

**Department of Energy, Office of Legacy Management
Monticello Mill Tailings Site Operable Unit III (MMTS OU III)**

Summary of Program Directive #: MNT-08-03

Subject: Aquatic macroinvertebrate sampling and analysis for OU III biomonitoring.

Directive/Task Changes: Continue macroinvertebrate sampling and analysis for selenium in 2008 in accordance with the attached procedures. The scope of work and methodology were developed in consultation with the Biological Technical Assistance Group (BTAG) for OU III biomonitoring. The attached procedures are similar to those described in previous program directives except that additional Hester-Dendy samplers will be placed in the sediment pond and monitored more frequently in May and June to optimize macroinvertebrate collections.

Affected Program Documents: Monticello Mill Tailings Operable Unit III Post-Record of Decision Monitoring Plan

Justification: Collection of aquatic macroinvertebrates for chemical analysis is a component of the OU III biomonitoring program in progress to evaluate selenium accumulation in the wetland environments at OU III, as required by the Record of Decision for Operable Unit III, signed in May 2004.

Effective Date: March 21, 2008

Expiration Date: September 30, 2008

Macroinvertebrate Sampling and Analysis Plan 2008 Field Season

I. Introduction

The U.S. Department of Energy (DOE) completed surface remediation at the Monticello Mill Tailings Site (MMTS), located near Monticello, Utah, under the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA). The remediated area currently supports three large manmade wetlands and a sediment retention pond, located approximately one mile east of the wetlands (map attached). The *MMTS Operable Unit III Post-Record of Decision Monitoring Plan* (DOE 2004) specifies post-remediation monitoring of groundwater, surface water, and sediment for selenium. Selenium has the ability to accumulate in the aquatic food chain and potentially harm organisms, particularly avian species that feed on macroinvertebrates with elevated levels. Therefore, biomonitoring has been conducted at the former MMTS since 2005, when selenium benchmark levels were exceeded for some surface water/sediment samples. The *Operable Unit III Remedial Investigation, Appendix M – Ecological Risk Assessment* (DOE 1998) discusses the potential receptors and exposure pathways in detail.

The locations and general approach for biomonitoring were determined in 2004 by the Biological Technical Assistance Group (BTAG), including representatives from DOE, the U.S. Environmental Protection Agency Region VIII, the U.S. Fish and Wildlife Service, and the Utah Department of Environmental Quality. Macroinvertebrate sampling was accomplished at the site in 2005, 2006, and 2007. Results indicate that, during these years, levels of selenium in macroinvertebrate tissues, while not exceeding toxicity levels (7 mg/kg Se dry weight), did exceed levels of concern (3 mg/kg Se dry weight) in Wetland 3 and the sediment retention pond. 2008 macroinvertebrate sampling will focus on these two wetlands, as the results from previous years exclude Wetland 1 and Wetland 2 from further sampling.

In previous monitoring years, the composition of species collected on artificial substrate (Hester-Dendy) samplers differed from the composition of species collected with kick nets. Therefore, characterization of selenium in macroinvertebrate tissue in the sediment retention pond is more complete with both methods than only one. Because previous years' sampling efforts have not yielded adequate sample sizes for full analysis for the Hester-Dendy samplers, particular attention will be paid to these collections. Additional samplers will be deployed at each sampling location utilizing two different orientations of the sampling devices.

In addition to macroinvertebrate sampling, sediment/surface water sampling and avian surveys, which are covered under separate program directives and plans (MNT-08-01 and MNT-08-02, respectively), will continue in Wetland 3 and the sediment retention pond as part of the biomonitoring effort.

II. Scope

Field sampling will be performed by S.M. Stoller personnel and will consist of procuring necessary field equipment, planning field activities, traveling to the field site, and collecting

macroinvertebrate samples. Kick net samples will be collected in May 2008 in three locations at Wetland 3 and three locations at the sediment retention pond. Also, three Hester-Dendy samplers will be deployed at each of 3 locations (for a total of 9 samplers) in the sediment retention pond. The samplers will be deployed in April 2008 and collected in May or June as necessary.

Sample identification and preparation will be performed by S.M. Stoller personnel and will consist of storing samples, cleaning, sorting and identifying macroinvertebrate species, and preparing samples for shipment to the laboratory.

Sample analysis will be performed by the Battelle Marine Sciences Laboratory in Sequim, Washington according to protocols established in previous monitoring years. The laboratory will provide results in an electronic data format.

A report documenting all the field activities and laboratory results will be prepared by S.M. Stoller personnel, and interpretation will consider results of sampling efforts in the past and current regulatory benchmarks. Finalization of the report will include incorporation of comments received from DOE and BTAG.

III. Field Sampling Procedures

The purpose of the field sampling effort is to collect macroinvertebrates for selenium analysis to determine if the macroinvertebrates in Wetland 3 and the sediment retention pond remain at or above levels of concern. Both wetlands will be sampled using kick nets. The deeper sediment retention pond will also be sampled using Hester-Dendy samplers. The use of both techniques will ensure that a greater diversity of macroinvertebrates in the pond is collected.

Each kick net or Hester-Dendy sample for selenium analysis will have a minimum mass of 3 g wet weight, although larger samples, up to 10 g wet weight, are preferred. A portable balance will be utilized to estimate the weight of each kick net sample in the field, and this weight will be recorded in field notes. If the goal of collecting a mass of 3 g wet weight per sample cannot be achieved, then samples for a location will be composited prior to analysis.

A. Kick net samples

Three replicate kick net samples will be collected at each of the wetlands in mid-May. Wetland 3 will be visually divided into three parts corresponding to the flow of water through the wetland from the selenium source (Seep 2). The up-gradient sample will be collected at the outlet of Seep 2; the mid-gradient sample will be collected half way between Seep 2 and the outlet of Wetland 3; and the down-gradient sample will be collected at the outlet. The sediment retention pond will be similarly divided into three sampling areas. The up-gradient sample will be collected near the inlet; the mid-gradient sample will be collected on the north edge of the sediment retention pond, approximately half way between the inlet and outlet; and the down-gradient sample will be collected near the outlet. The sampling locations correspond to those sampled for surface water and sediment under a separate Program Directive (MNT-08-01 [DOE 2008]).

An aquatic kick net with 500 um mesh netting will be used to collect macroinvertebrate samples. Prior to sampling each wetland, the net will be cleaned using a non-phosphate detergent, followed by 2-5% nitric acid wash, and three rinses with laboratory-grade deionized water. The pre-cleaning process is designed to minimize contamination prior to use. The net will be worked around the perimeter of the wetland and in areas where there is open water between the emergent vegetation. The traditional kick net technique may also be employed, where the sampler gently kicks the water and substrate up-gradient from the net, driving macroinvertebrates into it. If macroinvertebrates are observed on the submerged vegetation, they will also be collected. Because they may bias samples, exceptionally large macroinvertebrates, such as large crayfish and snails, will not be collected. Field personnel will wear Nitrile gloves during sample collection, and gloves will be changed between wetlands.

Macroinvertebrates will be removed from the kick nets with pre-cleaned plastic forceps and placed in sterile pre-weighed standard plastic collection bottles. Every attempt will be made to minimize detritus and other plant materials in the samples. To minimize potential predation between macroinvertebrate groups, water will not be added to the sample containers.

B. Artificial substrate samples

Three Hester-Dendy samplers will be deployed in each of three locations (for a total of 9 samplers) in the sediment retention pond. These locations will correspond to the kick net sampling locations described above. The samplers will be deployed in early- to mid-April and monitored in mid-May during kick net sampling. If necessary, they will be monitored on a weekly basis after this time to ensure that they are removed at the peak of colonization.

The Hester-Dendy samplers are artificial substrate systems that allow water-column sampling of macroinvertebrates. The device consists of 14 masonite plates spaced at varying widths, and it has a total surface area of 0.16 m². The samplers will remain in the pond for 4 to 8 weeks, during which time they will be colonized by periphyton and later by macroinvertebrates, which feed on the periphyton. Each sampler will be anchored to a cinder block or a metal post to prevent migration and excessive sedimentation. The plates of two of the samplers in each location will be oriented vertically (as in 2005 and 2006), and one will be oriented horizontally (the more typical orientation) to create a diversity of sampling substrates and maximize the diversity of the organisms collected.

The samplers will be retrieved by placing a collection bag around the sampler while still submerged, detaching the sampler from the anchoring device, decanting excess water, and sealing the collection bag. This method will minimize the loss of insects from the sampler while they are being retrieved. The samplers will be chilled in the field and transported to the laboratory, where organisms will be picked off the plates with pre-cleaned plastic forceps for identification and preparation.

C. Sample Preparation and Preservation, and Quality Control

The field samplers will make qualitative notes, as practicable, during the collection of samples. These notes may include information about the macroinvertebrates' functional feeding groups

(e.g., detritivores, predators, and filter feeders), the relative abundances of various groups, and/or field conditions. As soon as possible after collection, samples will be chilled in the field and transported to the laboratory where they will be identified, rinsed, and prepared for shipment. All samples will be kept at or below 4°C during storage and shipping. Standard chain of custody methods and labels will be used for all collected samples.

Macroinvertebrate samples will be rinsed, sorted, and identified by Stoller personnel. Individual macroinvertebrates will be removed from collection jars in the laboratory, rinsed with deionized water on filter paper, examined, and identified to Order and Family (when possible). The samples will then be chilled and prepared for shipment to the Battelle Marine Sciences Laboratory for selenium analysis. One sample will be split for quality control. Composite samples will not be prepared unless the wet weight for a particular wetland is less than 1 g.

To assess the quality of the field sampling technique, two types of quality control samples will be collected, field duplicates and equipment blanks. One field duplicate will be collected in the field. A second field duplicate will be a split of a composite sample prepared after identification and sorting. Care will be taken to make field duplicates indistinguishable to the lab so that personnel performing analyses cannot determine which samples are duplicates.

Equipment blanks are used to verify that selenium-contaminated equipment does not affect the quality of the samples. One field blank (deionized water rinse water that has rinsed all pre-cleaned field equipment to be used) will be prepared prior to sampling and submitted to the laboratory for analysis.

IV. Laboratory Test Analyses

The goal of the macroinvertebrate field sampling effort is to provide the following samples to the laboratory for selenium analysis:

- three samples collected with kick nets from wetland 3
- three samples collected with kick nets from the sediment retention pond
- three samples collected from Hester-Dendy samplers from the sediment retention pond
- one field duplicate (collected in the field)
- one split sample (prepared in the laboratory)
- one 1 equipment blank

The samples will be analyzed by Battelle Marine Sciences Laboratory according to the guidelines included in a separate Statement of Work. These guidelines are summarized below:

A. Percent Moisture Determination

The samples collected will arrive at the laboratory “as collected” and require that the percent moisture be determined to allow the selenium results to be reported on a dry weight basis. Percent moisture is determined as the percent ratio of wet to dry weight for the entire sample. Dry weights will be determined by placing the wet sample in a pre-tared, pre-cleaned sample

container, lyophilizing (freeze drying) the entire sample, and then recording the change in weights.

B. Low-Level Trace Metals Analysis

The required analytical method for analyzing selenium in the macroinvertebrate samples is EPA Method 270.3, gaseous hydride atomic absorption.

Sample preparation must include:

- lyophilizing the samples and then homogenizing them using a ball-mill prior to digestion
- digesting an aliquot of approximately 0.5 g of each dried, homogeneous sample by combining with nitric and hydrochloric acids (aqua regia) in a Teflon vessel and heating in an oven at 130°C ($\pm 10^\circ\text{C}$) for a minimum of eight hours
- diluting with deionized water to achieve analysis volume, then submittal of analysis.

The digested samples must be analyzed for selenium using hydride generation flow injection atomic absorption spectroscopy (HGAA-FIAS).

- All results will be determined and reported in units of mg/kg on a dry-weight basis.
- The detection limit for selenium in the macroinvertebrate samples will be based on a methods detection limit (MDL) and reporting limit (RL) study performed by the laboratory. MDLs for trace metals are determined in accordance with 40 CFR Part 136 Appendix B.
- The RL is calculated by multiplying the target analyte MDL by 3.18. The value 3.18 is based on the Student's-t value for 7 to 10 replicates, the number of replicates usually analyzed to generate the MDL.
- The MDL for tissues must be less than 0.02 mg/kg dry weight with an RL less than 0.07 mg/kg dry weight.

C. Laboratory Quality Control

Internal quality control (QC) is an important part of the measurement system to ensure that analytical results are reliable and that data integrity is maintained. Laboratory performance will be evaluated through analysis of laboratory quality control samples (in conjunction with field quality control samples, as appropriate).

The analytical performance of the laboratory will be validated by reviewing the results from analysis of the blank, matrix spike, duplicate, and quality control check samples. The following describes the batch preparative quality control samples that are required by the analytical method.

- **Method Blank (MB):** A Method Blank consists of Type II ASTM water that is subjected to the sample preparation or extraction procedures and analyzed as a sample. It serves to measure contamination associated with preparation and analysis. One MB is required for the 20 samples or fewer samples. If the analyte of interest is above the RL, corrective action must be taken.
- **Matrix Spike (MS):** A Matrix Spike is an aliquot of sample to which a known amount of analyte has been added. It is subjected to the sample preparation or extraction procedures and analyzed as a sample. The stock solutions used for spiking are purchased or prepared independently of calibration standards. One MS is required for every 20 or fewer samples analyzed. The spike recovery measures the effects of interferences in the sample matrix and reflects the accuracy of the determination.
- **Matrix Spike Duplicate (MSD):** A Matrix Spike Duplicate is an additional aliquot of sample to which known amounts of analyte have been added and subjected to the same preparation and analytical scheme as the original sample. The Relative Percent Difference (RPD) between MS and MSD measures the precision of a given analysis. One MSD will be required for every 20 or fewer samples analyzed.
- **Laboratory Control Sample (LCS):** Laboratory Control Sample is created from a standard reference material which is a material similar in nature to the sample being processed [traceable to the National Institute of Standards and Technology (NIST) or other agencies, to the extent possible]. A known amount of analyte is added to an aliquot of Type II ASTM water. The LCS is subjected to the sample preparation or extraction procedure and analyzed as a sample. One LCS will be required every 20 or fewer samples analyzed.
- **Laboratory Replicate Sample (LRS):** Laboratory Replicate Samples are used to assess the homogenization techniques. Samples are homogenized, and then divided into two equal parts for analysis. Care is taken to make both samples representative of materials present, including heterogeneities. If possible, at least one sample will be prepared and analyzed as a LRS.

Laboratory results will be available approximately 45 days after the samples have been received. The laboratory will provide the results to Stoller in electronic form.

V. Data Report

The data report will be prepared by S.M. Stoller and submitted to DOE by September 30, 2008. The report will include the following information.

- A summary of the dates, times and locations of the field sampling activities
- Any communications with federal and state agencies, and other professional biologists
- Any communications or direction from DOE
- A summary of the field activities, GPS data (if applicable) and any maps generated

- A summary of the laboratory test analyses including results, methods, detection limits, and laboratory qualifiers
- Education/qualifications of field samplers
- Comparison of results from macroinvertebrate samples to ecological risk guidelines and benchmarks
- Recommendation for follow-on activities
- References

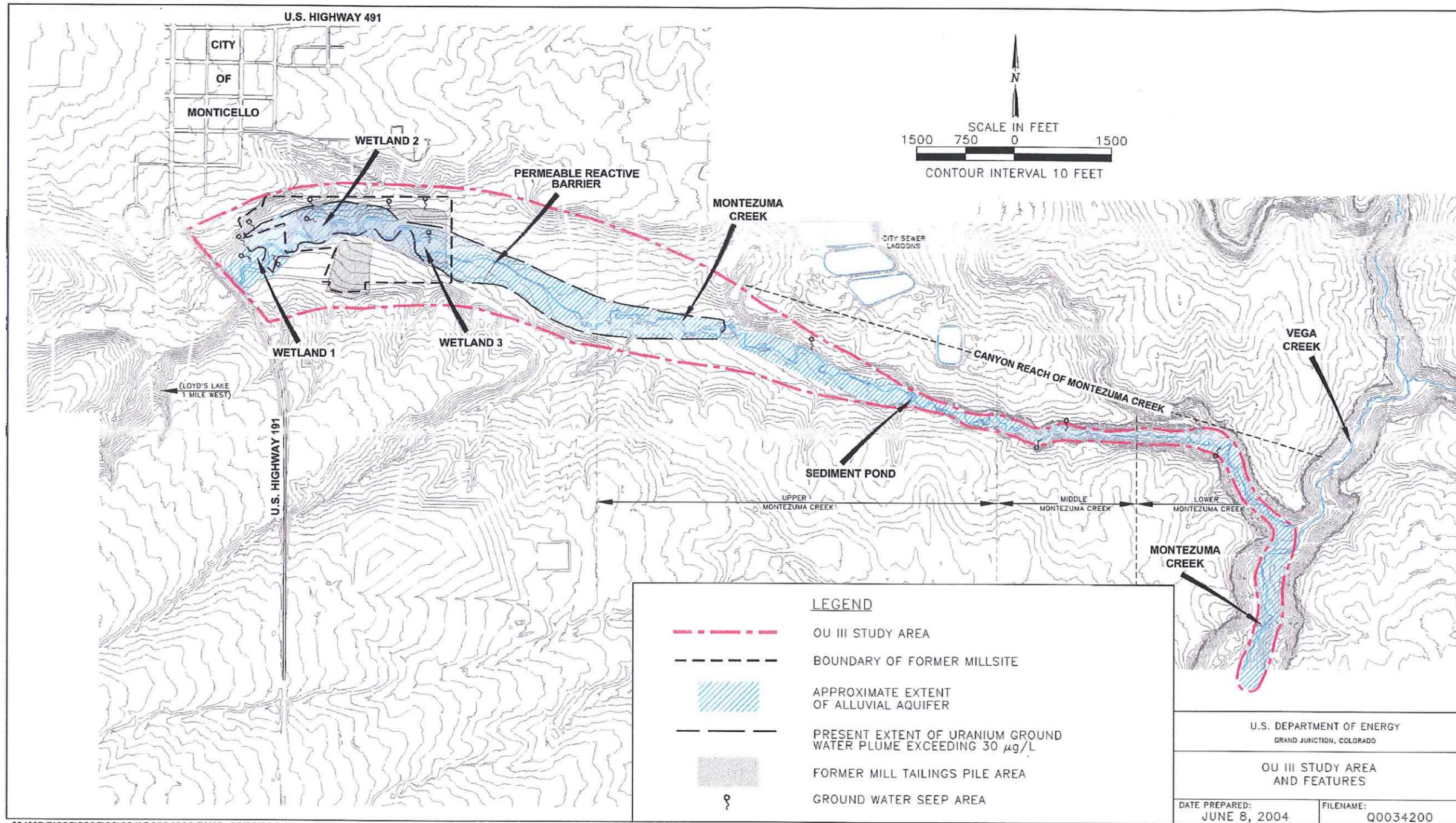
The report will be reviewed by DOE and transmitted to EPA and UDEQ by November 1, 2008.

VI. References

DOE (U.S. Department of Energy), 1998. *Operable Unit III Remedial Investigation, Appendix M – Ecological Risk Assessment*, GJO-97-9-TAR, U.S. Department of Energy Grand Junction Office, Grand Junction, Colorado, January.

DOE (U.S. Department of Energy), 2004. *MMTS Operable Unit III Post-Record of Decision Monitoring Plan*. DOE/LMGJ684-2004, U.S. Department of Energy, Office of Legacy Management, Grand Junction, Colorado, August..

DOE (U.S. Department of Energy), 2008. Program Directive MNT-08-01, *Biomonitoring: sediment and surface water sampling from former millsite Wetland 3 and the sediment retention pond*.



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