Department of Energy
Carlsbad Field Office
P. O. Box 3090
Carlsbad, New Mexico 88221
July 6, 2004

Dr. Amrit S. Boparai
Analytical Chemistry Laboratory
Argonne National Laboratory-East
9700 South Cass Avenue
Argonne, IL 60439-4837

Subject: Transmittal of Audit Report for Audit A-04-18

Dear Dr. Boparai:

The Carlsbad Field Office (CBFO) performed Audit A-04-18 of Argonne National Laboratory-East, on June 8 and 9, 2004. The audit team concluded that the overall status of the ANL-E Quality Assurance Program in its role as the sample preparation contractor for the Headspace Gas Performance Demonstration Program is, with the exception of the Corrective Action Report noted in the report, adequate, satisfactorily implemented, and effective. The details of the Audit as well as conclusions are within the enclosed audit report.

If you have any questions or comments, please contact me at (505) 234-7442.

Sincerely,

M. Lea Chism
Quality Assurance Specialist

Enclosure

cc: w/enclosure
A. Holland, CBFO
M. Navarrete, CBFO
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C. Watkins, CTAC
B. J. Verret, CTAC
A. Pangle, CTAC
L. Greene, WRES
WIPP Operating Records, WTS (MS486-06)
CBFO QA File
CBFO M&RC

*ED denotes electronic distribution
CBFO:QA:MLC:GS:04-1538:UFC 2300.00
SAMPLE PREPARATION SUPPORT FOR HEADSPACE GAS PERFORMANCE DEMONSTRATION PROGRAM

Prepared by: Norman C. Frank  
Audit Team Leader

Approved for Issue by: Ava L. Holland  
CBFO Quality Assurance Manager

Date: 6/21/04  
7/8/04
EXECUTIVE SUMMARY

CBFO Audit A-04-18 was conducted to evaluate the adequacy, implementation, and effectiveness of the Argonne National Laboratory (ANL) quality assurance and technical support activities for the preparation of samples needed to support the WIPP Headspace Gas (HSG) Performance Demonstration Program (PDP). The procedures covering document control, quality assurance records, procurement, training and qualification, laboratory notebooks, analytical sample preparation, PDP sample verification analyses, gas standards preparation, preparation and distribution of PDP samples, and canister cleaning activities were assessed for compliance with the CBFO Quality Assurance Program Document (CAO-94-1012) Revision 5, and the PDP Plan for Analysis of Simulated Headspace Gas (DOE/CAO-95-1076), Revision 4.

The audit was conducted at the ANL Chemical Technology Division (CMT) Analytical Chemistry Laboratory (ACL) in Argonne, Illinois on June 8-9, 2004. The audit team determined that the activities evaluated relating to quality assurance were adequate, satisfactorily implemented, and effective. The audit team determined that the HSG PDP implementing procedures and activities for sample preparation, verification and distribution remain adequate, satisfactorily implemented, and effective. The audit team identified four concerns, two of which were corrected during the audit (CDA) since they were isolated in nature and required only remedial corrective actions. One corrective action report (CAR) was issued concerning the review of Logbooks / Notebooks. One observation was issued as a result of this audit. There were no recommendations for management consideration. The CARs, CDAs, Observations, and Recommendations are described in Section 6.0 of this report.

2.0 SCOPE

CBFO Audit A-04-18 was conducted to evaluate the adequacy, implementation, and effectiveness of procedures and activities used by the ANL CMT ACL for the preparation of samples for the HSG PDP. The procedures covering the processes used in this role, and those for the procurement of the gas standards from which the PDP samples are prepared, were included.

AUDIT TEAM

CBFO AUDIT TEAM

Norman C. Frank  
Cliff Watkins  
B. J. Verret  
Audit Team Leader, CTAC  
Technical Specialist, CTAC  
Technical Specialist, CTAC

AUDIT PARTICIPANTS

A list of personnel contacted during the course of the audit is provided below:
### PERSONNEL CONTACTED DURING THE AUDIT

<table>
<thead>
<tr>
<th>NAME</th>
<th>TITLE/ORG</th>
<th>PRE AUDIT MEETING</th>
<th>CONTACTED DURING AUDIT</th>
<th>POST AUDIT MEETING</th>
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</thead>
<tbody>
<tr>
<td>Boparai, Amrit</td>
<td>Manager ACL/ANL</td>
<td>X</td>
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<tr>
<td>Graziano, Diane</td>
<td>Deputy Division Director CMT/ACL</td>
<td>X</td>
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<td>Kalensky, Michael</td>
<td>Technical Lead/ACL</td>
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<tr>
<td>Riel, Roberta</td>
<td>QA Representative, CMT/ACL</td>
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<tr>
<td>Streets, Elane</td>
<td>QA/QC Coordinator</td>
<td>X</td>
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<tr>
<td>Lopykinski, Susan</td>
<td>QA/QC Coordinator</td>
<td>X</td>
<td>X</td>
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### 5.0 SUMMARY OF AUDIT RESULTS

#### 5.1 Audit Activities

Details of audit activities, along with the specific objective evidence reviewed and the results of the reviews are contained within the audit checklists. The checklists are maintained as QA records.

The audit team verified ANL CMT ACL compliance with the PDP Plan for Analysis of Simulated Headspace Gas, DOE/CAO-95-1076, Revision 4. The audit team determined that the ANL CMT ACL Work Plan for HSG PDP Sample Preparation, Verification, and Distribution, Revision 8, adequately defined the current organizational structure of the project. As a result, the audit team concluded that the related QA program requirements, procedures, and activities were adequate, satisfactorily implemented, and effective. In addition, the audit team evaluated the HSG PDP sample preparation, verification analyses, canister cleaning, gas standards preparation, laboratory notebooks, quality assurance records, employee training, and the procurement of gas standards for the HSG PDP and concluded that these activities were adequate, satisfactorily implemented, and effective.
Organization

The audit team evaluated the adequacy of documents depicting the ANL CMT ACL organization. The ANL CMT ACL Work Plan for Headspace Gas Performance Demonstration Program (PDP) Sample Preparation, Verification and Distribution and the "Analytical Chemistry Laboratory Organization Chart" dated 5/10/04 adequately depict the current organizational structure and define the responsibilities of the ANL personnel involved in PDP sample preparation efforts.

Work Plan Implementation

The ANL CMT ACL prepared a Work Plan titled Work Plan for Headspace Gas (HSG) Performance Demonstration Program (PDP) Sample Preparation, Verification and Distribution, Revision 8, to meet the applicable requirements of the CBFO Quality Assurance Program Document (CAO-94-1012), Revision 5, and the PDP Plan for Analysis of Simulated Headspace Gas (DOE/CAO-95-1076), Revision 4. Activities related to the implementation of the Work Plan were reviewed, including the standard operating procedures, procurement, training and qualification, and QA records. No concerns were identified. The Work Plan was determined to be adequate, satisfactorily implemented, and effective.

Document Control

The audit team reviewed the ANL CMT ACL procedures SOP: ACL-033, Document Control, and SOP: ACL-022, Personnel Signature and Initials Record. One concern was noted where the document on the ANL Intranet was not the most current version. The current version was immediately placed on the Intranet (CDA 1). Controlled and current versions of ACL QA documents are accessible via the ACL Internet Home Page to authorized ANL-E employees. Superseded hardcopy and electronic versions are archived and made inaccessible for general use. The audit team also verified that the personnel signature and initial list existed and had been maintained current. This area was determined to be adequate, satisfactorily implemented, and effective.

5.1.4 Quality Assurance Records

The audit team reviewed the ANL CMT ACL procedure SOP: ACL-228, Disposition of Project Specific Quality Assurance Records. The audit team verified that the QA records identified in the Attachment 2 of the Work Plan were maintained and controlled. QA records were filed according to the QA Records elements identified in the Work Plan in a two-hour fire rated UL-certified cabinet located in the project record coordinator area (Room E-127 of Building 205). A list of personnel who have access to this file is posted on the cabinet, and the cabinet is locked when not in use. The audit team also verified that corrections to records were made with single line-through, not obliterating the prior entry, and initialed and dated by the originator. This procedure was determined to be adequate, satisfactorily implemented, and effective.
Laboratory Notebooks

The audit team reviewed SOP: ACL-107, *Review of Scientific Notebooks*. The audit team examined the records maintained by the QA Coordinator and verified that all tracking required by the SOP is completed. Objective evidence was obtained showing the effective implementation of the tracking requirements in the procedure. Laboratory Notebooks 1415 and 1787 were reviewed to ensure that the other requirements of the SOP were being implemented. One concern, (CAR 04-29) relating to the review of Logbooks / Notebooks by the QA Coordinator and the Group Leader, was identified. A second concern (OBS #1) related to the signing of the laboratory pages. SOP ACL-107 was determined to be adequate, marginally implemented, and effective.

5.1.6 Analytical Sample Preparation

One of the activities conducted by the ANL CMT ACL is verification of HSG PDP samples after they have been prepared. This requires the ANL CMT ACL to prepare one set of PDP canisters and analyze them as though they were samples of unknown concentration. The procedure for preparation of gas sample aliquots for analysis is SOP: ACL-159, *Preparation of Samples for Determination of Headspace - Volatile Organic Compounds (VOCs) in SUMMA® Canisters*. Interviews with the analyst, and review of objective evidence including Laboratory Notebook 1787, demonstrated effective implementation of the procedure. This procedure was determined to be adequate, satisfactorily implemented, and effective.

PDP Sample Verification Analyses

As stated above, one of the activities conducted by the ANL CMT ACL is the verification of HSG PDP samples after they have been prepared. This requires the ANL CMT ACL to prepare one set of PDP canisters and analyze them as though they were samples of unknown concentration. The procedure for sample analysis is SOP: ACL-168, *Analytical Method for the Determination of Volatile Organic Compounds in SUMMA® Canisters using Gas Chromatography/Mass Spectrometry*. During the on-site audit calibration and calibration verification of the GC/MS analytical system was discussed with, and demonstrated by, the responsible analyst. Processing of QC samples and generation of method performance data were also investigated. In general, the analyst was well trained and experienced in analytical duties, and the equipment and laboratory were adequate for the work performed. Overall, this procedure was determined to be adequate, satisfactorily implemented, and effective.

Gas Standards Preparation

Procedure SOP: ACL-169, *Preparation of Volatile Organic Compound Calibration Standards for use with SUMMA® Canisters*, was reviewed, prior to the on-site audit, to assess technical adequacy. Audit of the procedure included discussion with the analyst
and demonstration of critical steps in the process. Overall, this procedure was determined to be adequate, satisfactorily implemented, and effective.

**Preparation and Distribution of PDP Samples**

The audit team reviewed the ANL CMT ACL procedure SOP: ACL-211, "Preparation and Distribution of Simulated Headspace Gas Samples for the WIPP Headspace Gas Performance Demonstration Program." One concern identified that a certificate from a gas vendor did not include the notation "Received by ANL". The Group Leader immediately corrected this (CDA #2) by making the appropriate notation. This procedure was determined to be adequate, satisfactorily implemented, and effective.

### 5.1.10 Canister Cleaning

The audit team reviewed the ANL CMT ACL procedure SOP: ACL-158, "SUMMA® Canister Cleaning." This procedure was determined to be adequate, satisfactorily implemented, and effective.

## 6.0 CORRECTIVE ACTIONS REPORTS, CORRECTED DURING THE AUDIT, OBSERVATIONS, AND RECOMMENDATIONS

### 6.1 Corrective Action Report

#### 6.1.2 CAR Initiated as a Result of CBFO Audit A-04-18:

The following CAR, initiated as a result of Audit A-04-18, has been transmitted to the ANL-E under separate cover. A brief description of the CAR is provided below.

#### 6.1.2.1 CBFO CAR 04-29

Logbooks/Notebooks are not being reviewed as required by procedure.
- The QA Coordinator has not reviewed any logbooks/notebooks for WIPP-related work
- There is no objective evidence that notebook reviews conducted by the Group Leader are scheduled and that this schedule and completion of reviews are documented with the QA Coordinator. There is objective evidence in each notebook that these reviews had been performed periodically.

**Deficiencies Corrected During the Audit (CDA)**

**CDA #1**

The ANL-E Intranet had Revision 6 to ACL-033 as the current revision. Revision 7 had been effective since 8/3/03. ACL-033 concerns document control. A check of the files showed no other documents affected. The correct revision was immediately placed on the ANL Intranet.
CDA #2

SOP ACL-211 requires all certificates received from gas vendors to contain the notation “Received by ANL”. All but one of the certificates contained this notation. The certificate received 12/18/03 for the internal standards and surrogate cylinder lacked the notation.

Observations

The audit team identified one condition that, if left uncorrected, could lead to a future condition adverse to quality. This condition is reported as an Observation, and is being provided to cognizant ANL-E management for consideration.

Observation #1

Notebooks 1029 and 1415 were signed and dated for each entry. The procedure, ANL-107, requires a signature at the bottom of the page. The reason for the signature at the bottom of the page is to cover entries that run more than one page in length. No entry to date has gone more than one page in length. Thus, the signature for each entry is acceptable. If an entry goes longer than one page in length, the bottom of the pages must be signed.

Recommendation

No recommendations are provided as a result of this audit.

7.0 ATTACHMENTS

Attachment 1: Procedures Reviewed During the Audit
<table>
<thead>
<tr>
<th>NUMBER</th>
<th>PROCEDURE NUMBER/REVISION</th>
<th>TITLE</th>
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<tbody>
<tr>
<td>1</td>
<td>SOP: ACL-022, R1</td>
<td>Personnel Signature and Initials Record</td>
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<td>2</td>
<td>SOP: ACL-033, R7</td>
<td>Document Control</td>
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<td>3</td>
<td>SOP: ACL-107, R7</td>
<td>Review of Scientific Notebooks</td>
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<td>4</td>
<td>SOP: ACL-158, R6</td>
<td>SUMMA® Canister Cleaning</td>
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<td>5</td>
<td>SOP: ACL-159, R5</td>
<td>Preparation of Samples for Determination of Headspace-Volatile Organic Compounds (VOCs) in SUMMA® Canisters</td>
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<tr>
<td>6</td>
<td>SOP: ACL-162, R1</td>
<td>Qualification and Training of Analytical Chemistry Laboratory Analysts</td>
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<td>8</td>
<td>SOP: ACL-169, R4</td>
<td>Preparation of Volatile Organic Compound Calibration Standards for use with SUMMA® Canisters</td>
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<td>9</td>
<td>SOP: ACL-211, R5</td>
<td>Preparation and Distribution of Simulated Headspace Gases Samples for the WIPP Performance Demonstration Program</td>
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<tr>
<td>10</td>
<td>SOP: ACL-228, R3</td>
<td>Disposition of Project-Specific Quality Assurance Records</td>
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<td>11</td>
<td>No Number, R8</td>
<td>Work Plan for Headspace Gas Performance Demonstration Program Sample Preparation, Verification and Distribution</td>
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<td>12</td>
<td>C0030-0211, R10</td>
<td>Analytical Chemistry Laboratory Quality Assurance Plan</td>
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<tr>
<td>13</td>
<td>C0030-0362, R6</td>
<td>Preparing and Processing Standard Operating Procedures</td>
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