# ROCKY MOUNTAIN ARSENAL NATIONAL WILDLIFE REFUGE

## Sampling and Analysis Plan

## Bison Pesticide Residue Study

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# Signature Page
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**Approvals:**

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## ACRONYMS

| | |
|---|---|
| %R | Percent Recovery |
| ARER | Aquatic Residual Ecological Risk |
| ASTM | American Society for Testing and Materials |
| BCS | Biota Screening Criteria |
| c-o-c | Chain-of-Custody |
| COC | Contaminant of Concern |
| CQAP | Chemical Quality Assurance Plan |
| DBCP | Dibromochloropropane |
| DQCR | Daily Quality Control Report |
| DQI | Data Quality Indicator |
| DQO | Data Quality Objective |
| DQR | Data Quality Requirement |
| DSR | Data Summary Report |
| EPA | U.S. Environmental Protection Agency |
| ESD | Explanation of Significant Differences |
| HHE | Human Health Exceedance |
| HI | Hazard Index |
| HQ | Hazard Quotient |
| LCS | Laboratory Control Samples |
| OMC | Operation and Maintenance Contractor |
| oz | ounce |
| ppb | parts per billion |
| PBM | Performance-Based Method |
| PMRMA | Program Manager for the Rocky Mountain Arsenal |
| QA | Quality Assurance |
| QAPP | Quality Assurance Project Plan |
| QAR | Quality Assurance Representative |
| QED | Quod Erat Demonstratum (completion of the proof) |
| QC | Quality Control |
| RMA | Rocky Mountain Arsenal |
| RMAED | Rocky Mountain Arsenal Environmental Database |
| RMANWR | Rocky Mountain Arsenal National Wildlife Refuge |
| ROD | Record of Decision |
| RPD | Relative Percent Difference |
| RVO | Remediation Venture Office |
| SAP | Sampling and Analysis Plan |
| SEC | Site Evaluation Criteria |
| TRER | Terrestrial Residual Ecological Risk |

| | |
|---|---|
| ug/g | Micrograms per gram |
| ug/L | Micrograms per liter |
| USDA | United States Department of Agriculture |
| USFWS | United States Fish and Wildlife Service |

# PART I – Field Sampling Plan

## 1.0 INTRODUCTION

### *1.1 Purpose*

The purpose of this Sampling and Analysis Plan (SAP) is to demonstrate that dieldrin concentrations in bison tissues from the Rocky Mountain Arsenal National Wildlife Refuge (Refuge) are below the United States Department of Agriculture (USDA) action level of 300 parts per billion (ppb) (U.S. Department of Agriculture 2006). This SAP has been developed according to guidelines and requirements of the Rocky Mountain Arsenal Program Office. The following documents were also consulted in development of this plan: Guidance on Choosing a Sampling Design for Environmental Data Collection (U.S. Environmental Protection Agency 2002b); Guidance for Quality Assurance Project Plans (U.S. Environmental Protection Agency 2002a); Uniform Federal Policy for Quality Assurance Project Plans (Intergovernmental Data Quality Task Force 2005); Guidance on Systematic Planning Using the Data Quality Objectives Process (U.S. Environmental Protection Agency 2006).

The U.S. Fish and Wildlife Service (USFWS) and the U.S. Army will implement the requirements described in this SAP to collect and analyze samples of bison tissue from the Refuge in order to demonstrate compliance with the USDA action level for dieldrin (U.S. Department of Agriculture 2006). These data will also be compared to the EPA site-specific Tissue Screening Level. Data obtained from these investigations will be used in accordance with the Data Quality Objectives (DQOs) outlined in Section 4.1.

### *1.2 Scope*

This SAP provides the background for the Bison Pesticide Residue Study, the sampling and analytical quality requirements, the Data Quality Objectives, and data assessment processes for evaluating sample data,

## 2.0 BACKGROUND

Located approximately ten miles from downtown Denver, portions of the land within the acquisition boundary of the Refuge (15,988 acres) have a well-documented history of significant environmental disturbance and contamination. The primary causes of contamination were the manufacture of chemical weapons by the U.S. Army from the World War II through Vietnam eras and the production of pesticides by Shell Oil Company from 1950-1980. Common industrial and waste disposal practices resulted in contamination of structures, soil, surface water, and groundwater. As a result of this

contamination, in 1987 the Rocky Mountain Arsenal (RMA) was placed on the National Priorities List (NPL) for environmental cleanup under the Comprehensive Environmental Response Compensation, and Liability Act." As the remedy has been completed, portions of RMA have been deleted from the NPL site. Partial deletion from the NPL are based on the determination by EPA and the Colorado Department of Public Health and Environment (CDPHE), that all appropriate response actions under CERCLA have been completed (other than operation, maintenance, and five-year reviews) and there are no known hazardous substances above health-based levels remaining in the partial deletion areas, with respect to anticipated uses of and access to the site which are identified in the Federal Facility Agreement (FFA), On-Post ROD, and Public Law 102-402. Currently, over 14,700 acres have been transferred to the USFWS for establishment of the Rocky Mountain Arsenal Wildlife Refuge.

Remediation activities mandated under CERCLA will result in restoration of approximately 67% (10,739 acres) of Refuge lands to native short- and mixed-grass prairie. Other habitats that will be present on the Refuge include shrublands, forested lands, riparian areas, and numerous manmade features (irrigation lakes, ditches, homesteads, etc.), many of which are of cultural or historic importance.

The USFWS recently finalized a Habitat Management Plan for the Refuge (U.S. Fish and Wildlife Service 2013). This plan further reaffirms two high priorities for the Refuge will be: (1) to promote successful long-term establishment and maintenance of seeded restoration sites, existing native prairies and shrublands, and habitat for the resources of concern; and (2) maintain a bison (*Bison bison*) population that contributes to the Department of the Interior's Bison Conservation Initiative and helps maintain the structure and composition of native and restored prairies necessary to support priority grassland bird species (U.S. Fish and Wildlife Service 2013). Based upon an analysis of available forage and the habitat needs of all wildlife species, the USFWS developed the following objective for the Refuge bison herd:

> *Manage bison populations, in support of the Department of the Interior's Bison Conservation Initiative, at or below the carrying capacity for the refuge. At present, bison populations would range between 25-40 animals and should not exceed 42 animals. Once additional grazing units and opportunities are fully in place, long-term bison populations would range between 110-180 animals and should not exceed 209 animals (U.S. Fish and Wildlife Service 2013).*

In order to implement this objective and effectively manage the Refuge bison herd (Table 1), it is necessary to periodically remove animals from the Refuge. When appropriate and consistent with the Department of Interior's Bison Conservation

Initiative (U.S. Department of the Interior 2008), animals may be transferred to other national wildlife refuges. The USFWS may also reduce the herd by making animals available to Native American tribes or by auctioning surplus animals to the public. Whenever animals leave the Refuge, it becomes possible that they could be consumed by the public at some point in the future. Since consumption of RMA game is currently prohibited by the ROD, it is necessary to show that RMA bison are safe for human consumption and to eliminate or revise the game consumption prohibition. The purpose of this SAP is to obtain data for both of these objectives.

The decision-making process for this issue is necessarily sequential. USFWS will obtain tissue data to confirm that RMA bison are safe for human consumption as a due diligence effort. If the data are satisfactory, USFWS can transfer the animals without restrictions or tracking requirements while EPA, Army, and Shell work to revise or eliminate the RMA game consumption restriction in a timely manner.

**Table 1.** Bison population of the Rocky Mountain Arsenal NWR, 2007 to present

| | ***Bulls*** | ***Cows*** | ***Unknown*** | ***Calves*** | ***Import/Death*** | ***Total*** |
|---|---|---|---|---|---|---|
| 2007 | 3 | 13 | 0 | 3 | 16/0 | 19 |
| 2008 | 7 | 14 | 0 | 7 | 2/0 | 30 |
| 2009 | 17 | 20 | 2 | 7 | 10/1 | 46 |
| 2010 | 19 | 20 | 2 | 8 | 1/7 | 49 |
| 2011 | 18 | 20 | 9 | 11 | 3/1 | 59 |
| 2012 | 16 | 20 | 21 | 15 | 0/2 | 72 |
| 2013 | 14 | 20 | 36 | 16 | 0/2 | 86 |

In 2007, 16 bison were imported bison from the National Bison Range; 2008, 2 bison were imported from Sullys Hill NGP; 2009, 10 bison were imported from the National Bison Range; 2010, 1 bison was imported from the American Prairie Foundation; 2011, 3 bison were imported from Wichita Mountains National Wildlife Refuge.

Bison currently range on approximately 2,232 acres of the RMANWR in two pasture units. Subject to available funding, an additional pasture unit will be developed in 2013 and as more infrastructure (fences, water supplies, cattle guards, etc) is constructed, approximately 12,165 acres will eventually be available for bison grazing.
The following restriction is currently found in the Record of Decision:

> *The Rocky Mountain Arsenal National Wildlife Refuge Act of 1992 and the FFA restrict future land use, and prohibit certain activities such as agriculture, use of on-post groundwater as a drinking source, and consumption of fish and game taken at RMA (Foster Wheeler Environmental Corporation 1996).*

Parties have agreed to revise this restriction and to allow consumption of bison from Refuge if compliance to relevant standards for human consumption (USDA Action Levels, TSLs) can be demonstrated for bison. A site-specific EPA Tissue Screening Level will also be considered as part of the restriction revision process.

The primary chemical of potential concern that may still be present in Refuge soils at low concentrations is the organochlorine pesticide dieldrin (see Section 4.2). These low soil concentrations of dieldrin theoretically should not lead to detectable residues of dieldrin in Refuge bison meat. However, there are no data available for dieldrin concentrations in Refuge bison tissues. The purpose of this sampling and analysis plan is to demonstrate compliance of Refuge bison tissues with the USDA action level for dieldrin. The compliance demonstration will consist of two phases.

- In Phase 1, 15 historical tissues from Refuge bison that died from natural causes over the last several years will be analyzed for dieldrin.
- In Phase 2, muscle biopsy samples from twenty-two bison to be culled from the Refuge herd in 2013 will be collected and analyzed for organochlorine pesticides, including dieldrin.

The objective of this sampling and analysis plan is to show that the 95% upper confidence limit for mean dieldrin concentrations in bison meat for the current RMA herd is below the USDA action level for dieldrin.

# 3.0 PROJECT DESCRIPTION

*Phase 1 – Historical Samples*

In Phase 1, fifteen historical tissues from Refuge bison that died from natural causes over the last several years (Table 2) will be analyzed for dieldrin. Analysis of these historical samples will expand the total sample size and also demonstrate continuous compliance with the USDA action level over a six-year period since the bison first arrived on the Refuge. All available collection information for these historical samples will be summarized in the Data Summary Report (DSR).

**Table 2.** Current samples of bison tissue

| *Sample #* | *Date* | *Contents* | *Field ID* | *Site ID* |
|---|---|---|---|---|
| 001 | 20080818 | FRONT LEG | 08LG001 | 08LGBIEN |
| 011 | 20100410 | LOWER HEART | 10HE011 | 10HEBIEN |
| 013 | 20100410 | MUSCLE | 10MU013 | 10MUBIEN |

| | | | | |
|---|---|---|---|---|
| 014 | 20100526 | MUSCLE | 10MU014 | 10MUBIEN7A |
| 017 | 20100410 | TOP OF HEART | 10HE017 | 10HEBIEN |
| 006 | 20101006 | MUSCLE | 11MU006 | 11MUBIEN01 |
| 007 | 20101109 | MUSCLE | 11MU007 | 11MUBIEN03 |
| 008 | 20101122 | MUSCLE | 11MU008 | 11MUBIEN9A |
| 115 | 20120625 | MUSCLE | 12MU115 | 12MUBIEN88 |
| 116 | 20120705 | MUSCLE | 12MU116 | 12MUBIENCA |
| 001/001B | 20131007 | MUSCLE | 14MU001A/B | 14MUBIEN/1B |
| 002/002B | 20131007 | LIVER | 14LI002A/B | 14LIBIEN21/2B |
| 003 | 20131007 | LUNG | 14LU003 | 14LUBIEN03 |
| 006 CALF | 20131008 | MUSCLE | 14MU006 | 14MUBIEN06 |
| 007 CALF | 20131008 | LIVER | 14LI007 | 14LIBIEN07 |

*Phase 2 – 2013 Bison Roundup Samples*

In Phase 2, fat biopsy samples from each of the bison to be culled from the Refuge herd in 2013 will be collected and analyzed for dieldrin.

## *3.1 Project Organization*

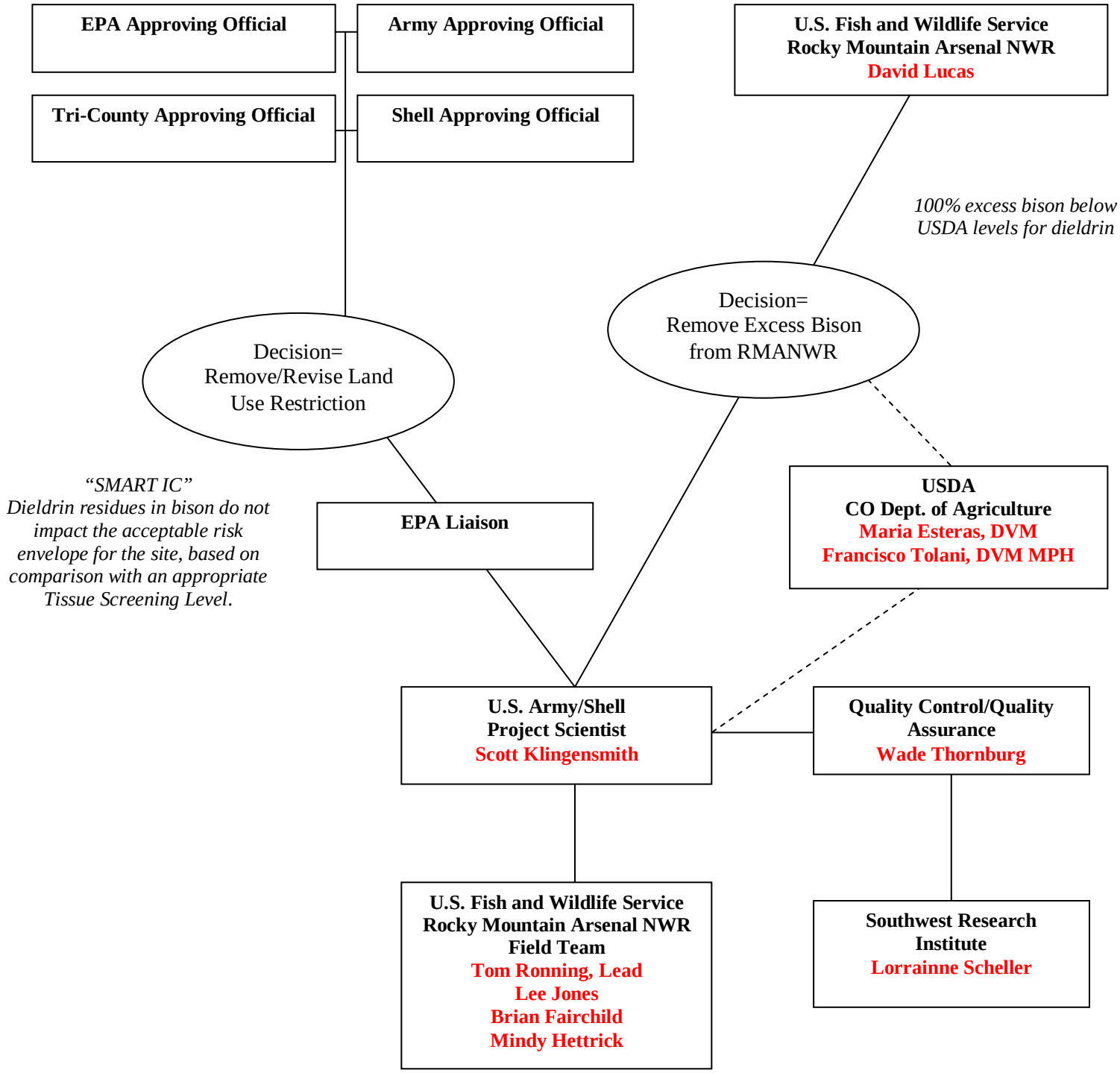
A table summarizing key staff participating in this study, their organizations, expertise, and primary roles is presented below. David Lucas is responsible for maintaining the official approved version of the SAP for this study.

**Table 3.** Table of key staff

| ***Name*** | ***Organization*** | ***Expertise*** | ***Role*** |
|---|---|---|---|
| David Lucas | USFWS | Program management | Decisionmaker |
| Dr. Scott Klingensmith | U.S. Army/Shell | Toxicology | Project Scientist |
| Tom Ronning | USFWS | Bison management | Field Team Leader |
| Dr. Lee Jones | USFWS | Veterinary medicine | Herd genetics/culling |
| Brian Fairchild | USFWS | Field biologist | Sample collection |
| Mindy Hetrick | USFWS | Field biologist | Sample collection |
| Sherry Skipper | USFWS | Contaminant biologist | EPA Liaison |
| Wade Thornburg | Navarro, Inc. | Environmental sampling | QA/QC/Lab Liaison |
| Lorraine Scheller | SWRI | Laboratory manager | Tissue analysis |

An organizational chart showing the relationships between participating staff and organizations is presented below.

**Organization Chart**



All personnel that conduct activities for this project must be properly trained and certified in sample collection, preservation, and storage techniques required by the SAP. The personnel involved in sampling activities for this project shall have education and experience necessary to conduct activities in a manner consistent with the sampling procedures described in Section 5.0.

# 4.0 OBJECTIVES

The objective of this SAP is to establish protocols to collect data for dieldrin concentrations in selected bison tissue samples, present analytical methods and requirements, and to describe the data evaluation process for the project. The samples obtained as part of this program will be analyzed for the standard suite of organochlorine pesticides with primary focus on dieldrin. Dieldrin is the only COPC identified for this SAP. Therefore, it is anticipated that statistical calculations will be performed only for dieldrin, unless the analytical data suggest additional evaluations are appropriate. The dieldrin data specifically will then be compared to the USDA action level for dieldrin to determine if the samples and therefore bison meat are acceptable for human consumption. A comparison of the dieldrin residue data will also be made to site-specific EPA Tissue Screening Level when they are developed.

## *4.1 Data Quality Objectives and Criteria for Measurement Data*

The DQOs and criteria for measurement data for this SAP are defined below using the seven-step process (Table 4) described in U.S. Environmental Protection Agency (EPA) Guidance for the Data Quality Objectives Process (U.S. Environmental Protection Agency 2000). This seven-step process clarifies the objectives, inputs, and decisions for the current project and helps define the data quality requirements. Below is a brief description of the outputs of each of the seven steps.

**Table 4.** Elements of Systematic Planning Process Corresponding Step in the DQO Process

| ***Elements of the Systematic Planning Process*** | ***Elements of Systematic Planning Process Corresponding Step in the DQO Process*** |
|---|---|
| Identifying and involving the project manager/decision maker, and project personnel | Step 1. Define the problem |
| Identifying the project schedule, resources, milestones, and requirements | Step 1. Define the problem |
| Describing the project goal(s) and objective(s) | Step 2. Identify the goal(s) of the study |
| Identifying the type of data needed | Step 3. Identify information needed for the decision |
| Identifying constraints to data collection | Step 4. Define the boundaries of the study |
| Determining the quality of the data needed | Step 5. Develop a decision rule<br>Step 6. Specify limits on decision errors |

| | |
|---|---|
| Determining the quantity of the data needed | Step 7. Optimize the design for obtaining data |
| Describing how, when, and where the data will be obtained | Step 7. Optimize the design for obtaining data |
| Specifying quality assurance and quality control activities to assess the quality performance criteria | Part B of QA Project Plan |
| Describing methods for data analysis, evaluation, and assessment against the intended use of the data and the quality performance criteria | Part D of QA Project Plan; DQA Process |

Table taken from Guidance for the Data Quality Objectives Process (U.S. Environmental Protection Agency 2000).

*Step 1. Define the problem*

- In order to achieve wildlife and habitat objectives for the Refuge, the USFWS must have the ability to remove surplus bison from the property.
- Bison that leave the Refuge may no longer be under the control of the USFWS and could be potentially consumed by humans.
- A restriction exists in the Record of Decision that prevents consumption of game taken on the Refuge.
- In order to confirm bison meat from the Refuge is safe for human consumption, dieldrin residue data in bison are required to demonstrate compliance with USDA action levels.
- Beginning in 2013, the USFWS has determined that twenty bison are surplus to the forage available and habitat goals for the Refuge. Annually thereafter, the USFWS anticipates a surplus of animals may exist.

*Step 2. Identify the goal(s) of the study*

- Test the hypothesis that the 95% confidence interval for mean dieldrin concentration in bison fat and other tissues from the RMA herd is below 300 ppb (U.S. Department of Agriculture 2006).

*Step 3. Identify Information Needed For the Decision*

- Historical bison tissue samples will be analyzed to provide an historical perspective of dieldrin concentrations for the RMA herd.
- Samples of fat will be collected from animals to be culled and analyzed to develop statistical estimates of the mean and 95% confidence intervals (CI) for dieldrin concentrations in meat for the current bison herd.
- Target practical quantification limit (PQL) for dieldrin is 100 ppb, and accuracy must be $\pm$ 100%.

*Step 4. Define the Boundaries of the Study*

- The historical bison samples span a time period from 2009-2013 during which the herd occupied both the north and south pastures at some point during that time interval. The additional samples to be collected for this SAP will be collected from October 2013 – December 2013 from bison that have grazed on both the north (Figure 1 – Bison Pilot Area Range) and south (Figure 2 – Visitor Center Range) pastures.


**Figure 1.** Bison management program, Bison Pilot Area Range “North Pasture” (1,460 acres)

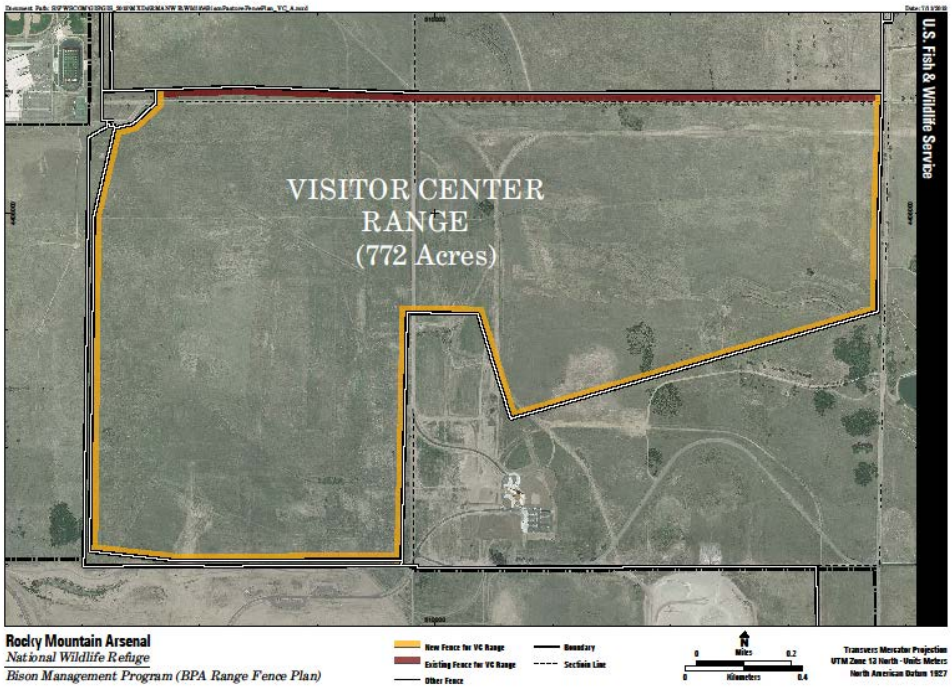



**Figure 2.** Bison management program, Visitor Center Range "South Pasture" (772 acres)

- It is expected that bison will graze approximately 12,165 acres (Figure 3) beginning in 2018. It is expected that samples will be taken during annual bison round-ups until it has been demonstrated that the RMA bison herd is safe for human consumption.

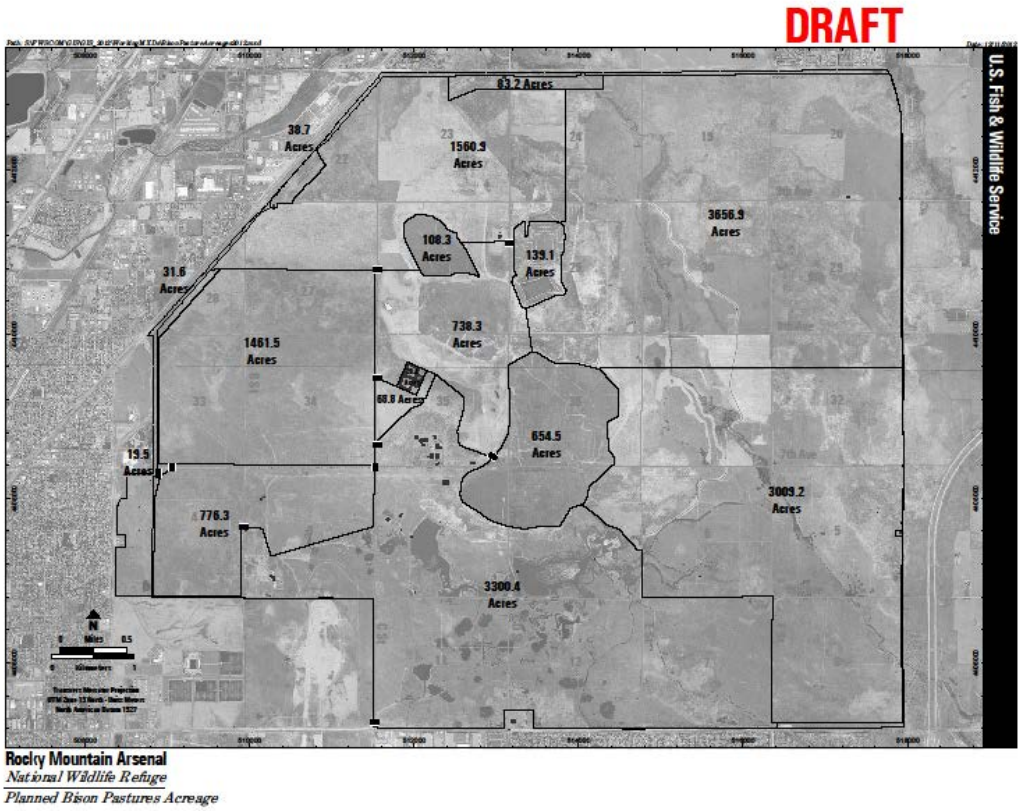



**Figure 3.** Bison management program, planned bison pasture acreage (12,165 acres)

## *Step 5. Develop a Decision Rule*

- If analytical data from the bison tissue sampling plan meet data requirements, then these data may be used to determine safety of RMA bison for human consumption. If the 95% upper confidence limit for the mean for dieldrin concentrations in bison fat is below the USDA action level, meat from the current bison herd will be determined to be in compliance with the USDA action level for dieldrin and therefore safe for human consumption. The approximately 20 animals to be culled from the herd in 2013 can be transferred without any restrictions or tracking requirements.
- If the 95% upper confidence limit for dieldrin is above the USDA action level, then the five bison to be transferred to other Refuges in 2013 will have to be tracked so that they are not made available for human consumption.
- If all samples are non-detect for dieldrin, mean tissue concentration will be identified as ½ the detection limit and compliance to the USDA action level will be considered to have been established *QED*. No confidence interval will be estimated.

*Step 6. Specify Acceptable Limits on Decision Error*

- The decision rule will be applied using valid test data from the samples. The sampling strategy is based on samples collected in sufficient quantity to yield data that will provide a reliable estimate of mean dieldrin concentrations in fat from the current RMA bison herd. A 95% confidence interval around the mean will encompass two standard deviations above the mean and provide an acceptable limit on decision error.

*Step 7. Optimize the Design*

- Mean dieldrin tissue concentrations are expected to be less than 10 ppb, standard deviations are expected to be small, and the upper 95% confidence limit is expected to be less than 100 ppb. The study design has been optimized to collect a representative number of samples that will provide a robust and reliable statistical estimate of mean dieldrin residues in bison meat and fat that can demonstrate current compliance with the USDA action level without excessive and unnecessary sample collections and analyses. It is expected that future statistically-based sampling of the RMA bison herd as it moves to new pastures will further strengthen and refine the residue estimates to the point that eventually no further sampling will be required at some point in the future. However, any subsequent sampling will be consistent with the terms of the ESD.
- The (DSR) prepared to summarize the results of the SAP will also compare the analytical data to the site-specific EPA Tissue Screening Level.

### *4.2 Selection of Analytes*

Selection of analytes for the Bison Pesticide Residue Study was based on review of the RMA Remedial Investigation (RI) (EBASCO 1989) and a FWS study of tissue contaminants in deer that was conducted before the remedy was initiated (Creekmore et al. 1999). In addition, a review of available soil contaminant data for the current bison pasture area was performed. A summary of those reviews is provided below.

RI

Based on a screening of documented RMA contaminants, the RMA Biota RI Report identified 39 contaminants of potential concern to RMA biota. Seven of these contaminants were determined to be major contaminants of concern based on considerations of toxicity, environmental persistence, spatial distribution, and other criteria. These chemicals were arsenic, mercury, dibromochloropropane (DBCP), aldrin, dieldrin, endrin, and isodrin. Of these chemicals, DBCP was eliminated from further analysis because it does not bioaccumulate and isodrin was eliminated because it is an

isomer of endrin that is converted to endrin metabolically in the body. DDE and DDT were added to the RI analyte list for selected species where these compounds were implicated in adverse effects (such as reproductive effects in birds).

The species evaluated in the RI considered to be most relevant for the bison evaluation was deer. Muscle and liver tissues were analyzed from RMA mule deer. No contaminants of concern (COCs) were detected in deer muscle (DL < 0.031 ug/g); one RMA deer sample reported a detection of dieldrin in liver (0.187 ug/g).

Creekmore et al., 1999

USFWS analyzed several tissues (brain, liver, kidney, muscle, and fat) from 18 radio-collared deer from RMA (13 mule deer, 5 white-tailed deer). The chemical analyte list for this study included two metals (arsenic and mercury) and eight organochlorine pesticides (aldrin, dieldrin, a-chlordane, g-chlordane, endrin, isodrin, DDE, and DDT). This study reported no detections for any of these analytes in deer meat, with the lone exception of a low concentration of DDE (0.02 ug/g) in 1/18 deer meat samples. Dieldrin was detected in 9/17 fat samples, 4/17 liver samples, and 1/18 brain samples. Mercury was detected in kidney samples from 10/12 deer sampled.

None of the deer evaluated in either of these reports were collected in the bison pasture area. The deer collected in the RI were taken primarily in the contaminated core area of RMA. Based on telemetry data, the mule deer collected for the Creekmore et al. study tended to occupy the highly contaminated areas that lie within close proximity of the manufacturing and disposal areas. In contrast, white-tailed deer frequented areas along riparian areas with less human activity.

Soil Data

An evaluation of soil contaminant data existing after the completion of the RMA cleanup program was performed based on queries directed to the Rocky Mountain Arsenal Environmental Database (RMAED) in August 2014. The data evaluated included soil data from the RI program, the TRER program, confirmatory sample program, and the tilling program. Soil samples in the dataset include multiple depths from 0-2 inches to 0-5 feet and represent both discrete and composite samples with a variety of detection limits for different analytes. These data are summarized in Table 5.

**Table 5.** Summary of ROD COCs for soil samples in current bison pasture

| *Compound* | | *No. of Samples* | *No. of Detections* | *Range* [1] | *Maximum Detection* [1] | *Indicator Level* [1] | *# Detections > IL* | *% Detections* [2] | *EPA Residential Soil Screening Levels* [3] *Carc ($1x10^{-6}$)* | *Noncarc (HI 1.0)* | *% Detections* [2] *> SSL* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1,1-Dichloroethene | 11DCE | 12 | 0 | LT 0.24 - LT 1.36 | NA | | | | | | |
| 1,2-Dichloroethane | 12DCLE | 18 | 0 | LT 0.085 - LT 1.24 | NA | | | | | | |
| Aldrin | ALDRN | 258 | 12 | LT 0.002 - 0.0258 | 0.0258 | | | 4.7% | 0.029 | 1.8 | 0% |
| Benzene | C6H6 | 16 | 0 | LT 0.085 - LT 0.3 | NA | | | | | | |
| Carbon tetrachloride | CCL4 | 18 | 0 | LT 0.12 - LT 1.2 | NA | | | | | | |
| Chlordane | CLDAN | 251 | 3 | LT 0.0139 - 0.232 | 0.232 | | | 1.2% | 1.6 | 35 | 0% |
| Chloroacetic acid | CLC2A | 2 | 0 | LT 35.5 | NA | | | | | | |
| Chlorobenzene | CLC6H5 | 18 | 0 | LT 0.1 - LT 1.0 | NA | | | | | | |
| Chloroform | CHCL3 | 18 | 0 | LT 0.068 - LT 1.32 | NA | | | | | | |
| DDE | PPDDE | 251 | 3 | LT 0.0022 - 0.0125 | 0.0125 | | | 1.2% | 1.4 | NA | 0% |
| DDT | PPDDT | 251 | 11 | LT 0.0023 - 0.0749 | 0.0749 | | | 4.4% | 1.7 | 36 | 0% |
| Dibromochloropropane | DBCP | 230 | 0 | LT 0.005 - LT 30 | NA | | | | | | |
| Dicyclopentadiene | DCPD | 210 | 0 | LT 0.3 - LT 40 | NA | | | | | | |
| Dieldrin | DLDRN | 258 | 34 | LT 0.0018 - 0.0897 | 0.0897 | | | 13.2% | 0.03 | 3.1 | 2.3% |
| Endrin | ENDRN | 251 | 3 | LT 0.002 - 0.0231 | 0.0231 | | | 1.2% | NA | 18 | 0% |
| Hexachlorocyclopentadiene | CL6CP | 251 | 0 | LT 0.0014 - LT 30 | NA | | | | | | |
| Isodrin | ISODR | 251 | 1 | LT 0.0017 - 0.0022 | 0.0022 | | | 0.4% | NA | NA | NA |
| Methylene chloride | CH2CL2 | 18 | 0 | LT 0.29 - LT 4.4 | NA | | | | | | |
| Toluene | MEC6HE | 0 | 0 | LT 0.1 - LT 1.14 | NA | | | | | | |
| 1,1,2,2-Tetrachloroethane | TCLEA | 9 | 0 | LT 0.2 - LT 0.29 | NA | | | | | | |
| Tetrachloroethylene | TCLEE | 18 | 0 | LT 0.16 - LT 1.07 | NA | | | | | | |
| Trichloroethylene | TRCLE | 18 | 0 | LT 0.14 - LT 1.16 | NA | | | | | | |
| Arsenic | AS | 217 | 16 | LT 2.5 - 19.3 | 19.3 | 10 | 4 | 1.8% | 0.61 | 34 | 1.8% |
| Cadmium | CD | 211 | 8 | LT 0.4 - 90.8 | 90.8 | 2 | 5 | 2.4% | 1800 | 70 | 0.5% |
| Chromium | CR | 211 | 143 | LT 5.2 - 27.4 | 27.4 | 40 | 0 | 0.0% | 0.29 | 230 | 0% |
| Lead | PB | 211 | 86 | LT 8.38 - 1700 | 1700 | 40 | 8 | 3.8% | NA | 400 | 0.5% |
| Mercury | HG | 210 | 7 | LT 0.0269 - 1.1 | 1.1 | 0.2 | 2 | 1.0% | NA | 23 | 0% |

[1]All values shown are mg/kg (ppm).

[2]For inorganic compounds, the percentage is shown for samples greater than the Indicator Levels.

[3]Values taken from EPA Regional Screening Levels Residential Soil, May 2013.

For organic chemicals, six of the RMA COCs were detected in bison pasture soils and all six were organochlorine pesticides (aldrin,dieldrin, endrin, isodrin, chlordane, and DDT/DDE). Only aldrin (4.7%), dieldrin (13.2%), and DDT/DDE (6.6%) were detected at a frequency of 5% or greater. Only dieldrin had any detections (2.3%) above the residential soil screening level (SSL). Since aldrin is converted metabolically to dieldrin almost instantly in mammalian systems, dieldrin was selected as an analyte for this SAP.

The only other organic analyte with a significant potential for bioaccumulation in bison meat was DDT/DDE. DDT/DDE was excluded from further consideration as a target analyte for this study due to the extremely low concentrations, relatively low human toxicity, and no detections above the residential SSL.

RMA COC metal concentrations detected in bison pasture soils were mostly below background levels (Table 5). Detections above background indicator levels (ILs) for arsenic, cadmium, lead, and mercury ranged from 2-8 detections for each metal out of over 200 samples. In addition, none of these metals has a high potential for bioaccumulation in meat at the soil concentrations and speciated forms found at RMA. Frequency of metal detections above SSLs ranged from 0 -1.8%. For these reasons, no metals were selected as analytes for this study.

No other RMA COCs were detected.

Basis for Selection of Analytes

Dieldrin was the only analyte detected in deer for the RI program. Dieldrin was also detected in deer in the Creekmore et al. study. Although mercury was detected in whitetail kidney tissue, mercury was not added to the analyte list for this study because it was likely derived from the RMA lakes prior to the initiation of the cleanup. Extensive remediation projects were conducted for the RMA South Lakes and the Aquatic Residual Ecological Risk (ARER) report concluded that all significant contamination was removed. In addition, the frequency of mercury detections in soil from the bison pasture area is less than 1%. Since all of these mercury detections were extremely low and likely in the less toxic form of mercuric chloride, rather than the presumed methylmercury species associated with lake sediments that the whitetails were exposed to, it is unlikely that there is any significant potential for mercury uptake by bison. Dieldrin was the only analyte detected in bison pasture soils with a significant bioaccumulation potential, detection frequency, and significant human toxicity.

**For these reasons, dieldrin was selected as the primary analyte for the Bison Pesticide Residue Study** Since the analytical method used for dieldrin provides results for a standard suite of organochlorine pesticides, the analytical results for the entire suite will be reported. However, statistical evaluations developed to demonstrate USDA compliance will be focused exclusively on dieldrin.

### *4.3 Data Collection Plan*

In order to meet the SAP objectives, this study will be conducted in two phases:

- Analysis of historical tissues from bison which died of natural causes
- Analysis of fat samples from the group of animals to be culled
- Calculation of the group mean and 95% confidence limit for each subsample of the RMA bison herd.

## 5.0 FIELD ACTIVITIES

### *5.1 Number and Source of Samples*

A total of approximately samples are to be collected for this SAP. This total is to include 15 historical samples from Refuge bison that died of natural causes since their arrival and 22 samples from the portion of the herd to be culled. Sample numbers and sources may be changed based on field conditions and other considerations.

### *5.2 Samples Collection and Procedures*

*Phase 1 – Historical Samples*

A subsample of up to approximately 4 grams of tissue will be retrieved from historical samples.

*Phase 2 – 2013 Bison Roundup Samples*

During the bison roundup, the information outlined in Table 6 will be collected.

**Table 6.** Data collected during each bison roundup sampling event

| ***Data/Sample*** | ***Item/Sample Size*** | ***Test*** | ***Comment*** |
|---|---|---|---|
| Chip | Record chip number | NA | |
| Origin | RMANWR/Bison Range/Sully's Hill | NA | Record bison's point of origin |
| Sex | Male/Female | NA | Prefer M/F with further age classification in next field |
| Age | Calf/Juvenile/Sub-adult/Adult | NA | |
| Weight | In grams (g) | NA | |
| | | | |
| Tail Hairs | 12 – 20 each with follicles | DNA | Immediately place in cooler after collection |
| | | | |
| Whole Blood | 4x 10ml (Annotate container i.e. purple top, red, etc) | Disease | Immediately place in cooler after collection |
| Blood Cards | Yes/No completed | DNA | 2x - Place in cooler after cards are dry |
| Plasma | 2x 4cc (Completed in lab following sampling event | DNA | 5ml whole blood is spun and plasma is immediately placed in cooler after separation and collection |
| Fat* | 1g minimum | Disease/Contaminant | *Additional if dispatched |
| | | | |
| Other | Specify | Specify | |

Field sample and site IDs must also be generated for each tissue sample following established c-o-c protocol.

For the purposes of the Bison Pesticide Residue Study, fat will be collected from all animals selected for culling. Samples will be collected as described below:

- All animals to be culled will be sampled.
- Animals will be biopsied to obtain either one or two 0.5 g fat samples.
- Samples will be retrieved with tweezers/forceps, placed into 2 ounce glass vials, and immediately placed on ice to begin the chain of custody process.

### *5.3 Health and Safety Plan*

In accordance with 29 CFR § 1910.133, A USFWS Job Hazard Analysis has been completed for the annual Bison Roundup and Corral Handling (December 17-18, 2013). In addition, sharps disposal containers will be used to safely dispose of needles and other sharps.

### *5.4 Sample Designation and Labeling*

*Phase 1 – Historical Samples*

Samples will be identified with the unique animal identification number (if available), date collected, and sample size.

*Phase2 – 2013 Bison Roundup Samples*

At the bison roundup, all animals will have a microchip or will have one inserted. Samples will be identified with the unique animal identification number, date collected, and sample size. As this microchip is scanned a barcode printer will print labels for each of the samples taken, and every sample will have a unique barcode that identifies which animal it was taken from.

### *5.5 Documentation, Preservation, Labeling and Packaging and Shipping*

Procedures described in this section are designed to ensure sample integrity through proper sample handling. Samples must be properly handled at the sampling site, during storage, and while being packaged and shipped to the laboratory

All samples will be preserved by chilling with blue, bagged, or dry ice to approximately 4 degrees Celsius (˚C), +/- 2˚C. Care will be taken to ensure that samples are not broken while being transported between sample sites. Samples should be transported to the USFWS onsite laboratory within four to eight hours of collection and placed in a designated freezer as soon as possible. Samples should be prepared for shipment to the contracted laboratory after returning from the field. Labels and the Chain-of-Custody (c-o-c) form will be properly completed and labels affixed to the sample containers.

Information shall be recorded on labels and chain of custody forms using the RMA C-O-C Entry computer program. Labels and c-o-c information may be recorded by hand with a permanent indelible pen if they are legible and complete.

Sample labels shall be completed immediately before or during collection of the corresponding sample. Handwritten labels must contain all required information. Labels shall be securely placed on appropriate sample containers. Label entries include.

- Field Sample ID - An eight character or less alphanumeric code assigned to each site ID for sample tracking. Field Sample Ids must be unique, as duplicates are not allowed in the Rocky Mountain Arsenal Environmental Database (RMAED).
- Site ID - A ten character or less alphanumeric code that relates the sample to a specific operating function or location.
- Date - Year (4 digits)/Month/Day of sample collection.
- Time - Time of sample collection. Time shall be entered using a 24-hour clock method.
- Method - The corresponding EPA or PMRMA analytical method number of the analysis or test to be performed on the sample.
- Bottle Type - The size and material type of the sample container.
- Treatments - The solution or method used to preserve the sample, e.g. hydrochloric acid, nitric acid, ice, etc.
- Lab - The two-digit code for the laboratory.
- Flag Code - A one-letter code used to denote various field quality control samples.
- Site Type - A four-letter code that describes the site from which the sample was collected.
- Test Name - The name of the analytical method corresponding to the method number.
- C-O-C Number - The number of the c-o-c corresponding to the label.
- Remarks - Any miscellaneous comments related to sample collection or analysis.

C-o-c records will be used to document the security and control process for samples from the time of collection until delivery to the laboratory. Copies of c-o-cs are included in analytical data packages from the laboratory and are available in the RMA C-O-C Entry program. C-o-c entries shall include the following information.

- Contracting Office Representative (COR) POC - The point of contact to resolve sampling issues.
- Contractor POC - OMC person/phone number responsible for sample collection and lab coordination.
- Project Name - A short description identifying the general location and frequency of the sampling event.
- Turn Around Time (TAT) - The time period between sample collection and delivery of analytical results. Standard TAT is 28 days, although an accelerated TAT can be arranged with the laboratory.
- Contractor Name - Navarro Research and Engineering, Inc.

- Lab Code - The two-character designation for the laboratory conducting the analysis. Acceptable entries are limited to current laboratories.
- Sampling Technique - A one-letter code used to designate the field sampling method, i.e. G for grab. Acceptable entries are listed in a pop-up window when the field is highlighted.
- Prime Contractor - A two-letter code to identify the prime contractor collecting the sample.
- Purchase Request Number - A four-digit task specific number assigned by the COR for samples going to off-post laboratories for analysis.
- Sampling Program - A three-letter code to designate the program under which the sample was authorized. The code utilized for this sampling program LNR (Litigation Navarro Research).
- Matrix Code - A two-letter code to denote the matrix of the sample. The code utilized for this sampling program is PT (plant and animal biota).
- Sampler's Initials - The initials of the person or persons collecting the samples.
- Sampler's Signature - The signature of a member of the field crew that collected the samples.
- File Type - A three-letter code identifying the type of data. Acceptable entries are CAT (chemical animal tissue) or CBT (chemical biota).
- QC Code - A one-letter code representing the type of QC sample, if applicable. QC samples will not be collected in conjunction with this task.
- Field Sample ID - An eight character or less alphanumeric code assigned to each site ID for sample tracking. Field Sample IDs must be unique, as duplicates are not allowed in the RMAED.
- Site ID - A ten character or less alphanumeric code that relates the sample to a specific operating function or location.
- Site Type - A four-letter code that describes the site from which the sample was collected. The code utilized for this task is BIOL.
- Sample Date - Year/Month/Day of sample collection.
- Sample Time - Time of sample collection. Time shall be entered using a 24-hour clock method.
- Flag Code - A one-letter code to denote certain field quality control samples.
- Methods - The analytical method number requested for the analysis. The "Test Name" field is contingent on the method number selected and will be completed automatically upon selection of a corresponding method number.
- Remarks - Enter any comments or other information needed to identify the sample, such as turnaround time, container size, or preservatives used, if other than ice.

Custody seals will be used to ensure that sample container integrity is not compromised. Custody seals are placed on individual sample containers or on the outside shipping container in such a manner that the container cannot be opened without compromising the custody seal. Once in place, either the sampler or their designee, or the laboratory can break custody seals. In order to transfer custody of the samples, one of the individuals collecting the samples will sign the c-o-c in the first "Relinquished By" box, located under the “Other Notes” box. The date and time of relinquishment is indicated in the "Date" and "Time" boxes. The person receiving the samples shall sign in the adjacent "Received By" box. Note that FedEx does not sign custody forms.

If the sample is placed in a locked storage location, such as a refrigerator, prior to shipment to the laboratory, the date and time of storage is entered in the first date and time boxes and the location is entered in the first "Received By" box. When the sample is removed from storage, the handler will initial the "Relinquished By" box indicating where the sample was received from, the time and date, and sign the "Received By" box following the date.

C-o-cs are submitted electronically to the laboratory. In addition, an original hardcopy of the c-o-c accompanies all shipments and deliveries.

The samples will be placed in a secure freezer at a temperature of approximately -20˚C (+/- 2˚C), or placed in a cooler maintained to the same temperature with ice and shipped to the laboratory.

For shipment, sample containers must be placed in a cooler with sufficient ice to preserve the correct sample temperature. The cooler should be packed to prevent sample breakage. Secure the cooler lid with shipping tape and affix signed and dated custody seals to the cooler box and lid.

All samples sent to the laboratory by the USFWS are labeled with a freezer-proof printed Mylar label documenting sample chain of custody information.

Samples will be shipped to the contract laboratory using Federal Express (FedEx) Priority Overnight® service in order for samples to arrive at the laboratory as soon as possible following sample collection. Coolers will be returned to RMA using FedEx 2Day® delivery when possible.

Sample collection is scheduled to begin in December 17, 2013 and be completed by December 18, 2013. The standard turnaround time (TAT) of 28 days will be requested for all samples.

### *5.6 Field Quality Control*

No field quality control samples will be collected for this study. Study matrix spikes will be performed by the lab.

### *5.7 Field Documentation*

USFWS staff will document and report field activities as required by USFWS procedures..

# PART II – Laboratory Plan

## 6.0 ANALYTICAL LABORATORY REQUIREMENTS

Analytical laboratory QA/Quality Control (QC) procedures are based on requirements specified in the RMA Chemical Quality Assurance Plan (CQAP) Revision 4, 2009 and the analytical methods approved by the OMC Analytical Manager, and are addressed in laboratory-specific QA plans. The CQAP describes the RMA management control systems that have been established to ensure the achievement of quality in a planned and systematic manner, and to ensure lab support compliance with applicable requirements of the RMA Quality Assurance Plan (QAP).

### *6.1 Laboratory Methods of Analysis*

Analysis of dieldrin in bison tissues will be performed using SwRI proprietary methods which are based on EPA Method 8081. SwRI procedure 01-0408-135 Extraction of Organochlorine Pesticides in Biota Matrices will be the extraction method and 01-0408-136 Determination of Organochlorine Pesticides in Biota by GC/MS will be used for the chemical analyses (Southwest Research Institute 2013). The suite of organochlorine analytes from this method (03 QM) is provided below in Table 7.

**Table 7.** List of analytes for Method 03QM

| |
|---|
| alpha-BHC |
| beta-BHC |
| gamma-BHC |
| delta-BHC |
| Heptachlor |
| Aldrin |
| Heptachlor epoxide |
| gamma-chlordane |
| alpha-chlordane |
| Endosulfan I |
| 4,4’ –DDE |
| Dieldrin |
| Endrin |
| Endosulfan II |
| 4,4’ –DDD |

| |
|---|
| Endrin aldehyde |
| Endosulfan sulfate |
| 4,4' –DDT |
| Endrin ketone |
| Methoxychlor |

The target Practical Quantification Limit (PQL) for dieldrin for the Bison SAP is 0.10 ug/g for tissues and fat. Lipid content will also be measured for each sample. Maximum sample holding time from sample collection to extraction and analysis for all bison tissue samples is arbitrarily set for project management purposes at one year.

### *6.2 Performance-Based Methods*

Analytical laboratories will perform an initial method proficiency demonstration prior to using a performance-based method (PBM) for the RMA. The laboratory will generate a series of standard matrix spikes at a range of concentrations in bison tissue representative of the samples to be collected. These recoveries will be plotted as the found concentrations versus the target concentrations. The accuracy correction factor for an analyte is the slope of the least squares linear curve fit line of this data set. If spike recoveries are 100 percent, the value of the accuracy correction factor will be 1.00. If the recoveries are less than 100 percent, the accuracy correction factor will be less than one.

When final results are entered into the Rocky Mountain Arsenal Environmental Database for a PBM, the laboratory's uncorrected (found) value will be divided by the accuracy correction factor, and the result posted as the final (reportable) result. The same adjustment will be made for the PQLs. Accuracy factors outside of a range of 50-125 percent should be noted in all results packages. (Wade, are we good with this change?

### *6.3 Analytical Equipment Calibration*

Calibration is a reproducible reference to which all sample measurements can be correlated. Accuracy of calibration standards is critical because all data will be in reference to these standards. A sound program for the laboratory includes documentation of calibration:

- Frequency
- Procedures
- Standards
- Records that reflect the calibration history of a measurement system

Tools, gauges, instruments, and other sampling, measuring, and test equipment that affects quality used for measurement activities, shall be calibrated. At specified periods, recalibration shall be performed to ensure accuracy is within specified limits. Calibration shall be conducted using certified equipment or standards that have a known valid relationship to nationally recognized measurement standards.

### *6.4 Analytical Equipment Maintenance, Testing, and Inspection*

The maintenance program for laboratory equipment shall provide documented long-term, in-depth maintenance on all measuring, sampling, and general laboratory equipment and support facilities. This program may include an in-house/on-site maintenance shop or full service maintenance agreement(s) with a commercial vendor(s).

### *6.5 Analytical Control*

Analytical QC is the systematic process of analytical protocols that controls the validity of analytical results by measuring the accuracy and precision for each method and matrix, developing expected control limits, using these Data Quality Indicators (DQIs) to identify anomalous events, and taking corrective action to prevent or minimize the recurrence of these events. QC checks are required for all laboratory measurement processes used to produce the final data package. Waivers to CQAP requirements may be allowed if documented properly and approval is obtained prior to implementation. In-control QC sample results do NOT ensure that final data is suitable for its intended purpose. Documented results obtained from laboratory QC samples must be evaluated against acceptance criteria per the specific laboratory SOP. QC checks verify that:

- Sample collection and preservation operations were conducted.
- Holding times were met.
- Values obtained from all QC samples have met method acceptance criteria per the analytical SOP.

Analytical laboratory QC samples may include method blanks, laboratory control samples, internal standards, calibration verifications, and matrix spike samples which will be analyzed in accordance with the RMA CQAP and the approved analytical methods.

### *6.6 Data Package Deliverables*

The term "data package" refers to paperwork (either manually or electronically generated) that pertains to a specific analysis for an analytical lot or batch of samples. Data packages will be assembled as specified by the RMA CQAP and/or the lab support contract, and are stand-alone documents. The lab support manager shall

require data package submission, so that independent data review can be performed prior to data package transfer and storage at the RMA Technical Information Center (RTIC).

### *6.7 Data Tracking and Control*

When the laboratory receives sample shipments, the original c-o-c and air bill forms should be signed and placed in a permanent project record or data package. If copies of originals are placed in data packages, the location of the original forms should be annotated on the copies.

Enclosed forms should be checked for completeness. Upon receipt of samples, the laboratory shall generate a sample condition form that will document the condition of samples as received. Any sample container breakage, documentation discrepancies, improper preservation, or other deficiencies will be noted on this form. This form must be signed and dated by the appropriate sample management personnel at the laboratory. Receipt of the samples should be logged into the laboratory information management/tracking system.

C-o-c procedures shall be followed for all samples submitted. C-o-c documentation must show samples were secure at all times and tampering could not have occurred. Documentation must also show hand-to-hand custody of samples. Laboratory personnel are responsible for all samples and documentation in their possession.

## 7.0 MEASUREMENT AND DATA ACQUISITION

### *7.1 Systems*

Measurement and data acquisition systems encompass the procedures by which environmental samples are collected and analyzed. The QC measures associated with the sampling process design and implementation are described in Sections 5, 6, and 7 of this SAP. The following discussion addresses procedures to be followed in the analytical laboratory

### *7.2 Laboratory Qualifications*

The laboratory will perform the analyses required for determination of sample constituents. Analyses will be conducted in accordance with OMC-approved methods and demonstrated method proficiency requirements stated in the OMC CQAP (RMA Remediation Venture Office 2009). The laboratory will also be expected to provide an internal QA Plan to the OMC for approval if one is not already on file.

All laboratories performing work in support of the Operations and Maintenance Contract at RMA must adhere to the requirements identified in the CQAP. Any variance from

CQAP requirements must be requested in writing and approved by the OMC prior to the performance of associated analytical work.

The OMC is responsible for ensuring that its subcontract laboratories perform the following:

- Participate in performance audits as required
- Participate in on site quality system and data audits by an authorized Navarro Research and Engineering audit team
- Correct any deficiencies identified during audits and other reviews and provide written reports of corrective actions to the OMPM for approval
- Satisfactorily perform Method Proficiency Demonstration (MPD) prior to performing analytical work
- Participate in the U.S. Army ALPES as applicable
- Ensure applicable SOPs are followed when performing all OMC work

The laboratory will be responsible for operating and maintaining all testing equipment as specified in the appropriate test methods. Only trained personnel will be allowed to operate, calibrate, and provide maintenance on the equipment or instruments, and only qualified laboratory personnel or service technicians will perform repairs. All maintenance and repair procedures will be documented in the instrument logs, which will be included in the laboratory project file.

# 8.0 DATA REVIEW, VERIFICATION, AND USABILITY

## *8.1 Data Review*

The OMC QA Validation Specialist will review sample data results. This process involves evaluating the analytical data to determine the certainty with which data may be used in making decisions. All data review and verification activities will be conducted in accordance with the RMA CQAP (RMA Remediation Venture Office 2009).

The purpose of the data review is to evaluate data quality with respect to the established data quality objectives. Components of the data review process include data verification, data validation and data usability.

**Data Verification**

Data verification is the process of evaluating the completeness, correctness, and conformance/ compliance of a specific data set against the method, procedural, or contractual requirements. When deficiencies in the data are identified, then those deficiencies should be documented for the data user's review and, where possible,

resolved by corrective action. Data verification applies to activities in the field as well as in the laboratory. Laboratory data verification is specified in the current revision of the RMA CQAP.

The components of field data verification include the following:

- Sampling and Analysis Plan (SAP) is current and has been approved.
- Field logbooks and documentation are complete.
- Equipment calibration data has been correctly recorded.
- Chain-of-Custody forms have been correctly completed.
- Shipping airbills have been correctly completed.
- Deviations from the SAP have been documented.

**Data Validation**

Data validation is an analyte- and sample-specific process that extends the evaluation of data beyond data verification to determine the analytical quality of a specific data set. Data validation includes a determination, where possible, of the reasons for any failure to meet method, procedural, or contractual requirements, and an evaluation of the impact of such failure on the overall data set. The scope of this document concerning data validation is limited to the analytical laboratory. Laboratory data validation is specified in the current revision of the RMA CQAP.

### *8.2 Data Usability*

The data usability process is the final assessment that will be performed to ensure that the implementation of the sampling and analysis program described in this SAP provides results that can be used to meet the data quality objectives. Components of the data review process include; evaluating the data against the data quality indicators of precision, accuracy/bias, representativeness, completeness, and comparability; review of field and laboratory QC results and evaluating the data for suitability based on the intended use. Deficiencies identified during this assessment will be reported to the PMC Project Manager, OMC QAR, and the Project Scientist, along with an indication of how the assessment will impact the use of the data.

#### *8.2.1 Precision*

Precision is a measure of agreement among measurements performed using the same test procedure. Precision values for an analytical system are generated by calculating the range of three consecutive spike samples (Laboratory Control Samples [LCS]) as follows:

- The accuracy (percent recovery [%R]) values from the LCS for the sample batch currently being evaluated and the previous two sample analysis batches are used to calculate precision. The highest and lowest values for these three batches will be used to calculate the percent range value by subtracting the lowest recovery value from the highest recovery value. This range value will be compared to the current control chart limits for that analyte/method combination. Since the range value for precision will never be less than zero, there will only be an upper control limit for this measurement. There is no lower control limit.

The procedures for calculating warning and control limits for range assessment are presented in the U.S. Army Toxic and Hazardous Materials Agency's Installation Restoration Program Quality Assurance Program (U.S. Army 1990).

### *8.2.2 Accuracy*

Accuracy is a measure of the bias in a measurement system, defined as the closeness of the reported value to the true value. Potential sources of error that could affect data accuracy include the sampling process, sample preservation and handling, sample matrix, equipment decontamination, and analytical techniques. Accuracy of the measurement system will be assessed by evaluating the results of quality control samples such as laboratory control spikes, and system monitoring compounds for organic analyses. Accuracy will be calculated as the percent recovery (%R) in the following manner:

$$\%R = \frac{\left(X_s - X_u\right)}{K} \times 100\%$$

Where: $X_S$ = measured value of the spiked sample
$X_U$ = measured value of the unspiked sample
K = known amount of spike in the sample

Accuracy of an analytical method is assessed by comparing the recovery of the LCS that is generated with every analysis batch, with the current control limits. Control limits are established from historical data by calculating 2-sigma ($\sigma$) [warning] and 3-sigma [control] limits around a central average recovery value.

Although accuracy values can be calculated from a number of QC sample types (sample matrix spikes/spike duplicates, LCSs, single-blind and double-blind field spikes), analytical control for RMA methods are assessed using the LCS recovery values for an analysis batch

### *8.2.3 Representativeness*

This parameter expresses the degree to which the sample data accurately and precisely represent a characteristic of the environmental condition. Representativeness is a qualitative parameter best addressed by ensuring that the proposed sampling techniques and the rationale used to select sampling locations are consistent with the overall project objectives

### *8.2.4 Comparability*

This qualitative parameter indicates the level of confidence with which one data set may be compared with another. Comparability will be achieved by using standard techniques to collect and analyze representative samples, and reporting chemical data in appropriate units.

### *8.2.5 Completeness*

This parameter is defined as the percentage of measurements made which are judged to be valid measurements compared to the total number of measurements planned in project-specific DQOs. Completeness may be calculated as follows:

$$PC = \frac{V}{n} * 100$$

Where: PC = relative percent completeness.
V = the number of valid measurements completed (or samples collected).
n = the total number of samples collected

Acceptable completeness requirement is a Percent Completeness value of 90% *(90% is the minimum acceptable value).*

## 9.0 AUDITS, SURVEILLANCES, AND OVERSIGHT REQUIREMENTS

No field audits are planned for this study. One or more surveillances of field sampling activities may be undertaken at the discretion of the program manager.

## 10.0 DOCUMENTATION AND RECORDS

### *10.1 Report*

At the conclusion of this study, a Data Summary Report (DSR) will be prepared to summarize the analytical results and to determine data usability. All project documents, records, and electronic files will be placed into a project file and stored in the RMA

Document Tracking Center and retained in perpetuity. All analytical data will be placed in the RMAED and retained in perpetuity

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