldehyde		19.64	330
15		Unknown alkane		20.18	1200
16		Unknown alkene		20.35	560
17		Unknown		21.07	240
18		Unknown		22.30	500
19		Unknown		23.84	760
20		Unknown		24.96	990

\* Estimated Concentration ( Response Factor = 1 )

Table 1.2 (Cont) Results of TIC for BNA in Soil  
 WA # 0-318 Range 17 (PNRR) Site

Sample #	318-0002				38.62
LabFile#	PNR2017		Con. Factor		Conc.*
	CAS#	Compound	Q	RT	µg/kg
1	79345	Ethane, 1,1,2,2-tetrachloro-	90	4.81	81
2	65850	Benzoic acid (CAS)	97	6.79	110
3		Unknown alcohol		13.36	170
4		Unknown aldehyde		14.13	100
5		Unknown		14.35	170
6		Unknown alcohol		14.50	510
7		Unknown aldehyde		15.51	120
8		Unknown alkane		15.95	180
9		Unknown alcohol		16.02	860
10		Unknown aldehyde		17.45	680
11		Unknown alkane		18.05	760
12		Unknown alcohol		18.18	870
13		Unknown		18.58	110
14		Unknown aldehyde		19.65	1700
15		Unknown alkane		20.19	940
16		Unknown		20.36	680
17		Unknown aldehyde		22.31	1200
18		Unknown alcohol		23.32	630
19		Unknown		24.97	770

\* Estimated Concentration ( Response Factor = 1 )

Table 1.2 (Cont) Results of TIC for BNA in Soil  
 WA # 0-318 Range 17 (PNRR) Site

Sample # 318-0003  
 LabFile# PNR2018  
 Con. Factor 51.28

	CAS#	Compound	Q	RT	Conc.* µg/kg
1	65850	Benzoic acid (CAS)	97	6.80	380
2		Unknown alcohol		13.36	910
3		Unknown		14.35	570
4		Unknown alcohol		14.49	2800
5		Unknown aldehyde		15.51	530
6		C27 Alkane		15.95	750
7		Unknown alcohol		16.02	5100
8		Unknown aldehyde		17.45	2200
9		C29 Alkane		18.06	3200
10		Unknown alcohol		18.18	2400
11		Unknown ketone		18.37	270
12		Unknown		18.58	500
13		Unknown aldehyde		19.65	6300
14		Unknown alkane		20.19	4200
15		Unknown		20.36	2300
16		Unknown ketone		20.57	930
17		Unknown aldehyde		22.32	3900
18		Unknown		23.60	1500
19		Unknown		24.98	2100
20		Unknown		26.06	2000

\* Estimated Concentration ( Response Factor = 1 )

Table 1.2 (Cont) Results of TIC for BNA in Soil  
 WA # 0-318 Range 17 (PNRR) Site

Sample #	318-0004				38.45
LabFile#	PNR2019		Con. Factor		
	CAS#	Compound	Q	RT	Conc.* µg/kg
1		Unknown		4.20	120
2	79345	Ethane, 1,1,2,2-tetrachloro-	95	4.81	130
3		Unknown		7.96	160
4		Unknown		13.36	230
5		Unknown		13.72	120
6		Unknown aldehyde		14.13	270
7		Unknown alcohol		14.50	330
8		Unknown aldehyde		14.77	130
9		Unknown aldehyde		15.51	92
10		Unknown alcohol		16.02	390
11		Unknown		17.45	200
12		Unknown alkane		18.06	450
13		Unknown aldehyde		19.64	220
14		Unknown		20.35	870
15		Unknown		21.08	370
16		Unknown		23.86	2200
17		Unknown		24.97	290

\* Estimated Concentration ( Response Factor = 1 )

Table 1.2 (Cont) Results of TIC for BNA in Soil  
 WA # 0-318 Range 17 (PNRR) Site

Sample # 318-0006  
 LabFile# PNR2020  
 Con. Factor 43.46

	CAS#	Compound	Q	RT	Conc.* µg/kg
1	80568	.alpha-Pinene	96	5.03	730
2	65850	Benzoic acid (CAS)	96	6.81	470
3		Unknown		7.96	250
4		Unknown		13.36	780
5		Unknown		13.72	290
6		Unknown aldehyde		14.13	360
7		Unknown alcohol		14.50	2400
8		Unknown aldehyde		15.51	240
9		Unknown alkane		15.95	330
10		Unknown alcohol		16.02	3100
11		Unknown aldehyde		17.45	780
12		C29 Alkane		18.06	2900
13		Unknown alkene		18.18	1200
14		Unknown aldehyde		19.65	1900
15		C31 Alkane		20.20	3600
16		Unknown alkene		20.36	920
17		Unknown ketone		20.58	820
18		Unknown		20.87	370
19		Unknown aldehyde		22.32	3000
20		Unknown ketone		23.61	1400

\* Estimated Concentration ( Response Factor = 1 )

Table 1.3 Results of the Analysis for Metals in Soil  
 WA # 0-318 Range 17 (PNRR) Site  
 Results Based on Dry Weight

Client ID Location % Solids	Method Blank Lab NA	318-0001 0150D 69		318-0002 100R100D 86		318-0003 100R50D 65		318-0004 150R100D 87		318-0005 150R50D 87			
Parameter	Analysis Method	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg
Aluminum	ICAP	U	10	5800	10	1700	8.6	8300	11	2200	8.7	1800	9.4
Antimony	ICAP	U	1.0	340	1.0	0.97	0.86	5.2	1.1	U	0.87	U	0.94
Arsenic	ICAP	U	1.0	220	1.0	2.9	0.86	12	1.1	2.8	0.87	2.1	0.94
Barium	ICAP	U	0.50	22	0.51	6.2	0.43	35	0.56	7.3	0.44	7.3	0.47
Beryllium	ICAP	U	0.50	U	0.51	U	0.43	U	0.56	U	0.44	U	0.47
Cadmium	ICAP	U	0.50	U	0.51	U	0.43	U	0.56	U	0.44	U	0.47
Calcium	ICAP	U	10	200	10	47	8.6	150	11	95	8.7	130	9.4
Chromium	ICAP	U	0.50	13	0.51	3.4	0.43	11	0.56	4.9	0.44	3.9	0.47
Cobalt	ICAP	U	0.50	3.7	0.51	U	0.43	6.4	0.56	0.5	0.44	0.52	0.47
Copper	ICAP	U	0.50	25	0.51	5.9	0.43	15	0.56	5.9	0.44	3.4	0.47
Iron	ICAP	U	4.0	13000	4.0	2500	3.4	10000	4.5	3700	3.5	2800	3.8
Lead	ICAP	U	1.0	44000	5.1	540	0.86	3000	1.1	260	0.87	270	0.94
Magnesium	ICAP	U	50	270	51	89	43	450	56	140	44	120	47
Manganese	ICAP	U	0.50	110	0.51	11	0.43	430	0.56	17	0.44	16	0.47
Mercury	Cold Vapor	U	0.040	0.072	0.038	0.099	0.032	0.098	0.040	0.037	0.033	U	0.033
Nickel	ICAP	U	0.50	4.7	0.51	1.1	0.43	5.9	0.56	1.8	0.44	1.5	0.47
Potassium	ICAP	U	50	270	51	92	43	360	56	100	44	84	47
Selenium	ICAP	1.1	0.50	1.9	0.51	0.80	0.43	1.9	0.56	0.89	0.44	0.68	0.47
Silver	ICAP	U	0.5	1.5	0.51	U	0.43	U	0.56	U	0.44	U	0.47
Sodium	ICAP	U	100	U	100	U	86	U	110	U	87	U	94
Thallium	ICAP	U	1.0	U	1.0	U	0.86	U	1.1	U	0.87	U	0.94
Vanadium	ICAP	U	0.50	27	0.51	5.7	0.43	22	0.56	9.6	0.44	7.0	0.47
Zinc	ICAP	1.7	1.0	28	1.0	8.6	0.86	35	1.1	11	0.87	12	0.94

Table 1.3 (Cont.) Results of the Analysis for Metals in Soil  
 WA # 0-318 Range 17 (PNRR) Site  
 Results Based on Dry Weight

Client ID	318-0006		
Location	Reference		
% Solids	77		
Parameter	Analysis Method	Conc mg/kg	MDL mg/kg
Aluminum	ICAP	3100	9.7
Antimony	ICAP	U	0.97
Arsenic	ICAP	3.0	0.97
Barium	ICAP	15	0.48
Beryllium	ICAP	U	0.48
Cadmium	ICAP	U	0.48
Calcium	ICAP	38	9.7
Chromium	ICAP	6.4	0.48
Cobalt	ICAP	1.3	0.48
Copper	ICAP	9.2	0.48
Iron	ICAP	5700	3.9
Lead	ICAP	46	0.97
Magnesium	ICAP	130	48
Manganese	ICAP	37	0.48
Mercury	Cold Vapor	0.056	0.036
Nickel	ICAP	1.8	0.48
Potassium	ICAP	230	48
Selenium	ICAP	0.94	0.48
Silver	ICAP	U	0.48
Sodium	ICAP	U	97
Thallium	ICAP	U	0.97
Vanadium	ICAP	15	0.48
Zinc	ICAP	15	0.97



Table 1.3 (Cont.) Results of the Analysis for Metals in Soil  
 WA # 0-318 Range 17 (PNRR) Site  
 Results Reported As Received

Client ID Location	Method Blank Lab	0-0-0 000		0-150D 0-150D		50L200D 050L200D		100L50D 100L050D		100L100D 100L100D			
Parameter	Analysis Method	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg		
Antimony	ICAP	U	1.0	U	1.0	190	1.0	5.0	1.0	23	1.0	2.3	1.0
Arsenic	ICAP	U	1.0	2.3	1.0	130	1.0	11	1.0	19	1.0	7.5	1.0
Copper	ICAP	U	0.50	17	0.50	22	0.50	51	0.50	8.9	0.50	7.4	0.50
Lead	ICAP	U	1.0	120	1.0	18000	1.0	2300	1.0	5800	1.0	2100	1.0

Client ID Location		100R150D 100R150D		150R50D 150R050D		150R100D 150R100D		200R0D 200R000D		250R50D 250R050D	
Parameter	Analysis Method	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg	Conc mg/kg	MDL mg/kg
Antimony	ICAP	1.3	0.99	U	0.98	U	1.0	U	0.97	U	0.98
Arsenic	ICAP	5.9	0.99	1.9	0.98	2.7	1.0	2.6	0.97	2.6	0.98
Copper	ICAP	20	0.5	3.6	0.49	5.0	0.50	4.5	0.49	12	0.49
Lead	ICAP	720	0.99	510	0.98	300	1.0	19	0.97	54	0.98

## QA/QC for BNA

### Results of the Internal Standard Areas for BNA in Soil

The internal standard areas (for 1,4-dichlorobenzene-d<sub>4</sub>, naphthalene-d<sub>8</sub>, acenaphthene-d<sub>10</sub>, phenanthrene-d<sub>10</sub>, chrysene-d<sub>12</sub>, perylene-d<sub>12</sub>) are listed in Table 2.1. All fifty-four areas were within the acceptable QC limits.

### Results of the Surrogate Percent Recoveries for BNA in Soil

Before extraction, each sample was spiked with a six component mixture of CLP surrogate standards consisting of 2-fluorophenol, phenol-d<sub>5</sub>, nitrobenzene-d<sub>5</sub>, 2-fluorobiphenyl, 2,4,6-tribromophenol and terphenyl-d<sub>14</sub>. The surrogate percent recoveries for the soil samples, listed in Table 2.2, ranged from 27 to 75. All fifty-four values were within the acceptable QC limits.

### Results of the MS/MSD Analysis for BNA in Soil

Sample 318-0005 was chosen for the matrix spike/matrix spike duplicate (MS/MSD) analysis for the soil samples. The percent recoveries, ranging from 30 to 71, are listed in Table 2.3. Twenty out of twenty-two values were within the acceptable QC limits. The relative percent differences, also listed in Table 2.3, ranged from 0 (zero) to 8. All eleven values were within the acceptable QC limits.

Table 2.1 Results of the Internal Standard Areas for BNA in Soil  
 WA # 0-318 Range 17 (PNRR) Site

Analysis Date 08/05/2003  
 Matrix Soil

Sample No.	File ID	IS 1	IS 2	IS 3	IS 4	IS 5	IS 6
SBLK073103	PNR2012.D	520761	2239174	1007492	1640843	1171211	856602
318-0001	PNR2013.D	459556	1993839	908490	1436525	875268	608869
318-0005	PNR2014.D	513246	2240882	1033038	1556549	934864	631112
318-0005 MS	PNR2015.D	533556	2320267	1095297	1622829	959135	671057
318-0005 MSD	PNR2016.D	544082	2358620	1090022	1627585	945964	670809
318-0002	PNR2017.D	520710	2241874	1009852	1500717	840221	625153
318-0003	PNR2018.D	453798	1916664	860403	1249323	586346	482402
318-0004	PNR2019.D	470868	1997050	889030	1280398	639130	526511
318-0006	PNR2020.D	473432	2019824	915979	1320212	625150	538833
Cal Check Area	PNR2011.D	453365	1862050	789437	1261670	802757	560215

IS 1 = d4-Dichlorobenzene  
 IS 2 = d8-Naphthalene  
 IS 3 = d10-Acenaphthene  
 IS 4 = d10-Phenanthrene  
 IS 5 = d12-Chrysene  
 IS 6 = d12-Perylene

Table 2.2 Results of the Surrogate Percent Recoveries for BNA in Soil  
WA # 0-318 Range 17 (PNRR) Site

Analysis Date 08/05/2003  
Matrix Soil

Sample No.	File ID	Surr. 1	Surr. 2	Surr. 3	Surr. 4	Surr. 5	Surr. 6
SBLK073103	PNR2012.D	34	39	37	38	39	58
318-0001	PNR2013.D	31	47	42	48	75	63
318-0005	PNR2014.D	32	46	39	44	71	63
318-0005 MS	PNR2015.D	35	49	40	43	72	64
318-0005 MSD	PNR2016.D	34	49	40	44	71	63
318-0002	PNR2017.D	29	42	36	43	66	64
318-0003	PNR2018.D	27	47	44	51	68	67
318-0004	PNR2019.D	34	46	40	44	69	67
318-0006	PNR2020.D	38	57	52	58	73	67

Surrogate Limits

	Soil
Surr 1 = 2-Fluorophenol	(25-121)
Surr 2 = Phenol-d5	(24-113)
Surr 3 = Nitrobenzene-d5	(23-120)
Surr 4 = 2-Fluorobiphenyl	(30-115)
Surr 5 = 2,4,6-Tribromophenol	(19-122)
Surr 6 = Terphenyl-d14	(18-137)

Table 2.3 Results of MS/MSD Analysis for BNA in Soil  
 WA # 0-318 Range 17 (PNRR) Site  
 Based On Dry Weight

Sample ID: 318-0005

Compound Name	Sample Conc. (µg/kg)	MS		MSD		MS % Rec.	MSD % Rec.	RPD	QC Limits			
		Spike Added (µg/kg)	Spike Added (µg/kg)	MS Conc. (µg/kg)	MSD Conc. (µg/kg)				RPD	RPD	% Rec.	
Phenol	U	3850	3850	1750	1700	46	44	3	35	26	-	90
2-Chlorophenol	U	3850	3850	1610	1580	42	41	2	50	25	-	102
1,4-Dichlorobenzene	U	1930	1930	620	574	32	30	8	27	28	-	104
N-Nitroso-Di-N-Propylamine	U	1930	1930	892	884	46	46	1	38	41	-	126
1,2,4-Trichlorobenzene	U	1930	1930	715	698	37	36	2	23	38	-	107
4-Chloro-3-Methylphenol	U	3850	3850	2320	2290	60	60	1	33	26	-	103
Acenaphthene	U	1930	1930	905	916	47	48	1	19	31	-	137
4-Nitrophenol	U	3850	3850	2450	2450	64	64	0	50	11	-	114
2,4-Dinitrotoluene	U	1930	1930	912	914	47	47	0	47	28	-	89
Pentachlorophenol	U	3850	3850	2750	2630	71	68	5	47	17	-	109
Pyrene	U	1930	1930	1170	1130	61	59	4	36	35	-	142

## QA/QC for Metals

### Results of the MS/MSD Analysis for Metals in Soil

Samples 318-0005 & 200R0D were chosen for the matrix spike/matrix spike duplicate analysis (MS/MSD). The percent recoveries, listed in Table 2.4, ranged from 26 to 192. Thirty-five out of forty calculated values were within the acceptable QC limits. Two values were not calculated (NC) as the spike concentration was <4x the sample concentration. The relative percent differences, also listed in Table 2.4, ranged from 0 (zero) to 42. Nineteen out of twenty calculated values were within the acceptable QC limits. One value was not calculated (NC) as the spike concentration was <4x the sample concentration.

### Results of the Blank Spike Analysis for Metals in Soil

The results of the blank spike analysis are reported in Table 2.5. The percent recoveries ranged from 87 to 117 and all twenty-four values were within the acceptable QC limits.

### Results of the Analysis of the Laboratory Control Sample for Metals in Soil

A laboratory control sample was analyzed for the metals in soil. The percent recoveries, listed in Table 2.6, ranged from 43 to 107 and all twenty-four concentrations were within the acceptable QC limits.

Table 2.4 Results of the MS/MSD Analysis for Metals in Soil  
 WA# 0-318 Range 17 (PNRR) Site  
 Results Based on Dry Weight

Sample ID: 318-0005

Metal	Sample Conc mg/kg	MS Spike Added mg/kg	MS Conc mg/kg	MS % Rec	MSD Spike Added mg/kg	MSD Conc mg/kg	MDS % Rec	RPD	Recommended QC Limits	
									%Rec	RPD
Antimony	U	8.69	3.43	39 *	9.25	3.76	41 *	3	75-125	20
Arsenic	2.06	8.69	10.7	99	9.25	11.7	104	5	75-125	20
Barium	7.31	8.69	16.7	108	9.25	16.7	102	6	75-125	20
Beryllium	U	8.69	8.8	101	9.25	9.5	103	1	75-125	20
Cadmium	U	8.69	8.67	100	9.25	9.26	100	0	75-125	20
Chromium	3.87	8.69	12.4	98	9.25	13.7	106	8	75-125	20
Cobalt	0.52	8.69	9.35	102	9.25	10	102	1	75-125	20
Copper	3.42	8.69	13	110	9.25	13.7	111	1	75-125	20
Lead	269	8.69	322	NC	9.25	309	NC	NC	75-125	20
Manganese	15.8	8.69	32.5	192 *	9.25	27.4	125	42 *	75-125	20
Mercury	U	0.335	0.318	95	0.345	0.383	111	16	75-125	20
Nickel	1.47	8.69	10.5	104	9.25	11.2	105	1	75-125	20
Selenium	0.68	8.69	8.93	95	9.25	9.71	98	3	75-125	20
Silver	U	8.69	8.36	96	9.25	9.04	98	2	75-125	20
Thallium	U	8.69	7.99	92	9.25	8.58	93	1	75-125	20
Vanadium	6.96	8.69	15.8	102	9.25	16.8	106	4	75-125	20
Zinc	12.4	8.69	21	99	9.25	22	104	5	75-125	20

Table 2.4 (Cont.) Results of the MS/MSD Analysis for Metals in Soil  
 WA# 0-318 Range 17 (PNRR) Site  
 Results Reported As Received

Sample ID: 200R0D

Metal	Sample Conc mg/kg	MS Spike Added mg/kg	MS Conc mg/kg	MS % Rec	MSD Spike Added mg/kg	MSD Conc mg/kg	MDS % Rec	RPD	Recommended QC Limits %Rec	RPD
Antimony	U	9.9	2.53	26 *	10	2.89	29 *	12	75-125	20
Arsenic	2.56	9.9	12.1	96	10	12.2	96	0	75-125	20
Copper	4.51	9.9	14.9	105	10	15.1	106	1	75-125	20
Lead	19.3	9.9	29.8	106	10	30.1	108	2	75-125	20



Table 2.5 Results of the Blank Spike Analysis for Metals in Soil  
WA# 0-318 Range 17 (PNRR) Site

Metal	Spiked Conc. mg/kg	Sand Blank Conc. mg/kg	Recovered Conc. mg/kg	% Recovery	Recommended QC Limits % Recovery
Aluminum	100	17.5	115	98	75-125
Antimony	10	U	10	100	75-125
Arsenic	10	U	10.5	105	75-125
Barium	10	U	10.4	104	75-125
Beryllium	10	U	10.2	102	75-125
Cadmium	10	U	10.4	104	75-125
Calcium	100	U	101	101	75-125
Chromium	10	U	10.6	106	75-125
Cobalt	10	U	10.4	104	75-125
Copper	10	U	10.5	105	75-125
Iron 2599	100	2.76	105	102	75-125
Iron 2714	100	U	105	105	75-125
Lead	10	U	10.8	108	75-125
Magnesium	100	U	98.1	98	75-125
Manganese	10	U	10.4	104	75-125
Mercury	0.392	U	0.341	87	75-125
Nickel	10	U	10.6	106	75-125
Potassium	100	U	94.5	95	75-125
Selenium	10	0.94	11.2	103	75-125
Silver	10	U	9.91	99	75-125
Sodium	800	U	782	98	75-125
Thallium	10	U	11	110	75-125
Vanadium	10	U	10.3	103	75-125
Zinc	10	U	11.7	117	75-125

Table 2.6 Results of the LCS Analysis for Metals in Soil  
WA# 0-318 Range 17 (PNRR) Site

Metal	Date Analyzed	LCS Standard (ERA Lot# )	Conc. Recovered mg/kg	Certified Value mg/kg	PALs mg/kg	% Recovery
Aluminum	08/05/03	0888	5990	6340	760 - 9920	94
Antimony	08/05/03	0888	14.6	34	DL - 77.5	43
Arsenic	08/05/03	0888	179	192	152 - 232	93
Barium	08/05/03	0888	384	417	332 - 502	92
Beryllium	08/05/03	0888	91.3	99.9	79.2 -121	91
Cadmium	08/05/03	0888	112	125	101 - 149	90
Calcium	08/05/03	0888	3060	3370	2550 - 4190	91
Chromium	08/05/03	0888	116	133	103 - 163	87
Cobalt	08/05/03	0888	51.5	56.8	45.0 - 68.7	91
Copper	08/05/03	0888	87.1	93.9	74.4 - 113	93
Iron 2599	08/05/03	0888	10400	11600	5500 - 17700	90
Iron 2714	08/05/03	0888	10800	11600	5500 - 17700	93
Lead	08/05/03	0888	147	160	124 - 196	92
Magnesium	08/05/03	0888	1830	2000	1410 - 2590	91
Manganese	08/05/03	0888	343	320	242 - 398	107
Mercury	08/07/03	0888	22.4	24	15.8 - 32.2	93
Nickel	08/05/03	0888	158	174	136-211	91
Potassium	08/05/03	0888	1570	1890	1200 - 2580	83
Selenium	08/05/03	0888	91.9	97	69.6 - 124	95
Silver	08/05/03	0888	108	115	63.3 - 167	94
Sodium	08/05/03	0888	177	241	122 - 360	73
Thallium	08/05/03	0888	65.5	79.1	58.4 - 100	83
Vanadium	08/05/03	0888	84.9	92.7	64.9 - 121	92
Zinc	08/05/03	0888	224	246	189 - 303	91

CHAIN OF CUSTODY RECORD

No: 318-0001

REAC, Edison, NJ  
EPA Contract #:

Site #: 318  
Contact Name: Jen Badner  
Contact Phone:

Lab: REAC Laboratory  
Lab Contact: Jerry Ingram  
Lab Phone: 732-321-4200

7/30/03

Lab #	Sample #	Location	Analyses	Matrix	Collected	Numb Cont	Container	Preservative	MS/MSD
3074	318-0001	0150D	TAL metals, BNAs, % Moisture	Soil	7/29/2003	1	8 oz glass	4 C	
3075	318-0002	100R100D	TAL metals, BNAs, % Moisture	Soil	7/29/2003	1	8 oz glass	4 C	
3076	318-0003	100R50D	TAL metals, BNAs, % Moisture	Soil	7/29/2003	1	8 oz glass	4 C	
3077	318-0004	150R100D	TAL metals, BNAs, % Moisture	Soil	7/29/2003	1	8 oz glass	4 C	
3078	318-0005	150R50D	TAL metals, BNAs, % Moisture	Soil	7/29/2003	1	8 oz glass	4 C	Y
3079	318-0006	Reference	TAL metals, BNAs, % Moisture	Soil	7/29/2003	1	8 oz glass	4 C	

027

Special Instructions:	SAMPLES TRANSFERRED FROM CHAIN OF CUSTODY #
-----------------------	--

Items/Reason	Relinquished by	Date	Received by	Date	Time	Items/Reason	Relinquished By	Date	Received by	Date	Time
All/Analyses	Jenny Badner	7/30/03	Jerry Ingram	7/30/03	11:30	All/Analyses	Jerry Ingram	7/31/03	Jerry Ingram	7-30-03	1:25
All/Analyses	Jerry Ingram	7-31-03	Jerry Ingram	7/31/03	14:20	All/TAL	Jerry Ingram	8/4/03	Jerry Ingram	8-4-03	9:05

318-DAR-090903

REAC, Edison.  
 (732) 321-4200  
 EPA Contract 68-C99-223

**CHAIN OF CUS /Y RECORD**

Project Name: Range 17  
 Project Number: RI000318  
 LM Contact: J. Badner Phone: 732-444-4004

No: 15364  
 Sheet **01** of **01** (Do not copy)  
 (for addnl. samples use new form)

8/4/05

**Sample Identification**

**Analyses Requested**

REAC#	Sample No	Sampling Location	Matrix	Date Collected	# of Bottles	Container/Preservative	Pb	As	Cu	Sb
3080	Ø-Ø-Ø	ØØØ	Soil	28-Jul-03	1	XRF cap/wat	✓	✓	✓	✓
3081	Ø-15ØD	Ø-15ØD		↓						
3082	5ØL2ØØD	Ø5ØL2ØØD		29-July 03						
3083	1ØØL5ØD	1ØØLØ5ØD		↓						
3084	1ØØL1ØØD	1ØØL1ØØD		↓						
3085	1ØØR15ØD	1ØØR15ØD		28-JUL-03						
3086	15ØR5ØD	15ØRØ5ØD		↓						
3087	15ØR1ØØD	15ØR1ØØD		↓						
3088	2ØØRØD	2ØØRØØØD		29-Jul-03						
3089	25ØR5ØD	25ØRØ5ØD		↓						

028

**Matrix:**

- A- Air
- AT- Animal Tissue
- DL- Drum Liquids
- DS- Drum Solids
- GW- Groundwater
- O- Oil
- PR- Product
- PT- Plant Tissue
- PW- Potable Water
- S- Soil
- SD- Sediment
- SL- Sludge
- SW- Surface Water
- TX- TCLP Extract
- W- Water
- X- Other

**Special Instructions:**

**SAMPLES TRANSFERRED FROM  
 CHAIN OF CUSTODY #:**

318-DAR-090903

Items/Reason	Relinquished by	Date	Received by	Date	Time	Items/Reason	Relinquished by	Date	Received by	Date	Time
10/analysis	R. Kalinsky	8/4/03	J. Badner	8/4/03	1045	11/1001	J. Badner	8/4/03	Chasler	8/4/03	1105

ANALYTICAL REPORT

Prepared by  
Lockheed Martin, Inc.

Range 17 (PNRR) Site  
Laurel, MD

December, 2003

EPA Work Assignment No. 0-318  
LOCKHEED MARTIN Work Order No. R1A00318  
EPA Contract No. 68-C99-223

Submitted to  
D. Charters  
EPA-ERTC

Jennif Badner 12/3/03  
J. Badner Date  
Task Leader

Analysis by:  
REAC

Vinod Kansal 12/8/03  
V. Kansal Date  
Analytical Section Leader

Prepared by:  
A. LoSurdo

Dennis Miller 12/8/03  
D. Miller Date  
Project Manager

Reviewed by:  
J. Soroka

## Table of Contents

<u>Topic</u>		<u>Page Number</u>
Introduction		Page 1
Case Narrative		Page 1
Summary of Abbreviations		Page 2
<b>Section I</b>		
Analytical Procedure for Pesticides/PCBs in Soil		Page 3
Results of the Analysis for Pesticides/PCBs in Soil	Table 1.1	Page 5
<b>Section II</b>		
<b>QA/QC for Pesticides/PCBs</b>		Page 6
Results of the Surrogate Percent Recoveries for Pesticides/PCBs In Soil	Table 2.1	Page 7
Results of the MS/MSD Analysis for Pesticides/PCBs in Soil	Table 2.2	Page 8
<b>Section III</b>		
Chains of Custody		Page 9
Appendix A Data for Pesticides/PCBs in Soil		Page M 215 001
Appendix will be furnished on request.		

## Introduction

REAC, in response to WA # 0-318, provided analytical support for environmental samples collected at the Range 17 (PNRR) Site located in Laurel, MD as described in the following table. This support included the QA/QC, data review and the preparation of a report summarizing the analytical methods, results, and the QA/QC results.

The samples were treated with procedures consistent with those described in SOP # 1008.

COC #	Number of Samples	Sampling Date	Date Received	Matrix	Analysis	Laboratory	Data Package
06057	2	7/29/03	10/21/03	Soil	Pest/ PCB	REAC	M 215

## Case Narrative

The data in this report have been validated to two significant figures. Any other representation of the data is the responsibility of the user.

### Pesticides/PCBs in Soil Package M 215

The samples holding time was exceeded by 91 days. The non detected results for samples 318-0004 and 318-0005 should be regarded as unusable. The dieldrin result for sample 318-0004 should be considered estimated.

The acceptable QC limits for the percent difference were exceeded for the end of sequence (EOS) calibration check standard of 11/20/03 for p,p'-DDT (42%), endrin aldehyde (34%), endosulfan sulfate (33%) and TCMX (29%). No analytes were detected in the method blank, the only sample analyzed with the EOS; the data are not affected.

The acceptable QC limits for the percent difference were exceeded for the end of sequence calibration check standard of 11/21/03 (19:41) for p,p'-DDD (40%), p,p'-DDT (62%) and methoxychlor (50%). These compounds were not detected in the associated samples 318-0004 and 318-0005; the data are not affected.

## Summary of Abbreviations

AA	Atomic Absorption
B	The analyte was found in the blank
BFB	Bromofluorobenzene
C	Centigrade
cont.	Continued
D	(Surrogate Table) this value is from a diluted sample and was not calculated (Result Table) this result was obtained from a diluted sample
Dioxin and/or	
PCDD and PCDF	denotes Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans
CLP	Contract Laboratory Protocol
COC	Chain of Custody
CONC	Concentration
CRDL	Contract Required Detection Limit
CRQL	Contract Required Quantitation Limit
DFTPP	Decafluorotriphenylphosphine
DL	Detection Limit
E	The value is greater than the highest linear standard and is estimated
EMPC	Estimated maximum possible concentration
ICAP	Inductively Coupled Argon Plasma
ISTD	Internal Standard
J	The value is below the method detection limit and is estimated
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
MDL	Method Detection Limit
MI	Matrix Interference
MS (BS)	Matrix Spike (Blank Spike)
MSD (BSD)	Matrix Spike Duplicate (Blank Spike Duplicate)
MW	Molecular Weight
NA	either Not Applicable or Not Available
NC	Not Calculated
NR	Not Requested
NS	Not Spiked
% D	Percent Difference
% REC	Percent Recovery
PPB	Parts per billion
PPBV	Parts per billion by volume
PPMV	Parts per million by volume
PQL	Practical Quantitation Limit
QA/QC	Quality Assurance/Quality Control
QL	Quantitation Limit
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SIM	Selected Ion Monitoring
TCLP	Toxic Characteristic Leaching Procedure
U	Denotes not detected
W	Weathered analyte; Aroclor pattern displays a degradation of earlier eluting peaks
m <sup>3</sup>	cubic meter      kg                      kilogram              µg                      microgram
L	liter                      g                      gram                      pg                      picogram
mL	milliliter              mg                      milligram              ng                      nanogram
µL	microliter
*	denotes a value that exceeds the acceptable QC limit Abbreviations that are specific to a particular table are explained in footnotes on that table

Revision 7/26/01



## Analytical Procedure for Pesticides/PCBs in Soil

### Extraction Procedure

The soil samples were extracted by the Soxhlet method. A thirty gram aliquot was spiked with a surrogate solution consisting of tetrachloro-m-xylene and decachlorobiphenyl, mixed with 30 g anhydrous sodium sulfate and extracted for 16 hours with 300 mL of acetone/hexane (1:1). The extract was concentrated to 5 mL.

### Gas Chromatographic Analysis

The extract was analyzed for pesticide/PCBs using simultaneous dual column injections. The analysis was done on an HP 6890 GC/ECD system equipped with an HP 6890 automatic sampler. The systems were controlled with an HP-ChemStation. The following conditions were employed:

First Column	Rtx-CLPesticides II, 30 meter, 0.32 mm fused silica capillary, 0.25 µm film thickness
Second Column	Rtx-CLPesticides I, 30 meter, 0.32 mm fused silica capillary, 0.50 µm film thickness
Injector Temperature	250°C
Detector Temperature	300°C
Temperature Program	120°C for 1 minute 9°C/min to 285°C, hold for 10 minutes
Injection Volume	1 µL

The gas chromatographs were calibrated using 5 pesticide standards at 20, 50, 100, 200, and 500 µg/L. The results from each mixture were used to calculate the response factor (RF) of each analyte and the average RF was used to calculate the concentration of pesticide in the sample. Quantification was based on the Rtx-CLPesticides II column (signal 1) and the identity of the analyte was confirmed using the Rtx-CLPesticides I column (signal 2). A fingerprint chromatogram was run using each of the seven Aroclor mixtures and toxaphene; the calibration curves were run only if a particular PCB or toxaphene were found in the sample.

The pesticide results, listed in Table 1.1, are calculated by using the following formula:

$$C_u = \frac{(DF)(A_u)(V_t)}{(RF_{ave})(V_i)(W)(D)}$$

where;

$C_u$	= Concentration of analyte (µg/kg)
DF	= Dilution Factor
$A_u$	= Area or peak height
$V_t$	= Volume of sample (mL)
$RF_{ave}$	= Average response factor
$V_i$	= Volume of extract injected (µL)
W	= Weight of sample (g)
D	= Decimal percent solids

Response Factor calculation:

The response factor for each specific analyte is calculated using the peak area (peak height) from the continuing calibration check as follows:

$$RF = \frac{A_u}{\text{total pg injected}}$$

where;

$A_u$  = Area or peak height

and

$$RF_{ave} = \frac{RF_1 + \dots + RF_n}{n}$$

where;

n = number of samples.

Revision 3/9/00

Table 1.1 Results of the Analysis for Pesticide/PCBs in Soil  
 WA# 0-318 Range 17 (PNRR) Site  
 Based on Dry Weight

Client ID	SBLK110403		318-0004		318-0005	
Location	-		150R100D		150R50D	
Percent Solid	100		88		86	
Analyte	Conc. µg/kg	MDL µg/kg	Conc. µg/kg	MDL µg/kg	Conc. µg/kg	MDL µg/kg
a-BHC	U	3.3	U	3.8	U	3.9
g-BHC	U	3.3	U	3.8	U	3.9
b-BHC	U	3.3	U	3.8	U	3.9
Heptachlor	U	3.3	U	3.8	U	3.9
d-BHC	U	3.3	U	3.8	U	3.9
Aldrin	U	3.3	U	3.8	U	3.9
Heptachlor Epoxide	U	3.3	U	3.8	U	3.9
g-Chlordane	U	3.3	U	3.8	U	3.9
a-Chlordane	U	3.3	U	3.8	U	3.9
Endosulfan (I)	U	3.3	U	3.8	U	3.9
p,p'-D D E	U	3.3	0.9 J	3.8	U	3.9
Dieldrin	U	3.3	U	3.8	U	3.9
Endrin	U	3.3	U	3.8	U	3.9
p,p'-D D D	U	3.3	U	3.8	U	3.9
Endosulfan (II)	U	3.3	U	3.8	U	3.9
p,p'-D D T	U	3.3	U	15	U	15
Endrin Aldehyde	U	3.3	U	3.8	U	3.9
Endosulfan Sulfate	U	3.3	U	3.8	U	3.9
Methoxychlor	U	3.3	U	15	U	15
Endrin Ketone	U	3.3	U	3.8	U	3.9
Toxaphene	U	83	U	95	U	97
Aroclor 1016	U	42	U	47	U	48
Aroclor 1221	U	83	U	95	U	97
Aroclor 1232	U	42	U	47	U	48
Aroclor 1242	U	42	U	47	U	48
Aroclor 1248	U	42	U	47	U	48
Aroclor 1254	U	42	U	47	U	48
Aroclor 1260	U	42	U	47	U	48
Aroclor 1268	U	42	U	47	U	48

## QA/QC for Pesticides/PCBs

### Results of the Surrogate Percent Recoveries for Pesticides in Soil

Each sample was spiked with a solution of tetrachloro-m-xylene and decachlorobiphenyl as surrogates. The percent recoveries ranged from 65 to 114 and are listed in Table 2.1. All ten values were within the acceptable QC limits.

### Results of the MS/MSD Analysis for Pesticides in Soil

Samples 318-0004 was chosen for the matrix spike/matrix spike duplicate (MS/MSD) analyses. The percent recoveries ranged from 69 to 103 and are listed in Table 2.2. All twelve values were within the acceptable QC limits. The relative percent differences (RPDs), also listed in Table 2.2, ranged from zero (0) to 9 and all six values were within the acceptable QC limits.

Table 2.1 Results of the Surrogate Percent Recoveries for Pesticide/PCBs in Soil  
WA# 0-318 Range 17 (PNRR) Site

Sample ID	Percent Recovery	
	TCMX	DCBP
SBLK110403	65	69
318-0004	84	114
318-0004MS	78	111
318-0004MSD	75	109
318-0005	82	124

TCMX denotes Tetrachloro-m-xylene  
DCBP denotes Decachlorobiphenyl

	Advisory
	QC
	Limits
TCMX	30-150
DCBP	30-150

Table 2.2 Results of the MS/MSD Analysis for Pesticide/PCB in Soil  
 WA# 0-318 Range 17 (PNRR) Site  
 Results Based on Dry Weight

Sample ID: 318-0004

Compound	Sample Conc µg/kg	MS			MSD			RPD	Advisory QC Limits	
		Spike Added µg/kg	MS Conc µg/kg	MS % Rec	Spike Added µg/kg	MSD Conc µg/kg	MSD % Rec		% Rec	RPD
g-BHC	U	23.7	16.9	71	23.7	16.3	69	3	46-127	50
Heptachlor	U	23.7	18.5	78	23.7	17.0	71	9	35-130	31
Aldrin	U	23.7	22.9	96	23.7	22.7	96	0	34-132	43
Dieldrin	U	47.5	48.0	101	47.5	48.6	103	2	31-134	38
Endrin	U	47.5	46.5	98	47.5	46.4	98	0	42-139	45
p,p'-DDT	U	47.5	36.4	77	47.5	36.0	76	1	23-134	50



# Total Organic Content

Method: Loss on Ignition AASHTO T-267-86

Site Name	Range 17
	R1A00318
Date Performed	10/07/03
Technician	Chris French

	450	
	240	

318-0001	63.03g	60.74g	29.22g	6.77%
318-0002	51.49g	50.95g	22.17g	1.84%
318-0003	57.52g	55.00g	29.72g	9.06%
318-0004	60.32g	59.38g	30.16g	3.12%
318-0005	61.23g	60.29g	30.33g	3.04%
318-0006	48.97g	47.52g	22.61g	5.21%

## Definitions:

A = Mass of Crucible with Moisture Free Sample

B = Mass of Crucible with Furnace Treated Sample

C = Mass of Crucible

$$TOC = (A-B)/(A-C) \times 100$$

Prepared by Lockheed Martin - REAC

Technician Signature: Chris French

Date: 11/18/03



**PARTICLE SIZE ANALYSIS**

ASTM Method D-420

Technician's name:	Chris French
Date:	04/14/03
Site name:	Range 17
Sample No.:	318-0001

Mass of sample split on No. 10 sieve (g):	598.41
Mass retained on No. 10 sieve (g):	0
Mass passing No. 10 sieve (g):	
<b>Percent passing No. 10 sieve (g):</b>	

Mass used in Hydrometer test (g):	104
Specific gravity of soil:	2.65
Correction factor:	1
<b>Corrected mass of soil used in hydrometer test (g):</b>	

Wet mass of hygroscopic test sample (g):	15
Oven-dry mass of test sample (g):	14.12
Percent hygroscopic moisture:	
<b>Corrected mass of soil used in hydrometer test (g):</b>	

Hydrometer type:	
Hydrometer correction:	0.003
Average temperature (C):	20
Temperature correction factor:	0
<b>Total Hydrometer correction:</b>	

K:	0.01365
W:	
F:	

Results 318-0001

Sieve Analysis

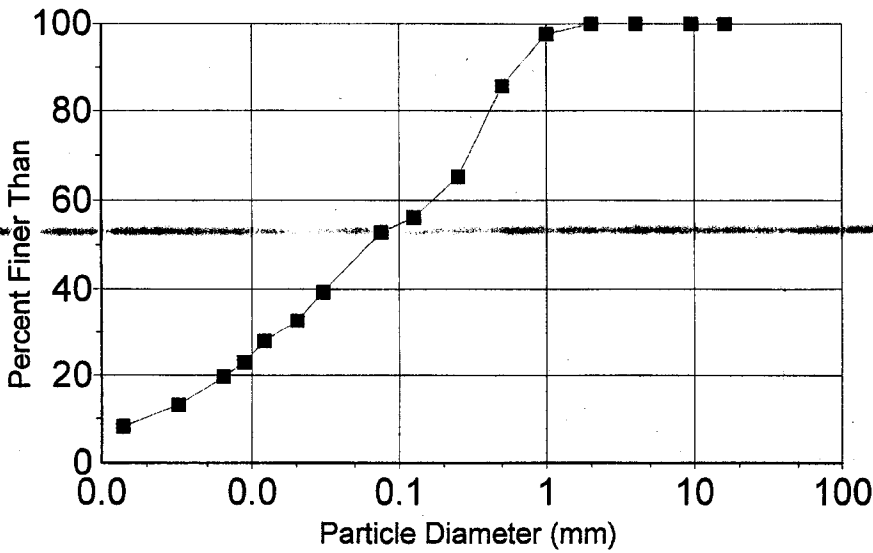
Sieve Size (mm)	Mass Retained (g)	Hygroscopic Corrected Mass Retained (g)	Mass Retained Corrected for F (g)	Mass Passing (g)	Percent Finer Than
16	0.00	0.00	0.00	97.90	100.00
9.5	0.00	0.00	0.00	97.90	100.00
	0.00				100.00
2	0.00	0.00	0.00	97.90	100.00

Hydrometer Test Analysis

Time (Min)	Hydrometer Reading	Corrected Reading	Length, L (cm)	Diameter (mm)	Percent Suspended
1	1.027	0.22	10	0.075	39.37
2	1.023	0.18	10	0.054	32.81
5	1.020	0.15	10	0.038	27.89
10	1.017	0.12	10	0.027	22.97
15	1.015	0.10	10	0.020	19.69
20	1.011	0.07	10	0.015	13.12
30	1.008	0.05	10	0.011	8.20

Sieve Analysis <No.10

Sieve Size	Mass Retained (g)	Percent Retained
2.38		
11.71		
19.97		
8.85		
3.24		



ASTM Grain Size	Particle Diameter (mm)	Percent Finer
Fine Gravel	4.75	100.00
Course Sand	0.425	100.00
Medium Sand	0.250	97.57
Fine Sand	0.075	85.61
	0.054	65.21
	0.038	56.17
	0.027	52.86
Silt	0.0304	39.37
	0.0203	32.81
	0.0121	27.89
	0.0088	22.97
Clay	0.0064	19.69
	0.0033	13.12
	0.0014	8.20

**PARTICLE SIZE ANALYSIS**

ASTM Method D-420

Technician's name:

Chris French

Date:

09/23/03

Site name:

Range 17

Sample No.:

318-0002

Mass of sample split on No. 10 sieve (g):  
Mass retained on No. 10 sieve (g):  
Mass passing No. 10 sieve (g):  
**Percent passing No. 10 sieve (g):**

689.19

32.57

Mass used in Hydrometer test (g):  
Specific gravity of soil:  
Correction factor:  
**Corrected mass of soil used  
in hydrometer test (g):**

104

2.65

1

Wet mass of hygroscopic test sample (g):  
Oven-dry mass of test sample (g):  
Percent hygroscopic moisture:  
**Corrected mass of soil  
used in hydrometer test (g):**

15

14.34

Hydrometer type:  
Hydrometer correction:  
Average temperature (C):  
Temperature correction factor:  
**Total Hydrometer correction:**

0.003

20

0

K: 0.01365  
W:  
F:

Results: 318-0002

Sieve Analysis

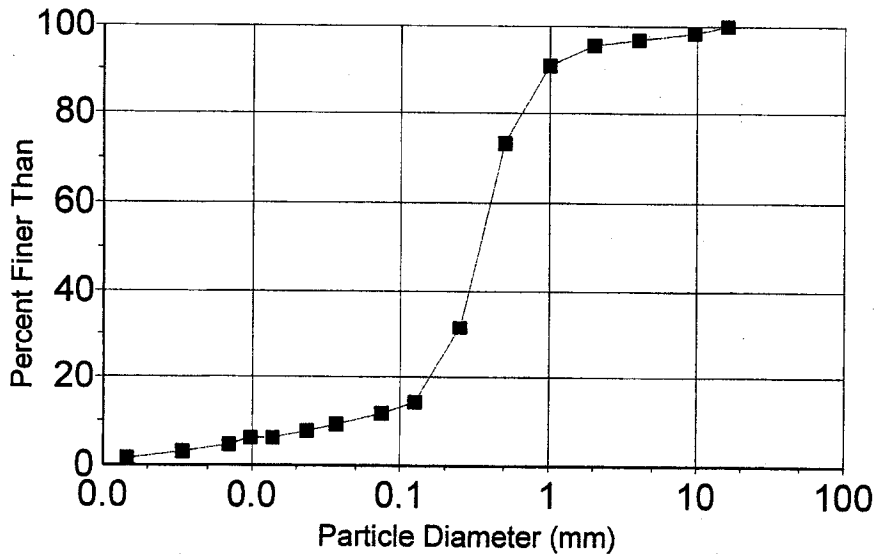
Sieve Size (mm)	Mass Retained (g)	Hygroscopic Corrected Mass Retained (g)	Mass Retained Corrected for F (g)	Mass Passing (g)	Percent Finer Than
16	0.00	0.00	0.00	100.00	100.00
9.5	11.92	11.40	1.78	102.64	98.35
4.75	11.15				
2	9.50	3.08	3.89	99.64	95.48

Hydrometer Test Analysis

Time (min)	Hydrometer Reading	Corrected Reading	Length L (cm)	Diameter (mm)	Percent Standard
	1.009				
	1.008				
	1.007				
30	1.007				
60	1.006				
90	1.005				
120	1.004				

Sieve Analysis <No.10

Sieve Size (mm)	Mass Retained (g)	Mass Retained Corrected for F (g)	Percent Finer Than
4.75	4.69		
2.0	18.28		
0.85	43.53		
0.425	18.04		
0.25	2.68		



ASTM Grain Size	Mass Retained (g)	Percent Finer Than (%)
Fine Gravel		100.00
Course Sand		98.35
Medium Sand		96.80
Fine Sand		95.48
Silt	0.0370	92.30
	0.0236	77.00
	0.0138	61.60
	0.0097	61.60
	0.0069	46.20
Clay	0.0034	31.55
	0.0014	1.54

**PARTICLE SIZE ANALYSIS**

ASTM Method D-420

Technician's name:

Chris French

Date:

09/23/03

Site name:

Range 17

Sample No.:

318-0003

Mass of sample split on No. 10 sieve (g):

398.26

Mass retained on No. 10 sieve (g):

6.89

Mass passing No. 10 sieve (g):

Percent passing No. 10 sieve (g):

Mass used in Hydrometer test (g):

104

Specific gravity of soil:

2.65

Correction factor:

1

Corrected mass of soil used  
in hydrometer test (g):

Wet mass of hygroscopic test sample (g):

15

Oven-dry mass of test sample (g):

13.99

Percent hygroscopic moisture:

Corrected mass of soil

used in hydrometer test (g):

Hydrometer type:

Hydrometer correction:

0.003

Average temperature (C):

20

Temperature correction factor:

0

Total Hydrometer correction:

K:

0.01365

W:

F:

Results: 318-0003

Sieve Analysis

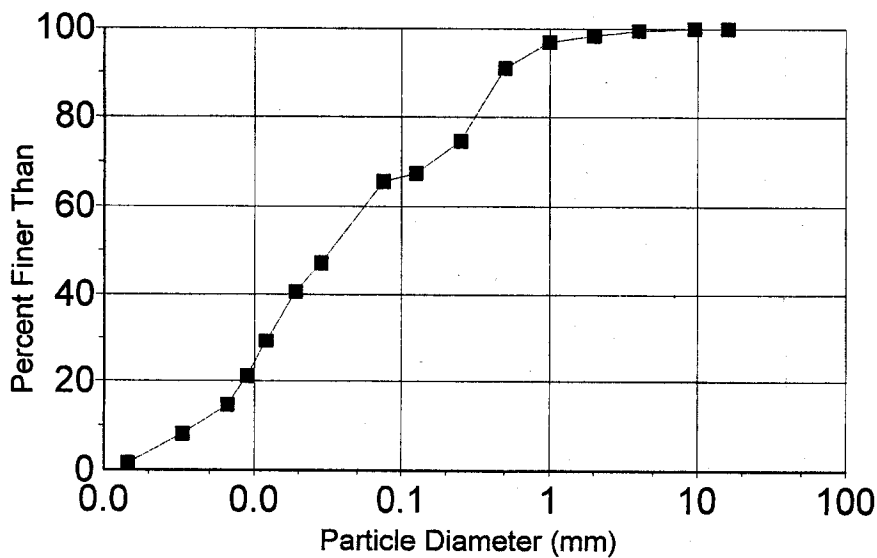
Sieve Size (mm)	Mass Retained (g)	Hygroscopic Corrected Mass Retained (g)	Mass Retained Corrected for F (g)	Mass Passing (g)	Percent Finer Than
16	0.00	0.00	0.00	98.70	100.00
9.5	0.00	0.00	0.00	98.70	100.00
	2.07				
2	4.82	4.50		97.71	98.39

Hydrometer Test Analysis

Time (Minutes)	Hydrometer Reading	Corrected Reading	Length L (cm)	Diameter (mm)	Percent Suspended
	1.032				
	1.028				
	1.021				
30	1.016				
60	1.012				
150	1.008				
300	1.004				

Sieve Analysis <No.10

Size (mm)	Mass Passing (g)	Percent Finer Than
	1.35	
	5.86	
	16.08	
	7.12	
	1.83	



ASTM Grain Size	Particle Diameter (mm)	Percent Finer Than (%)
Fine Gravel		100.00
Course Sand		99.52
Medium Sand		98.39
Fine Sand		90.97
Silt	0.0283	47.19
	0.0190	40.68
	0.0120	29.29
	0.0089	21.15
	0.0066	14.64
Clay	0.0033	8.14
	0.0014	1.63

**PARTICLE SIZE ANALYSIS**

ASTM Method D-420

Technician's name:

Chris French

Date:

09/23/03

Site name:

Range 17

Sample No.:

318-0004

Mass of sample split on No. 10 sieve (g):

649.47

Mass retained on No. 10 sieve (g):

73.82

Mass passing No. 10 sieve (g):

Percent passing No. 10 sieve (g):

Mass used in Hydrometer test (g):

104

Specific gravity of soil:

2.65

Correction factor:

1

Corrected mass of soil used  
in hydrometer test (g):

Wet mass of hygroscopic test sample (g):

15

Oven-dry mass of test sample (g):

14.08

Percent hygroscopic moisture:

Corrected mass of soil

used in hydrometer test (g):

Hydrometer type:

Hydrometer correction:

0.003

Average temperature (C):

20

Temperature correction factor:

0

Total Hydrometer correction:

K:

0.01365

W:

F:

Results 318-0004

Sieve Analysis

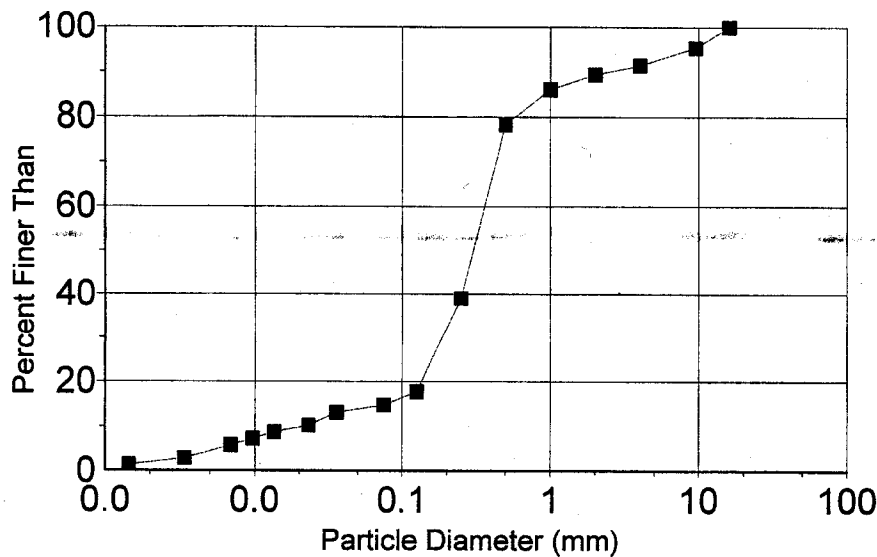
Sieve Size (mm)	Mass Retained (g)	Hygroscopic Corrected Mass Retained (g)	Mass Retained Corrected for F (g)	Mass Passing (g)	Percent Finer Than
16	0.00	0.00	0.00	110.14	100.00
9.5	32.38	30.39	5.15	104.99	95.32
	27.49				91.35
2	13.95	13.09	2.22	98.99	89.33

Hydrometer Test Analysis

Time (min)	Reading	Corrected Reading	Length (cm)	Diameter (mm)	Percent Suspended
	1.012				
	1.010				
	1.009				
	1.008				
	1.007				
	1.005				
	1.004				

Sieve Analysis <No.10

	2.90
	8.49
	43.17
	23.51
	3.28



ASTM Grain Size	ASTM No.	Percent
Fine Gravel		100.00
Course Sand		95.32
Medium Sand		91.35
Fine Sand		89.33
Silt	0.0360	13.12
	0.0232	10.21
	0.0135	8.75
	0.0096	7.29
	0.0069	5.83
Clay	0.0034	2.92
	0.0014	1.46



**PARTICLE SIZE ANALYSIS**

ASTM Method D-420

Technician's name: 

Chris French
--------------

  
Date: 

09/23/03
----------

  
Site name: 

Range 17
----------

  
Sample No.: 

318-0005
----------



Mass of sample split on No. 10 sieve (g): 

715.37
--------

  
Mass retained on No. 10 sieve (g): 

53.07
-------

  
Mass passing No. 10 sieve (g): 

--

  
Percent passing No. 10 sieve (g): 

--

Mass used in Hydrometer test (g): 

104
-----

  
Specific gravity of soil: 

2.65
------

  
Correction factor: 

1
---

  
Corrected mass of soil used  
in hydrometer test (g): 

--



Wet mass of hygroscopic test sample (g): 

15
----

  
Oven-dry mass of test sample (g): 

14.23
-------

  
Percent hygroscopic moisture: 

--

  
Corrected mass of soil  
used in hydrometer test (g): 

--



Hydrometer type: 

--

  
Hydrometer correction: 

0.003
-------

  
Average temperature (C): 

20
----

  
Temperature correction factor: 

0
---

  
Total Hydrometer correction: 

--



K: 

0.01365
---------

  
W: 

--

  
F: 

--

Results 318-0005

Sieve Analysis

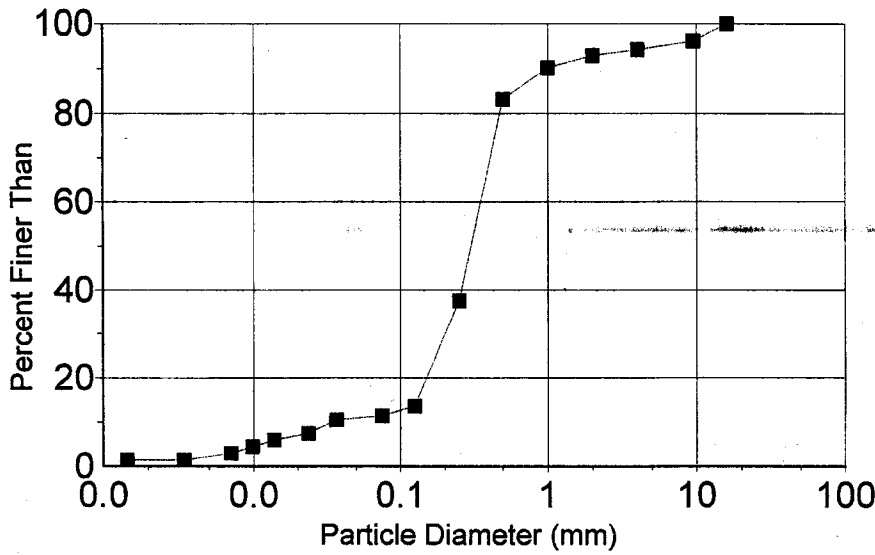
Sieve Size (mm)	Mass Retained (g)	Hygroscopic Corrected Mass Retained (g)	Mass Retained Corrected for F (g)	Mass Passing (g)	Percent Finer Than
16	0.00	0.00	0.00	106.57	100.00
9.5	28.96	27.47	4.09	102.47	96.16
	14.42	13.83	0.59	101.88	94.25
2	9.69	9.19	1.37	99.07	92.96

Hydrometer Test Analysis

Time (min)	Specific Gravity	Corrected Specific Gravity	Particle Size (mm)	Percent Finer Than
	1.010			
	1.008			
	1.007			
	1.006			
	1.005			
	1.004			
	1.004			

Sieve Analysis <No.10

	2.53
	7.50
	48.60
	25.47
	2.33



ASTM Grain Size	Percent Finer Than (%)	
Fine Gravel	100.00	
Course Sand	96.16	
Medium Sand	94.25	
Fine Sand	92.96	
Silt	0.0367	10.55
	0.0236	7.54
	0.0138	6.03
	0.0098	4.52
	0.0070	3.01
Clay	0.0035	1.51
	0.0014	1.51

**PARTICLE SIZE ANALYSIS**

ASTM Method D-420

Technician's name:

Chris French

Date:

09/23/03

Site name:

Range 17

Sample No.:

318-0006

Mass of sample split on No. 10 sieve (g):  
Mass retained on No. 10 sieve (g):  
Mass passing No. 10 sieve (g):  
Percent passing No. 10 sieve (g):

605.03
5.29

Mass used in Hydrometer test (g):  
Specific gravity of soil:  
Correction factor:  
Corrected mass of soil used  
in hydrometer test (g):

104
2.65
1

Wet mass of hygroscopic test sample (g):  
Oven-dry mass of test sample (g):  
Percent hygroscopic moisture:  
Corrected mass of soil  
used in hydrometer test (g):

15
13.98

Hydrometer type:  
Hydrometer correction:  
Average temperature (C):  
Temperature correction factor:  
Total Hydrometer correction:

0.003
20
0

K: 0.01365  
W:  
F:

0.01365

Results: 318-0006

Sieve Analysis

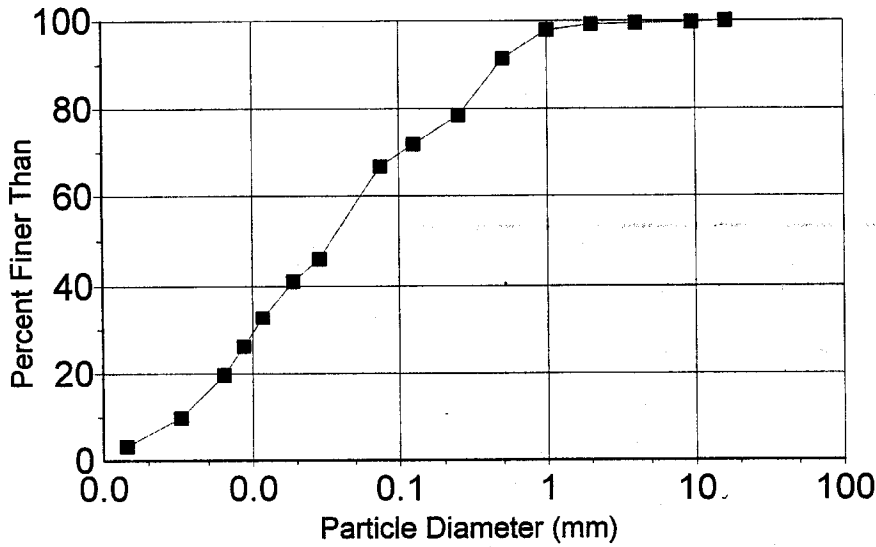
Sieve Size (mm)	Mass Retained (g)	Hygroscopic Corrected Mass Retained (g)	Mass Retained Corrected for F (g)	Mass Passing (g)	Percent Finer Than
16	0.00	0.00	0.00	97.78	100.00
9.5	1.96	1.86	0.30	97.49	99.70
	1.44	1.34	1.04	97.27	99.48
2	1.89	1.76	1.28	96.99	99.19

Hydrometer Test Analysis

Time (min)	Hydrometer Reading	Corrected Reading	Specific Gravity (G)	Diameter (mm)	Percent Suspended
	1.031				100.00
	1.028				99.70
	1.023				99.48
30	1.019				97.91
60	1.015				91.40
150	1.009				66.84
300	1.005				45.99

Sieve Analysis <No.10

	1.19
	6.36
	12.64
	6.43
	4.95



ASTM Grain Size	Percent Finer (%)	Percent Finer (%)
Fine Gravel		100.00
Course Sand		99.70
Medium Sand		99.48
Fine Sand		97.91
		91.40
		78.48
		71.90
		66.84
Silt	0.0288	45.99
	0.0190	41.06
	0.0117	32.85
	0.0087	26.28
	0.0064	19.71
Clay	0.0033	9.85
	0.0014	3.28

Appendix D  
Toxicity Evaluation Report  
Range 17 –Patuxent Research Refuge  
Laurel, MD  
March 2004

# **Aqua Survey, Inc.**

## **FINAL REPORT**

### **THE 28 DAY TOXICITY AND BIOACCUMULATION ASSAY USING THE EARTHWORM *EISENIA FOETIDA***

**ASI PROJECT NO.: 23-144**

**REAC PROJECT NO.: 0-318**

**October 21, 2003**

*469 Point Breeze Road  
Flemington, NJ 08822*

*Phone: 908/788-8700  
Fax: 908/788-9165  
mail@aquasurvey.com  
aquasurvey.com*



**THE 28 DAY TOXICITY AND  
BIOACCUMULATION ASSAY USING  
THE EARTHWORM *EISENIA FOETIDA***

**DATA REQUIREMENT**

**USEPA 600/3-88/1029 and ASTM E1676-97**

**AUTHOR**

**York Terrell**

**STUDY COMPLETION DATE**

**October 21, 2003**

**PERFORMING LABORATORY**

**Aqua Survey, Inc.  
469 Point Breeze Road  
Flemington, New Jersey 08822**

**SPONSOR**

**Lockheed Martin/REAC  
2890 Woodbridge Avenue  
Edison, NJ 08837**

**LABORATORY PROJECT NO.**

**ASI: 23-144**

**REAC: 0-318**

## TABLE OF CONTENTS

<u>Section</u>	<u>Page</u>
<b>I. EXECUTIVE SUMMARY.....</b>	<b>5</b>
<b>II. INTRODUCTION.....</b>	<b>6</b>
<b>III. TEST ADMINISTRATION.....</b>	<b>6</b>
A. Sponsor .....	6
B. Testing Facility .....	6
C. Dates of Experiment .....	6
D. Study Participants .....	6
<b>IV. TEST AND CONTROL SUBSTANCES .....</b>	<b>7</b>
A. Test Substance .....	7
B. Control Substance .....	7
<b>V. MATERIALS AND METHODS .....</b>	<b>7</b>
A. Test System .....	7
B. Source of Organisms.....	7
C. Hydration Water.....	8
D. Acclimation Procedure.....	8
E. Diet.....	8
F. Characterization of Age and Size .....	8
G. Collection of Organisms for Testing.....	8
H. Apparatus and Test Conditions.....	8
I. Preparation of Test Solution .....	8
J. Test Procedures.....	9
K. Data Analysis .....	9
<b>VI. TEST RESULTS .....</b>	<b>9</b>
<b>VII. TEST VALIDITY .....</b>	<b>9</b>
<b>VIII. DISCUSSION .....</b>	<b>10</b>
<b>IX. REFERENCES.....</b>	<b>10</b>
<b>SIGNATURE PAGE.....</b>	<b>11</b>



<u>Tables</u>	<u>Page</u>
1. Survival .....	12
2. Weight Loss/Gain .....	13
3. Soil pH .....	14
4. Soil Hydration.....	15

<u>Appendices</u>	<u>Page</u>
A. Chain of Custody .....	A
B. Statistical Analysis for Mortality .....	B
C. Statistical Analysis for Weight .....	C
D. Raw Data.....	D
E. Standard Reference Toxicant.....	E
F. Continuous Temperature Recording.....	F

## **THE 28 DAY TOXICITY AND BIOACCUMULATION ASSAY USING THE EARTHWORM *EISENIA FOETIDA***

### **I. EXECUTIVE SUMMARY**

Aqua Survey, Inc. (ASI) conducted a 28-day toxicity and bioaccumulation assay on six soil samples supplied by Lockheed Marin/REAC. The soil samples were identified as samples No. 318-0001, 318-0002, 318-0003, 318-0004, 318-0005 and 318-0006 (location 0150D, 100R100D, 100R50D, 150R100D, 150R50D and Reference, respectively). In addition, two control soil samples were used. One control was ASTM artificial soil while the second control was a natural soil believed to be free of contamination. A total of 80 earthworms were exposed to each soil sample. Tests were performed in replicates of four (20 organisms per replicate). Each replicate exposure vessel contained 400 grams of dry weight soil.

Earthworms used for this test were mature adults with an average weight of approximately 300 mg each. The test temperature was  $23 \pm 1$  °C. Test illumination was continuous, fluorescent, 50 to 100 ft-candles. The worms were weighed at the beginning and end of the test. Observation for mortality was made on day 14 and 28. After 28 days, mortality and weight loss/gain of the test exposures to the laboratory controls using an appropriate statistical method (i.e., analysis of variance or t-Test) was measured. Test exposures, where no statistical difference for mortality is observed, may be analyzed for bioaccumulation of soil contaminant and submitted under separate covers.

After 28 days a statistical difference was observed for survival for all test soil with the exception of 318-0006 when compared to the controls. All test soil exhibited a statistical difference for weight loss when compared to the controls.

A previous test was initiated on August 8, 2003 and was terminated on August 22, 2003 due to mortality observed in the controls after 14-days. In this test greater than 10% mortality was observed in both the ASTM artificial soil control and the natural soil control (24 and 33%, respectively). This mortality may have been the result of poor organism health.

## II. INTRODUCTION

Earthworms were exposed to soil samples in a 28-day, static, terrestrial effect and bioaccumulation test in order to permit a more accurate and complete assessment of its environmental impact. Exposures were prepared by the addition of appropriate aliquots of soil to replicate test vessels. The earthworm, *Eisenia foetida* was chosen for this test based on its ecological importance as a representative terrestrial organism, which can be reared within the laboratory. The objectives of the test were:

- 1) To determine if the soils would effect survival;
- 2) To determine if the soils would effect growth; and
- 3) If appropriate, determine if the contaminants of potential concern would accumulate in worm tissue.

The results of this test may be used to determine the likelihood of an adverse effect of the contaminants to the terrestrial environment.

## III. TEST ADMINISTRATION

### A. Sponsor

Lockheed Marin/REAC  
2890 Woodbridge Avenue  
Edison, NJ 08837

### B. Testing Facility

Aqua Survey, Inc.  
469 Point Breeze Road  
Flemington, NJ 08822

### C. Dates of Experiment

Date of Study Initiation:	July 31, 2003
Date of Soil Exposure:	August 8-22, 2003 and August 28-September 26, 2003

### D. Study Participants

Tom Dolce	Laboratory Manager
York Terrell	Study Director
Jon Doi, Ph.D.	Executive Vice President, Laboratory Operations

#### IV. TEST AND CONTROL SUBSTANCES

##### A. Test Substance

Test matrix used for this test was soil and was supplied by the sponsor. The samples were received on July 30, 2003 and identified as samples No. 318-0001, 318-0002, 318-0003, 318-0004, 318-0005 and 318-0006. The chain of custody record as well as the sample receiving form and any other sample information is presented in Appendix A.

##### B. Control Substance(s)

Control Soil: Two control soils were required for this test.

1. The first control soil was ASTM artificial soil prepared from the following constituents on a dry weight basis:
  - a. Sphagnum Peat Moss 10%
  - b. EPK Kaolin Clay 20%
  - c. Silica Sand (grade 70) 70%
  - d. Calcium Carbonate 0.4%
2. The second control was a natural soil believed to be free of contamination. The soil was obtained from Farm Ubel's Stand, Flemington, NJ. The soil was identified as Soil King Leaf Soil, supplied by BANFF Products, Inc. Barrington, NJ 08007.

##### C. Reference Toxicant

A standard reference toxicant using KCl was conducted concurrent with the test to see if the organisms would respond to a toxicant in the expected manner.

#### V. MATERIALS AND METHODS

##### A. Test System

The earthworm, *Eisenia foetida*, was used for this test.

##### B. Source of Organism

Earthworms used in this test were purchased from Aquatic Research Organisms, One Lafayette Road, Hampton, NH 03842. The organisms were received by this laboratory on August 27, 2003 and assigned culture Log No. 23-0134. The age of the organisms was listed as adults.

C. Hydration Water

Water used for soil hydration was reagent grade (deionized) water prepared by passing well water through a mixed-bed deionization system.

D. Acclimation Procedure

The test organisms were held at test condition for 24-hours prior to test initiation.

E. Diet

The organisms were not fed during acclimation or testing.

F. Characterization of Age and Size

No attempt was made to characterize the organisms' exact age. The organisms were mature fully clitella. The organisms were weighed prior to test initiation and determined to be approximately 300 mg each.

G. Collection of Organisms for Testing

Worms of relatively uniform size were collected and transferred to test vessels. Sequential randomization was accomplished by allocating to each container no more than 20 percent of any one set of test organisms at a time. The worms were depurated for 24-hours prior to test initiation, and the biomass added to each test vessel was recorded. All worms were rinsed of debris prior to weighing.

H. Apparatus and Test Conditions

This test was performed in 2 L glass jars, each containing 400 grams of dry weight soil. The photoperiod was continuous, fluorescent, 50 to 100 ft-candles. Test temperature was at  $23 \pm 1$  °C. The soil was hydrated to a level approximating the appearance of the control.

I. Preparation of Soil for Testing

Prior to test initiation the moisture fraction of the soils was determined. Four hundred (400) grams dry weight soil was added to each test vessel using the following formula: Wet sample weight (g) = [400 g dry sample] + [moisture fraction x 400 g dry weight sample]. The soil was hydrated to a level approximating the appearance of the control using deionized water. Tests were prepared in replicates of four. Replicate controls were also prepared.

## J. Test Procedures

The procedures used in this test were based on accepted methodologies<sup>1-3</sup>. Test vessels were weighted periodically for water loss. Daily observations for mortality, appearance and behavior were made when possible. Mortality counts were made at day 14 and test termination. The temperature was measured continuously (hourly) in a surrogate vessel during the entire study using a Ryan RL100 Temperature Recorder. The pH of the test and control soils was measured prior to test initiation.

The test was started when 20 test organisms were placed into each of four replicate exposure vessels for each test soil and control. All exposures were 28 day static.

A standard reference toxicant using KCl was conducted concurrent with this test to see if the organisms would respond to a toxicant in the expected manner. Test concentrations of 1250, 2500, 5000, 10000 and 20000 ppm were prepared by adding appropriate volumes of a KCl stock solution to 200 grams dry weight ASTM artificial soil. The solution was added as part of the total of water used to hydrate the soil. This test was started when 10 organisms were placed in each of two replicate test vessels. This expose was 14 day static.

## K. Data Analysis

The TOXSTAT computer program was used to compare mortality and weight loss/gain of test exposures to laboratory controls.

## VI. TEST RESULTS

Mortality is presented in Table 1. Weight gain/loss is presented in Table 2. Soil pH is presented in Table 3. Soil hydration is presented in Table 4. Statistical analysis for mortality is presented in Appendix B. Statistical analysis for weight gain/loss is presented in Appendix C. Raw data is presented in Appendix D.

## VII. TEST VALIDITY

The following criteria for a valid test were met during the study:

- A. The control effect was not greater than 10%.
- B. No abnormal occurrences (i.e., laboratory accidents) that might have influenced the outcome of the test were noted.

## VIII. DISCUSSION

During this 28-day test no noteworthy deviations from the protocol were observed. Control survival in the ASTM artificial soil and natural soil was greater than 90%. The organisms responded in the expected manner when exposed to a standard reference toxicant (see Appendix E). Statistical difference was observed for survival in all test soil with the exception of 318-0006 when compared to the controls. All test organisms exhibited a statistical difference for weight loss when compared to the controls.

It should be noted that this test was initiated on August 28, 2003 and terminated on September 26, 2003. It should also be noted that a previous test was initiated on August 8, 2003 and was terminated on August 22, 2003 due to mortality observed in the controls after 14-days. In this test greater than 10% mortality was observed in both the ASTM artificial soil control and the natural soil control (24 and 33%, respectively). This mortality may have been the result of poor organism health. However, a standard reference toxicant conducted concurrent with this test, under the same condition as the test and using the same ASTM artificial soil used as a control in the test, exhibited 90% survival in the control after 14-days. However, a trend deviation in mortality was observed and the results were at the lower limit of the control chart.

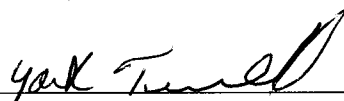
New organisms and control soils were employed for the second test. No definitive explanation is provided for the mortality observed in the first test, although the test organisms did not appear that healthy upon setting up the toxicity and bioaccumulation assay.

## IX. REFERENCES

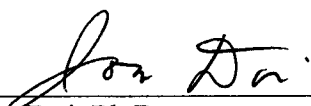
1. American Public Health Association/American Water Works Association/Water Pollution Control Federation. 1989. Standard Methods for the Examination of Water and Wastewater, 17th Ed. American Public Health Association, Washington, D.C.
2. American Society for Testing and Materials (ASTM). Annual Book of ASTM Standards. 1997. Volume 11.05: Biological Effects and Environmental Fate; Biotechnology; Pesticides. Standard Practice for Conducting Laboratory Soil Toxicity or Bioaccumulation Test with the Lumbricid Earthworm *Eisenia Foetida*. E1676-97; American Society for Testing and Materials, Philadelphia, Pennsylvania.
3. Protocol For Short Toxicity Screen Of Hazardous Waste Sites. USEPA 600/3-88/1029.

SIGNATURE PAGE

THE 28 DAY TOXICITY AND BIOACCUMULATION  
ASSAY USING THE EARTHWORM *EISENIA FOETIDA*

  
York Terrell  
Study Director

10/31/03  
Date

  
Jon Doi, Ph.D.  
Executive Vice President, Laboratory Operations

10-21-03  
Date



**TABLE 1**  
**THE 28 DAY TOXICITY AND BIOACCUMULATION**  
**ASSAY USING THE EARTHWORM *EISENIA FOETIDA***  
**SURVIVAL**

Sample No.	ASI Sample ID No.	Initial Count	Day 14	Day 28
318-0001	20031063	80	0	0
318-0002	20031064	80	0	0
318-0003	20031065	80	0	0
318-0004	20031066	80	80	66
318-0005	20031067	80	74	49
318-0006	20031068	80	78	78
ASTM Control	20031293	80	80	78
Natural Control	20031294	80	80	79

**TABLE 2**  
**THE 28 DAY TOXICITY AND BIOACCUMULATION**  
**ASSAY USING THE EARTHWORM *EISENIA FOETIDA***  
**WEIGHT LOSS/GAIN**

Sample No.	Rep.	Mean Initial Weight (gm)	Mean Final Weight (gm)	Mean Weight Loss/Gain (gm)
Natural Soil Control ASI #20031294	1	0.370	0.320	-0.050
	2	0.310	0.290	-0.020
	3	0.295	0.335	0.040
	4	0.270	0.355	0.085
ASTM Soil Control ASI #20031293	1	0.290	0.255	-0.035
	2	0.320	0.258	-0.062
	3	0.330	0.245	-0.085
	4	0.350	0.242	-0.108
318-0001 ASI #20031063	1	0.300	-	-
	2	0.290	-	-
	3	0.340	-	-
	4	0.300	-	-
318-0002 ASI #20031064	1	0.320	-	-
	2	0.325	-	-
	3	0.320	-	-
	4	0.275	-	-
318-0003 ASI #20031065	1	0.285	-	-
	2	0.300	-	-
	3	0.305	-	-
	4	0.290	-	-
318-0004 ASI #20031066	1	0.300	0.124	-0.176
	2	0.375	0.126	-0.249
	3	0.295	0.165	-0.130
	4	0.355	0.123	-0.232
318-0005 ASI #20031067	1	0.275	0.125	-0.150
	2	0.300	0.121	-0.179
	3	0.320	0.123	-0.197
	4	0.270	0.130	-0.140
318-0006 ASI #20031068	1	0.295	0.200	-0.095
	2	0.330	0.215	-0.115
	3	0.325	0.235	-0.090
	4	0.300	0.226	-0.074

**TABLE 3**

**THE 28 DAY TOXICITY AND BIOACCUMULATION  
ASSAY USING THE EARTHWORM *EISENIA FOETIDA***

**SOIL pH**

Sample No.	ASI Sample ID No.	Initial pH
318-0001	20031063	5.4
318-0002	20031064	5.0
318-0003	20031065	5.0
318-0004	20031066	4.6
318-0005	20031067	4.5
318-0006	20031068	4.3
ASTM Control	20031293	6.2
Natural Control	20031294	7.7

**TABLE 4****THE 28 DAY TOXICITY AND BIOACCUMULATION  
ASSAY USING THE EARTHWORM *EISENIA FOETIDA*****SOIL HYDRATION**

Sample ID	Initial Hydration				
	Sample Dry Wt. (g)	Sample Wet Wt. (g)	Moisture Fracture (%)	Water Added (ml)	Initial Hydration (%)
318-0001 ASI #20031063	400	500.8	25.2	10	28.0
318-0002 ASI #20031064	400	450.0	12.5	30	20.0
318-0003 ASI #20031065	400	543.2	35.8	N/A	35.8
318-0004 ASI #20031066	400	455.2	13.8	30	21.3
318-0005 ASI #20031067	40	456.0	14.0	30	21.5
318-0006 ASI #20031068	400	496.8	24.2	10	26.7
ASTM SOIL ASI #20031293	400	407.2	1.8	150.4	39.4
Natural Soil ASI #20031294	400	557.6	39.4	N/A	39.4

**Chain  
Of  
Custody**

**CHAIN OF CUSTODY RECORD**

23-144

<b>FACILITY/LOCATION:</b> LOCKHEED MARTIN/REAC EDISON, NJ ASI, Flemington, NJ	<b>METHOD OF SHIPMENT:</b> UPS OVERNIGHT TO LOCKHEED MARTIN/REAC EDISON, NJ FROM ASI, Flemington, NJ
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<b>SAMPLING AND ANALYSES AUTHORIZED BY:</b> LOCKHEED MARTIN/REAC	<b>DATE:</b> 9/30/03
---	-------------------------

SAMPLE #	SAMPLING LOCATION AND DESCRIPTION	DATE	TIME	SAMPLE TYPE			# OF CONT.	ANALYSES REQUIRED
				C	G	SOLID		
20031329	E. foetida tissue PRETEST A	8/29/03	—			Tissue	1	ANALYTICAL
20031330	E. foetida tissue B	8/29/03	—			↓	1	↓
20031331	E. foetida tissue C	8/29/03	—			↓	1	↓
20031332	E. foetida tissue D	8/29/03	—			↓	1	↓

<b>SAMPLE COLLECTED BY:</b> ASI	<b>EXACT SAMPLING LOCATION:</b> ASI LABORATORY
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<b>SAMPLE RELINQUISHED BY:</b> Tom Doherty 9/30/03, 500	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>
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<b>SAMPLE RELINQUISHED BY:</b>	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>
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<b>SAMPLE RELINQUISHED BY:</b>	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>
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<b>SAMPLE RELINQUISHED BY:</b>	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>
--------------------------------	----------------------------	-------------	-------------

<b>SAMPLE RELINQUISHED AFTER ANALYSES:</b>	<b>ANALYZED SAMPLE RECEIVED BY:</b>		
		<b>DATE</b>	<b>TIME</b>

<b>SAMPLE DESCRIPTION:</b>	<b># OF CONTAINERS:</b>
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**CHAIN OF CUSTODY RECORD**

23-144

<b>FACILITY/LOCATION:</b> LOCKHEED MARTIN/REAC EDISON, NJ ASI, Flemington, NJ	<b>METHOD OF SHIPMENT:</b> UPS OVERNIGHT TO LOCKHEED MARTIN/REAC EDISON, NJ FROM ASI, Flemington, NJ
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<b>SAMPLING AND ANALYSES AUTHORIZED BY:</b> Lockheed Martin / REAC	<b>DATE:</b> 9/30/03
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SAMPLE #	SAMPLING LOCATION AND DESCRIPTION	DATE	TIME	SAMPLE TYPE			# OF CONT.	ANALYSES REQUIRED
				C	G	SOLID		
20031497	E. foetida tissue CULTURE CONTROL JAR 31	9/27/03	-			TISSUE	1	ANALYTICAL
20031498	E. foetida tissue CULTURE CONTROL JAR 24		-					
20031499	E. foetida tissue CULTURE CONTROL JAR 12		-					
20031500	E. foetida tissue CULTURE CONTROL JAR 10		-					
20031501	E. foetida tissue ASTM CONTROL JAR 19		-					
20031502	E. foetida tissue ASTM CONTROL JAR 23		-					
20031503	E. foetida tissue ASTM CONTROL JAR 1		-					
20031504	E. foetida tissue ASTM CONTROL JAR 25		-					
20031505	E. foetida tissue 318-004 JAR 16	v	-			v	v	v

<b>SAMPLE COLLECTED BY:</b> ASI	<b>EXACT SAMPLING LOCATION:</b> ASI LABORATORY
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<b>SAMPLE RELINQUISHED BY:</b> Tom Dole 9/30/03 1500	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>
<b>SAMPLE RELINQUISHED BY:</b>	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>
<b>SAMPLE RELINQUISHED BY:</b>	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>
<b>SAMPLE RELINQUISHED BY:</b>	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>

<b>SAMPLE RELINQUISHED AFTER ANALYSES:</b>	<b>ANALYZED SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>
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<b>SAMPLE DESCRIPTION:</b>	<b># OF CONTAINERS:</b>
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**CHAIN OF CUSTODY RECORD**

23-144

<b>FACILITY/LOCATION:</b> LOCKHEED MARTIN/BEAC - EDISON, NJ ASI, FLEMINGTON, NJ	<b>METHOD OF SHIPMENT:</b> UPS OVERNIGHT TO LOCKHEED MARTIN/BEAC EDISON, NJ FROM ASI, Flemington, NJ
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<b>SAMPLING AND ANALYSES AUTHORIZED BY:</b> Lockheed Martin/BEAC	<b>DATE:</b> 9/30/03
---	-------------------------

SAMPLE #	SAMPLING LOCATION AND DESCRIPTION	DATE	TIME	SAMPLE TYPE			# OF CONT.	ANALYSES REQUIRED
				C	G	SOLID		
20031506	E. foetida tissue 318-004 JAR 26	9/27/03	-			TISSUE	1	AV. 2477C.H
20031507	E. foetida tissue 318-004 JAR 4		-					
20031508	E. foetida tissue 318-004 JAR 11		-					
20031509	E. foetida tissue 318-005 JAR 29		-					
20031510	E. foetida tissue 318-005 JAR 27		-					
20031511	E. foetida tissue 318-005 JAR 5		-					
20031512	E. foetida tissue 318-005 JAR 20		-					
20031513	E. foetida tissue 318-006 JAR 7		-					
20031514	E. foetida tissue 318-006 JAR 28	↓	-			↓	↓	↓

<b>SAMPLE COLLECTED BY:</b> ASI	<b>EXACT SAMPLING LOCATION:</b> ASI LABORATORY
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<b>SAMPLE RELINQUISHED BY:</b> Tom Dale 9/30/03 1500	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>
<b>SAMPLE RELINQUISHED BY:</b>	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>
<b>SAMPLE RELINQUISHED BY:</b>	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>
<b>SAMPLE RELINQUISHED BY:</b>	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>

<b>SAMPLE RELINQUISHED AFTER ANALYSES:</b>	<b>ANALYZED SAMPLE RECEIVED BY:</b>			
	<table style="width:100%; border-collapse: collapse;"> <tr> <td style="width:70%; border-bottom: 1px solid black;"></td> <td style="width:15%; border-bottom: 1px solid black;">DATE</td> <td style="width:15%; border-bottom: 1px solid black;">TIME</td> </tr> </table>		DATE	TIME
	DATE	TIME		

<b>SAMPLE DESCRIPTION:</b>	<b># OF CONTAINERS:</b>
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**CHAIN OF CUSTODY RECORD**

25-112

<b>FACILITY/LOCATION:</b> LOCKHEED MARTIN/REAL - EDISON NJ ASI, Flemington, NJ	<b>METHOD OF SHIPMENT:</b> UPS OVERNIGHT TO LOCKHEED MARTIN/REAL EDISON, NJ FROM ASI, Flemington, NJ
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<b>SAMPLING AND ANALYSES AUTHORIZED BY:</b> Lockheed Martin / <i>legk</i>	<b>DATE:</b> 9/30/03
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SAMPLE #	SAMPLING LOCATION AND DESCRIPTION	DATE	TIME	SAMPLE TYPE			# OF CONT.	ANALYSES REQUIRED
				C	G	SOLID		
20031515	E. Fortida Field 318-006 JAR 2	9/27/03	-			TISSUE	1	ANALYTICAL
20031516	E. Fortida Field 318-006 JAR 21	9/27/03	-			↓	↓	↓

<b>SAMPLE COLLECTED BY:</b> ASI	<b>EXACT SAMPLING LOCATION:</b> ASI LABORATORY
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<b>SAMPLE RELINQUISHED BY:</b> <i>Tom Dole</i> 9/30/03	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>
<b>SAMPLE RELINQUISHED BY:</b>	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>
<b>SAMPLE RELINQUISHED BY:</b>	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>
<b>SAMPLE RELINQUISHED BY:</b>	<b>SAMPLE RECEIVED BY:</b>	<b>DATE</b>	<b>TIME</b>

<b>SAMPLE RELINQUISHED AFTER ANALYSES:</b>	<b>ANALYZED SAMPLE RECEIVED BY:</b>			
	<table style="width:100%; border: none;"> <tr> <td style="width:70%; border: none;"> </td> <td style="width:15%; border: none;"><b>DATE</b></td> <td style="width:15%; border: none;"><b>TIME</b></td> </tr> </table>		<b>DATE</b>	<b>TIME</b>
	<b>DATE</b>	<b>TIME</b>		

<b>SAMPLE DESCRIPTION:</b>	<b># OF CONTAINERS:</b>
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**CHAIN OF CUSTODY RECORD**

REAC, Edison, NJ  
EPA Contract #:

Site #: 318  
Contact Name: John Johnson  
Contact Phone: 732-321-4248

No: 318-0003  
Lab: Aqua Survey  
Lab Contact: Jon Doi  
Lab Phone:

Lab #	Sample #	Location	Analyses	Matrix	Collected	Numb Cont	Container	Preservative	MS/MSD
	318-0001	0150D	E. Foetida Toxicity Test	Soil	7/29/2003	1	2.5 gal bucket	4 C	
	318-0002	100R100D	E. Foetida Toxicity Test	Soil	7/29/2003	1	2.5 gal bucket	4 C	

**SAMPLES TRANSFERRED FROM  
CHAIN OF CUSTODY #**

Special Instructions:

Items/Reason	Relinquished by	Date	Received by	Date	Time
All Tox Test	Jerry Bottom	7/30/03	Yak T. Leed	7/30/03	12:50
All ITPX Test	Jerry Bottom	7/30/03	Yak T. Leed	7/30/03	12:50

**CHAIN OF CUSTODY RECORD**

**No: 318-0004**  
 Lab: Aqua Survey  
 Lab Contact: Jon Doi  
 Lab Phone:

Site #: 318  
 Contact Name: John Johnson  
 Contact Phone: 732-321-4248

REAC, Edison, NJ  
 EPA Contract #:

Lab #	Sample #	Location	Analyses	Matrix	Collected	Numb Cont	Container	Preservative	MS/MSD
318-0003		100R50D	E. Foetida Toxicity Test	Soil	7/29/2003	1	2.5 gal bucket	4 C	
318-0004		150R100D	E. Foetida Toxicity Test	Soil	7/29/2003	1	2.5 gal bucket	4 C	

**SAMPLES TRANSFERRED FROM  
 CHAIN OF CUSTODY #**

Special Instructions:

Items/Reason	Relinquished by	Date	Received by	Date	Time
All Toxic Test	Jenny Bedon	7/30/03	Yak Trivedi	7/30/03	12:50

**CHAIN OF CUSTODY RECORD**

**No: 318-0005**  
 Lab: Aqua Survey  
 Lab Contact: Jon Doi  
 Lab Phone:

Site #: 318  
 Contact Name: John Johnson  
 Contact Phone: 732-321-4248

REAC, Edison, NJ  
 EPA Contract #:

Lab #	Sample #	Location	Analyses	Matrix	Collected	Numb Cont	Container	Preservative	MS/MSD
	318-0005	150R50D	E. Foetida Toxicity Test	Soil	7/29/2003	1	2.5 gal bucket	4 C	
	318-0006	Reference	E. Foetida Toxicity Test	Soil	7/29/2003	1	2.5 gal bucket	4 C	

**SAMPLES TRANSFERRED FROM  
 CHAIN OF CUSTODY #**

Special Instructions:

Items/Reason	Relinquished by	Date	Received by	Date	Time
All Toxic Test	Summy Bedina	7/30/03			
	Arthur Test	7/30/03			
	Jon Doi	7/30/03			

ASI, INC.  
SAMPLE RECEIVING FORM

Client: <b>LHM</b>				Job #: <b>23-144</b>			
Shipped Via: <b>COURIER</b>				# of Shipping Containers: <b>3</b>			
Type of Shipping Container: <b>3 coolers</b>				Custody Seal: Present ___ Absent <input checked="" type="checkbox"/> Broken ___		Condition of Shipping Containers: Acceptable <input checked="" type="checkbox"/> Unacceptable ___	
ASI #	Sample ID	Type of Container	Number of Containers	Condition of Samples †	Temp. °C	Ice +	Type of Sample *
1. <b>20031063</b>	<b>318-0001</b>	<b>2.5 gal plastic bucket</b>	<b>1</b>	<b>A</b>	<b>40</b>	<b>I</b>	<b>S</b>
2. <b>20031064</b>	<b>318-0002</b>	↓	<b>1</b>	↓	↓	↓	↓
3. <b>20031065</b>	<b>318-0003</b>	↓	<b>1</b>	↓	↓	↓	↓
4. <b>20031066</b>	<b>318-0004</b>	↓	<b>1</b>	↓	↓	↓	↓
5. <b>20031067</b>	<b>318-0005</b>	↓	<b>1</b>	↓	↓	↓	↓
6. <b>20031068</b>	<b>318-0006</b>	↓	<b>1</b>	↓	↓	↓	↓
7.							
8.							
9.							
10.							
NOTES: (Discrepancies Between Sample Label and COC Record) <b>① cooler environment</b>							

OPENED/ RECEIVED BY: 1. <b>Y. T. ...</b>	DATE/ TIME: <b>7/30/03 1250</b>
2.	
3.	
4.	
5.	
6.	
7.	
8.	
9.	
10.	

\*  
S= Soil  
SD= Sediment  
SL= Sludge  
W= Water

†  
A= Acceptable  
U= Unusable or Contaminated

+  
I= Ice  
B= Blue Ice  
D= Dry Ice  
N= None

**Statistical Analysis**

**For**

**Mortality**

23-144 controls AST v. Culture survival  
File: 144con.s Transform: ARC SINE(SQUARE ROOT(Y))

Shapiro - Wilk's test for normality

---

D = 0.023

W = 0.828

Critical W (P = 0.05) (n = 8) = 0.818

Critical W (P = 0.01) (n = 8) = 0.749

---

Data PASS normality test at P=0.01 level. Continue analysis.

23-144 controls AST v. Culture survival  
 File: 144con.s Transform: ARC SINE(SQUARE ROOT(Y))

F-Test for equality of two variances

GROUP	IDENTIFICATION	VARIANCE	F
1	culture	0.003	
2	ASTM	0.004	1.333

Critical F = 47.50 (P=0.01, 3, 3)

Since F <= Critical F, FAIL TO REJECT Ho: Equal Variances.

TITLE: 23-144 controls AST v. Culture survival  
 FILE: 144con.s  
 TRANSFORM: ARC SINE(SQUARE ROOT(Y)) NUMBER OF GROUPS: 2

GRP	IDENTIFICATION	REP	VALUE	TRANS VALUE
1	culture	1	0.9500	1.3453
1	culture	2	1.0000	1.4588
1	culture	3	1.0000	1.4588
1	culture	4	1.0000	1.4588
2	ASTM	1	1.0000	1.4588
2	ASTM	2	0.9500	1.3453
2	ASTM	3	1.0000	1.4588
2	ASTM	4	0.9500	1.3453



23-144 controls AST v. Culture survival  
File: 144con.s Transform: ARC SINE(SQUARE ROOT(Y))

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 1 of 2

---

GRP	IDENTIFICATION	N	MIN	MAX	MEAN
1	culture	4	1.345	1.459	1.430
2	ASTM	4	1.345	1.459	1.402

---

23-144 controls AST v. Culture survival  
File: 144con.s Transform: ARC SINE(SQUARE ROOT(Y))

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 2 of 2

---

GRP	IDENTIFICATION	VARIANCE	SD	SEM	C.V. %
1	culture	0.003	0.057	0.028	3.97
2	ASTM	0.004	0.066	0.033	4.67

---

23-144 controls AST v. Culture survival

File: 144con.s

Transform: ARC SINE(SQUARE ROOT(Y))

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	1	0.002	0.002	0.429
Within (Error)	6	0.023	0.004	
Total	7	0.024		

Critical F value = 5.99 (0.05,1,6)

Since  $F < \text{Critical } F$  FAIL TO REJECT  $H_0$ : All equal

13

23-144 controls AST v. Culture survival  
 File: 144con.s Transform: ARC SINE(SQUARE ROOT(Y))

EQUAL VARIANCE t-TEST - TABLE 1 OF 2 Ho:Control<Treatment

---

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	culture	1.430	0.988		
2	ASTM	1.402	0.975	0.655	

---

2 Sample t table value = 1.94 (1 Tailed Value, P=0.05, df=6,1)

UNEQUAL VARIANCE t-TEST Ho:Control<Treatment

---

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	culture	1.430	0.988		
2	ASTM	1.402	0.975	0.655	

---

2 Sample t table value = 2.01 (1 Tailed Value, P=0.05, df=5,1)

23-144 controls AST v. Culture survival  
 File: 144con.s Transform: ARC SINE(SQUARE ROOT(Y))

EQUAL VARIANCE t-TEST - TABLE 2 OF 2 Ho:Control<Treatment

---

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	culture	4			
2	ASTM	4	0.030	3.0	0.012

---

UNEQUAL VARIANCE t-TEST Ho:Control<Treatment

---

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	culture	4			
2	ASTM	4	0.031	3.2	0.012

---

23-144 E. foetida survival (Culture control)  
File: 144ef.s Transform: ARC SINE(SQUARE ROOT(Y))

Shapiro - Wilk's test for normality

---

D = 0.130

W = 0.947

Critical W (P = 0.05) (n = 16) = 0.887

Critical W (P = 0.01) (n = 16) = 0.844

---

Data PASS normality test at P=0.01 level. Continue analysis.

23-144 E. foetida survival (Culture control)  
File: 144ef.s Transform: ARC SINE(SQUARE ROOT(Y))

-----  
Bartlett's test for homogeneity of variance  
Calculated B1 statistic = 4.01

-----  
Table Chi-square value = 11.34 (alpha = 0.01, df = 3)  
Table Chi-square value = 7.81 (alpha = 0.05, df = 3)

Data PASS B1 homogeneity test at 0.01 level. Continue analysis.

TITLE: 23-144 E. foetida survival (Culture control)

FILE: 144ef.s

TRANSFORM: ARC SINE(SQUARE ROOT(Y))

NUMBER OF GROUPS: 4

GRP	IDENTIFICATION	REP	VALUE	TRANS VALUE
1	con (culture)	1	0.9500	1.3453
1	con (culture)	2	1.0000	1.4588
1	con (culture)	3	1.0000	1.4588
1	con (culture)	4	1.0000	1.4588
2	318-0004	1	0.8500	1.1731
2	318-0004	2	0.9500	1.3453
2	318-0004	3	0.8500	1.1731
2	318-0004	4	0.6500	0.9377
3	318-0005	1	0.6000	0.8861
3	318-0005	2	0.7000	0.9912
3	318-0005	3	0.6500	0.9377
3	318-0005	4	0.5000	0.7854
4	318-0006	1	0.9500	1.3453
4	318-0006	2	1.0000	1.4588
4	318-0006	3	1.0000	1.4588
4	318-0006	4	0.9500	1.3453



23-144 E. foetida survival (Culture control)  
 File: 144ef.s Transform: ARC SINE(SQUARE ROOT(Y))

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 1 of 2

GRP	IDENTIFICATION	N	MIN	MAX	MEAN
1	con (culture)	4	1.345	1.459	1.430
2	318-0004	4	0.938	1.345	1.157
3	318-0005	4	0.785	0.991	0.900
4	318-0006	4	1.345	1.459	1.402

23-144 E. foetida survival (Culture control)  
 File: 144ef.s Transform: ARC SINE(SQUARE ROOT(Y))

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 2 of 2

GRP	IDENTIFICATION	VARIANCE	SD	SEM	C.V. %
1	con (culture)	0.003	0.057	0.028	3.97
2	318-0004	0.028	0.167	0.084	14.46
3	318-0005	0.008	0.088	0.044	9.74
4	318-0006	0.004	0.066	0.033	4.67

23-144 E. foetida survival (Culture control)  
File: 144ef.s Transform: ARC SINE(SQUARE ROOT(Y))

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	3	0.735	0.245	22.665
Within (Error)	12	0.130	0.011	
Total	15	0.864		

Critical F value = 3.49 (0.05,3,12)  
Since  $F > \text{Critical } F$  REJECT  $H_0$ : All equal



23-144 E. foetida survival (Culture control)  
 File: 144ef.s Transform: ARC SINE(SQUARE ROOT(Y))

DUNNETT'S TEST - TABLE 1 OF 2 Ho:Control<Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	con (culture)	1.430	0.988		
2	318-0004	1.157	0.825	3.716	*
3	318-0005	0.900	0.613	7.215	*
4	318-0006	1.402	0.975	0.386	

Dunnett table value = 2.29 (1 Tailed Value, P=0.05, df=12,3)

23-144 E. foetida survival (Culture control)  
 File: 144ef.s Transform: ARC SINE(SQUARE ROOT(Y))

DUNNETT'S TEST - TABLE 2 OF 2 Ho:Control<Treatment

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	con (culture)	4			
2	318-0004	4	0.073	7.4	0.162
3	318-0005	4	0.073	7.4	0.375
4	318-0006	4	0.073	7.4	0.012

**Statistical Analysis**

**For**

**Weight**

23-144 Control ASTM v. culture dry weight  
File: 144con.dw Transform: NO TRANSFORMATION

Shapiro - Wilk's test for normality

---

D = 0.002

W = 0.943

Critical W (P = 0.05) (n = 8) = 0.818

Critical W (P = 0.01) (n = 8) = 0.749

---

Data PASS normality test at P=0.01 level. Continue analysis.

23-144 Control ASTM v. culture dry weight  
File: 144con.dw Transform: NO TRANSFORMATION

F-Test for equality of two variances

---

GROUP	IDENTIFICATION	VARIANCE	F
1	culture con	0.001	
2	ASTM con	0.000	12.640

---

Critical F = 47.50 (P=0.01, 3, 3)

Since  $F \leq \text{Critical } F$ , FAIL TO REJECT  $H_0$ : Equal Variances.

23-144 Control ASTM v. culture dry weight  
File: 144con.dw Transform: NO TRANSFORMATION

---

Bartlett's test for homogeneity of variance

Calculated B1 statistic = 3.35

---

Table Chi-square value = 6.63 (alpha = 0.01, df = 1)

Table Chi-square value = 3.84 (alpha = 0.05, df = 1)

Data PASS B1 homogeneity test at 0.01 level. Continue analysis.

TITLE: 23-144 Control ASTM v. culture dry weight

FILE: 144con.dw

TRANSFORM: NO TRANSFORMATION

NUMBER OF GROUPS: 2

---

GRP	IDENTIFICATION	REP	VALUE	TRANS VALUE
1	culture con	1	0.3200	0.3200
1	culture con	2	0.2900	0.2900
1	culture con	3	0.3350	0.3350
1	culture con	4	0.3550	0.3550
2	ASTM con	1	0.2550	0.2550
2	ASTM con	2	0.2580	0.2580
2	ASTM con	3	0.2450	0.2450
2	ASTM con	4	0.2420	0.2420

---



23-144 Control ASTM v. culture dry weight  
File: 144con.dw Transform: NO TRANSFORMATION

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 1 of 2

---

GRP	IDENTIFICATION	N	MIN	MAX	MEAN
1	culture con	4	0.290	0.355	0.325
2	ASTM con	4	0.242	0.258	0.250

---

23-144 Control ASTM v. culture dry weight  
File: 144con.dw Transform: NO TRANSFORMATION

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 2 of 2

---

GRP	IDENTIFICATION	VARIANCE	SD	SEM	C.V. %
1	culture con	0.001	0.027	0.014	8.43
2	ASTM con	0.000	0.008	0.004	3.08

---

23-144 Control ASTM v. culture dry weight  
File: 144con.dw Transform: NO TRANSFORMATION

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	1	0.011	0.011	27.801
Within (Error)	6	0.002	0.000	
Total	7	0.014		

Critical F value = 5.99 (0.05,1,6)  
Since  $F > \text{Critical F}$  REJECT  $H_0$ : All equal

23-144 Control ASTM v. culture dry weight  
 File: 144con.dw Transform: NO TRANSFORMATION

EQUAL VARIANCE t-TEST - TABLE 1 OF 2 Ho:Control<Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	culture con	0.325	0.325		
2	ASTM con	0.250	0.250	5.273	*

2 Sample t table value = 1.94 (1 Tailed Value, P=0.05, df=6,1)

UNEQUAL VARIANCE t-TEST Ho:Control<Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	culture con	0.325	0.325		
2	ASTM con	0.250	0.250	5.273	*

2 Sample t table value = 2.35 (1 Tailed Value, P=0.05, df=3,1)

23-144 Control ASTM v. culture dry weight  
 File: 144con.dw Transform: NO TRANSFORMATION

EQUAL VARIANCE t-TEST - TABLE 2 OF 2 Ho:Control<Treatment

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	culture con	4			
2	ASTM con	4	0.028	8.5	0.075

UNEQUAL VARIANCE t-TEST Ho:Control<Treatment

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	culture con	4			
2	ASTM con	4	0.033	10.3	0.075



23-144 E. foetida wet wt.  
File: 144cc.dw Transform: NO TRANSFORMATION

Shapiro - Wilk's test for normality

D = 0.004

W = 0.961

Critical W (P = 0.05) (n = 16) = 0.887

Critical W (P = 0.01) (n = 16) = 0.844

Data PASS normality test at P=0.01 level. Continue analysis.

23-144 E. foetida wet wt.  
File: 144cc.dw Transform: NO TRANSFORMATION

Bartlett's test for homogeneity of variance  
Calculated B1 statistic = 7.05

Table Chi-square value = 11.34 (alpha = 0.01, df = 3)

Table Chi-square value = 7.81 (alpha = 0.05, df = 3)

Data PASS B1 homogeneity test at 0.01 level. Continue analysis.

TITLE: 23-144 E. foetida wet wt.  
FILE: 144cc.dw  
TRANSFORM: NO TRANSFORMATION

NUMBER OF GROUPS: 4

GRP	IDENTIFICATION	REP	VALUE	TRANS VALUE
1	con (culture)	1	0.3200	0.3200
1	con (culture)	2	0.2900	0.2900
1	con (culture)	3	0.3350	0.3350
1	con (culture)	4	0.3550	0.3550
2	318-0004	1	0.1240	0.1240
2	318-0004	2	0.1260	0.1260
2	318-0004	3	0.1650	0.1650
2	318-0004	4	0.1230	0.1230
3	318-0005	1	0.1250	0.1250
3	318-0005	2	0.1210	0.1210
3	318-0005	3	0.1230	0.1230
3	318-0005	4	0.1300	0.1300
4	318-0006	1	0.2000	0.2000
4	318-0006	2	0.2150	0.2150
4	318-0006	3	0.2350	0.2350
4	318-0006	4	0.2260	0.2260

C-7

23-144 E. foetida wet wt.

File: 144cc.dw

Transform: NO TRANSFORMATION

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 1 of 2

GRP	IDENTIFICATION	N	MIN	MAX	MEAN
1	con (culture)	4	0.290	0.355	0.325
2	318-0004	4	0.123	0.165	0.135
3	318-0005	4	0.121	0.130	0.125
4	318-0006	4	0.200	0.235	0.219

23-144 E. foetida wet wt.

File: 144cc.dw

Transform: NO TRANSFORMATION

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 2 of 2

GRP	IDENTIFICATION	VARIANCE	SD	SEM	C.V. %
1	con (culture)	0.001	0.027	0.014	8.43
2	318-0004	0.000	0.020	0.010	15.15
3	318-0005	0.000	0.004	0.002	3.10
4	318-0006	0.000	0.015	0.008	6.88

23-144 E. foetida wet wt.

File: 144cc.dw

Transform: NO TRANSFORMATION

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	3	0.104	0.035	98.295
Within (Error)	12	0.004	0.000	
Total	15	0.108		

Critical F value = 3.49 (0.05,3,12)

Since  $F > \text{Critical } F$  REJECT  $H_0$ : All equal

RF

23-144 E. foetida wet wt.

File: 144cc.dw

Transform: NO TRANSFORMATION

BONFERRONI t-TEST - TABLE 1 OF 2

Ho:Control<Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	con (culture)	0.325	0.325		
2	318-0004	0.135	0.135	14.363	*
3	318-0005	0.125	0.125	15.098	*
4	318-0006	0.219	0.219	7.992	*

Bonferroni t table value = 2.40 (1 Tailed Value, P=0.05, df=12,3)

23-144 E. foetida wet wt.

File: 144cc.dw

Transform: NO TRANSFORMATION

BONFERRONI t-TEST - TABLE 2 OF 2

Ho:Control<Treatment

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	con (culture)	4			
2	318-0004	4	0.032	9.8	0.191
3	318-0005	4	0.032	9.8	0.200
4	318-0006	4	0.032	9.8	0.106

23-144 E. foetida wet wt.

File: 144astm.dw Transform: NO TRANSFORMATION

Shapiro - Wilk's test for normality

---

D = 0.002

W = 0.940

Critical W (P = 0.05) (n = 16) = 0.887

Critical W (P = 0.01) (n = 16) = 0.844

---

Data PASS normality test at P=0.01 level. Continue analysis.

23-144 E. foetida wet wt.

File: 144astm.dw

Transform: NO TRANSFORMATION

-----  
Bartlett's test for homogeneity of variance

Calculated B1 statistic = 6.62

-----  
Table Chi-square value = 11.34 (alpha = 0.01, df = 3)

Table Chi-square value = 7.81 (alpha = 0.05, df = 3)

Data PASS B1 homogeneity test at 0.01 level. Continue analysis.

TITLE: 23-144 E. foetida wet wt.

FILE: 144astm.dw

TRANSFORM: NO TRANSFORMATION

NUMBER OF GROUPS: 4

---

GRP	IDENTIFICATION	REP	VALUE	TRANS VALUE
1	con (ASTM)	1	0.2550	0.2550
1	con (ASTM)	2	0.2580	0.2580
1	con (ASTM)	3	0.2450	0.2450
1	con (ASTM)	4	0.2420	0.2420
2	318-0004	1	0.1240	0.1240
2	318-0004	2	0.1260	0.1260
2	318-0004	3	0.1650	0.1650
2	318-0004	4	0.1230	0.1230
3	318-0005	1	0.1250	0.1250
3	318-0005	2	0.1210	0.1210
3	318-0005	3	0.1230	0.1230
3	318-0005	4	0.1300	0.1300
4	318-0006	1	0.2000	0.2000
4	318-0006	2	0.2150	0.2150
4	318-0006	3	0.2350	0.2350
4	318-0006	4	0.2260	0.2260

---

23-144 E. foetida wet wt.

File: 144astm.dw

Transform: NO TRANSFORMATION

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 1 of 2

---

GRP	IDENTIFICATION	N	MIN	MAX	MEAN
1	con (ASTM)	4	0.242	0.258	0.250
2	318-0004	4	0.123	0.165	0.135
3	318-0005	4	0.121	0.130	0.125
4	318-0006	4	0.200	0.235	0.219

---

23-144 E. foetida wet wt.

File: 144astm.dw

Transform: NO TRANSFORMATION

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 2 of 2

---

GRP	IDENTIFICATION	VARIANCE	SD	SEM	C.V. %
1	con (ASTM)	0.000	0.008	0.004	3.08
2	318-0004	0.000	0.020	0.010	15.15
3	318-0005	0.000	0.004	0.002	3.10
4	318-0006	0.000	0.015	0.008	6.88

---



23-144 E. foetida wet wt.

File: 144astm.dw

Transform: NO TRANSFORMATION

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	3	0.046	0.015	85.791
Within (Error)	12	0.002	0.000	
Total	15	0.048		

Critical F value = 3.49 (0.05,3,12)

Since  $F > \text{Critical } F$  REJECT  $H_0$ : All equal

23-144 E. foetida wet wt.

File: 144astm.dw

Transform: NO TRANSFORMATION

BONFERRONI t-TEST - TABLE 1 OF 2

Ho:Control<Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	con (ASTM)	0.250	0.250		
2	318-0004	0.135	0.135	12.204	*
3	318-0005	0.125	0.125	13.234	*
4	318-0006	0.219	0.219	3.275	*

Bonferroni t table value = 2.40 (1 Tailed Value, P=0.05, df=12,3)

23-144 E. foetida wet wt.

File: 144astm.dw

Transform: NO TRANSFORMATION

BONFERRONI t-TEST - TABLE 2 OF 2

Ho:Control<Treatment

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	con (ASTM)	4			
2	318-0004	4	0.023	9.1	0.115
3	318-0005	4	0.023	9.1	0.125
4	318-0006	4	0.023	9.1	0.031

# Raw Data

23-144 Earthworm Bioaccumulation Study

	C2	C3	C4
	Position	Sample	Code
1	31	Culture Soil Control	0.1
2	24	# 2003-1074	0.2
3	12	1294	0.3
4	10		0.4
5	19	ASTM Soil Control	1.1
6	23	ASI # 20031069	1.2
7	1	1293	1.3
8	25		1.1
9	8	318-0001	2.1
10	30	ASI # 20031063	2.2
11	14		2.3
12	3		2.4
13	6	318-0002	3.1
14	32	ASI # 20031064	3.2
15	9		3.3
16	17		3.4
17	22	318-0003	4.1
18	18	ASI # 20031065	4.2
19	15		4.3
20	13		4.4
21	16	318-004	5.1
22	26	ASI # 20031066	5.2
23	4		5.3
24	11		5.4
25	29	318-0005	6.1
26	27	ASI # 20031067	6.2
27	5		6.3
28	20		6.4
29	7	318-0006	7.1
30	28	ASI #20031068	7.2
31	2		7.3
32	21		7.4

**AQUA SURVEY, INC.**  
28-Day Earthworm Survival and Growth

Job #: 23-144

Client: LHM

Test Start Date: 8/29/03

Organism: E. foetida

Chamber No.	Initial Count/Weight			14-Day Count	28-Day Count/Weight		
	Live Count	Wet Wt. (gm)	Mean Wt. (gm)		Live Count	Wet Wt. (gm)	Mean Wt. (gm)
1	20	6.6	0.330	20	20	4.9	0.245
2	20	6.5	0.325	20	20	4.7	0.235
3	20	6.0	0.300	0	—	—	—
4	20	5.9	0.295	20	17	2.8	0.165
5	20	6.4	0.320	20	13	1.6	0.123
6	20	6.4	0.320	0	—	—	—
7	20	5.9	0.295	19	19	3.8	0.200
8	20	6.0	0.300	0	—	—	—
9	20	6.4	0.320	0	—	—	—
10	20	5.4	0.270	20	20	7.1	0.355
11	20	7.1	0.355	20	13	1.6	0.123
12	20	5.9	0.295	20	20	6.7	0.335
13	20	5.8	0.290	0	—	—	—
14	20	6.8	0.340	0	—	—	—
15	20	6.1	0.305	0	—	—	—
16	20	6.0	0.300	20	17	2.1	0.124
17	20	5.5	0.275	0	—	—	—
18	20	6.0	0.300	0	—	—	—
19	20	5.8	0.290	20	20	5.1	0.255
20	20	5.4	0.270	18	10	1.3	0.130
21	20	6.6	0.300	19	19	4.3	0.226
22	20	5.7	0.285	0	—	—	—
23	20	6.4	0.320	20	19	4.9	0.258
24	20	6.2	0.310	20	20	5.8	0.290
25	20	7.0	0.350	20	19	4.6	0.242
Date/Init.	8/29/03 YT	8/29/03 YT	8/29/03 YT	9/12/03 YT	9/10/03 YT	9/17/03 YT	9/17/03 YT

Initial Depuration Date: 8/28/03 to 8/29/03.

28-Day Depuration Date: 9/24/03 to 9/27/03



**AQUA SURVEY, INC.**  
28-Day Earthworm (Observations)

Job #: 23-144

Client: LHM

Test Start Date: 8/29/03

Organism: E. Coetida

Chamber No.	Test Day									
	Initial	4	7	11	14	19	24			
1	N	N	N	N	N	N	N			
2	N	N	N	N	N	N	N			
3	N	N	D	D	D	-	-	-	-	-
4	N	N	N	N	N	N	N			
5	N	N	N	N	N	N	N			
6	N	N	N	D	D	-	-	-	-	-
7	N	N	N	N	N	N	N			
8	N	S <sup>3</sup>	D	D	D	-	-	-	-	-
9	N	N	N	D	D	-	-	-	-	-
10	N	N	N	N	N	N	N			
11	N	N	N	N	N	N	N			
12	N	N	N	N	N	N	N			
13	N	S <sup>2</sup>	S <sup>1</sup>	D	D	-	-	-	-	-
14	N	N	D	D	D	-	-	-	-	-
15	N	S <sup>1</sup>	S <sup>1</sup> D	D	D	-	-	-	-	-
16	N	N	N	N	N	N	N			
17	N	N	N	D	D	-	-	-	-	-
18	N	S <sup>2</sup>	D	D	D	-	-	-	-	-
Date/Init.	8/29/03 Y1	9/2/03 Y1	9/5/03 Y1	9/9/03 Y1	9/12/03 Y1	9/17/03 Y1	9/22/03 Y1			

D = Dead

S = Surface

N = Nothing Unusual

Comments: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
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 \_\_\_\_\_  
 \_\_\_\_\_

**AQUA SURVEY, INC.**  
28-Day Earthworm (Observations)

Job #: 23-144

Client: LHM

Test Start Date: 8/29/03

Organism: E-foetida

Chamber No.	Test Day							
	Initial	4	7	11	14	17	21	
19	N	N	N	N	N	N	N	
20	N	N	N	N	N	N	N	
21	N	N	N	N	N	N	N	
22	N	N	D	D	D	-	-	-
23	N	N	N	N	N	N	N	
24	N	N	N	N	N	N	N	
25	N	N	N	N	N	N	N	
26	N	N	N	N	N	N	N	
27	N	N	N	N	N	N	N	
28	N	N	N	N	N	N	N	
29	N	N	N	N	N	N	N	
30	N	S <sup>2</sup>	D	D	D	-	-	-
31	N	N	N	N	N	N	N	
32	N	N	N	D	D	-	-	-
Date/Init.	8/23/03 YT	9/12/03 YT	9/15/03 YT	9/19/03 YT	10/12/03 YT	9/17/03 YT	9/22/03 YT	

D = Dead                      S = Surface                      N = Nothing Unusual

Comments: \_\_\_\_\_  
 \_\_\_\_\_  
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**AQUA SURVEY, INC.**  
**28-Day Earthworm (Chambers Hydrated Weight)**

Job #: 23-144

Client: LHM

Test Start Date: 8/29/03

Organism: E. Foetida

Chamber No.	Weight (gm)			Chamber No.	Weight (gm)		
	Initial	Day 28 <sup>1</sup> / <sub>4</sub>	Day 28 <sup>2</sup> / <sub>4</sub>		Initial	Day 28 <sup>1</sup> / <sub>4</sub>	Day 28 <sup>2</sup> / <sub>4</sub>
1	1295.3	1282.9	1295.3	26	1225.8	1215.1	1225.8
2	1266.9	1258.1	1266.9	27	1242.0	1232.9	1242.0
3	1279.2	—	—	28	1268.9	1263.5	1268.9
4	1233.9	1222.1	1233.9	29	1235.2	1225.0	1235.2
5	1216.7	1210.1	1216.7	30	1253.6	—	—
6	1231.8	—	—	31	1300.2	1293.6	1300.2
7	1274.3	1265.2	1274.3	32	1234.0	—	—
8	1259.6	—	—				
9	1235.7	—	—				
10	1308.1	1296.9	1308.1				
11	1231.0	1226.1	1231.0				
12	1303.9	1297.6	1303.9				
13	1281.7	—	—				
14	1260.3	—	—				
15	1281.5	—	—				
16	1229.5	1219.9	1229.5				
17	1224.6	—	—				
18	1288.8	—	—				
19	1311.6	1299.6	1311.6				
20	1243.8	1233.9	1243.8				
21	1277.8	1270.7	1277.8				
22	1285.0	—	—				
23	1301.8	1290.9	1301.8				
24	1301.8	1292.0	1301.8				
25	1361.0	1288.2	1301.0				
Date/Init.	8/29/03 YT	9/22/03 YT	9/22/03 YT		8/29/03 YT	9/22/03 YT	9/22/03 YT

- ① weight of sample prior to adding water
- ② weight of sample after adding water
- ③ wrong date was recorded YT 9/24/03

**AQUA SURVEY, INC.**  
**PERCENT MOISTURE (SOIL)**

Job #: 23-174

Client: LHM

Start Date: 7/31/03

Balance: Acculab

Date last calibrated 7/31/03

Drying Oven: \_\_\_\_\_

Temperature 105°C Date/Time Samples in 7/31/03 1200 Date/Time Samples out 8/1/03 1200

Sample No.	(a) Pan Wt. (gm)	(b) Wet Wt. + Pan (gm)	(c) Dry Wt. + Pan (gm)	% Moisture $\frac{[(b - c)] \times 100}{[b - a]}$
20031063	64.1	252.1	204.7	25.2
20031064	64.1	229.2	208.8	12.5
20031065	71.9	247.1	184.4	35.8
20031066	72.9	238.8	215.9	13.8
20031067	63.1	228.0	204.9	14.0
20031068	62.1	252.8	206.7	24.2
20031069	71.8	<del>229.3</del> 223.3	221.1	1.4
20031074	73.8	229.2	158.0	59
Date/Initial	7/31/03 yr	7/31/03 yr	8/1/03 yr	8/1/03 yr

**AQUA SURVEY, INC.**  
**PERCENT MOISTURE (SOIL)**

Job #: 23144      Client: LHM      Start Date: 8/26/03

Balance: Acculab      Date last calibrated 8/26/03      Drying Oven: \_\_\_\_\_

Temperature 105°C      Date/Time Samples in 8/26/03 17:00      Date/Time Samples out 8/27/03 17:00

Sample No.	(a) Pan Wt. (gm)	(b) Wet Wt. + Pan (gm)	(c) Dry Wt. + Pan (gm)	% Moisture [(b - c) / (b - a)] x 100
20031293	73.3	176.1	174.3	1.8
20031294	64.0	210.0	152.5	39.4
Date/Initial	8/26/03 YI	8/26/03 YI	8/28/03 YI	8/28/03 YI

**AQUA SURVEY, INC.**  
Earthworm Soil Hydration

Job #: 23-144

Client: LHM

Test Start Date: 8/28/03

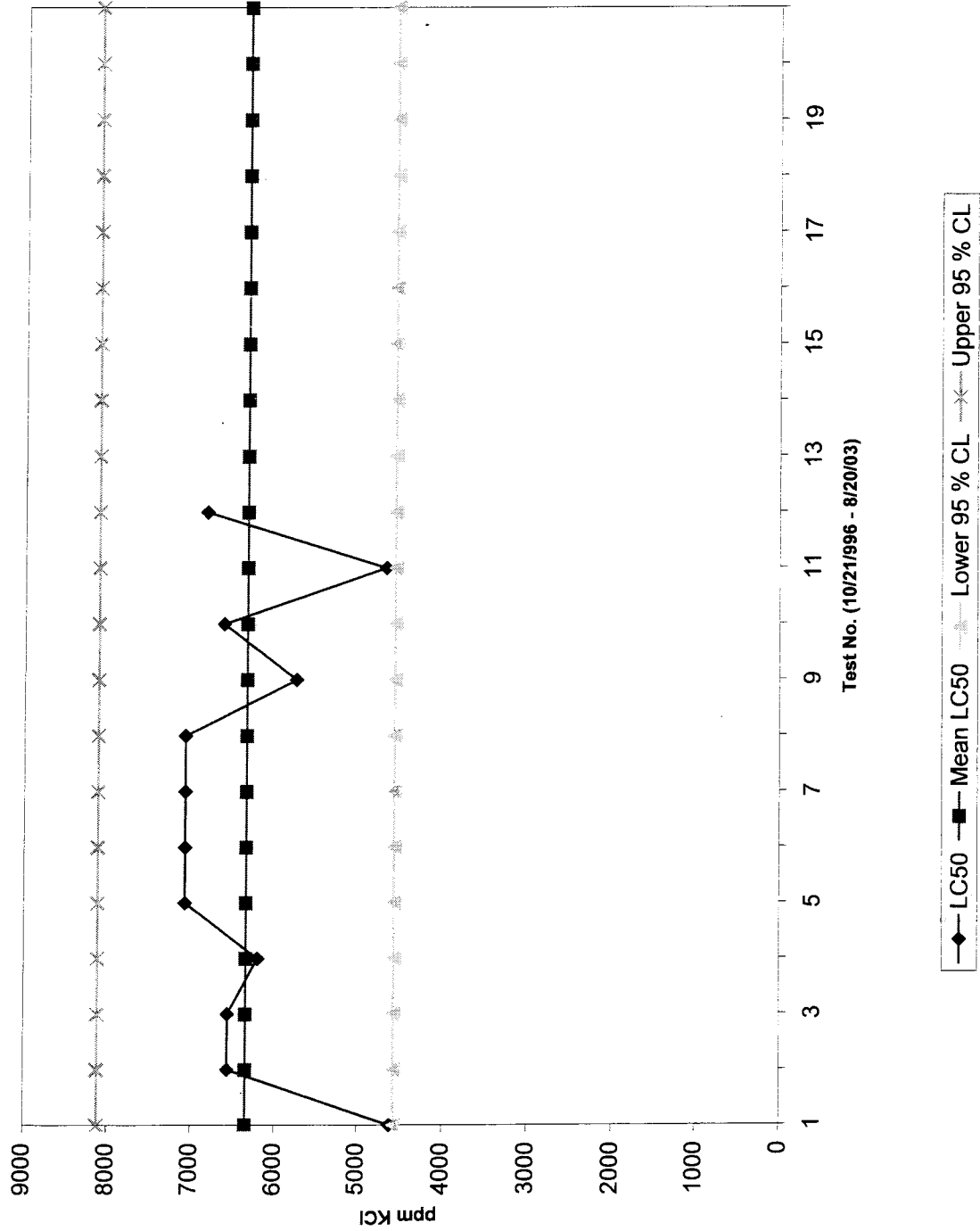
Organism: E. Foetida

Sample ID	Initial Hydration				
	Sample Dry Wt. (g)	Sample Wet Wt. (g)	Moisture Fraction (%)	Water Added (ml)	Initial Hydration (%)
20031063	400	500.8	25.2	10	28.0
20031064	400	450.0	12.5	30	20.0
20031065	400	543.2	35.8	N/A	35.8
20031066	400	455.2	13.8	30	21.3
20031067	400	456.0	14.0	30	21.5
20031068	400	496.8	24.2	10	26.7
20031293	400	407.2	1.8	150.4	39.4
20031294	400	557.6	39.4	N/A	39.4
Date/Init.	8/28/03 YI	8/28/03 YI	8/28/03 YI	8/28/03 YI	8/28/03 YI

Comments: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

# **Standard Reference Toxicant**

Control Chart of LC50 Values for E. foetida Using KCl



Control Chart of LC50 values for *E. foetida* using KCI

Date	Test No	ppm LC50	Mean LC50	SD	Lower 95% CL	Upper 95% CL
10/21/96	1	4613	6341	887	4566.787	8116.05
2/19/97		6559	6341		4566.787	8116.05
10/16/98	3	6559	6341		4566.787	8116.05
3/5/99		6202	6341		4566.787	8116.05
4/16/99	5	7071	6341		4566.787	8116.05
9/13/99		7071	6341		4566.787	8116.05
10/25/99	7	7071	6341		4566.787	8116.05
11/16/99		7071	6341		4566.787	8116.05
11/28/00	9	5748	6341		4566.787	8116.05
6/28/01		6622	6341		4566.787	8116.05
8/8/03	11	4680	6341		4566.787	8116.05
8/29/03		6830	6341		4566.787	8116.05



ACUTE 14 DAY STANDARD REFERENCE TOXICANT

Stock and Treatments Preparation Sheet

\*\*\*\* ALL UNITS ARE MG/L UNLESS OTHERWISE SPECIFIED\*\*\*\*


Source of Dilution Water: *ASI DI*  
 Date: *8/8/03*  
*29*

Species: *E. foetida or L. terrestris*  
 Location: *ASI Flemington, NJ*

TOXICANT: Potassium chloride

Source/Lot No.: *10616J0L0*

Prepare Stock Solution (mg/L) by adding  
 50 g to 500 ml volumetric flask with DI water

<u>Conc. ppm</u>	<u><i>E. foetida</i> ml Stock solution</u>	<u><i>L. terrestris</i> ml of Stock solution</u>	<u>Total Volume, ml</u>
0	0	0	Hydration volume
1250	2.5	6.25	<i>90 ml</i> 
2500	5.0	12.5	
5000	10.0	25.0	
10000	20.0	50.0	
20000	40.0	100.0	

Each volume of stock solution is added as part of total hydration water, which is then added to 200 g dry weight of artificial soil for each *E. foetida* replicate, and 500 g dry weight of artificial soil for *L. terrestris* replicate.

Prepared by: *YT 8/29/03*

AQUA SURVEY, INC.  
EARTHWORM LIVE COUNT

Start Date: 8/8/03

Start Time: 1200

Job #: SCT

Sample ID: KC1

Sample ID	Rep	0	7	14
Control	1	10	10	9
	2	10	10	9
	<del>3</del>			
pH			xx	xx
1250	1	10	10	9
	2	10	9	7
pH			xx	xx
2500	1	10	8	7
	2	10	9	8
pH			xx	xx
5000	1	10	10	9
	2	10	10	10
pH			xx	xx
10000	1	10	0 <sup>10</sup>	—
	2	10	0 <sup>10</sup>	—
pH			xx	xx
20,000	1	10	0 <sup>10</sup>	—
	2	10	0 <sup>10</sup>	—
pH			xx	xx
Initials		YT	YT	YT
Date		8/8/03	8/15/03	8/22/03

	Temperature	Initials
Day 0:	_____	_____
Day 1:	_____	_____
Day 2:	_____	_____
Day 3:	_____	_____
Day 4:	_____	_____
Day 5:	_____	_____
Day 6:	_____	_____
Day 7:	_____	_____
Day 8:	_____	_____
Day 9:	_____	_____
Day 10:	_____	_____
Day 11:	_____	_____
Day 12:	_____	_____
Day 13:	_____	_____
Day 14:	_____	_____

Notes:

CT-TOX: BINOMIAL, MOVING AVERAGE, PROBIT, AND SPEARMAN METHODS

SPEARMAN-KARBER

TRIM: 16.67%  
 LC50: 6.598  
 95% CONFIDENCE LIMITS  
 ARE UNRELIABLE.

CONC. ppb	NUMBER EXPOSED	NUMBER DEAD	PERCENT DEAD	BINOMIAL PROB. (%)
1.25	20.	4.	20.00	.5909D+00
2.50	20.	5.	25.00	.2069D+01
5.00	20.	1.	5.00	.2003D-02
10.00	20.	20.	100.00	.9537D-04
20.00	20.	20.	100.00	.9537D-04

THE BINOMIAL TEST SHOWS THAT 5.00 AND 10.00 CAN BE USED AS STATISTICALLY SOUND CONSERVATIVE 95 PERCENT CONFIDENCE LIMITS SINCE THE ACTUAL CONFIDENCE LEVEL ASSOCIATED WITH THESE LIMITS IS 99.9979 PERCENT.  
 AN APPROXIMATE LC50 FOR THIS DATA SET IS 6.756

RESULTS USING MOVING AVERAGE

SPAN	G	LC50	95% CONFIDENCE LIMIT
4	.098	4.68	3.43 6.29

\*\*\*\*\* RESULTS CALCULATED BY PROBIT METHOD

ITERATIONS	G	H	GOODNESS OF FIT
7	2.754	9.95	.00

A PROBABILITY OF 0 MEANS LESS THAN 0.001

SLOPE = 2.59  
 95% CONFIDENCE LIMITS: -1.71 AND 6.90

LC50= 4.72  
 95% CONFIDENCE LIMITS: 0 AND + INFINITY

LC1 = .60  
 95% CONFIDENCE LIMITS: 0 AND 2.93

DATE: 8/8/03 TEST NUMBER: DURATION: 14 days  
 SAMPLE: KCl SPECIES: E. foetida

METHOD	LC50	CONFIDENCE LIMITS		
		LOWER	UPPER	SPAN
BINOMIAL	6.756	5.000	10.000	5.000
MAA	4.680	3.434	6.286	2.852
PROBIT	4.724	*****	*****	*****
SPEARMAN	6.598	*****	*****	*****

NOTE: MORTALITY PROPORTIONS WERE NOT MONOTONICALLY INCREASING.  
 ADJUSTMENTS WERE MADE PRIOR TO SPEARMAN-KARBER ESTIMATION.  
 \*\*\*\* = LIMIT DOES NOT EXIST

AQUA SURVEY, INC.

CULTURE LAB DISTRIBUTION FORM

DATE: 8/17/03  
TEST JOB#: 23-144/SRT CLIENT: LHM  
TEST LOCATION: IN-LAB [] FIELD [  
TEST SPECIES: E. foetida  
TOTAL NUMBER ORGANISMS TRANSFERRED: 1000+  
AQUA SURVEY, INC. CULTURE LAB INVESTIGATORS: BK

A. ORGANISMS

1. ASI CULTURE/HOLDING UNIT: Worm Holding tray  
2. RECEIVING LOG #: 23-086 Carolina Bto  
3. CULTURE LOG #: 23-0120  
4. AGE/SIZE INFORMATION: Adults

B. HOLDING [] CULTURE [] WATER PARAMETERS

1. TEMPERATURE: 22°C  
2. SALINITY: N/A  
3. WATER SOURCE: N/A

C. TRANSFER CUSTODY & TRANSFER

1. LIVESTOCK RELINQUISHMENT DATE: 8/17/03  
TIME: 0930 hrs.  
BY: BK  
2. LIVESTOCK RECEIVING DATE: 8/17/03  
TIME: 0930 hrs.  
BY: VT  
3. CULTURE SUPERVISOR OR SENIOR TECH. INITIALS: BK

REMARKS: \_\_\_\_\_  
\_\_\_\_\_

AQUA SURVEY, INC.

CULTURE LABORATORY RECEIVING FORM

RECEIVING LOG #: 23-086 DATE: 8/6/03

SHIPPING CARRIER: FedEx CARRIER LOG #: N/A

SPECIES: E. foetida NUMBER SHIPPED: 1000+

LIVESTOCK SOURCE/SHIPPER: Carolina Bio Supply

SHIPPER INVOICE #: N/A PACKER'S NAME: N/A

ASI ORDER REF. DATE: 7/30/03 ASI REF. INITIALS: BK

AGE/CHARACTERISTICS: Adults

TAXONOMIC VERIFICATION LOG #: N/A DATE: N/A

RECEIVING/WATER QUALITY PARAMETERS

D.O: \* TEMP: 22.0°C NH<sub>3</sub>/NO<sub>2</sub>: \*/\*

SALINITY/HARDNESS: \* ALK: \* pH: \*

WATER - CLEAR/CLOUDY CONTAINER SIZE/NUMBER: (2) large deli cups

# OF BLUE ICE®: 0 TYPE OF PACKING: Cardboard box

OBSERVATION/CONDITION OF LIVESTOCK: Appears healthy

-Added to holding tray with receiving substrate.

RECEIVING TECH INT.: BK SUPERVISOR'S INT.: BK

\* shipped in moist peat moss. BK  
E-7

Date	07/31/03
Time	15:41:27
Page	1 of 1

# PICK SLIP

**IF APPLICABLE, NOTIFY YOUR  
PAYING OFFICE ON RECEIPT OF  
MERCHANDISE.**

BILL TO: 111511

AQUA SURVEY  
ACCOUNTS PAYABLE  
469 POINT BREEZE RD  
FLEMINGTON NJ 08822

FROM: CAROLINA BIOLOGICAL SUPPLY COMPANY  
2700 YORK ROAD  
BURLINGTON, NC 27215-3398

Tel: 800-334-5551  
Fax: 336-584-3399

SHIP TO: 900491 YORK TERRELL  
AQUA SURVEY  
499 POINT BREEZE RD  
FRENCHTOWN NJ 08825

Dept.: 300

B/P: 3

Quote No.	Pick List No.	Requested Delivery Date	Ship Date	Ship Method	Our Order No.	Customer Purchase Order No.		
	185915	07/31/03	07/31/03	FEF	2650575 SO 00010	TERRELL		
Line	Qty No.	Ordered	Shipped	B/O	Catalog No.	Description	Unit	Location
1.000			20		141650	L409-REDWORMS	EA	
<p>ATTN: TIM WOODY SHIP HEAVIEST AVAILABLE. PRICING PER TIM WOODY</p>								

*Steve Wilson  
6353*

**AQUA SURVEY, INC.  
EARTHWORM LIVE COUNT**

Start Date: 8/29/03

Job #: 23-174/SRT

Start Time: 1200

Sample ID: KCL

Sample ID	Rep	0	DAY 7	14
PPM				
Control	1	10	10	10
	2	10	10	10
	3			
pH			xx	xx
1250	1	10	10	10
	2	10	10	10
pH			xx	xx
2500	1	10	10	10
	2	10	10	10
pH			xx	xx
5000	1	10	10	10
	2	10	9	9
pH			xx	xx
10000	1	10	0	—
	2	10	0	—
pH			xx	xx
20000 P 10/10/03	1	10	0	—
	2	10	0	—
pH			xx	xx
Initials		YT	YT	YT
Date		8/29/03	9/5/03	9/12/03

	Temperature	Initials
Day 0:	_____	_____
Day 1:	_____	_____
Day 2:	_____	_____
Day 3:	_____	_____
Day 4:	_____	_____
Day 5:	_____	_____
Day 6:	_____	_____
Day 7:	_____	_____
Day 8:	_____	_____
Day 9:	_____	_____
Day 10:	_____	_____
Day 11:	_____	_____
Day 12:	_____	_____
Day 13:	_____	_____
Day 14:	_____	_____

Notes:

CT-TOX: BINOMIAL, MOVING AVERAGE, PROBIT, AND SPEARMAN METHODS

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SPEARMAN-KARBER

TRIM: .00%  
 LC50: 6.830  
 95% LOWER CONFIDENCE: 6.384  
 95% UPPER CONFIDENCE: 7.308

---

CONC. <i>ppb. ppm</i>	NUMBER EXPOSED	NUMBER DEAD	PERCENT DEAD	BINOMIAL PROB. (%)
1.25	20.	0.	.00	.9537D-04
2.50	20.	0.	.00	.9537D-04
5.00	20.	1.	5.00	.2003D-02
10.00	20.	20.	100.00	.9537D-04
20.00	20.	20.	100.00	.9537D-04

THE BINOMIAL TEST SHOWS THAT 5.00 AND 10.00 CAN BE USED AS STATISTICALLY SOUND CONSERVATIVE 95 PERCENT CONFIDENCE LIMITS SINCE THE ACTUAL CONFIDENCE LEVEL ASSOCIATED WITH THESE LIMITS IS 99.9979 PERCENT.  
 AN APPROXIMATE LC50 FOR THIS DATA SET IS 6.756

---

WHEN THERE ARE LESS THAN TWO CONCENTRATIONS AT WHICH THE PERCENT DEAD IS BETWEEN 0 AND 100, NEITHER THE MOVING AVERAGE NOR THE PROBIT METHOD CAN GIVE ANY STATISTICALLY SOUND RESULTS.

---

DATE: 8/29/03                      TEST NUMBER:                      DURATION: 14 days  
 SAMPLE: KCl                          SPECIES: E. foetida

METHOD	LC50	CONFIDENCE LIMITS		
		LOWER	UPPER	SPAN
BINOMIAL	6.756	5.000	10.000	5.000
MAA	*****	*****	*****	*****
PROBIT	*****	*****	*****	*****
SPEARMAN	6.830	6.384	7.308	.924

\*\*\*\* = LIMIT DOES NOT EXIST

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AQUA SURVEY, INC.

CULTURE LAB DISTRIBUTION FORM

DATE: 8/28/03  
TEST JOB#: 23-144/SRT CLIENT: LHM  
TEST LOCATION: IN-LAB [] FIELD [  
TEST SPECIES: E. foetida  
TOTAL NUMBER ORGANISMS TRANSFERRED: 1000+  
AQUA SURVEY, INC. CULTURE LAB INVESTIGATORS: BK

A. ORGANISMS

1. ASI CULTURE/HOLDING UNIT: Warm holding tray  
2. RECEIVING LOG #: 23-097 ARO  
3. CULTURE LOG #: 23-0134  
4. AGE/SIZE INFORMATION: Adults

B. HOLDING [] CULTURE [] WATER PARAMETERS

1. TEMPERATURE: 23.0°C  
2. SALINITY: N/A  
3. WATER SOURCE: N/A

C. TRANSFER CUSTODY & TRANSFER

1. LIVESTOCK RELINQUISHMENT DATE: 8/28/03  
TIME: 0900 hrs.  
BY: BK  
2. LIVESTOCK RECEIVING DATE: 8/28/03  
TIME: 0900 hrs.  
BY: VT  
3. CULTURE SUPERVISOR OR SENIOR TECH. INITIALS: BK

REMARKS: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

AQUA SURVEY, INC.

CULTURE LABORATORY RECEIVING FORM

RECEIVING LOG #: 23-097      DATE: 8/27/03  
SHIPPING CARRIER: FedEx      CARRIER LOG #: N/A  
SPECIES: E. foetida      NUMBER SHIPPED: 1000+  
LIVESTOCK SOURCE/SHIPPER: Aquatic Research Organisms

SHIPPER INVOICE #: N/A      PACKER'S NAME: N/A  
ASI ORDER REF. DATE: 8/22/03      ASI REF. INITIALS: BK  
AGE/CHARACTERISTICS: Adults  
TAXONOMIC VERIFICATION LOG #: N/A      DATE: N/A

RECEIVING/WATER QUALITY PARAMETERS

D.O: \*      TEMP: 22.0°C      NH<sub>3</sub>/NO<sub>2</sub>: \*/\*  
SALINITY/HARDNESS: \*      ALK: \*      pH: \*  
WATER - CLEAR/CLOUDY      CONTAINER SIZE/NUMBER: (2) mesh bags w/ soil  
# OF BLUE ICE®: 1      TYPE OF PACKING: Styrofoam  
OBSERVATION/CONDITION OF LIVESTOCK: Appear healthy  
\* Shipped in moist topsoil and peat moss.

RECEIVING TECH. INIT.: BK      SUPERVISOR'S INIT.: BK



# Aquatic Research Organisms

## DATA SHEET

### I. Organism History

Species: EISENIA foetida

Source: Lab reared \_\_\_\_\_ Hatchery reared  Field collected \_\_\_\_\_

Hatch date mixed age Adults Receipt date \_\_\_\_\_  
> 90 DAYS

Lot number 082603EF Strain \_\_\_\_\_

Brood Origination F1

### II. Water Quality NOT applicable

Temperature \_\_\_\_\_ °C Salinity \_\_\_\_\_ ppt DO \_\_\_\_\_

pH \_\_\_\_\_ Hardness \_\_\_\_\_ ppm

### III. Culture Conditions

System: TOPSOIL/PEAT MASS

Diet: Flake Food \_\_\_\_\_ Phytoplankton \_\_\_\_\_ Trout Chow \_\_\_\_\_

Brine Shrimp \_\_\_\_\_ Rotifers \_\_\_\_\_ Other \_\_\_\_\_

Prophylactic Treatments: \_\_\_\_\_

Comments: KEEP WORMS MOIST, NOT WET  
KEEP COOL, PLACE FEED ON TOP OF SOIL

### IV. Shipping Information

Client: AQUASURVEY # of Organisms: 1000

Carrier: FED. EX Date Shipped: 08-26-2003

Biologist: Stan Simitzer

1 - 800 - 927 - 1650

PO Box 1271 • One Lafayette Road • Hampton, NH 03842 • (603) 926-1650

# **Continuous Temperature Recording**

Reporting time: Wednesday, October 08, 2003, 08:38

Recorder ID: 7010260      Deploy No: 6  
State: Run  
Span: 61 days, 20 hours  
Interval: one hour  
Samples: 1483  
Delay: 0 seconds  
Start: 8/7/2003 2:22:35 PM

Recover: 10/8/2003 08:27  
Data source: Unit '7010260'  
Trip Average:  
73.4°F= 23.0°C

Trip Std Dev:  
1.8°F= 1.0°C  
Window: 71.6°F= 22.0°C      75.2°F= 24.0°C  
Extremes: 68.0°F= 20.0°C      79.7°F= 26.5°C  
Description: 8/7/2003 13:21 Mini lab A 23-144

E.foetida

Notes:

Daily summary

Date	Samples	Min	Max	Under	Over	Daily Average
8/7/2003	9	71.6°F= 22.0°C	72.5°F= 22.5°C	0	0	72.5°F= 22.5°C
8/8/2003	24	71.6°F= 22.0°C	74.3°F= 23.5°C	0	0	72.5°F= 22.5°C
8/9/2003	24	73.4°F= 23.0°C	73.4°F= 23.0°C	0	0	73.4°F= 23.0°C
8/10/2003	24	73.4°F= 23.0°C	73.4°F= 23.0°C	0	0	73.4°F= 23.0°C
8/11/2003	24	73.4°F= 23.0°C	73.4°F= 23.0°C	0	0	73.4°F= 23.0°C
8/12/2003	24	72.5°F= 22.5°C	73.4°F= 23.0°C	0	0	73.4°F= 23.0°C
8/13/2003	24	73.4°F= 23.0°C	73.4°F= 23.0°C	0	0	73.4°F= 23.0°C
8/14/2003	24	72.5°F= 22.5°C	74.3°F= 23.5°C	0	0	73.4°F= 23.0°C
8/15/2003	24	73.4°F= 23.0°C	73.4°F= 23.0°C	0	0	73.4°F= 23.0°C
8/16/2003	24	72.5°F= 22.5°C	73.4°F= 23.0°C	0	0	73.4°F= 23.0°C
8/17/2003	24	72.5°F= 22.5°C	76.1°F= 24.5°C	0	8	74.3°F= 23.5°C
8/18/2003	24	73.4°F= 23.0°C	75.2°F= 24.0°C	0	0	74.3°F= 23.5°C
8/19/2003	24	73.4°F= 23.0°C	77.0°F= 25.0°C	0	7	75.2°F= 24.0°C
8/20/2003	24	73.4°F= 23.0°C	76.1°F= 24.5°C	0	4	75.2°F= 24.0°C
8/21/2003	24	73.4°F= 23.0°C	73.4°F= 23.0°C	0	0	73.4°F= 23.0°C
8/22/2003	24	73.4°F= 23.0°C	73.4°F= 23.0°C	0	0	73.4°F= 23.0°C
8/23/2003	24	73.4°F= 23.0°C	76.1°F= 24.5°C	0	9	75.2°F= 24.0°C
8/24/2003	24	73.4°F= 23.0°C	75.2°F= 24.0°C	0	0	74.3°F= 23.5°C
8/25/2003	24	72.5°F= 22.5°C	76.1°F= 24.5°C	0	2	74.3°F= 23.5°C
8/26/2003	24	72.5°F= 22.5°C	73.4°F= 23.0°C	0	0	72.5°F= 22.5°C
8/27/2003	24	72.5°F= 22.5°C	73.4°F= 23.0°C	0	0	72.5°F= 22.5°C
8/28/2003	24	71.6°F= 22.0°C	76.1°F= 24.5°C	0	2	73.4°F= 23.0°C
8/29/2003	24	70.7°F= 21.5°C	73.4°F= 23.0°C	5	0	72.5°F= 22.5°C
8/30/2003	24	72.5°F= 22.5°C	73.4°F= 23.0°C	0	0	73.4°F= 23.0°C
8/31/2003	24	72.5°F= 22.5°C	76.1°F= 24.5°C	0	3	74.3°F= 23.5°C
9/1/2003	24	71.6°F= 22.0°C				

			75.2°F= 24.0°C	0	73.4°F= 23.0°C
9/2/2003	24	71.6°F= 22.0°C			
			75.2°F= 24.0°C	0	74.3°F= 23.5°C
9/3/2003	24	72.5°F= 22.5°C			
			75.2°F= 24.0°C	0	74.3°F= 23.5°C
9/4/2003	24	72.5°F= 22.5°C			
			72.5°F= 22.5°C	0	72.5°F= 22.5°C
9/5/2003	24	72.5°F= 22.5°C			
			76.1°F= 24.5°C	7	74.3°F= 23.5°C

Page: 2

9/6/2003	24	71.6°F= 22.0°C 75.2°F= 24.0°C	0	73.4°F= 23.0°C
9/7/2003	24	71.6°F= 22.0°C 75.2°F= 24.0°C	0	73.4°F= 23.0°C
9/8/2003	24	71.6°F= 22.0°C 76.1°F= 24.5°C	3	73.4°F= 23.0°C
9/9/2003	24	71.6°F= 22.0°C 75.2°F= 24.0°C	0	73.4°F= 23.0°C
9/10/2003	24	73.4°F= 23.0°C 74.3°F= 23.5°C	0	74.3°F= 23.5°C
9/11/2003	24	72.5°F= 22.5°C 74.3°F= 23.5°C	0	73.4°F= 23.0°C
9/12/2003	24	72.5°F= 22.5°C 75.2°F= 24.0°C	0	74.3°F= 23.5°C
9/13/2003	24	71.6°F= 22.0°C 74.3°F= 23.5°C	0	72.5°F= 22.5°C
9/14/2003	24	72.5°F= 22.5°C 75.2°F= 24.0°C	0	73.4°F= 23.0°C
9/15/2003	24	72.5°F= 22.5°C 75.2°F= 24.0°C	0	73.4°F= 23.0°C
9/16/2003	24	72.5°F= 22.5°C 75.2°F= 24.0°C	0	74.3°F= 23.5°C
9/17/2003	24	73.4°F= 23.0°C 75.2°F= 24.0°C	0	74.3°F= 23.5°C
9/18/2003	24	71.6°F= 22.0°C 75.2°F= 24.0°C	0	73.4°F= 23.0°C
9/19/2003	24	71.6°F= 22.0°C 75.2°F= 24.0°C	0	72.5°F= 22.5°C
9/20/2003	24	71.6°F= 22.0°C 72.5°F= 22.5°C	0	71.6°F= 22.0°C
9/21/2003	24	71.6°F= 22.0°C 76.1°F= 24.5°C	3	74.3°F= 23.5°C
9/22/2003	24	72.5°F= 22.5°C 76.1°F= 24.5°C	3	74.3°F= 23.5°C
9/23/2003	24	71.6°F= 22.0°C 72.5°F= 22.5°C	0	72.5°F= 22.5°C
9/24/2003	24	71.6°F= 22.0°C 75.2°F= 24.0°C	0	74.3°F= 23.5°C
9/25/2003	24	70.7°F= 21.5°C 71.6°F= 22.0°C	8	71.6°F= 22.0°C
9/26/2003	24	71.6°F= 22.0°C 72.5°F= 22.5°C	0	71.6°F= 22.0°C
9/27/2003	24	72.5°F= 22.5°C 79.7°F= 26.5°C	12	75.2°F= 24.0°C
9/28/2003	24	75.2°F= 24.0°C 79.7°F= 26.5°C	23	77.9°F= 25.5°C
9/29/2003	24	73.4°F= 23.0°C 76.1°F= 24.5°C	1	74.3°F= 23.5°C
9/30/2003	24	72.5°F= 22.5°C 74.3°F= 23.5°C	0	73.4°F= 23.0°C
10/1/2003	24	72.5°F= 22.5°C 74.3°F= 23.5°C	0	73.4°F= 23.0°C
10/2/2003	24	72.5°F= 22.5°C 74.3°F= 23.5°C	0	72.5°F= 22.5°C
10/3/2003	24	71.6°F= 22.0°C 72.5°F= 22.5°C	0	72.5°F= 22.5°C
10/4/2003	24	71.6°F= 22.0°C 72.5°F= 22.5°C	0	71.6°F= 22.0°C
10/5/2003	24	71.6°F= 22.0°C 72.5°F= 22.5°C	0	72.5°F= 22.5°C
10/6/2003	24	70.7°F= 21.5°C 77.9°F= 25.5°C	6	72.5°F= 22.5°C
10/7/2003	24	68.0°F= 20.0°C 78.8°F= 26.0°C	9	73.4°F= 23.0°C
10/8/2003	10	70.7°F= 21.5°C 73.4°F= 23.0°C	3	71.6°F= 22.0°C





Readings

Sample	Date	Time	°F	°C	Sample	Date	Time	°F	°C
1	8/7/2003	14:22:35	71.6	22.0	51	8/9/2003	16:22:35	73.4	23.0
2	8/7/2003	15:22:35	71.6	22.0	52	8/9/2003	17:22:35	73.4	23.0
3	8/7/2003	16:22:35	72.5	22.5	53	8/9/2003	18:22:35	73.4	23.0
4	8/7/2003	17:22:35	72.5	22.5	54	8/9/2003	19:22:35	73.4	23.0
5	8/7/2003	18:22:35	72.5	22.5	55	8/9/2003	20:22:35	73.4	23.0
6	8/7/2003	19:22:35	72.5	22.5	56	8/9/2003	21:22:35	73.4	23.0
7	8/7/2003	20:22:35	72.5	22.5	57	8/9/2003	22:22:35	73.4	23.0
8	8/7/2003	21:22:35	72.5	22.5	58	8/9/2003	23:22:35	73.4	23.0
9	8/7/2003	22:22:35	72.5	22.5	59	8/10/2003	00:22:35	73.4	23.0
10	8/7/2003	23:22:35	72.5	22.5	60	8/10/2003	01:22:35	73.4	23.0
11	8/8/2003	00:22:35	71.6	22.0	61	8/10/2003	02:22:35	73.4	23.0
12	8/8/2003	01:22:35	71.6	22.0	62	8/10/2003	03:22:35	73.4	23.0
13	8/8/2003	02:22:35	71.6	22.0	63	8/10/2003	04:22:35	73.4	23.0
14	8/8/2003	03:22:35	71.6	22.0	64	8/10/2003	05:22:35	73.4	23.0
15	8/8/2003	04:22:35	71.6	22.0	65	8/10/2003	06:22:35	73.4	23.0
16	8/8/2003	05:22:35	71.6	22.0	66	8/10/2003	07:22:35	73.4	23.0
17	8/8/2003	06:22:35	71.6	22.0	67	8/10/2003	08:22:35	73.4	23.0
18	8/8/2003	07:22:35	71.6	22.0	68	8/10/2003	09:22:35	73.4	23.0
19	8/8/2003	08:22:35	72.5	22.5	69	8/10/2003	10:22:35	73.4	23.0
20	8/8/2003	09:22:35	72.5	22.5	70	8/10/2003	11:22:35	73.4	23.0
21	8/8/2003	10:22:35	73.4	23.0	71	8/10/2003	12:22:35	73.4	23.0
22	8/8/2003	11:22:35	74.3	23.5	72	8/10/2003	13:22:35	73.4	23.0
23	8/8/2003	12:22:35	74.3	23.5	73	8/10/2003	14:22:35	73.4	23.0
24	8/8/2003	13:22:35	74.3	23.5	74	8/10/2003	15:22:35	73.4	23.0
25	8/8/2003	14:22:35	74.3	23.5	75	8/10/2003	16:22:35	73.4	23.0
26	8/8/2003	15:22:35	74.3	23.5	76	8/10/2003	17:22:35	73.4	23.0
27	8/8/2003	16:22:35	73.4	23.0	77	8/10/2003	18:22:35	73.4	23.0
28	8/8/2003	17:22:35	73.4	23.0	78	8/10/2003	19:22:35	73.4	23.0
29	8/8/2003	18:22:35	73.4	23.0	79	8/10/2003	20:22:35	73.4	23.0
30	8/8/2003	19:22:35	73.4	23.0	80	8/10/2003	21:22:35	73.4	23.0
31	8/8/2003	20:22:35	73.4	23.0	81	8/10/2003	22:22:35	73.4	23.0
32	8/8/2003	21:22:35	73.4	23.0	82	8/10/2003	23:22:35	73.4	23.0
33	8/8/2003	22:22:35	73.4	23.0	83	8/11/2003	00:22:35	73.4	23.0
34	8/8/2003	23:22:35	73.4	23.0	84	8/11/2003	01:22:35	73.4	23.0
35	8/9/2003	00:22:35	73.4	23.0	85	8/11/2003	02:22:35	73.4	23.0
36	8/9/2003	01:22:35	73.4	23.0	86	8/11/2003	03:22:35	73.4	23.0
37	8/9/2003	02:22:35	73.4	23.0	87	8/11/2003	04:22:35	73.4	23.0
38	8/9/2003	03:22:35	73.4	23.0	88	8/11/2003	05:22:35	73.4	23.0
39	8/9/2003	04:22:35	73.4	23.0	89	8/11/2003	06:22:35	73.4	23.0
40	8/9/2003	05:22:35	73.4	23.0	90	8/11/2003	07:22:35	73.4	23.0
41	8/9/2003	06:22:35	73.4	23.0	91	8/11/2003	08:22:35	73.4	23.0
42	8/9/2003	07:22:35	73.4	23.0	92	8/11/2003	09:22:35	73.4	23.0
43	8/9/2003	08:22:35	73.4	23.0	93	8/11/2003	10:22:35	73.4	23.0
44	8/9/2003	09:22:35	73.4	23.0	94	8/11/2003	11:22:35	73.4	23.0
45	8/9/2003	10:22:35	73.4	23.0	95	8/11/2003	12:22:35	73.4	23.0
46	8/9/2003	11:22:35	73.4	23.0	96	8/11/2003	13:22:35	73.4	23.0
47	8/9/2003	12:22:35	73.4	23.0	97	8/11/2003	14:22:35	73.4	23.0
48	8/9/2003	13:22:35	73.4	23.0	98	8/11/2003	15:22:35	73.4	23.0
49	8/9/2003	14:22:35	73.4	23.0	99	8/11/2003	16:22:35	73.4	23.0
50	8/9/2003	15:22:35	73.4	23.0	100	8/11/2003	17:22:35	73.4	23.0

Sample	Date	Time	°F	°C	Sample	Date	Time	°F	°C
101	8/11/2003	18:22:35	73.4	23.0	151	8/13/2003	20:22:35	73.4	23.0
102	8/11/2003	19:22:35	73.4	23.0	152	8/13/2003	21:22:35	73.4	23.0
103	8/11/2003	20:22:35	73.4	23.0	153	8/13/2003	22:22:35	73.4	23.0
104	8/11/2003	21:22:35	73.4	23.0	154	8/13/2003	23:22:35	73.4	23.0
105	8/11/2003	22:22:35	73.4	23.0	155	8/14/2003	00:22:35	73.4	23.0
106	8/11/2003	23:22:35	73.4	23.0	156	8/14/2003	01:22:35	73.4	23.0
107	8/12/2003	00:22:35	73.4	23.0	157	8/14/2003	02:22:35	73.4	23.0
108	8/12/2003	01:22:35	73.4	23.0	158	8/14/2003	03:22:35	73.4	23.0
109	8/12/2003	02:22:35	73.4	23.0	159	8/14/2003	04:22:35	73.4	23.0
110	8/12/2003	03:22:35	72.5	22.5	160	8/14/2003	05:22:35	72.5	22.5
111	8/12/2003	04:22:35	72.5	22.5	161	8/14/2003	06:22:35	72.5	22.5
112	8/12/2003	05:22:35	72.5	22.5	162	8/14/2003	07:22:35	72.5	22.5
113	8/12/2003	06:22:35	72.5	22.5	163	8/14/2003	08:22:35	73.4	23.0
114	8/12/2003	07:22:35	73.4	23.0	164	8/14/2003	09:22:35	73.4	23.0
115	8/12/2003	08:22:35	73.4	23.0	165	8/14/2003	10:22:35	73.4	23.0
116	8/12/2003	09:22:35	73.4	23.0	166	8/14/2003	11:22:35	73.4	23.0
117	8/12/2003	10:22:35	73.4	23.0	167	8/14/2003	12:22:35	73.4	23.0
118	8/12/2003	11:22:35	73.4	23.0	168	8/14/2003	13:22:35	73.4	23.0
119	8/12/2003	12:22:35	73.4	23.0	169	8/14/2003	14:22:35	73.4	23.0
120	8/12/2003	13:22:35	73.4	23.0	170	8/14/2003	15:22:35	73.4	23.0
121	8/12/2003	14:22:35	73.4	23.0	171	8/14/2003	16:22:35	73.4	23.0
122	8/12/2003	15:22:35	73.4	23.0	172	8/14/2003	17:22:35	73.4	23.0
123	8/12/2003	16:22:35	73.4	23.0	173	8/14/2003	18:22:35	73.4	23.0
124	8/12/2003	17:22:35	73.4	23.0	174	8/14/2003	19:22:35	73.4	23.0
125	8/12/2003	18:22:35	73.4	23.0	175	8/14/2003	20:22:35	73.4	23.0
126	8/12/2003	19:22:35	73.4	23.0	176	8/14/2003	21:22:35	74.3	23.5
127	8/12/2003	20:22:35	73.4	23.0	177	8/14/2003	22:22:35	73.4	23.0
128	8/12/2003	21:22:35	73.4	23.0	178	8/14/2003	23:22:35	73.4	23.0
129	8/12/2003	22:22:35	73.4	23.0	179	8/15/2003	00:22:35	73.4	23.0
130	8/12/2003	23:22:35	73.4	23.0	180	8/15/2003	01:22:35	73.4	23.0
131	8/13/2003	00:22:35	73.4	23.0	181	8/15/2003	02:22:35	73.4	23.0
132	8/13/2003	01:22:35	73.4	23.0	182	8/15/2003	03:22:35	73.4	23.0
133	8/13/2003	02:22:35	73.4	23.0	183	8/15/2003	04:22:35	73.4	23.0
134	8/13/2003	03:22:35	73.4	23.0	184	8/15/2003	05:22:35	73.4	23.0
135	8/13/2003	04:22:35	73.4	23.0	185	8/15/2003	06:22:35	73.4	23.0
136	8/13/2003	05:22:35	73.4	23.0	186	8/15/2003	07:22:35	73.4	23.0
137	8/13/2003	06:22:35	73.4	23.0	187	8/15/2003	08:22:35	73.4	23.0
138	8/13/2003	07:22:35	73.4	23.0	188	8/15/2003	09:22:35	73.4	23.0
139	8/13/2003	08:22:35	73.4	23.0	189	8/15/2003	10:22:35	73.4	23.0
140	8/13/2003	09:22:35	73.4	23.0	190	8/15/2003	11:22:35	73.4	23.0
141	8/13/2003	10:22:35	73.4	23.0	191	8/15/2003	12:22:35	73.4	23.0
142	8/13/2003	11:22:35	73.4	23.0	192	8/15/2003	13:22:35	73.4	23.0
143	8/13/2003	12:22:35	73.4	23.0	193	8/15/2003	14:22:35	73.4	23.0
144	8/13/2003	13:22:35	73.4	23.0	194	8/15/2003	15:22:35	73.4	23.0
145	8/13/2003	14:22:35	73.4	23.0	195	8/15/2003	16:22:35	73.4	23.0
146	8/13/2003	15:22:35	73.4	23.0	196	8/15/2003	17:22:35	73.4	23.0
147	8/13/2003	16:22:35	73.4	23.0	197	8/15/2003	18:22:35	73.4	23.0
148	8/13/2003	17:22:35	73.4	23.0	198	8/15/2003	19:22:35	73.4	23.0
149	8/13/2003	18:22:35	73.4	23.0	199	8/15/2003	20:22:35	73.4	23.0
150	8/13/2003	19:22:35	73.4	23.0	200	8/15/2003	21:22:35	73.4	23.0

Sample	Date	Time	°F	°C	Sample	Date	Time	°F	°C
201	8/15/2003	22:22:35	73.4	23.0	251	8/18/2003	00:22:35	75.2	24.0
202	8/15/2003	23:22:35	73.4	23.0	252	8/18/2003	01:22:35	75.2	24.0
203	8/16/2003	00:22:35	73.4	23.0	253	8/18/2003	02:22:35	75.2	24.0
204	8/16/2003	01:22:35	73.4	23.0	254	8/18/2003	03:22:35	74.3	23.5
205	8/16/2003	02:22:35	73.4	23.0	255	8/18/2003	04:22:35	74.3	23.5
206	8/16/2003	03:22:35	73.4	23.0	256	8/18/2003	05:22:35	74.3	23.5
207	8/16/2003	04:22:35	73.4	23.0	257	8/18/2003	06:22:35	74.3	23.5
208	8/16/2003	05:22:35	72.5	22.5	258	8/18/2003	07:22:35	74.3	23.5
209	8/16/2003	06:22:35	72.5	22.5	259	8/18/2003	08:22:35	74.3	23.5
210	8/16/2003	07:22:35	72.5	22.5	260	8/18/2003	09:22:35	74.3	23.5
211	8/16/2003	08:22:35	72.5	22.5	261	8/18/2003	10:22:35	74.3	23.5
212	8/16/2003	09:22:35	73.4	23.0	262	8/18/2003	11:22:35	74.3	23.5
213	8/16/2003	10:22:35	73.4	23.0	263	8/18/2003	12:22:35	75.2	24.0
214	8/16/2003	11:22:35	73.4	23.0	264	8/18/2003	13:22:35	75.2	24.0
215	8/16/2003	12:22:35	73.4	23.0	265	8/18/2003	14:22:35	75.2	24.0
216	8/16/2003	13:22:35	73.4	23.0	266	8/18/2003	15:22:35	74.3	23.5
217	8/16/2003	14:22:35	73.4	23.0	267	8/18/2003	16:22:35	74.3	23.5
218	8/16/2003	15:22:35	73.4	23.0	268	8/18/2003	17:22:35	73.4	23.0
219	8/16/2003	16:22:35	73.4	23.0	269	8/18/2003	18:22:35	73.4	23.0
220	8/16/2003	17:22:35	73.4	23.0	270	8/18/2003	19:22:35	73.4	23.0
221	8/16/2003	18:22:35	73.4	23.0	271	8/18/2003	20:22:35	73.4	23.0
222	8/16/2003	19:22:35	73.4	23.0	272	8/18/2003	21:22:35	73.4	23.0
223	8/16/2003	20:22:35	73.4	23.0	273	8/18/2003	22:22:35	73.4	23.0
224	8/16/2003	21:22:35	73.4	23.0	274	8/18/2003	23:22:35	73.4	23.0
225	8/16/2003	22:22:35	73.4	23.0	275	8/19/2003	00:22:35	73.4	23.0
226	8/16/2003	23:22:35	73.4	23.0	276	8/19/2003	01:22:35	73.4	23.0
227	8/17/2003	00:22:35	73.4	23.0	277	8/19/2003	02:22:35	73.4	23.0
228	8/17/2003	01:22:35	73.4	23.0	278	8/19/2003	03:22:35	73.4	23.0
229	8/17/2003	02:22:35	73.4	23.0	279	8/19/2003	04:22:35	73.4	23.0
230	8/17/2003	03:22:35	72.5	22.5	280	8/19/2003	05:22:35	74.3	23.5
231	8/17/2003	04:22:35	72.5	22.5	281	8/19/2003	06:22:35	74.3	23.5
232	8/17/2003	05:22:35	72.5	22.5	282	8/19/2003	07:22:35	74.3	23.5
233	8/17/2003	06:22:35	72.5	22.5	283	8/19/2003	08:22:35	74.3	23.5
234	8/17/2003	07:22:35	72.5	22.5	284	8/19/2003	09:22:35	74.3	23.5
235	8/17/2003	08:22:35	73.4	23.0	285	8/19/2003	10:22:35	75.2	24.0
236	8/17/2003	09:22:35	74.3	23.5	286	8/19/2003	11:22:35	75.2	24.0
237	8/17/2003	10:22:35	74.3	23.5	287	8/19/2003	12:22:35	75.2	24.0
238	8/17/2003	11:22:35	75.2	24.0	288	8/19/2003	13:22:35	75.2	24.0
239	8/17/2003	12:22:35	75.2	24.0	289	8/19/2003	14:22:35	76.1	24.5
240	8/17/2003	13:22:35	75.2	24.0	290	8/19/2003	15:22:35	76.1	24.5
241	8/17/2003	14:22:35	76.1	24.5	291	8/19/2003	16:22:35	76.1	24.5
242	8/17/2003	15:22:35	76.1	24.5	292	8/19/2003	17:22:35	77.0	25.0
243	8/17/2003	16:22:35	76.1	24.5	293	8/19/2003	18:22:35	77.0	25.0
244	8/17/2003	17:22:35	76.1	24.5	294	8/19/2003	19:22:35	77.0	25.0
245	8/17/2003	18:22:35	76.1	24.5	295	8/19/2003	20:22:35	76.1	24.5
246	8/17/2003	19:22:35	76.1	24.5	296	8/19/2003	21:22:35	75.2	24.0
247	8/17/2003	20:22:35	76.1	24.5	297	8/19/2003	22:22:35	74.3	23.5
248	8/17/2003	21:22:35	76.1	24.5	298	8/19/2003	23:22:35	74.3	23.5
249	8/17/2003	22:22:35	75.2	24.0	299	8/20/2003	00:22:35	73.4	23.0
250	8/17/2003	23:22:35	75.2	24.0	300	8/20/2003	01:22:35	74.3	23.5

Sample	Date	Time	°F	°C	Sample	Date	Time	°F	°C
301	8/20/2003	02:22:35	74.3	23.5	351	8/22/2003	04:22:35	73.4	23.0
302	8/20/2003	03:22:35	75.2	24.0	352	8/22/2003	05:22:35	73.4	23.0
303	8/20/2003	04:22:35	75.2	24.0	353	8/22/2003	06:22:35	73.4	23.0
304	8/20/2003	05:22:35	75.2	24.0	354	8/22/2003	07:22:35	73.4	23.0
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313	8/20/2003	14:22:35	76.1	24.5	363	8/22/2003	16:22:35	73.4	23.0
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316	8/20/2003	17:22:35	74.3	23.5	366	8/22/2003	19:22:35	73.4	23.0
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322	8/20/2003	23:22:35	73.4	23.0	372	8/23/2003	01:22:35	73.4	23.0
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329	8/21/2003	06:22:35	73.4	23.0	379	8/23/2003	08:22:35	74.3	23.5
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343	8/21/2003	20:22:35	73.4	23.0	393	8/23/2003	22:22:35	75.2	24.0
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345	8/21/2003	22:22:35	73.4	23.0	395	8/24/2003	00:22:35	75.2	24.0
346	8/21/2003	23:22:35	73.4	23.0	396	8/24/2003	01:22:35	74.3	23.5
347	8/22/2003	00:22:35	73.4	23.0	397	8/24/2003	02:22:35	74.3	23.5
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349	8/22/2003	02:22:35	73.4	23.0	399	8/24/2003	04:22:35	74.3	23.5
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403	8/24/2003	08:22:35	74.3	23.5	453	8/26/2003	10:22:35	72.5	22.5
404	8/24/2003	09:22:35	74.3	23.5	454	8/26/2003	11:22:35	72.5	22.5
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408	8/24/2003	13:22:35	74.3	23.5	458	8/26/2003	15:22:35	73.4	23.0
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410	8/24/2003	15:22:35	75.2	24.0	460	8/26/2003	17:22:35	73.4	23.0
411	8/24/2003	16:22:35	75.2	24.0	461	8/26/2003	18:22:35	73.4	23.0
412	8/24/2003	17:22:35	75.2	24.0	462	8/26/2003	19:22:35	73.4	23.0
413	8/24/2003	18:22:35	75.2	24.0	463	8/26/2003	20:22:35	73.4	23.0
414	8/24/2003	19:22:35	75.2	24.0	464	8/26/2003	21:22:35	73.4	23.0
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416	8/24/2003	21:22:35	74.3	23.5	466	8/26/2003	23:22:35	73.4	23.0
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419	8/25/2003	00:22:35	72.5	22.5	469	8/27/2003	02:22:35	72.5	22.5
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421	8/25/2003	02:22:35	73.4	23.0	471	8/27/2003	04:22:35	72.5	22.5
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426	8/25/2003	07:22:35	74.3	23.5	476	8/27/2003	09:22:35	72.5	22.5
427	8/25/2003	08:22:35	74.3	23.5	477	8/27/2003	10:22:35	72.5	22.5
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430	8/25/2003	11:22:35	75.2	24.0	480	8/27/2003	13:22:35	72.5	22.5
431	8/25/2003	12:22:35	75.2	24.0	481	8/27/2003	14:22:35	72.5	22.5
432	8/25/2003	13:22:35	75.2	24.0	482	8/27/2003	15:22:35	73.4	23.0
433	8/25/2003	14:22:35	76.1	24.5	483	8/27/2003	16:22:35	73.4	23.0
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443	8/26/2003	00:22:35	73.4	23.0	493	8/28/2003	02:22:35	72.5	22.5
444	8/26/2003	01:22:35	72.5	22.5	494	8/28/2003	03:22:35	72.5	22.5
445	8/26/2003	02:22:35	72.5	22.5	495	8/28/2003	04:22:35	72.5	22.5
446	8/26/2003	03:22:35	72.5	22.5	496	8/28/2003	05:22:35	72.5	22.5
447	8/26/2003	04:22:35	72.5	22.5	497	8/28/2003	06:22:35	72.5	22.5
448	8/26/2003	05:22:35	72.5	22.5	498	8/28/2003	07:22:35	73.4	23.0
449	8/26/2003	06:22:35	72.5	22.5	499	8/28/2003	08:22:35	73.4	23.0
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Sample	Date	Time	°F	°C	Sample	Date	Time	°F	°C
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502	8/28/2003	11:22:35	75.2	24.0	552	8/30/2003	13:22:35	73.4	23.0
503	8/28/2003	12:22:35	75.2	24.0	553	8/30/2003	14:22:35	73.4	23.0
504	8/28/2003	13:22:35	75.2	24.0	554	8/30/2003	15:22:35	73.4	23.0
505	8/28/2003	14:22:35	76.1	24.5	555	8/30/2003	16:22:35	73.4	23.0
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512	8/28/2003	21:22:35	72.5	22.5	562	8/30/2003	23:22:35	72.5	22.5
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521	8/29/2003	06:22:35	70.7	21.5	571	8/31/2003	08:22:35	74.3	23.5
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524	8/29/2003	09:22:35	71.6	22.0	574	8/31/2003	11:22:35	74.3	23.5
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526	8/29/2003	11:22:35	72.5	22.5	576	8/31/2003	13:22:35	75.2	24.0
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529	8/29/2003	14:22:35	73.4	23.0	579	8/31/2003	16:22:35	75.2	24.0
530	8/29/2003	15:22:35	73.4	23.0	580	8/31/2003	17:22:35	75.2	24.0
531	8/29/2003	16:22:35	73.4	23.0	581	8/31/2003	18:22:35	76.1	24.5
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603	9/1/2003	16:22:35	75.2 24.0	653	9/3/2003	18:22:35	75.2 24.0
604	9/1/2003	17:22:35	75.2 24.0	654	9/3/2003	19:22:35	74.3 23.5
605	9/1/2003	18:22:35	75.2 24.0	655	9/3/2003	20:22:35	73.4 23.0
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611	9/2/2003	00:22:35	75.2 24.0	661	9/4/2003	02:22:35	72.5 22.5
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621	9/2/2003	10:22:35	75.2 24.0	671	9/4/2003	12:22:35	72.5 22.5
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625	9/2/2003	14:22:35	74.3 23.5	675	9/4/2003	16:22:35	72.5 22.5
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627	9/2/2003	16:22:35	74.3 23.5	677	9/4/2003	18:22:35	72.5 22.5
628	9/2/2003	17:22:35	73.4 23.0	678	9/4/2003	19:22:35	72.5 22.5
629	9/2/2003	18:22:35	72.5 22.5	679	9/4/2003	20:22:35	72.5 22.5
630	9/2/2003	19:22:35	72.5 22.5	680	9/4/2003	21:22:35	72.5 22.5
631	9/2/2003	20:22:35	72.5 22.5	681	9/4/2003	22:22:35	72.5 22.5
632	9/2/2003	21:22:35	71.6 22.0	682	9/4/2003	23:22:35	72.5 22.5
633	9/2/2003	22:22:35	72.5 22.5	683	9/5/2003	00:22:35	72.5 22.5
634	9/2/2003	23:22:35	72.5 22.5	684	9/5/2003	01:22:35	72.5 22.5
635	9/3/2003	00:22:35	73.4 23.0	685	9/5/2003	02:22:35	72.5 22.5
636	9/3/2003	01:22:35	73.4 23.0	686	9/5/2003	03:22:35	72.5 22.5
637	9/3/2003	02:22:35	73.4 23.0	687	9/5/2003	04:22:35	72.5 22.5
638	9/3/2003	03:22:35	73.4 23.0	688	9/5/2003	05:22:35	72.5 22.5
639	9/3/2003	04:22:35	74.3 23.5	689	9/5/2003	06:22:35	72.5 22.5
640	9/3/2003	05:22:35	74.3 23.5	690	9/5/2003	07:22:35	73.4 23.0
641	9/3/2003	06:22:35	74.3 23.5	691	9/5/2003	08:22:35	73.4 23.0
642	9/3/2003	07:22:35	74.3 23.5	692	9/5/2003	09:22:35	73.4 23.0
643	9/3/2003	08:22:35	74.3 23.5	693	9/5/2003	10:22:35	73.4 23.0
644	9/3/2003	09:22:35	74.3 23.5	694	9/5/2003	11:22:35	74.3 23.5
645	9/3/2003	10:22:35	74.3 23.5	695	9/5/2003	12:22:35	74.3 23.5
646	9/3/2003	11:22:35	74.3 23.5	696	9/5/2003	13:22:35	74.3 23.5
647	9/3/2003	12:22:35	75.2 24.0	697	9/5/2003	14:22:35	75.2 24.0
648	9/3/2003	13:22:35	75.2 24.0	698	9/5/2003	15:22:35	76.1 24.5
649	9/3/2003	14:22:35	75.2 24.0	699	9/5/2003	16:22:35	76.1 24.5
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702	9/5/2003	19:22:35	76.1	24.5	752	9/7/2003	21:22:35	72.5	22.5
703	9/5/2003	20:22:35	76.1	24.5	753	9/7/2003	22:22:35	72.5	22.5
704	9/5/2003	21:22:35	76.1	24.5	754	9/7/2003	23:22:35	72.5	22.5
705	9/5/2003	22:22:35	75.2	24.0	755	9/8/2003	00:22:35	72.5	22.5
706	9/5/2003	23:22:35	75.2	24.0	756	9/8/2003	01:22:35	71.6	22.0
707	9/6/2003	00:22:35	75.2	24.0	757	9/8/2003	02:22:35	71.6	22.0
708	9/6/2003	01:22:35	75.2	24.0	758	9/8/2003	03:22:35	71.6	22.0
709	9/6/2003	02:22:35	75.2	24.0	759	9/8/2003	04:22:35	71.6	22.0
710	9/6/2003	03:22:35	75.2	24.0	760	9/8/2003	05:22:35	71.6	22.0
711	9/6/2003	04:22:35	75.2	24.0	761	9/8/2003	06:22:35	72.5	22.5
712	9/6/2003	05:22:35	75.2	24.0	762	9/8/2003	07:22:35	72.5	22.5
713	9/6/2003	06:22:35	75.2	24.0	763	9/8/2003	08:22:35	73.4	23.0
714	9/6/2003	07:22:35	74.3	23.5	764	9/8/2003	09:22:35	73.4	23.0
715	9/6/2003	08:22:35	74.3	23.5	765	9/8/2003	10:22:35	73.4	23.0
716	9/6/2003	09:22:35	74.3	23.5	766	9/8/2003	11:22:35	74.3	23.5
717	9/6/2003	10:22:35	75.2	24.0	767	9/8/2003	12:22:35	74.3	23.5
718	9/6/2003	11:22:35	74.3	23.5	768	9/8/2003	13:22:35	75.2	24.0
719	9/6/2003	12:22:35	73.4	23.0	769	9/8/2003	14:22:35	75.2	24.0
720	9/6/2003	13:22:35	73.4	23.0	770	9/8/2003	15:22:35	75.2	24.0
721	9/6/2003	14:22:35	72.5	22.5	771	9/8/2003	16:22:35	75.2	24.0
722	9/6/2003	15:22:35	72.5	22.5	772	9/8/2003	17:22:35	76.1	24.5
723	9/6/2003	16:22:35	72.5	22.5	773	9/8/2003	18:22:35	76.1	24.5
724	9/6/2003	17:22:35	72.5	22.5	774	9/8/2003	19:22:35	76.1	24.5
725	9/6/2003	18:22:35	72.5	22.5	775	9/8/2003	20:22:35	75.2	24.0
726	9/6/2003	19:22:35	72.5	22.5	776	9/8/2003	21:22:35	74.3	23.5
727	9/6/2003	20:22:35	72.5	22.5	777	9/8/2003	22:22:35	73.4	23.0
728	9/6/2003	21:22:35	72.5	22.5	778	9/8/2003	23:22:35	73.4	23.0
729	9/6/2003	22:22:35	71.6	22.0	779	9/9/2003	00:22:35	72.5	22.5
730	9/6/2003	23:22:35	71.6	22.0	780	9/9/2003	01:22:35	72.5	22.5
731	9/7/2003	00:22:35	71.6	22.0	781	9/9/2003	02:22:35	72.5	22.5
732	9/7/2003	01:22:35	71.6	22.0	782	9/9/2003	03:22:35	72.5	22.5
733	9/7/2003	02:22:35	72.5	22.5	783	9/9/2003	04:22:35	72.5	22.5
734	9/7/2003	03:22:35	72.5	22.5	784	9/9/2003	05:22:35	71.6	22.0
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736	9/7/2003	05:22:35	73.4	23.0	786	9/9/2003	07:22:35	71.6	22.0
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738	9/7/2003	07:22:35	73.4	23.0	788	9/9/2003	09:22:35	72.5	22.5
739	9/7/2003	08:22:35	73.4	23.0	789	9/9/2003	10:22:35	73.4	23.0
740	9/7/2003	09:22:35	73.4	23.0	790	9/9/2003	11:22:35	73.4	23.0
741	9/7/2003	10:22:35	73.4	23.0	791	9/9/2003	12:22:35	74.3	23.5
742	9/7/2003	11:22:35	74.3	23.5	792	9/9/2003	13:22:35	74.3	23.5
743	9/7/2003	12:22:35	74.3	23.5	793	9/9/2003	14:22:35	74.3	23.5
744	9/7/2003	13:22:35	74.3	23.5	794	9/9/2003	15:22:35	74.3	23.5
745	9/7/2003	14:22:35	75.2	24.0	795	9/9/2003	16:22:35	75.2	24.0
746	9/7/2003	15:22:35	75.2	24.0	796	9/9/2003	17:22:35	75.2	24.0
747	9/7/2003	16:22:35	75.2	24.0	797	9/9/2003	18:22:35	75.2	24.0
748	9/7/2003	17:22:35	74.3	23.5	798	9/9/2003	19:22:35	75.2	24.0
749	9/7/2003	18:22:35	73.4	23.0	799	9/9/2003	20:22:35	75.2	24.0
750	9/7/2003	19:22:35	73.4	23.0	800	9/9/2003	21:22:35	75.2	24.0



Sample	Date	Time	°F	°C	Sample	Date	Time	°F	°C
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802	9/9/2003	23:22:35	74.3	23.5	852	9/12/2003	01:22:35	73.4	23.0
803	9/10/2003	00:22:35	74.3	23.5	853	9/12/2003	02:22:35	73.4	23.0
804	9/10/2003	01:22:35	74.3	23.5	854	9/12/2003	03:22:35	74.3	23.5
805	9/10/2003	02:22:35	74.3	23.5	855	9/12/2003	04:22:35	74.3	23.5
806	9/10/2003	03:22:35	74.3	23.5	856	9/12/2003	05:22:35	74.3	23.5
807	9/10/2003	04:22:35	74.3	23.5	857	9/12/2003	06:22:35	74.3	23.5
808	9/10/2003	05:22:35	74.3	23.5	858	9/12/2003	07:22:35	74.3	23.5
809	9/10/2003	06:22:35	74.3	23.5	859	9/12/2003	08:22:35	74.3	23.5
810	9/10/2003	07:22:35	74.3	23.5	860	9/12/2003	09:22:35	74.3	23.5
811	9/10/2003	08:22:35	74.3	23.5	861	9/12/2003	10:22:35	74.3	23.5
812	9/10/2003	09:22:35	74.3	23.5	862	9/12/2003	11:22:35	74.3	23.5
813	9/10/2003	10:22:35	74.3	23.5	863	9/12/2003	12:22:35	74.3	23.5
814	9/10/2003	11:22:35	74.3	23.5	864	9/12/2003	13:22:35	74.3	23.5
815	9/10/2003	12:22:35	74.3	23.5	865	9/12/2003	14:22:35	74.3	23.5
816	9/10/2003	13:22:35	74.3	23.5	866	9/12/2003	15:22:35	74.3	23.5
817	9/10/2003	14:22:35	74.3	23.5	867	9/12/2003	16:22:35	75.2	24.0
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819	9/10/2003	16:22:35	73.4	23.0	869	9/12/2003	18:22:35	75.2	24.0
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821	9/10/2003	18:22:35	73.4	23.0	871	9/12/2003	20:22:35	75.2	24.0
822	9/10/2003	19:22:35	73.4	23.0	872	9/12/2003	21:22:35	75.2	24.0
823	9/10/2003	20:22:35	74.3	23.5	873	9/12/2003	22:22:35	74.3	23.5
824	9/10/2003	21:22:35	74.3	23.5	874	9/12/2003	23:22:35	74.3	23.5
825	9/10/2003	22:22:35	74.3	23.5	875	9/13/2003	00:22:35	74.3	23.5
826	9/10/2003	23:22:35	74.3	23.5	876	9/13/2003	01:22:35	74.3	23.5
827	9/11/2003	00:22:35	74.3	23.5	877	9/13/2003	02:22:35	73.4	23.0
828	9/11/2003	01:22:35	74.3	23.5	878	9/13/2003	03:22:35	72.5	22.5
829	9/11/2003	02:22:35	74.3	23.5	879	9/13/2003	04:22:35	72.5	22.5
830	9/11/2003	03:22:35	74.3	23.5	880	9/13/2003	05:22:35	72.5	22.5
831	9/11/2003	04:22:35	74.3	23.5	881	9/13/2003	06:22:35	71.6	22.0
832	9/11/2003	05:22:35	74.3	23.5	882	9/13/2003	07:22:35	71.6	22.0
833	9/11/2003	06:22:35	74.3	23.5	883	9/13/2003	08:22:35	71.6	22.0
834	9/11/2003	07:22:35	74.3	23.5	884	9/13/2003	09:22:35	71.6	22.0
835	9/11/2003	08:22:35	74.3	23.5	885	9/13/2003	10:22:35	71.6	22.0
836	9/11/2003	09:22:35	74.3	23.5	886	9/13/2003	11:22:35	71.6	22.0
837	9/11/2003	10:22:35	74.3	23.5	887	9/13/2003	12:22:35	71.6	22.0
838	9/11/2003	11:22:35	74.3	23.5	888	9/13/2003	13:22:35	71.6	22.0
839	9/11/2003	12:22:35	74.3	23.5	889	9/13/2003	14:22:35	71.6	22.0
840	9/11/2003	13:22:35	73.4	23.0	890	9/13/2003	15:22:35	71.6	22.0
841	9/11/2003	14:22:35	73.4	23.0	891	9/13/2003	16:22:35	71.6	22.0
842	9/11/2003	15:22:35	72.5	22.5	892	9/13/2003	17:22:35	71.6	22.0
843	9/11/2003	16:22:35	72.5	22.5	893	9/13/2003	18:22:35	71.6	22.0
844	9/11/2003	17:22:35	72.5	22.5	894	9/13/2003	19:22:35	72.5	22.5
845	9/11/2003	18:22:35	72.5	22.5	895	9/13/2003	20:22:35	72.5	22.5
846	9/11/2003	19:22:35	72.5	22.5	896	9/13/2003	21:22:35	72.5	22.5
847	9/11/2003	20:22:35	72.5	22.5	897	9/13/2003	22:22:35	72.5	22.5
848	9/11/2003	21:22:35	72.5	22.5	898	9/13/2003	23:22:35	72.5	22.5
849	9/11/2003	22:22:35	72.5	22.5	899	9/14/2003	00:22:35	72.5	22.5
850	9/11/2003	23:22:35	72.5	22.5	900	9/14/2003	01:22:35	72.5	22.5

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902	9/14/2003	03:22:35	72.5	22.5	952	9/16/2003	05:22:35	72.5	22.5
903	9/14/2003	04:22:35	72.5	22.5	953	9/16/2003	06:22:35	72.5	22.5
904	9/14/2003	05:22:35	72.5	22.5	954	9/16/2003	07:22:35	72.5	22.5
905	9/14/2003	06:22:35	72.5	22.5	955	9/16/2003	08:22:35	73.4	23.0
906	9/14/2003	07:22:35	72.5	22.5	956	9/16/2003	09:22:35	73.4	23.0
907	9/14/2003	08:22:35	73.4	23.0	957	9/16/2003	10:22:35	74.3	23.5
908	9/14/2003	09:22:35	73.4	23.0	958	9/16/2003	11:22:35	74.3	23.5
909	9/14/2003	10:22:35	74.3	23.5	959	9/16/2003	12:22:35	74.3	23.5
910	9/14/2003	11:22:35	75.2	24.0	960	9/16/2003	13:22:35	75.2	24.0
911	9/14/2003	12:22:35	75.2	24.0	961	9/16/2003	14:22:35	75.2	24.0
912	9/14/2003	13:22:35	75.2	24.0	962	9/16/2003	15:22:35	75.2	24.0
913	9/14/2003	14:22:35	75.2	24.0	963	9/16/2003	16:22:35	75.2	24.0
914	9/14/2003	15:22:35	75.2	24.0	964	9/16/2003	17:22:35	75.2	24.0
915	9/14/2003	16:22:35	74.3	23.5	965	9/16/2003	18:22:35	75.2	24.0
916	9/14/2003	17:22:35	74.3	23.5	966	9/16/2003	19:22:35	75.2	24.0
917	9/14/2003	18:22:35	74.3	23.5	967	9/16/2003	20:22:35	75.2	24.0
918	9/14/2003	19:22:35	74.3	23.5	968	9/16/2003	21:22:35	75.2	24.0
919	9/14/2003	20:22:35	73.4	23.0	969	9/16/2003	22:22:35	75.2	24.0
920	9/14/2003	21:22:35	73.4	23.0	970	9/16/2003	23:22:35	74.3	23.5
921	9/14/2003	22:22:35	73.4	23.0	971	9/17/2003	00:22:35	74.3	23.5
922	9/14/2003	23:22:35	73.4	23.0	972	9/17/2003	01:22:35	74.3	23.5
923	9/15/2003	00:22:35	73.4	23.0	973	9/17/2003	02:22:35	74.3	23.5
924	9/15/2003	01:22:35	72.5	22.5	974	9/17/2003	03:22:35	74.3	23.5
925	9/15/2003	02:22:35	72.5	22.5	975	9/17/2003	04:22:35	74.3	23.5
926	9/15/2003	03:22:35	72.5	22.5	976	9/17/2003	05:22:35	74.3	23.5
927	9/15/2003	04:22:35	72.5	22.5	977	9/17/2003	06:22:35	74.3	23.5
928	9/15/2003	05:22:35	72.5	22.5	978	9/17/2003	07:22:35	74.3	23.5
929	9/15/2003	06:22:35	72.5	22.5	979	9/17/2003	08:22:35	74.3	23.5
930	9/15/2003	07:22:35	72.5	22.5	980	9/17/2003	09:22:35	74.3	23.5
931	9/15/2003	08:22:35	72.5	22.5	981	9/17/2003	10:22:35	74.3	23.5
932	9/15/2003	09:22:35	72.5	22.5	982	9/17/2003	11:22:35	75.2	24.0
933	9/15/2003	10:22:35	73.4	23.0	983	9/17/2003	12:22:35	74.3	23.5
934	9/15/2003	11:22:35	73.4	23.0	984	9/17/2003	13:22:35	73.4	23.0
935	9/15/2003	12:22:35	74.3	23.5	985	9/17/2003	14:22:35	73.4	23.0
936	9/15/2003	13:22:35	74.3	23.5	986	9/17/2003	15:22:35	73.4	23.0
937	9/15/2003	14:22:35	75.2	24.0	987	9/17/2003	16:22:35	74.3	23.5
938	9/15/2003	15:22:35	74.3	23.5	988	9/17/2003	17:22:35	74.3	23.5
939	9/15/2003	16:22:35	73.4	23.0	989	9/17/2003	18:22:35	75.2	24.0
940	9/15/2003	17:22:35	73.4	23.0	990	9/17/2003	19:22:35	75.2	24.0
941	9/15/2003	18:22:35	73.4	23.0	991	9/17/2003	20:22:35	75.2	24.0
942	9/15/2003	19:22:35	73.4	23.0	992	9/17/2003	21:22:35	75.2	24.0
943	9/15/2003	20:22:35	72.5	22.5	993	9/17/2003	22:22:35	75.2	24.0
944	9/15/2003	21:22:35	72.5	22.5	994	9/17/2003	23:22:35	74.3	23.5
945	9/15/2003	22:22:35	72.5	22.5	995	9/18/2003	00:22:35	74.3	23.5
946	9/15/2003	23:22:35	72.5	22.5	996	9/18/2003	01:22:35	74.3	23.5
947	9/16/2003	00:22:35	72.5	22.5	997	9/18/2003	02:22:35	75.2	24.0
948	9/16/2003	01:22:35	72.5	22.5	998	9/18/2003	03:22:35	75.2	24.0
949	9/16/2003	02:22:35	72.5	22.5	999	9/18/2003	04:22:35	75.2	24.0
950	9/16/2003	03:22:35	72.5	22.5	1000	9/18/2003	05:22:35	75.2	24.0

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1002	9/18/2003	07:22:35	75.2	24.0	1052	9/20/2003	09:22:35	71.6	22.0
1003	9/18/2003	08:22:35	75.2	24.0	1053	9/20/2003	10:22:35	71.6	22.0
1004	9/18/2003	09:22:35	75.2	24.0	1054	9/20/2003	11:22:35	71.6	22.0
1005	9/18/2003	10:22:35	75.2	24.0	1055	9/20/2003	12:22:35	71.6	22.0
1006	9/18/2003	11:22:35	74.3	23.5	1056	9/20/2003	13:22:35	71.6	22.0
1007	9/18/2003	12:22:35	73.4	23.0	1057	9/20/2003	14:22:35	72.5	22.5
1008	9/18/2003	13:22:35	73.4	23.0	1058	9/20/2003	15:22:35	72.5	22.5
1009	9/18/2003	14:22:35	72.5	22.5	1059	9/20/2003	16:22:35	72.5	22.5
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1017	9/18/2003	22:22:35	71.6	22.0	1067	9/21/2003	00:22:35	71.6	22.0
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1021	9/19/2003	02:22:35	71.6	22.0	1071	9/21/2003	04:22:35	73.4	23.0
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1028	9/19/2003	09:22:35	71.6	22.0	1078	9/21/2003	11:22:35	75.2	24.0
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1049	9/20/2003	06:22:35	71.6	22.0	1099	9/22/2003	08:22:35	74.3	23.5
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1103	9/22/2003	12:22:35	75.2	24.0	1153	9/24/2003	14:22:35	75.2	24.0
1104	9/22/2003	13:22:35	75.2	24.0	1154	9/24/2003	15:22:35	75.2	24.0
1105	9/22/2003	14:22:35	76.1	24.5	1155	9/24/2003	16:22:35	75.2	24.0
1106	9/22/2003	15:22:35	76.1	24.5	1156	9/24/2003	17:22:35	74.3	23.5
1107	9/22/2003	16:22:35	76.1	24.5	1157	9/24/2003	18:22:35	73.4	23.0
1108	9/22/2003	17:22:35	75.2	24.0	1158	9/24/2003	19:22:35	72.5	22.5
1109	9/22/2003	18:22:35	74.3	23.5	1159	9/24/2003	20:22:35	72.5	22.5
1110	9/22/2003	19:22:35	73.4	23.0	1160	9/24/2003	21:22:35	71.6	22.0
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1113	9/22/2003	22:22:35	72.5	22.5	1163	9/25/2003	00:22:35	71.6	22.0
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1115	9/23/2003	00:22:35	72.5	22.5	1165	9/25/2003	02:22:35	71.6	22.0
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1117	9/23/2003	02:22:35	72.5	22.5	1167	9/25/2003	04:22:35	71.6	22.0
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1122	9/23/2003	07:22:35	72.5	22.5	1172	9/25/2003	09:22:35	70.7	21.5
1123	9/23/2003	08:22:35	72.5	22.5	1173	9/25/2003	10:22:35	70.7	21.5
1124	9/23/2003	09:22:35	72.5	22.5	1174	9/25/2003	11:22:35	70.7	21.5
1125	9/23/2003	10:22:35	72.5	22.5	1175	9/25/2003	12:22:35	70.7	21.5
1126	9/23/2003	11:22:35	72.5	22.5	1176	9/25/2003	13:22:35	70.7	21.5
1127	9/23/2003	12:22:35	72.5	22.5	1177	9/25/2003	14:22:35	70.7	21.5
1128	9/23/2003	13:22:35	72.5	22.5	1178	9/25/2003	15:22:35	70.7	21.5
1129	9/23/2003	14:22:35	72.5	22.5	1179	9/25/2003	16:22:35	71.6	22.0
1130	9/23/2003	15:22:35	72.5	22.5	1180	9/25/2003	17:22:35	71.6	22.0
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1144	9/24/2003	05:22:35	74.3	23.5	1194	9/26/2003	07:22:35	71.6	22.0
1145	9/24/2003	06:22:35	74.3	23.5	1195	9/26/2003	08:22:35	71.6	22.0
1146	9/24/2003	07:22:35	74.3	23.5	1196	9/26/2003	09:22:35	71.6	22.0
1147	9/24/2003	08:22:35	74.3	23.5	1197	9/26/2003	10:22:35	71.6	22.0
1148	9/24/2003	09:22:35	75.2	24.0	1198	9/26/2003	11:22:35	71.6	22.0
1149	9/24/2003	10:22:35	75.2	24.0	1199	9/26/2003	12:22:35	71.6	22.0
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1204	9/26/2003	17:22:35	72.5	22.5
1205	9/26/2003	18:22:35	72.5	22.5
1206	9/26/2003	19:22:35	72.5	22.5
1207	9/26/2003	20:22:35	72.5	22.5
1208	9/26/2003	21:22:35	71.6	22.0
1209	9/26/2003	22:22:35	71.6	22.0
1210	9/26/2003	23:22:35	72.5	22.5
1211	9/27/2003	00:22:35	72.5	22.5
1212	9/27/2003	01:22:35	72.5	22.5
1213	9/27/2003	02:22:35	72.5	22.5
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1215	9/27/2003	04:22:35	72.5	22.5
1216	9/27/2003	05:22:35	72.5	22.5
1217	9/27/2003	06:22:35	72.5	22.5
1218	9/27/2003	07:22:35	72.5	22.5
1219	9/27/2003	08:22:35	72.5	22.5
1220	9/27/2003	09:22:35	73.4	23.0
1221	9/27/2003	10:22:35	75.2	24.0
1222	9/27/2003	11:22:35	76.1	24.5
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1224	9/27/2003	13:22:35	77.0	25.0
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1226	9/27/2003	15:22:35	77.9	25.5
1227	9/27/2003	16:22:35	78.8	26.0
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1248	9/28/2003	13:22:35	77.0	25.0
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1252	9/28/2003	17:22:35	77.0	25.0
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1255	9/28/2003	20:22:35	76.1	24.5
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1262	9/29/2003	03:22:35	74.3	23.5
1263	9/29/2003	04:22:35	74.3	23.5
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1305	9/30/2003	22:22:35	72.5	22.5
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1356	10/3/2003	01:22:35	72.5	22.5
1357	10/3/2003	02:22:35	72.5	22.5
1358	10/3/2003	03:22:35	72.5	22.5
1359	10/3/2003	04:22:35	72.5	22.5
1360	10/3/2003	05:22:35	72.5	22.5
1361	10/3/2003	06:22:35	71.6	22.0
1362	10/3/2003	07:22:35	71.6	22.0
1363	10/3/2003	08:22:35	71.6	22.0
1364	10/3/2003	09:22:35	71.6	22.0
1365	10/3/2003	10:22:35	71.6	22.0
1366	10/3/2003	11:22:35	71.6	22.0
1367	10/3/2003	12:22:35	71.6	22.0
1368	10/3/2003	13:22:35	71.6	22.0
1369	10/3/2003	14:22:35	72.5	22.5
1370	10/3/2003	15:22:35	72.5	22.5
1371	10/3/2003	16:22:35	72.5	22.5
1372	10/3/2003	17:22:35	72.5	22.5
1373	10/3/2003	18:22:35	72.5	22.5
1374	10/3/2003	19:22:35	72.5	22.5
1375	10/3/2003	20:22:35	71.6	22.0
1376	10/3/2003	21:22:35	71.6	22.0
1377	10/3/2003	22:22:35	71.6	22.0
1378	10/3/2003	23:22:35	71.6	22.0
1379	10/4/2003	00:22:35	71.6	22.0
1380	10/4/2003	01:22:35	71.6	22.0
1381	10/4/2003	02:22:35	71.6	22.0
1382	10/4/2003	03:22:35	71.6	22.0
1383	10/4/2003	04:22:35	71.6	22.0
1384	10/4/2003	05:22:35	71.6	22.0
1385	10/4/2003	06:22:35	71.6	22.0
1386	10/4/2003	07:22:35	71.6	22.0
1387	10/4/2003	08:22:35	71.6	22.0
1388	10/4/2003	09:22:35	71.6	22.0
1389	10/4/2003	10:22:35	71.6	22.0
1390	10/4/2003	11:22:35	71.6	22.0
1391	10/4/2003	12:22:35	71.6	22.0
1392	10/4/2003	13:22:35	71.6	22.0
1393	10/4/2003	14:22:35	71.6	22.0
1394	10/4/2003	15:22:35	71.6	22.0
1395	10/4/2003	16:22:35	72.5	22.5
1396	10/4/2003	17:22:35	72.5	22.5
1397	10/4/2003	18:22:35	72.5	22.5
1398	10/4/2003	19:22:35	72.5	22.5
1399	10/4/2003	20:22:35	72.5	22.5
1400	10/4/2003	21:22:35	72.5	22.5

Sample	Date	Time	°F	°C	Sample	Date	Time	°F	°C
1401	10/4/2003	22:22:35	72.5	22.5	1451	10/7/2003	00:22:35	71.6	22.0
1402	10/4/2003	23:22:35	72.5	22.5	1452	10/7/2003	01:22:35	70.7	21.5
1403	10/5/2003	00:22:35	72.5	22.5	1453	10/7/2003	02:22:35	69.8	21.0
1404	10/5/2003	01:22:35	72.5	22.5	1454	10/7/2003	03:22:35	69.8	21.0
1405	10/5/2003	02:22:35	72.5	22.5	1455	10/7/2003	04:22:35	68.9	20.5
1406	10/5/2003	03:22:35	72.5	22.5	1456	10/7/2003	05:22:35	68.9	20.5
1407	10/5/2003	04:22:35	72.5	22.5	1457	10/7/2003	06:22:35	68.0	20.0
1408	10/5/2003	05:22:35	72.5	22.5	1458	10/7/2003	07:22:35	68.0	20.0
1409	10/5/2003	06:22:35	72.5	22.5	1459	10/7/2003	08:22:35	68.9	20.5
1410	10/5/2003	07:22:35	72.5	22.5	1460	10/7/2003	09:22:35	70.7	21.5
1411	10/5/2003	08:22:35	72.5	22.5	1461	10/7/2003	10:22:35	73.4	23.0
1412	10/5/2003	09:22:35	71.6	22.0	1462	10/7/2003	11:22:35	75.2	24.0
1413	10/5/2003	10:22:35	71.6	22.0	1463	10/7/2003	12:22:35	77.0	25.0
1414	10/5/2003	11:22:35	71.6	22.0	1464	10/7/2003	13:22:35	77.9	25.5
1415	10/5/2003	12:22:35	71.6	22.0	1465	10/7/2003	14:22:35	77.9	25.5
1416	10/5/2003	13:22:35	71.6	22.0	1466	10/7/2003	15:22:35	77.9	25.5
1417	10/5/2003	14:22:35	72.5	22.5	1467	10/7/2003	16:22:35	78.8	26.0
1418	10/5/2003	15:22:35	72.5	22.5	1468	10/7/2003	17:22:35	78.8	26.0
1419	10/5/2003	16:22:35	72.5	22.5	1469	10/7/2003	18:22:35	78.8	26.0
1420	10/5/2003	17:22:35	72.5	22.5	1470	10/7/2003	19:22:35	77.9	25.5
1421	10/5/2003	18:22:35	72.5	22.5	1471	10/7/2003	20:22:35	77.0	25.0
1422	10/5/2003	19:22:35	71.6	22.0	1472	10/7/2003	21:22:35	75.2	24.0
1423	10/5/2003	20:22:35	71.6	22.0	1473	10/7/2003	22:22:35	74.3	23.5
1424	10/5/2003	21:22:35	71.6	22.0	1474	10/7/2003	23:22:35	73.4	23.0
1425	10/5/2003	22:22:35	71.6	22.0	1475	10/8/2003	00:22:35	72.5	22.5
1426	10/5/2003	23:22:35	71.6	22.0	1476	10/8/2003	01:22:35	72.5	22.5
1427	10/6/2003	00:22:35	71.6	22.0	1477	10/8/2003	02:22:35	71.6	22.0
1428	10/6/2003	01:22:35	71.6	22.0	1478	10/8/2003	03:22:35	71.6	22.0
1429	10/6/2003	02:22:35	71.6	22.0	1479	10/8/2003	04:22:35	70.7	21.5
1430	10/6/2003	03:22:35	71.6	22.0	1480	10/8/2003	05:22:35	70.7	21.5
1431	10/6/2003	04:22:35	71.6	22.0	1481	10/8/2003	06:22:35	70.7	21.5
1432	10/6/2003	05:22:35	71.6	22.0	1482	10/8/2003	07:22:35	71.6	22.0
1433	10/6/2003	06:22:35	70.7	21.5	1483	10/8/2003	08:22:35	72.5	22.5
1434	10/6/2003	07:22:35	70.7	21.5					
1435	10/6/2003	08:22:35	70.7	21.5					
1436	10/6/2003	09:22:35	70.7	21.5					
1437	10/6/2003	10:22:35	70.7	21.5					
1438	10/6/2003	11:22:35	70.7	21.5					
1439	10/6/2003	12:22:35	71.6	22.0					
1440	10/6/2003	13:22:35	71.6	22.0					
1441	10/6/2003	14:22:35	71.6	22.0					
1442	10/6/2003	15:22:35	72.5	22.5					
1443	10/6/2003	16:22:35	72.5	22.5					
1444	10/6/2003	17:22:35	77.9	25.5					
1445	10/6/2003	18:22:35	77.9	25.5					
1446	10/6/2003	19:22:35	76.1	24.5					
1447	10/6/2003	20:22:35	75.2	24.0					
1448	10/6/2003	21:22:35	73.4	23.0					
1449	10/6/2003	22:22:35	72.5	22.5					
1450	10/6/2003	23:22:35	71.6	22.0					