Mr. Steve Zappe, Project Leader  
Hazardous Waste Bureau  
New Mexico Environment Department  
2905 Rodeo Park Drive East, Bldg. 1  
Santa Fe, New Mexico 87505-6303

Subject: Transmittal of the Re-certification Audit Report for the Idaho National Engineering and Environmental Laboratory (INEEL), Analytical Laboratories Sampling and Analysis Program, Audit A-04-17

June 30, 2004

Dear Mr. Zappe:

This letter transmits the Audit Report for the INEEL analytical sampling and analysis program developed to comply with the WAP requirements for an independent analytical laboratory. The program was implemented to support the certified characterization programs of other generator sites. The subject audit was conducted at the INEEL near Idaho Falls, Idaho, on May 25-27, 2004. This audit report fulfills the requirements of Section II.C.2.c of the WIPP Hazardous Waste Facility Permit. The report contains the results of the certification audit and addresses the services that the INEEL analytical laboratories will perform, including, headspace gas analysis, generation level data verification and validation, solids sampling (coring), solids sample analysis, VE of solids, and supplying certified SUMMA® canisters for other generator site use.

I certify under penalty of law that this document and all enclosures were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fines and imprisonment for knowing violations.

Please contact the CBFO Quality Assurance Manager, Ava L. Holland, at (505) 234-7423 should you have any questions concerning this audit report.

Sincerely,

R. Paul Detwiler  
Acting Manager

Enclosure
cc: w/o enclosure
A. Holland, CBFO *ED
D. Miehls, CBFO *ED
M. Navarrete, CBFO *ED
K. Watson, CBFO *ED
R. Knerr, CBFO *ED
E. Feltcorn, EPA *ED
R. Joglekar, EPA *ED
M. Eagle, EPA *ED
E. Ziemianski, DOE-ID *ED
J. Wells, DOE-ID *ED
S. Sailer, INEEL *ED
C. Crowder, INEEL *ED
D. Winter, DNFSB *ED
L. Greene, WRES *ED
S. Lee, ANL-W *ED

cc: w/enclosure
S. Holmes, NMED *ED
C. Walker, Techlaw *ED
WTS Operating Record (MS486-06)
CBFO QA File
CBFO M&RC

*ED denotes electronic distribution
CBFO:QA/MPN/JS:04-1537:UFC 2300.00
U. S. DEPARTMENT OF ENERGY
CARLSBAD FIELD OFFICE

FINAL AUDIT REPORT

OF THE

IDAHO NATIONAL ENGINEERING AND
ENVIRONMENTAL LABORATORY (INEEL)

IDAHO FALLS, IDAHO

AUDIT NUMBER A-04-17

MAY 25-27, 2004

FINAL AUDIT REPORT OF THE INEEL
ANALYTICAL LABORATORIES SAMPLING AND ANALYSIS PROGRAM
IN
ACCORDANCE WITH THE HAZARDOUS WASTE FACILITY PERMIT

Prepared by: [Signature] Date: 6/29/2004
A. Earl Bradford
Audit Team Leader, CTAC

Approved by: [Signature] Date: 6/29/04
Ava L. Holland
Quality Assurance Manager
1.0 EXECUTIVE SUMMARY

The Carlsbad Field Office (CBFO) Audit A-04-17 was conducted to reevaluate the adequacy, implementation, and effectiveness of the technical and quality assurance (QA) activities related to the Idaho National Engineering and Environmental Laboratory (INEEL) analytical laboratory, which functions as an independent sampling and analysis laboratory. The INEEL analytical laboratory program provides sampling and analysis services to the generator sites in accordance with Waste Analysis Plan (WAP) requirements.

The INEEL analytical laboratory program is managed by the current INEEL management and operating (M&O) contractor Bechtel BWXT Idaho, LLC. The sampling of the homogeneous solids and visual examination (VE) of solids will be conducted at the Argonne National Laboratory – West (ANL-W) facilities at the INEEL. The analytical laboratories will manage the sampling and analysis efforts and the ANL-W activities will be conducted under the analytical laboratories program. The generator sites using the services of the INEEL retain responsibility for waste certification under their respective certification programs.

The INEEL activities evaluated included: headspace gas analysis, sampling of homogeneous solid waste at ANL-W, analysis of homogeneous solid waste, generation-level data verification and validation (V&V), SUMMA® sample canister preparation, and the VE of homogeneous solid waste at ANL-W.

The audit was conducted at the INEEL facilities May 25 through May 27, 2004. The audit team concluded that, overall, the INEEL technical and QA procedures remain adequate relative to the flow-down of requirements from the CBFO Quality Assurance Program Document (QAPD); the Waste Isolation Pilot Plant (WIPP) Hazardous Waste Facility Permit (HWFP); and the Contact-Handled Transuranic Waste Acceptance Criteria for the Waste Isolation Pilot Plant (WAC). The audit team also concluded that the defined QA program is still satisfactorily implemented in accordance with the CBFO contract and statement of work, as well as the INEEL implementing procedures. Additionally, the audit team determined that the INEEL technical areas evaluated remain adequate, satisfactorily implemented, and effective.

The audit team did not identify any concerns, document any observations, or make any recommendations for improvement of the INEEL processes during the audit.

2.0 SCOPE AND PURPOSE

2.1 Scope

The audit team reevaluated the adequacy, implementation, and effectiveness of technical and QA processes related to the INEEL analytical laboratories activities associated with headspace gas (HSG) analysis, solids sampling and analysis, SUMMA® canister preparation and certification for use by other generator sites, VE of homogeneous solid wastes, and generation-level data verification and validation (V&V).
The following INEEL program elements were evaluated in accordance with the WIPP HWFP:

**Quality:**

- Organization and Quality Assurance Program (Program Interfaces and Statement of Work)
- Personnel Qualification and Training
- Nonconformance's and Corrective Action
- Documents and Records
- Work Processes
- Procurement
- Control of Measuring and Test Equipment used for Characterization
- Audit/Assessments
- Sample Control
- Software QA

**Technical:**

- Solids analysis (volatile organic compounds [VOCs], non-halogenated volatile organic compounds [NHVOCs], semi-volatile organic compounds [SVOCs] and metals)
- HSG analysis
- Solids coring
- VE of homogeneous solids
- Generation-level data V&V

The evaluation of INEEL analytical laboratories waste analysis activities and documents was based on current revisions of the following documents:

- **Quality Assurance Program Document (QAPD),** DOE-CBFO-94-1012

- **Hazardous Waste Facility Permit Waste Isolation Pilot Plant EPA No. NM4890139088-TSDF,** by the New Mexico Environment Department, dated October 27, 1999, including all applicable modifications

- **Hazardous Waste Facility Permit Waste Isolation Pilot Plant EPA No. NM4890139088-TSDF,** by the New Mexico Environment Department, dated October 27, 1999, including all applicable modifications

- **Contact-Handled Transuranic Waste Acceptance Criteria for the Waste Isolation Pilot Plant (WAC),** DOE/WIPP-02-3122
• Related INEEL technical and quality assurance implementing procedures and Argonne National Laboratory – West (ANL-W) technical implementing procedures (see Attachment 3)

2.2 Purpose

Audit A-04-17 was conducted to reassess whether the INEEL analytical laboratory services related to headspace analysis, sampling and analysis of homogeneous solids, preparation and certification of SUMMA® canisters for use by other generator sites, and the performance of VE of homogeneous solid wastes, and generation-level data verification and validation (V&V) still complied with the WIPP HWFP requirements.

3.0 AUDIT TEAM AND OBSERVERS

AUDITORS/TECHNICAL SPECIALISTS

Earl Bradford
Steve Calvert
Tammy Bowden
Norman Frank
Porf Martinez
BJ Verret
Wayne Ledford
Audit Team Leader, CBFO Technical Assistance Contractor (CTAC)
Auditor, CTAC
Auditor, CTAC
Auditor, CTAC
Auditor, CTAC
Technical Specialist, CTAC
Technical Specialist, CTAC

OBSERVERS

Steve Holmes
Dorothy Gill
Brian English
New Mexico Environment Department (NMED)
NMED
Idaho Department of Environmental Quality (DEQ)

4.0 AUDIT PARTICIPANTS

A pre-audit conference was held at ANL-W in Building 714, room 107 on May 25, 2004. During the audit, daily meetings were held with INEEL management and staff to discuss audit progress. The audit was concluded with a post-audit conference held at the INEEL on May 27, 2004. INEEL individuals involved in the audit process are identified in Attachment 1.

5.0 SUMMARY OF AUDIT RESULTS

5.1 Program Adequacy and Implementation

The audit team concluded that the INEEL technical and QA procedures adequately reflect the requirements of the CBFO QAPD and the HWFP. The audit team also concluded that
the defined QA program remains satisfactorily implemented in accordance with the INEEL QA Plan and implementing procedures. The INEEL technical processes evaluated by the audit team remain satisfactorily implemented and effective.

5.2 Technical Activities

Each technical area audited is discussed in detail in the following sections. The method used to select objective evidence is discussed, the objective evidence used to assess compliance with the HWFP is briefly cited (and shown in detail on the appropriate B6 checklist), and the result of the assessment is provided. If B6 checklists questions cannot be satisfactorily answered during an audit, an audit concern must be generated. The audit team did not identify any concerns, document any observations, or make any recommendations for improvement of the INEEL processes during the audit.

5.2.1 Table B6-1 WAP Checklist

The B6-1 WAP checklist addresses program requirements from an overall management perspective. It documents the verification that the waste characterization activities and requirements as defined in the WAP are planned and implemented by using controlled procedures. This audit was performed to reassess the INEEL analytical laboratories’ ability to provide adequate analytical services for user sites and therefore ensure that user sites will have credible laboratory analysis results to finalize their TRU waste characterization and certification activities. The INEEL analytical laboratory program provides headspace gas analysis services, core sampling of organic and inorganic homogeneous solids waste at ANL-W, analysis of organic and inorganic homogeneous solids samples, VE of homogeneous solid wastes at ANL-W, and certified SUMMA® canisters for use by other generator sites. Objective evidence to reevaluate the implementation of the associated sampling and analysis, as well as the VE activities, was selected and reviewed. Batch data reports (BDRs), sampling records, and training documentation for INEEL laboratory personnel were evaluated during the audit. The audit included direct observation of actual analysis activities, e.g., gas analysis and solids analysis, as well as evaluation of records associated with solids coring and VE of solids. Each characterization or analysis process involves:

- Collecting raw data
  - Collecting quality assurance/quality control (QA/QC) samples or information
  - Reducing the data to a useable format, including a standard report
  - Review of the generated data

The data generated for each characterization/analysis technique was reviewed to ensure that all applicable requirements were captured in the site operating procedures. The material in this section is also addressed in more detail in the following sections, which provide the specific procedures audited and the objective evidence reviewed.
During the audit, the INEEL analytical laboratories demonstrated compliance with the HWFP by performing analysis and providing documentation for solids coring and VE activities. The INEEL analytical laboratories provided the following:

- Headspace gas analytical BDRs ECL04013M and ECL04015G (containing gas analysis information)
- Analytical BDRs ALD0410V, ALD0413N, ALD03020S and ALD04017M (containing VOCs, NHVOCs, SVOCs and metals analysis results)
  - VE BDRs WCV-RFD67011, WCV-LAS831581 and WCV-RFD68254.
- Solids sampling BDRs WCS-0306, WCS-0307, WCS-0308, WCS-0309 and WCS-0310.

Copies of these BDRs are included in Attachment 2. The audit team concluded that the BDRs reviewed and the sampling processes observed were acceptable.

5.2.2 Table B6-2 Solids and Soils/Gravel Sampling Checklist

This audit was performed to reassess the ability of the INEEL analytical laboratories program to ensure the proper management and performance of solids coring at ANL-W and solids sample analysis in accordance with WAP requirements.

The audit team reevaluated the core sampling capabilities performed at ANL-W. There were no sampling activities ongoing during the audit, so sampling was not observed. The audit team examined the coring area, coring equipment, and the sample storage refrigerator. Based on interviews with the sampling team at ANL-W and review of BDRs, no significant changes had occurred in the coring and sampling processes since the previous certification audit. The audit team reviewed five sampling BDRs and the documentation for the collection and analysis of equipment blanks. The audit team evaluated equipment storage and determined the storage practices to be acceptable. The overall solids sampling procedures were determined to be adequate. The audit team concluded that the solids sampling process is still satisfactorily implemented and effective.

The process for sample handling was evaluated at both the sampling facility and the laboratory facility. The evaluation verified that handling of samples was performed in accordance with procedures. The samples are stored correctly after collection and receipt and are correctly tracked as they move through the collection and analysis processes. It was concluded that the sample handling procedures are adequate and satisfactorily implemented and the process is effective. The chain-of-custody initiation process at the sample facility was examined for samples taken and being sent to the laboratory facility. The overall chain-of-custody program and procedures were determined to be adequate and satisfactorily implemented and the process remains effective.
Solid sample preparation and analysis for metals (mercury and ICP elements), VOCs, NHVOCs, and SVOCs were evaluated. The audit team conducted document and record reviews, and interviews with analysts, and reviewed selected analysis BDRs. The audit team determined that the procedures used to control the processes were adequate in meeting the requirements of the WAP. All laboratory areas inspected were well organized, and the analysts and supervisors were knowledgeable with regard to their analytical duties. BDRs ALD0410V, ALD04013N, ALD03020S and ALD04017M (analytical batches) were reviewed to evaluate analysis results against HWFP requirements.

The audit team did not identify any deficiencies and concluded that the solids sampling and analysis processes were still adequate, satisfactorily implemented, and effective.

5.2.3 Table B6-4 Headspace Gas Checklist

This audit was performed to reassess the ability of the INEEL's analytical laboratories program to provide headspace gas analysis services and provide certified SUMMA® canisters for use by other generator sites. Headspace gas analysis operations at the INEEL were observed during actual analysis of samples. The following procedures were evaluated:

- ACLP 4.10, Determination of Method Detection Limits for Gas Analysis
- ACLP-4.25, Sample Receiving, Custody and Storage
- ACLP-4.40, Summa Canister Cleaning
- ACLP 4.45, Gas Transfer Manifold Systems
- ACMM-9910, Analysis of Gas Samples for VOCs by GC/FID
- ACMM-9925, Analysis of Gas Samples for Hydrogen and Methane by Micro GC/TCD
- ACMM-9930, Analysis of Gas Samples for VOCs by GC/MS

The audit team evaluated the HSG analysis processes including sample receipt and storage, standard and sample analysis (initial and continuing calibrations, QC analyses, minimum detection limit [MDL] studies and performance and assessment [P&A] studies), data review at the data-generation level, and sample disposal.

The audit team performed a walkthrough of the laboratory to verify acceptable operations and the physical presence of required instrumentation and equipment. The audit team reviewed BDRs ECL04013M (containing analytical data for all VOCs except methanol) and ECL04015G (for methanol). The audit team verified calibrated temperature and pressure monitoring instrumentation for sample receipt, storage, and analysis. Documentation associated with these activities (e.g., calibration records, maintenance logbooks, and instrument logbooks) was reviewed to ensure that laboratory operations met the QA requirements specified in the HWFP. In addition, the audit team verified that the INEEL HSG processes had successfully completed the HSG Performance Demonstration Plan (PDP) Cycle 18A. Copies of the applicable documentation reviewed were contained in the BDRs and are included in Attachment 2.
The audit team completed Table B6-4, the headspace gas checklist, while assessing implementation of the procedures. The audit team verified that generation-level data reviews were conducted to ensure that analysis results met program requirements and that the reviews were controlled in accordance with MCP-2008, Analytical Data Recording, Review and Reporting.

The audit team evaluated the processes used to clean, leak check, and certify SUMMA® canisters and verified that the processes adequately met HWFP requirements.

The audit team did not identify any deficiencies and concluded that the headspace gas analysis process and SUMMA® canister certification processes used by the INEEL analytical laboratories remain adequate, satisfactorily implemented, and effective per HWFP requirements.

5.2.4 B6-6 VE Checklist

This audit was performed to reassess the ability of the INEEL's analytical laboratory program to perform VE of homogeneous solid wastes. The VE of solids is performed at ANL-W under the umbrella of the analytical laboratory program. The laboratory program provides the VE services for other generator sites. VE activities are recorded on audio/videotape and documented on standard forms.

The audit team evaluated the solids VE program at the ANL-W. The VE procedures are HFEF-OI-6890, TWCP Visual Examination, and NT-AP-09, TWCP Visual Examination Expert Functions and Process. The procedures were found to be adequate in meeting the HWFP requirements. There was no visual examination ongoing at ANL-W during the audit, so visual examination was not directly observed. The audit team reviewed VE BDRs WCV-RFD67011, WCV-LAS831581 and WCV-RFD68254, along with the associated videotape records. The audit team reviewed the training records for the operators and the VE experts and found them acceptable. The BDRs and training records are included in Attachment 2.

The audit team did not identify any deficiencies and determined that the solids VE processes continue to be adequate, satisfactorily implemented, and effective.

6.0 SUMMARY OF DEFICIENCIES

The audit team did not identify any conditions adverse to quality during the audit. The audit team did not identify any isolated conditions that could have been corrected during the audit. The audit team did not observe any conditions that could result in future deficiencies, nor did it make any recommendations for improvement of the laboratory processes.
7.0 LIST OF ATTACHMENTS

Attachment 1: Personnel Contacted During the Audit
Attachment 2: Objective Evidence
Attachment 3: Audited INEEL Documents / Implementing Procedures
<table>
<thead>
<tr>
<th>NAME</th>
<th>TITLE/ORG</th>
<th>PREAUDIT MEETING</th>
<th>CONTACTED DURING AUDIT</th>
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