DEPARTMENT OF THE ARMY



CORPS OF ENGINEERS
HTRW CENTER OF EXPERTISE
12565 WEST CENTER ROAD
OMAHA, NEBRASKA 68144-3869

28 September 2000

Hazardous, Toxic and Radioactive Waste Center of Expertise

HISS (Hazelwood Interim Storage Site) On-Site Laboratory 8945 Latty Avenue Berkeley, MO 63134

Gentlemen:

This correspondence addresses the recent evaluation of HISS (Hazelwood Interim Storage Site) On-Site Laboratory of Berkeley, MO by the U.S. Army Corps of Engineers (USACE) for chemical analysis in support of the USACE Hazardous, Toxic and Radioactive Waste Program.

Your laboratory is now validated for the parameters listed below:

METHOD (SOP) PAR	AMETERS		MATRIX ⁽¹⁾
ML-003	Gan	ma Spectroscopy		Water ⁽²⁾
ML-003	Gan	ma Spectroscopy		Solids ⁽²⁾
ML-004	Alp	ha Spectroscopy		NA
ML-005	Isc	topic Thorium		Water ⁽²⁾
ML-005	Isc	topic Thorium		Solids ⁽²⁾
ML-006	Rad	lium-226		Water ⁽²⁾
ML-006	Rad	lium-226		Solids ⁽²⁾
ML-015	Isc	topic Uranium		Water ⁽²⁾
ML-015	Isc	topic Uranium		Solids ⁽²⁾
ML-018	Gro	ss Alpha and/or	Gross Beta	Water ⁽²⁾
	Act	ivity		

Remarks: 1) 'Solids' includes soils, sediments, and solid waste.

2) Approval for this parameter is based on review of SOPs only.

Enclosed for your information is a copy of the Laboratory Inspection and Evaluation Report. Your laboratory has responded to the deficiencies as noted in the report. No further responses are necessary.

Based on the successful analysis of the performance evaluation samples and the results of the laboratory inspection, your laboratory will be validated for sample analysis by the methods listed above. The period of validation is 24 months and expires on September 28, 2002.

The USACE reserves the right to conduct additional laboratory inspections or to suspend validation status for any or all of the listed parameters if deemed necessary. It should be noted that your laboratory may not subcontract USACE analytical work to any other laboratory location without the approval of this office. This laboratory validation does not guarantee the delivery of any analytical samples from a USACE Contracting Officer Representative.

Any questions or comments can be directed to Dr. Jan W. Dunker at (402) 697-2566. General questions regarding laboratory validation may be directed to the Laboratory Validation Coordinator at (402) 697-2574.

Sincerely,

Marcia C. Davies, Ph.D.
Director, USACE Hazardous,
Toxic and Radioactive Waste

Center of Expertise

CENWO-HX-C (200)

28 September 2000

MEMORANDUM THRU

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FOR FILES (CENWO-HX-C)

SUBJECT: Laboratory Inspection and Evaluation Report - HISS (Hazelwood Interim Storage Site) On-Site Laboratory of Berkeley, MO

1. General:

- a. Date of inspection: 02 03 February 2000
- b. Contract for which the laboratory will be used: Saint Louis District FUSRAP projects
- c. Description of contract: Radiochemical analysis of water and soil samples for:
 - Gamma Spectroscopy
 - Alpha Spectroscopy
 - Isotopic Thorium
 - Isotopic Uranium
 - Gross Alpha / Gross Beta in water

d. General information of laboratory inspected:

Business name: HISS (Hazelwood Interim Storage

Site) On-Site Laboratory

Street Address: 8945 Latty Avenue City and State: Berkeley, MO 63134

Phone: 314-260-3902

Fax: 314-260-3903

How long in business: 3 years

Number employed: 6

Services Offered: Radiochemical analyses

SUBJECT: Laboratory Inspection and Evaluation Report - HISS (Hazelwood Interim Storage Site) On-Site Laboratory of Berkeley, MO

Additional Information: The laboratory is a medium-sized on-site laboratory, and has the following major instruments:

- one (1) gamma spectrometer system
- one (1) gas flow proportional counters
- one (2) alpha spectrometer system
- one (1) Kinetic Phosphorescence Analyzer (KPA)

The laboratory does not have a Laboratory Information Management System.

The laboratory does not hold certificates and licenses for analytical work in any state. The laboratory participates in the following performance evaluation sample program: USDOE Mixed Analyte Performance Evaluation Program (MAPEP)

- Summary of Inspection/Evaluation Results:
- a. HISS (Hazelwood Interim Storage Site) On-Site Laboratory's QA/QC policy is consistent with USACE and EPA policy and guidance.
- b. HISS (Hazelwood Interim Storage Site) On-Site Laboratory is a medium-sized on-site laboratory that has adequate personnel and instrumentation to support USACE projects.
- c. Based on the information gathered during the current onsite inspection, HISS (Hazelwood Interim Storage Site) On-Site Laboratory is considered to be qualified to perform chemical analyses for the U. S. Army Corps of Engineers.
- d. HISS (Hazelwood Interim Storage Site) On-Site Laboratory was inspected by Dr. Jan Dunker, CENWO-HX-C, of the U.S. Army Corps of Engineers (USACE) Hazardous, Toxic, and Radioactive Waste Center of Expertise (HTRW CX). Detailed inspection results are addressed below.

SUBJECT: Laboratory Inspection and Evaluation Report - HISS (Hazelwood Interim Storage Site) On-Site Laboratory of Berkeley, MO

3. Interviews:

a. The following persons were present during the Entrance Interview:

Brad Wilson HISS (Hazelwood Interim

Storage Site) On-Site

Laboratory, SEC

Brian Sparks HISS (Hazelwood Interim

Storage Site) On-Site

Laboratory, Manager

Amy Gibson HISS (Hazelwood Interim

Storage Site) On-Site

Laboratory, SAIC

Alice Thompson HISS (Hazelwood Interim

Storage Site) On-Site

Laboratory, SEC

Jan Dunker Chemist, CENWO-HX-C Ron Frerker Chemist, CEMVS-ED-HQ

The general topics discussed during the entrance interview were: (1) the USACE validation procedure and QA program and (2) the Saint Louis District FUSRAP projects.

b. At the conclusion of the inspection, an Exit Interview was held with the following persons:

Brian Sparks HISS (Hazelwood Interim

Storage Site) On-Site

Laboratory, Manager Victor Samargian HISS (Hazelwood International HISS (HIS) HISP (HIS) (

HISS (Hazelwood Interim Storage Site) On-Site

Laboratory, SAIC

Jan Dunker Chemist, CENWO-HX-C Ron Frerker Chemist, CEMVS-ED-HQ

Deficiencies noted during the inspection, recommendations and required corrective actions to be taken were discussed. The laboratory was requested to respond with corrective actions taken/proposed, with supporting documentation, within ten (10) working days after receipt of the inspection findings.

SUBJECT: Laboratory Inspection and Evaluation Report - HISS (Hazelwood Interim Storage Site) On-Site Laboratory of Berkeley, MO

4. Conclusions:

a. SOP evaluation - The following parameters were evaluated:

SOP Number	SOP Title
ML-001	Sample Preparation
ML-003	Operation of the Gamma Spectroscopy System
ML-004	Operation of the Alpha Spectroscopy System
ML-005	Isotopic Determination of Thorium (Fusion)
ML-006	Determination of Radium-226 (Fusion)
ML-007	Balance Calibration Check
ML-008	Glassware Washing and Storage
ML-009	Waste Disposal
ML-010	Segregation of Out-of-Tolerance Instruments
ML-011	Use and Maintenance of Mechanical Pipette
ML-012	Deionized (DI) Water System
ML-013	Samples Management
ML-014	Data package Assembly, Transmittal and
	Generation of Electronic Deliverable
ML-015	Isotopic Determination of Uranium (Fusion)
ML-016	Determination of Self Adsorption Factors
ML-017	Radioactive Reference Standard Solutions &
	Records
ML-018	Determination of Gross Alpha and/or Gross Beta
	Activity
ML-019	Operation of the Gas Flow Proportional Gross
	Alpha / Beta Counting System
ML-020	Measurement of Settable Solids

b. Laboratory Quality Management Plan (LQMP): The laboratory submitted one document entitled "Science Applications International Corporation Quality Assurance Program (QAP) and Quality Assurance Administrative Procedures (QAAPs)" dated 30 April 1999 and another document entitled "Science Application International Corporation FUSRAP St Louis Laboratory Quality Assurance Plan and Laboratory Procedures Manual" dated 31 March 1999 for review. All laboratory QA/QC generally agrees with USACE and EPA's policy and guidance.

SUBJECT: Laboratory Inspection and Evaluation Report - HISS (Hazelwood Interim Storage Site) On-Site Laboratory of Berkeley, MO

- c. Deficiencies (D)/Recommendations (R)/Observations (O): Several deficiencies or concerns that could adversely affect HISS (Hazelwood Interim Storage Site) On-Site Laboratory's ability to conduct the required chemical analyses were noted. See the enclosed Report of Findings.
 - d. Laboratory Concerns/Questions: None were raised.
- 5. Action Items for the Laboratory: No specific action items remain.

JAN W. DUNKER, Ph.D.

Chemist, Chemical Data Quality

Management Branch

Enclosure

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U.S. Army Corps of Engineers HTRW Laboratory Inspection Findings

Lab Name / Location:	HISS On-Site Laboratory, Hazelwood, MO
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On-Site Inspection Findings:

Deficiencies/Recommendations/Observations:

A. General

- 1. (D) The laboratory does not assign unique IDs to method blanks, LCSs, and replicates. The sample ID for the method blank is "Blank"; for the LCS is "Control Spike", and for the replicate is "repl". (Observed on bench worksheets for Gross Alpha Beta; KPA Total Uranium, Isotopic radium, thorium and uranium, and in the run logs for gamma spectroscopy and alpha spectroscopy.) Unique IDs should be assigned to all QC samples.
- 2. (R) The laboratory should consider incorporating a data review checklist to document the peer review. See the USACE Shell for Analytical Chemistry Requirements; Section 13.2.1 Level 1 Analyst Review.

B. Sample Receipt

1. (O) Samples are logged into an Excel spreadsheet. This spreadsheet is printed, but the laboratory does not have a bound sample receipt logbook. The information from the Excel spreadsheet is transferred to a Microsoft Access database.

C. Sample Preparation

- 1. (R) It was stated that soil samples usually arrive in paint cans which are weighed and then dried overnight. The SOP states: "Weigh and record the tare weight of a drying pan, and place sample in pan. ...Place sample in drying oven until dry". The SOP should be changed to reflect the laboratory's actual practice.
- 2. (R) It was stated that the usual method of grinding/homogenizing soil samples is to place the sealed paint can (which contains the sample) on a paint can shaker. Other procedures are used if necessary. The SOP states: "homogenize the sample using the Bico pulverizer, Grinder, Paint can shaker, or mortar and pestle." The SOP should be changed to reflect the laboratory's actual practice. Note the SOP should describe the normal route of sample preparation along with the logic which will be used to select an alternate procedure.
- 3. (R) The balance used in the sample preparation area should have the calibration check performed over the range of use. The balance is currently checked at 100 g, 500 g, and 1000 g. It was stated that the wet weight of samples can range up to 1200 g.

- 4. (R) It was stated that a mortar and pestle is used if the soil sample is not ground fine enough in the paint can shaker. The SOP should describe how it is determined if this additional grinding is to be performed.
- 5. (R) A 50 g aliquot of water is used for sample preparation (observed for isotopic thorium analysis). Results are reported on a volumetric basis (pCi/L), with the assumption that the density of the aqueous samples is exactly 1.00 g/mL. Normally a laboratory should verify the correctness of this assumption on a sample-specific basis. However, since the aqueous samples processed by this laboratory are from limited and well-known sources, this is probably not necessary. However, the laboratory must demonstrate that this assumption is valid, and should confirm the validity of the assumption on a periodic basis (i.e., once per quarter).
- 6. (O) The 50 g aliquot of water samples is initially weighed in a centrifuge tube. The aliquot is poured from the centrifuge tube into to a platinum crucible, and then evaporated to dryness. It was stated that the aliquot was not rinsed from the centrifuge tube into the crucible. The question then arises as to whether this is quantitative transfer from the centrifuge tube into the crucible.
- 7. (D) The reagents used in sample preparation (i.e. fluoride fusion flux, sulfuric acid) do not have unique IDs, and are not recorded on the bench worksheets.
- 8. (R) The fluoride fusion flux was stated as being prepared in a mortar and pestle. The SOP states: "Blend thoroughly, using jar mill, mortar and pestle or similar method" The SOP should be changed to reflect the laboratory's actual practice.
- 9. (R) The meter on the Barnstead DI water purification system has not had a calibration check.
- 10. (R) The SOP for gross alpha and/or gross beta (ML-018) does not describe how the aqueous samples are transferred into centrifuge tubes three 10-mL aliquots measured using a Eppendorf-type pipette to transfer from the 1 L bottle to the centrifuge tubes. Note that the SOP also states that the aliquot is transferred into a beaker. The SOP should be changed to reflect the laboratory's actual practice.
- 11. (R) The SOP for gross alpha and/or gross beta (ML-018) should state what sensitivity is needed for determining the mass of the residue remaining after the aqueous sample has been evaporated to dryness. (i.e. Determine the mass of the residue to the nearest 0.1 mg)
- 12. (R) For gross alpha by the coprecipitation method (SM 7110C), a 250 g aliquot of water is used for sample preparation. Results are reported on a volumetric basis (pCi/L), with the assumption that the density of the aqueous samples is exactly 1.00 g/mL. Normally a laboratory should verify the correctness of this assumption on a sample-specific basis. However, since the aqueous samples processed by this laboratory are from limited and well-known sources, this is probably not necessary. However, the laboratory must demonstrate that this assumption is valid, and should confirm the validity of the assumption on a periodic basis (i.e., once per quarter).

- 13. (R) The SOP for gross alpha by the coprecipitation method (SM 7110C) indicates that a "detergent" is used. The SOP should be changed to reflect the actual reagent used by the laboratory (NaOH solution).
- 14. (R) The SOP for gross alpha by the coprecipitation method (SM 7110C) indicates that all samples are filtered. During the inspection it was stated that clear aqueous samples are not filtered. The SOP should be changed to reflect the laboratory's actual practice.
- 15. (R) The SOP for gross alpha by the coprecipitation method (SM 7110C) should indicate how the method blank and LCS are prepared.
- 16. (R) The SOP for radiochemical standards preparation (ML-017) indicates that standards are prepared using volumetric glassware. During the inspection it was stated that these standards are prepared gravimetrically. The SOP should be changed to reflect the laboratory's actual practice.
- 17. (O) The standards log is individual sheets in a three-ring binder.
- 18. (R) The standards preparation sheets (and also the bench worksheets) should have reference to the SOP (SOP number and version) which governs their preparation.
- 19. (R) The sample preparation bench worksheets should have entries for the volume of spiking solutions added.

D. Instrumental Analysis (Gamma Spectroscopy, Alpha Spectroscopy, Proportional Counter, KPA)

- 1. (R) The maintenance log for the alpha spectrometer had some entries that were not initialed. Also some pages were blank (left hand pages). Blank pages should be Z'd out, dated, and initialed.
- 2. (R) The maintenance log for the proportional counter had some pages which were blank (left hand pages). Blank pages should be Z'd out, dated, and initialed.
- 3. (R) The maintenance log for the KPA had some pages which were blank (left hand pages). Blank pages should be Z'd out, dated, and initialed. Also the maintenance log should have an entry for the date placed in service.
- 4. (R) In gamma spectroscopy an empty detector chamber is counted for the method blank.

 A more appropriate method blank should be used DI water for aqueous samples;
 a suitable matrix for soil samples. See the USACE Shell for Analytical Chemistry
 Requirements; Section 10.2.1 Method Blank.

E. Quality Assurance Function

1. (R) The laboratory should simplify the SOP review and approval process. As described during the inspection, a SOP change requires review and approval by the corporate QA function at SAIC.

Cataloging Form
{Technical/Project Managers fill in C through G, K through Q. RM completes other fields}

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