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CONTRACT NO. DE-AC05-86OR21548

# **SAMPLING PLAN FOR THE FEASIBILITY STUDY FOR THE QUARRY RESIDUALS OPERABLE UNIT AT THE WELDON SPRING SITE, WELDON SPRING, MISSOURI**

Weldon Spring Site Remedial Action Project  
Weldon Spring, Missouri

**JUNE 1997**

**REV. 0**

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U.S. Department of Energy  
Oak Ridge Operations Office  
Weldon Spring Site Remedial Action Project

Prepared by MK-Ferguson Company and Jacobs Engineering Group

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Contract No. DE-AC05-86OR21548

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Operable Unit at the Weldon Spring Site, Weldon Spring, Missouri

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Weldon Spring Site Remedial Action Project

Sampling Plan for the Feasibility Study for the Quarry Residuals Operable Unit  
at the Weldon Spring Site, Weldon Spring, Missouri

June 1997

Revision 0

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U.S. DEPARTMENT OF ENERGY  
Oak Ridge Operations Office  
Under Contract DE-AC05-86OR21548

## ABSTRACT

The Quarry Residuals Operable Unit is one of four operable units comprising the Weldon Spring Site Remedial Action Project. The components of this operable unit are: (1) the residual material remaining at the Weldon Spring quarry after removal of the bulk waste; (2) other media located in the surrounding vicinity of the quarry, including adjacent soil, surface water, and sediment in the Femme Osage Slough and several surrounding creeks; and (3) groundwater at the quarry that extends south of the Femme Osage Slough. This sampling plan describes the soil, groundwater, and surface water sampling that will be conducted at the Weldon Spring quarry to provide data to support the Feasibility Study for the Quarry Residuals Operable Unit. The two tasks are (1) determination of distribution coefficients (Kds) in the alluvial materials, and (2) isotopic oxygen ( $^{18}\text{O}$  and  $^{16}\text{O}$ ) and isotopic hydrogen ( $^2\text{H}$  and  $^1\text{H}$ ) comparisons for groundwater in the bedrock, alluvium, and Missouri River. This plan will outline the sampling methods, locations, and analysis for each task. Quality control and standard operating procedures will also be discussed.

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## 1. INTRODUCTION

This plan describes the soil, groundwater, and surface water sampling that will be conducted at the Weldon Spring Quarry to provide data for the evaluation of remedial alternatives for groundwater south of the quarry. The purpose and methodology for this additional data are outlined under this plan.

### 1.1 Purpose and Scope

The purpose of this plan is to outline the objectives for the two tasks which are needed to support the *Feasibility Study for the Quarry Residuals Operable Unit (QROU)*. The two tasks are (1) determination of distribution coefficients (Kds) in the alluvial materials, and (2) isotopic oxygen ( $^{18}\text{O}$  and  $^{16}\text{O}$ ) and isotopic hydrogen ( $^2\text{H}$  and  $^1\text{H}$ ) comparisons for groundwater in the bedrock, alluvium, and Missouri River. This plan will outline the sampling methods, locations, and analyses for each task. Quality control and standard operating procedures will also be discussed.

The scope of the first task is to obtain groundwater and soil samples using a vehicle-mounted, hydraulically-powered, soil probing machine, in order to obtain soil and groundwater samples from the same interval at each location. These samples will be used to determine site-specific distribution coefficients for uranium.

The scope of the second task is to sample groundwater utilizing existing monitoring wells and surface water for the determination of a mixing zone in the alluvium south of the quarry. This mixing boundary will be determined by comparing the oxygen isotope ratios ( $^{18}\text{O}$ :  $^{16}\text{O}$ ) and hydrogen isotope ( $^2\text{H}$ :  $^1\text{H}$ ) ratios at selected bedrock and alluvial wells in the quarry area and the Missouri River.

## 1.2 Previous Sampling Activities

### 1.2.1 Distribution Coefficients

Site-specific Kds have not been established for any of the quarry media. A summary of Kds that have been used for the quarry is presented in Table 1-1. Many of these Kds were derived from sorption coefficients calculated using Kds from literature for rock and soil; however, the actual Kds were not presented in the original report (Ref. 1). Kds for the upland soils were estimated from studies performed at the chemical plant on unconsolidated materials in that area (Ref. 2).

Table 1-1 Distribution Coefficients for Uranium

MEDIA	Kd (ml/g)	SORPTION COEFFICIENT	REFERENCE
Fractured Limestone	0.024	1.1	BGA 1984
Alluvium	1.1	3.6	BGA 1984
Clay	6.9	36.4	BGA 1984
Upland Soils	10.7 - 437	—	Schumacher 1993

Sorption coefficients (a) were derived using the following equation (BGA 1984):

$$a = \frac{\rho_b}{1-\theta} K_d$$

where: Kd = distribution coefficient  
 $\rho_b$  = bulk density  
 $\theta$  = effective porosity

### 1.2.2 $^{18}\text{O}$ : $^{16}\text{O}$ and $^2\text{H}$ : $^1\text{H}$ Ratios

Site-specific oxygen and hydrogen isotope data have not been collected for any of the quarry media.

### 1.3 Sampling Objectives

The objectives for each sampling task are presented below.

#### 1.3.1 Distribution Coefficients

Groundwater and soil sampling will be conducted to meet the following objectives:

- Determine the uranium concentrations in soils at selected locations and depths.
- Determine the uranium concentrations in groundwater from the same locations and depths as the above soil samples.
- Establish the relationship between the uranium levels in soil and groundwater at each location and depth interval.
- Determine if these relationships correlate to soil types and/or sample locations.

#### 1.3.2 $^{18}\text{O}$ : $^{16}\text{O}$ and $^2\text{H}$ : $^1\text{H}$ Ratios

Groundwater sampling will be conducted to meet the following objectives:

- Determine the  $^{18}\text{O}$ ,  $^{16}\text{O}$ ,  $^2\text{H}$ , and  $^1\text{H}$  levels in groundwater in the bedrock, alluvium north of the slough, and Missouri River alluvium.
- Determine the  $^{18}\text{O}$ ,  $^{16}\text{O}$ ,  $^2\text{H}$ , and  $^1\text{H}$  levels in the Missouri River.
- Establish the ratios for  $^{18}\text{O}$  and  $^{16}\text{O}$  and  $^2\text{H}$  and  $^1\text{H}$  for the upland groundwater (bedrock), alluvial groundwater, and the Missouri River.

- Compare the  $^{18}\text{O}$  and  $^{16}\text{O}$  and  $^2\text{H}:^1\text{H}$  ratios to determine if they can be used to calculate mixing ratios for the upland groundwater and Missouri River.

## 2. SAMPLING REQUIREMENTS

This section outlines the sampling method and locations for the two tasks. The volume of sample required and sample preparation for specific analyses are also summarized in this section.

### 2.1 Distribution Coefficient Determination

#### 2.1.1 Sampling Method

A vehicle-mounted hydraulically-powered soil probing machine will be utilized to obtain soil and groundwater samples at each location. Equipment to be utilized will be *Geoprobe*® or equivalent. Standard operating procedures (SOPs) for the *Geoprobe*® equipment will be followed. The applicable SOPs are provided in Appendix A.

##### 2.1.1.1 Soils

Samples will be collected from specified depths using a discrete sampler. Liners (clear plastic) will be used to maintain and store samples. The SOP for the *Geoprobe*® *Large Bore Soil Sampler - Discrete Interval Soil Sampler* (Ref. 3) will be followed during sampling activities.

##### 2.1.1.2 Groundwater

Samples from north of the slough will be obtained by using temporary well points. These well points will be constructed of 1-in. diameter polyvinyl chloride (PVC) (Schedule 40) tubing with a 36-in. screened section pushed by the sampler into the boring used to obtain the soil sample. A steel drive point will be attached to the end and the push rods installed through the PVC for installation. Temporary well points are necessary due to the low hydraulic conductivity of the materials in this area and some development will be necessary to obtain water. Several samplings may be necessary to obtain the necessary sample volume. These well points will be removed prior to 30 days in accordance with 10 CSR 23.

South of the slough, samples will be obtained by using a screen point groundwater sampler. Due to the higher conductivities of the alluvium, temporary well points will not be required. The SOP for either the *Geoprobe® Screen Point Groundwater Sampler* (Ref. 4) or the *Geoprobe® Screen Point 15 Groundwater Sampler* (Ref. 5) will be followed during sampling activities. The water will be obtained adjacent to the location of the soil boring.

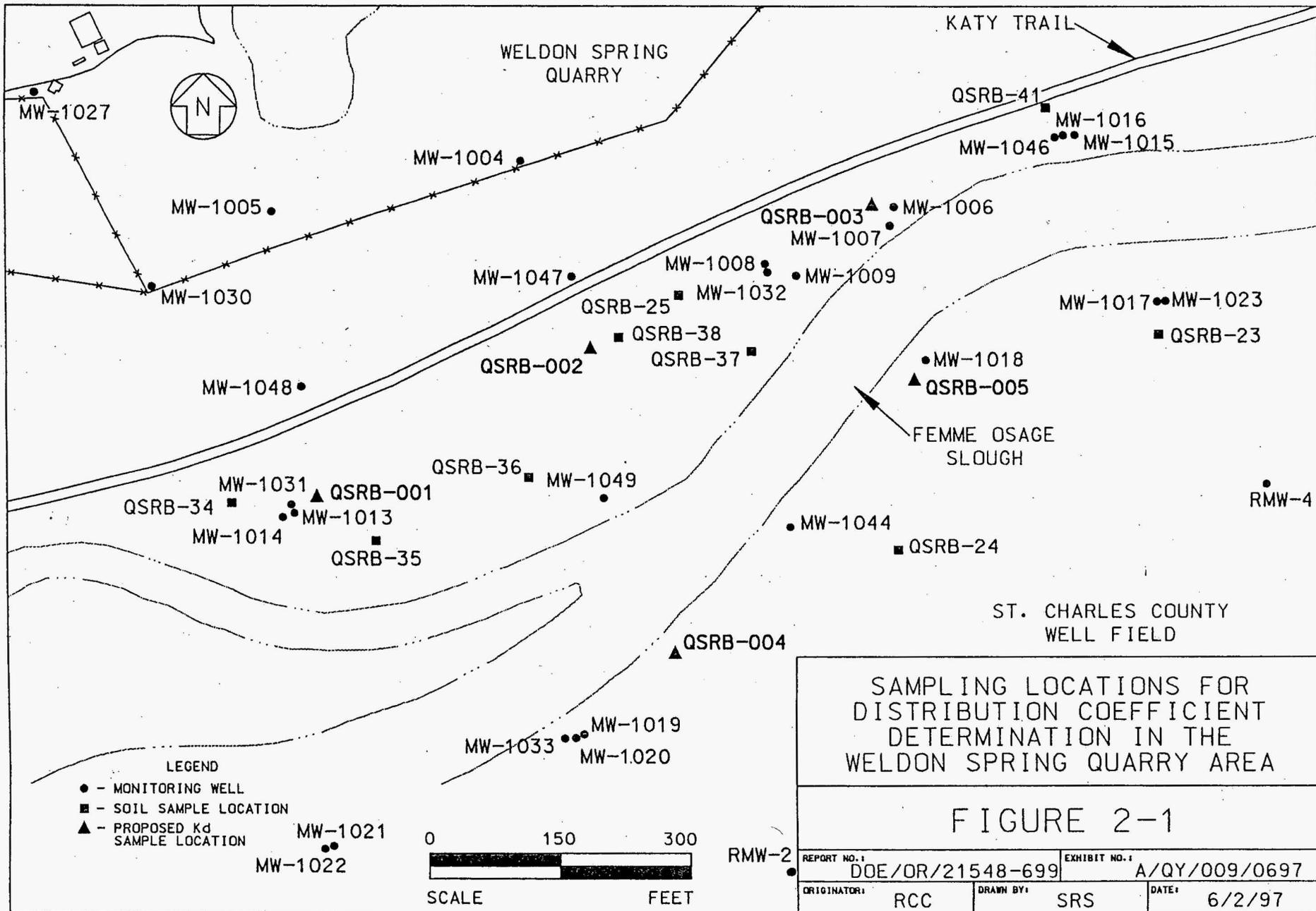
### 2.1.2 Sample Locations

Soil and groundwater samples will be obtained from five locations north and south of the slough (Figure 2-1). These locations have been selected to represent the width of the uranium plume north of the slough and the potential migration pathways south of the potential migration pathway south of the slough. These locations and the sampling intervals are summarized in Table 2-1. Depths and sampling intervals were determined from previous investigations and current groundwater data.

Table 2-1 Sampling Locations and Intervals for the Determination of Kds

LOCATION	DEPTH TO TOP OF ROCK (ft)	DEPTH TO GROUNDWATER	SAMPLE INTERVAL 1	SAMPLE INTERVAL 2
QRSB-001	20	8	8 - 10	18 - 20
QRSB-002	15	5	8 - 10	13 - 15
QRSB-003	12	3	3 - 5	10 - 12
QRSB-004	70	15	14 - 16	48 - 50
QRSB-005	50	14	14 - 16	48 - 50

Sample intervals are selected based on lithology in the soil boring, screened interval of nearby wells, uranium concentrations in nearby soil borings, depth to the top of bedrock, and



SAMPLING LOCATIONS FOR  
DISTRIBUTION COEFFICIENT  
DETERMINATION IN THE  
WELDON SPRING QUARRY AREA

FIGURE 2-1

depth to groundwater. Sample intervals are selected to meet the following requirements for each boring and should represent:

- Different soil types (i.e., clay, silt, or sand) in each boring.
- The uranium concentrations monitored in nearby wells in at least one sample interval.
- The highest uranium concentrations monitored in a nearby soil boring in at least one sample interval.
- The soil type located directly above the bedrock in the lowest sample interval.
- Saturated conditions.

Soil boring locations are approximate; exact locations will be determined in the field based on accessibility. These locations will be surveyed after sampling activities have been completed.

### 2.1.3 Sample Preparation

Sample collection and labeling will be performed in accordance with procedures ES&H 4.4.1, *Groundwater Sampling* and ES&H 4.4.5, *Soil/Sediment Sampling*.

Soil and groundwater sample containers, volumes, and preservation requirements are listed in Table 2-2.

Table 2-2 Kd Determination Sampling Requirements

MEDIA	VOLUME	CONTAINER	PRESERVATION
Groundwater	1 liter	plastic	store at 4°C
Soil	100 grams	plastic	store at 4°C

Groundwater samples will be filtered using a 0.45 micron in-line filter to remove sediments and colloids.

Additional groundwater (approximately 100 ml to 200 ml) will be required to obtain field measurements of temperature, pH, and Eh. These measurements will be taken in accordance with the Procedure ES&H 4.4.1, *Groundwater Sampling*.

Soil and groundwater samples will be kept at 4°C immediately after collection and during shipment.

## 2.2 $^{18}\text{O}$ : $^{16}\text{O}$ and $^2\text{H}$ : $^1\text{H}$ Ratios

### 2.2.1 Sampling Method

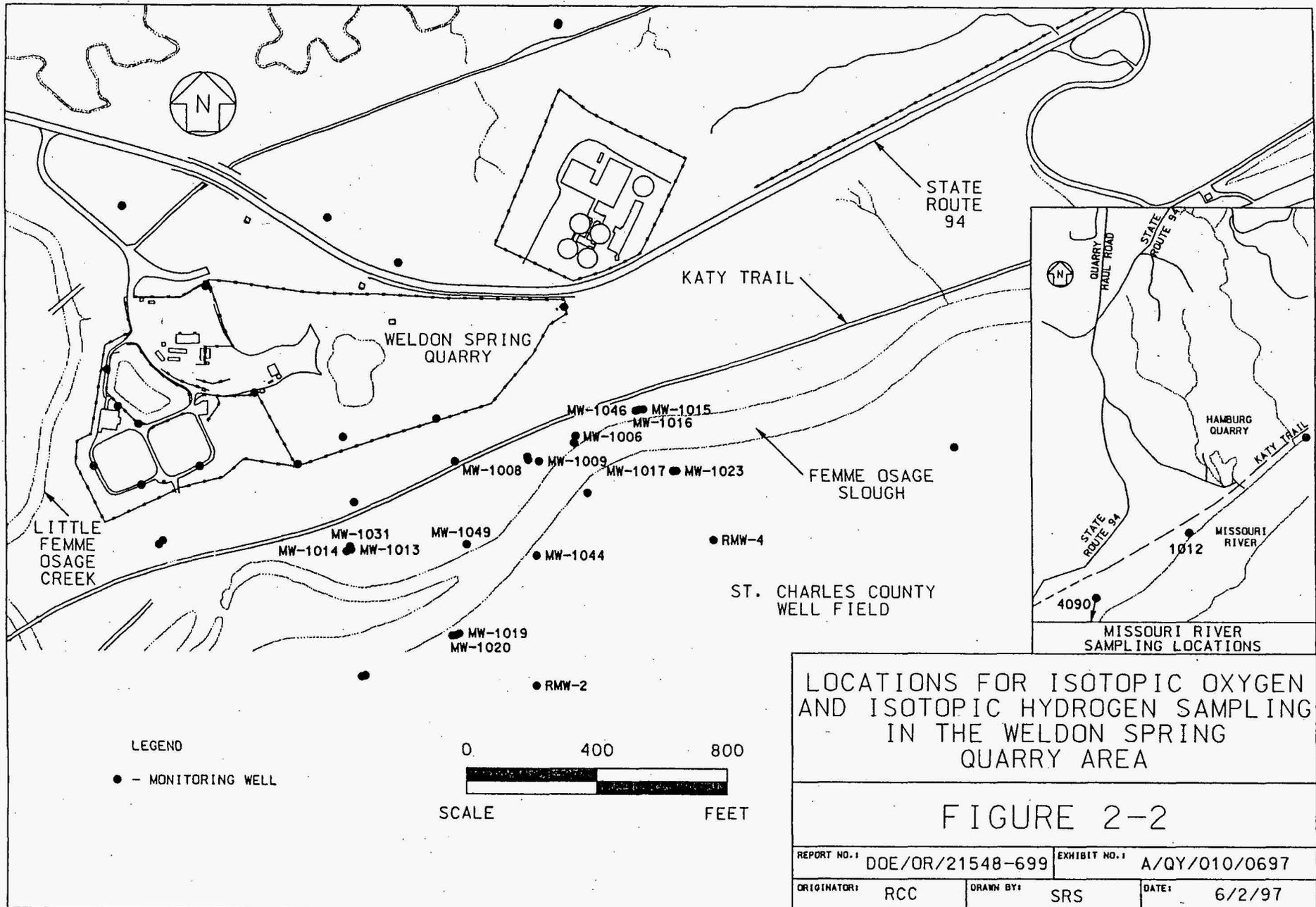
Groundwater samples will be collected in accordance with Procedure ES&H 4.4.1, *Groundwater Sampling*. Surface water samples will be collected in accordance with Procedure ES&H 4.3.1, *Surface Water Sampling*.

### 2.2.2 Sampling Locations

Samples will be obtained from 19 existing monitoring wells and two locations in the Missouri River (Figure 2-2). Groundwater or surface water sampling are summarized in Table 2-3.

Sampling locations were selected to establish isotopic oxygen and isotopic hydrogen ratios:

- In the bedrock aquifer discharging to the Missouri River Alluvium.
- In the shallow alluvium north of the slough.



LOCATIONS FOR ISOTOPIC OXYGEN  
AND ISOTOPIC HYDROGEN SAMPLING  
IN THE WELDON SPRING  
QUARRY AREA

FIGURE 2-2

REPORT NO.:	DOE/OR/21548-699	EXHIBIT NO.:	A/QY/010/0697
ORIGINATOR:	RCC	DRAWN BY:	SRS
		DATE:	6/2/97

Table 2-3 Oxygen and Hydrogen Isotope Sampling Locations

LOCATION	UNIT	LOCATION	UNIT
GROUNDWATER			
MW-1006	Alluvium (s)	MW-1023	Alluvium (s)
MW-1008	Alluvium (s)	MW-1031	Plattin
MW-1009	Alluvium (s)	MW-1044	Alluvium (s)
MW-1013	Decorah	MW-1046	Plattin
MW-1014	Alluvium (s)	MW-1049	Alluvium (s)
MW-1015	Decorah	MW-RMW2	Alluvium (d)
MW-1016	Alluvium (s)	MW-RMW4	Alluvium (d)
MW-1017	Alluvium (d)	MW-PW08	Alluvium (d)
MW-1019	Alluvium (d)	MW-PW09	Alluvium (d)
MW-1020	Alluvium (s)		
SURFACE WATER			
SW=4090	Missouri River	SW-1012	Missouri River

d Deep alluvium  
 s Shallow alluvium

- 
- In the Missouri River.
  - Across the Missouri River Alluvium.

### 2.2.3 Sample Preparation

Sample collection and labeling will be performed in accordance with Procedure ES&H 4.4.1, *Groundwater Sampling* and ES&H 4.3.1, *Surface Water Sampling*.

Groundwater sample containers, volumes, and preservations requirements are listed in Table 2-4.

Table 2-4 Oxygen and Hydrogen Isotopic Sample Requirements

MEDIA	VOLUME	CONTAINER	PRESERVATION
Groundwater	40 ml*	plastic	store at 4°C

- Obtain 3 - 40 ml samples

---

Groundwater samples will be filtered in accordance with Procedure ES&H 4.5.8, *Water Sample Filtering*. No head space should be left in the sample container by filling to form a negative meniscus.

Field measurements of temperature, pH, and Eh will be taken in accordance with Procedure ES&H 4.4.1, *Groundwater Sampling*.

Samples will be kept at 4°C immediately after collection and during shipment.

### 3. STANDARD OPERATING PROCEDURES

This section outlines the procedures that will be followed during these sampling events. Some deviations will be made, as noted in the appropriate sections, because the laboratory coordination, data archival and use will be performed by Argonne National Laboratory (ANL), the data requester.

#### 3.1 Sample Identification

Sample numbers will be assigned according to Procedure ES&H 4.1.1, *Numbering System for Environmental Samples and Sample Locations*. A summary of the sample identification number for each location is provided in Table 3-1.

LOCATION	SAMPLE ID	SAMPLE TYPE
QRSB-001	SO-197001-(01.02)	Soil
	IS-197001-(01.0)	Groundwater
QRSB-002	SO-197002-(01.02)	Soil
	IS-197002-(01.02)	Groundwater
QRSB-003	SO-197003-(01.02)	Soil
	IS-197003-(01.02)	Groundwater
QRSB-004	SO-197004-(01.02)	Soil
	IS-197004-(01.02)	Groundwater
QRSB-005	SO-197005-(01.02)	Soil
	IS-1970059(01.02)	Groundwater
MW-1006	GW-1006-(date)	Groundwater
MW-1008	GW-1008-(date)	Groundwater
MW-1009	GW-1009-(date)	Groundwater
MW-1013	GW-1013-(date)	Groundwater
MW-1014	GW-1014-(date)	Groundwater
MW-1015	GW-1015-(date)	Groundwater
MW-1016	GW-1016-(date)	Groundwater
MW-1017	GW-1017-(date)	Groundwater

Table 3-1 Sample Identification Numbers

LOCATION	SAMPLE ID	SAMPLE TYPE
MW-1019	GW-1019-(date)	Groundwater
MW-1020	GW-1020-(date)	Groundwater
MW-1023	GW-1023-(date)	Groundwater
MW-1031	GW-1031-(date)	Groundwater
MW-1044	GW-1044-(date)	Groundwater
MW-1046	GW-1046-(date)	Groundwater
MW-1049	GW-1049-(date)	Groundwater
MW-RMW2	GW-RMW2-(date)	Groundwater
MW-RMW4	GW-RMW8-(date)	Groundwater
MW-PW08	GW-OW04-(date)	Groundwater
MW-PW09	GW-PW09-(date)	Groundwater
SW-1012	SW-1012-(date)	Surface water
SW-4090	SW-4090-(date)	Surface water

### 3.2 Chain-of-Custody Requirements

Sample chain-of-custody will be maintained in accordance with Procedure ES&H 4.1.2, *Initiation, Generation, and Transfer of Environmental Chain of Custody*. Chain-of-custody forms (COCs) for laboratory samples will be completed and placed in the sample coolers. Sample coolers prepared for shipment will be sealed with chain-of-custody control seals signed and dated by the shipper.

The exception to Procedure ES&H 4.1.2 is that the laboratory coordinator will not generate the COCs or coordinate sample shipment to an off-site laboratory. These tasks will be performed by the sampler. Samples will be shipped to laboratories specified by ANL.

### 3.3 Sample Shipment

Samples will be packaged and transported to an off-site laboratory, as specified by ANL, in accordance with site procedures. Samples will be shipped per ECDI-3, *HMSTA Operations*. A separate custody record must accompany each sample cooler.

### **3.4 Equipment Decontamination**

All equipment and tools used to collect or transfer samples will be cleaned and decontaminated between each sample. Decontamination will be performed in accordance with Procedure ES&H 4.1.3, *Sampling Equipment Decontamination*. Decontamination wastes will be handled per ECDI-17, *Handling and Disposition of Site-Generated Waste*. Equipment and tools will be stored during sampling activities to maintain cleanliness. This may include use of plastic sheeting, boxes, or other appropriate methods. Where applicable, disposable sampling devices will be employed.

### **3.5 Borehole Abandonment**

Boreholes will be backfilled and abandoned as specified in Procedure ES&H 4.4.4, *Subsurface Monitoring Device Plugging and Abandonment*.

### **3.6 Documentation**

Sample locations, samples collected, and all related data will be recorded in a logbook at the time of collection, as outlined in Procedure ES&H 1.1.4, *Logbook Procedure*.

Field sampling forms generated as a result of this plan will be maintained in accordance with the requirements of Procedure SQP-7, *Quality Assurance Records*.

## 4. ANALYTICAL METHODS

### 4.1 Distribution Coefficients

Distribution coefficients (Kds) for uranium will be determined by a method developed at Argonne National Laboratory that is based on a study of complex interactions between dissolved metals and their environment observed in natural systems (Ref. 6). Typically, 20 g to 25 g of soil are contacted with approximately 15 ml to 20 ml of surface water for three weeks at room temperature. For the soil samples located in the aquifer region, filtered groundwater from the soil sample locations will be used. At the end of the contact period, the sample is centrifuged to obtain about 5 ml for the determination of uranium concentration. A separate aliquot of the remaining soil is used for uranium concentration determination in that phase.

### 4.2 $^{18}\text{O}$ : $^{16}\text{O}$ and $^2\text{H}$ : $^1\text{H}$ Ratios

Oxygen isotopes ( $^{18}\text{O}$  and  $^{16}\text{O}$ ) and hydrogen isotopes ( $^2\text{H}$  and  $^1\text{H}$ ) will be determined by Stable Isotope Ratio Analysis (SIRA). The procedure for sample preparation, analytical method, and instrument calibration are provided in Appendix B.

## 5. QUALITY CONTROL

MK-Ferguson Company, the Project Management Contractor at the Weldon Spring Site Remedial Action Project (WSSRAP) has developed the *Environmental Quality Assurance Project Plan* (EQAPjP) (Ref. 7) to guide all environmental activities conducted at the WSSRAP in accordance with U.S. Environmental Protection Agency guidelines. The *Sample Management Guide* (Ref. 8) has been developed following the guidelines listed in the EQAPjP. This guide established the approach to sample planning, collection, and data analysis.

Quality control samples will be collected to ensure consistent and accurate performance of sample collection and laboratory analysis. Table 5-1 provides a summary list of the quality control samples that will be collected to support this effort.

Table 5-1 Field Quality Control Sample Summary

QC SAMPLE TYPE	FREQUENCY	PURPOSE
Field Replicate	1 per matrix type (or 1 per 20)	Assess matrix and inter-laboratory variability
Equipment Blank (non-dedicated equipment only)	1 per matrix type (or 1 per 20)	Assess effectiveness of decontamination.

## 6. REFERENCES

1. Schumacher, J.G. *Geochemistry and Migration of Contaminants at the Weldon Spring Chemical Plant Site, St. Charles County, Missouri-1989-91*. U.S. Geological Survey, Open File Report 93-422, 102 pp.
2. Berkeley Geosciences Associates. *Characterization and Assessment for the Weldon Spring Quarry Low-Level Radioactive Waste Storage Site*. DOE/OR/-853; DE850005424. Prepared for the U.S. Department of Energy, Oak Ridge Operations Office. September 1984.
3. Geoprobe® Systems, A Division of Kejr Engineering, Inc. *Geoprobe® Screen Point Groundwater Sampler, Standard Operating Procedure*, Technical Bulletin No. 94-440, Reprinted January 1995.
4. Geoprobe® Systems, A Division of Kejr Engineering, Inc. *Geoprobe® Screen Point 15 Groundwater Sampler, Standard Operating Procedure*, Technical Bulletin No. 95-1500, prepared October 1995.
5. Geoprobe® Systems, A Division of Kejr Engineering, Inc. *Geoprobe® AT=660 Series Large Bore Soil Sampler, Standard Operating Procedure*, Technical Bulletin No. 93-660, revised June 1995.
6. Honeyman, B.D. and Santschi, P.H. *Metals in Aquatic Systems*. Environmental Science and Technology, Volume 22 No. 8 pp 862-871, 1988.
7. MK-Ferguson Company and Jacobs Engineering Group. *Sample Management Guide*. Rev. 0. DOE/OR/21548-499. Prepared for the U. S. Department of Energy, Oak Ridge Operations Office. St. Charles, MO. March 1995.
8. MK-Ferguson Company and Jacobs Engineering Group. *Environmental Quality Assurance Project Plan*, Rev. 2. DOE/OR/21548-352. Prepared for the U.S. Department of Energy, Oak Ridge Operations Office. St. Charles, MO. May 1996.

## **PROCEDURES**

ECDI-17, *Handling and Disposition of Site-Generated Waste*

ES&H 1.1.4, *Logbook Procedure*

ES&H 4.1.1, *Numbering System for Environmental Samples and Sample Locations*

ES&H 4.1.2, *Initiation, Generation, and Transfer of Environmental Chain of Custody*

ES&H 4.1.3, *Sampling Equipment Decontamination*

ES&H 4.3.1, *Surface Water Sampling*

ES&H 4.4.1, *Groundwater Sampling*

ES&H 4.4.4, *Subsurface Monitoring Device Plugging and Abandonment*

ES&H 4.4.5, *Soil/Sediment Sampling*

SQP-7, *Quality Assurance Records*

## **CODE OF STATE REGULATIONS**

10 CSR 23, *Division of Geology and Land Survey, Well Construction*

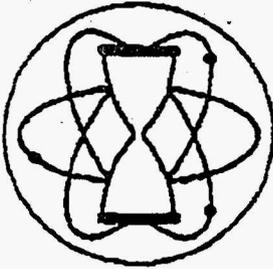
APPENDIX A  
Geoprobe® Standard Operating Procedures

**The following documents are protected by copyright laws and cannot be reproduced:**

1. Geoprobe Large Bore Soil Sampler Discrete Interval Soil Sampler Standard Operating Procedure, Technical Bulletin No. 93-600, September, 1996
2. Geoprobe Screen Point Ground Water Sampler Standard Operating Procedure, Technical Bulletin No. 94-440, Reprinted: January, 1995
3. Geoprobe Screen Point 15 Groundwater Sampler Standard Operating Procedure, Technical Bulletin No. 95-1500, Prepared: October, 1995

They may be obtained from Geoprobe Systems a Division of Kejr Engineering

APPENDIX B  
Oxygen and Hydrogen Iostope Analysis of Water



**GEOCHRON LABORATORIES** a division of  
**KRUEGER ENTERPRISES, INC.**

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**STABLE ISOTOPE RATIO ANALYSIS (SIRA) LABORATORY**

**OXYGEN ISOTOPE ANALYSIS OF WATER**

**PREPARATION**

For oxygen SIRA, water samples are prepared using the CO<sub>2</sub>/water equilibration method of Epstein and Mayeda (see *Geochimica et Cosmochimica Acta* 4, 213-224, 1953). In this technique, a known volume of water sample is combined in a sealed tube with a known volume of CO<sub>2</sub> gas and allowed to equilibrate for at least 24 hours at 25 °C. At Geochron, we normally combine 2 ml of water sample (if available) with 5 ccSTP of Tank CO<sub>2</sub> gas in a standard 7 ml vial. (Smaller samples (eg. mineral water of hydration) can be accommodated using a Micro-CO<sub>2</sub>/water equilibration technique developed here at Geochron.) After equilibration, the CO<sub>2</sub> gas is cryogenically purified by passing it through a dry ice/alcohol trap (-78.5 °C) to remove H<sub>2</sub>O, and then trapped in two liquid nitrogen traps (-196 °C). The frozen CO<sub>2</sub> is pumped-on to a vacuum of at least 100 mtorr to remove any non-condensable gases, and then cryogenically transferred to a sample flask and sealed to await analysis on the mass spectrometer.

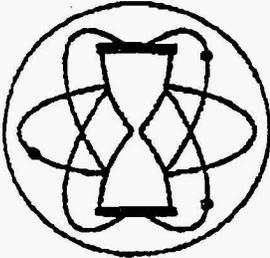
**ANALYSIS**

The oxygen-18/oxygen-16 ratio of the equilibrated CO<sub>2</sub> gas is determined on a VG Micromass gas source stable isotope ratio mass spectrometer (Model 903). In practice, the actual ratio is not measured directly, but rather is determined by comparison of the sample gas with a reference CO<sub>2</sub> gas. This method avoids many possible sources of instrumental error since both the reference and sample gases are affected and the errors are cross-cancelling in the calculation of the results.

Three measurements each of the reference and sample gases are made allowing four <sup>18</sup>O/<sup>16</sup>O ratios to be calculated for statistical comparison. If the instrumental precision on the measured oxygen ratios exceeds 0.08 ‰, the analysis is rejected and the gas is rerun.

The oxygen isotopic difference between the Tank CO<sub>2</sub> and the equilibrated CO<sub>2</sub> is directly related to the oxygen isotope composition of the sample water. The latter can be calculated by mass balance considerations knowing the volumes of sample water and Tank CO<sub>2</sub> involved, the <sup>18</sup>O/<sup>16</sup>O ratio of the Tank CO<sub>2</sub>, and the CO<sub>2</sub> - water oxygen isotope fractionation factor at 25 °C.

The final SIRA results are reported in terms of the relative difference (delta value denoted by the symbol δ) in parts per thousand (termed per mil, and denoted by the symbol ‰) of the <sup>18</sup>O/<sup>16</sup>O ratio of the sample relative to that of the internationally



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**STABLE ISOTOPE RATIO ANALYSIS (SIRA) LABORATORY**

**OXYGEN ISOTOPE ANALYSIS OF WATER**  
 (cont.)

ANALYSIS (cont.)

accepted standard Standard Mean Ocean Water (SMOW). The method precision for oxygen SIRA on water is about  $\pm 0.2$  ‰.

Duplicate preparations and analyses are routinely done on about 1 in 10 samples, or when an anomalous result is suspected. If the results of duplicate analyses are not within 0.3 ‰ of each other, the sample is prepared and analyzed again.

REFERENCE GAS/INSTRUMENT CALIBRATION

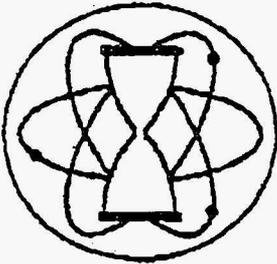
Two aliquots (Ref I and Ref II) of reference CO<sub>2</sub> are produced periodically by reaction of Geochron's in-house carbonate (marble) standard (COR-0101) with 100% orthophosphoric acid in an evacuated reaction vessel at 50 °C. After both reference gases have been analyzed with respect to the previous reference gas (if available), they are analyzed against each other so that their relative compositions are well characterized. One of the reference gases (usually Ref I) is then put into use, and its composition is monitored by periodically analyzing the other reference, providing an internal check on its composition. If the difference in composition between Ref I and Ref II changes by  $\pm 0.2$  ‰, two new reference gases are produced.

The calibration of the CO<sub>2</sub> reference gases is monitored by periodically analyzing various international reference materials, including SMOW ( $\delta^{18}\text{O} = 0.0$  ‰), and Standard Light Antarctic Precipitation (SLAP -  $\delta^{18}\text{O} = -55.5$  ‰). However, in order to conserve these standard materials, an in-house reference water (OR-76664) is maintained which has a  $\delta^{18}\text{O}$  composition of -6.9 ‰. If the results of these analyses are consistently  $> 0.2$  or  $< -0.2$  ‰ off from the accepted values, two new reference gases are produced.

Zero enrichment measurements are accomplished on a weekly basis. Three or more measurements are taken and averaged. If the average exceeds  $\pm 0.05$  ‰, a zero enrichment is input into the program.

Updated 2 OCT 1996

Marshall L. Otter  
 SIRA Laboratory Manager



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**STABLE ISOTOPE RATIO ANALYSIS (SIRA) LABORATORY**

**HYDROGEN ISOTOPE ANALYSIS OF WATER**  
Uranium Reduction

**PREPARATION**

For hydrogen SIRA, hydrogen gas is generated from each water sample by the uranium reduction method. The water (about three microliters) is introduced into the preparation line, doubly distilled and passed through metallic uranium heated to 800 °C. At this temperature, the water reacts with the uranium producing H<sub>2</sub> gas. As the gas is produced, it is reacted to uranium hydride (UH<sub>3</sub>) in an absorption reservoir of metallic uranium at 80 °C. This ensures that the reduction of water to H<sub>2</sub> goes to completion. The UH<sub>3</sub> is then quantitatively decomposed back to H<sub>2</sub> gas at 800 °C and the H<sub>2</sub> is introduced directly into the mass spectrometer for analysis.

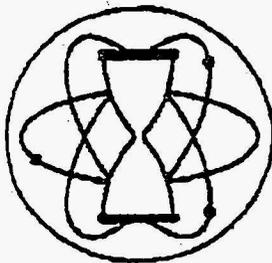
The memory effect caused by adsorption of the H<sub>2</sub> onto the uranium metal has a magnitude of about 3% for samples of natural compositions. For differences of < 50 ‰ from one sample to the next, the memory effect is less than the analytical uncertainty of the method which is +/- 2 ‰. If a difference of > 50 ‰ is found between two samples, additional aliquots are prepared until equilibrium is obtained.

**ANALYSIS**

The Deuterium/Hydrogen (D/H) ratio is determined using a VG Micromass gas source stable isotope ratio mass spectrometer (Model 502D). In practice, the actual ratio is not measured directly, but rather is determined by comparison of the sample H<sub>2</sub> gas with a reference H<sub>2</sub> gas. This method avoids many possible sources of instrumental error since both the reference and sample gases are affected and the errors are cross-cancelling in the calculation of the results.

Four measurements each of the reference and sample gases are made allowing six D/H ratios to be calculated for statistical comparison. If the instrumental precision on the measured hydrogen ratios exceeds 1 ‰, the analysis is rejected and the gas is rerun.

The raw data are corrected for the production of H<sub>3</sub><sup>+</sup> in the analyzer which is checked daily.



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**STABLE ISOTOPE RATIO ANALYSIS (SIRA) LABORATORY**

**HYDROGEN ISOTOPE ANALYSIS OF WATER**  
Uranium Reduction  
(cont.)

ANALYSIS (cont.)

The final SIRA results are reported in terms of the relative difference ( $\delta$  value denoted by the symbol  $\delta$ ) in parts per thousand (termed per mil, and denoted by the symbol ‰) of the D/H ratio of the sample relative to that of the internationally accepted standard Standard Mean Ocean Water (SMOW). The method precision for hydrogen SIRA on water is about  $\pm 2$  ‰.

Duplicate preparations and analyses are routinely done on about 1 in 10 samples, or when an anomalous result is suspected. If the results of duplicate analyses are not within 3 ‰ of each other, the sample is prepared and analyzed again.

REFERENCE GAS/INSTRUMENT CALIBRATION

The reference gas is produced from an aliquot of local tap water by the uranium reduction method.

The reference H<sub>2</sub> gas is calibrated with respect to Standard Mean Ocean Water (SMOW -  $\delta D = 0$  ‰) and Standard Light Antarctic Precipitation (SLAP -  $\delta D = -428$  ‰) using the method recommended by the International Atomic Energy Agency (see Nature 271, 534-536, 1978). Replicate analyses of each standard are run against the reference gas and the SMOW/SLAP scale is determined.

The calibration of the reference gas is checked periodically by preparing and analyzing additional aliquots of SMOW and SLAP, as well as other reference materials including Greenland Ice Sheet Precipitation (GISP -  $\delta D = -190$  ‰).

Updated 2 OCT 1996

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APPENDIX C  
Document Hierarchy

LEVEL I  
DOCUMENTS HAVING  
THE FORCE OF  
LAW OR CONTRACT

DEPARTMENT OF  
ENERGY CONTRACT  
DE-AC05-86OR21548

DEPARTMENT OF  
ENERGY ORDERS

FEDERAL AND STATE  
LAWS AND  
REGULATIONS

FEDERAL FACILITY  
AGREEMENT AND  
AMENDMENTS

RECORD OF  
DECISION  
ROD/RAD

LEVEL 2  
PROJECT GUIDANCE  
DOCUMENTS

PMC  
QUALITY ASSURANCE  
PROGRAM  
DOE/OR/21548-333

ENVIRONMENTAL  
QUALITY ASSURANCE  
PROJECT PLAN  
DOE/OR/21548-352

PMC  
PROJECT  
MANAGEMENT PLAN  
DOE/OR/21548-048

LEVEL 3  
DEPARTMENT PLANS

COMPLIANCE  
DEPARTMENT  
PLAN  
DOE/OR/21548-331

LEVEL 4  
OPERATIONS PLANS

SAMPLE MANAGEMENT  
GUIDE  
DOE/OR/21548-499

LEVEL 5  
PROCEDURES AND  
INSTRUCTIONS

LEVEL 6  
REPORTS AND  
PERFORMANCE INDICATORS

SAMPLING PLAN FOR THE  
FEASIBILITY STUDY FOR THE  
QUARRY RESIDUALS OPERABLE  
UNIT AT THE WELDON SPRING  
SITE, WELDON SPRING, MO

DOCUMENT NUMBER DOE/OR/21548-699	DATE 6/25/97
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