

United States Government

Department of Energy

memorandum

Carlsbad Field Office
Carlsbad, New Mexico 88221

DATE: August 8, 2001

REPLY TO
ATTN OF: CBFO:QA:SAV:VW:01-1404:UFC:2300

SUBJECT: Surveillance (S-01-15) of the Argonne National Laboratory Sample Preparation Support for Headspace Gas Performance Demonstration Program



TO: Amrit Boparai, ANL-E

The Carlsbad Field Office (CBFO) conducted a surveillance of the Argonne National Laboratory-East (ANL-E) Sample Preparation Support for Headspace Gas Performance Demonstration Program on July 10-11, 2001. The surveillance team determined that the activities evaluated relating to quality assurance were adequate, satisfactorily implemented, and effective. The surveillance team also concluded that the ANL-E procedures covering quality assurance records, laboratory notebooks, analytical sample preparation, PDP sample verification analyses, gas standards preparation, preparation and distribution of PDP samples, and canister cleaning activities were adequate, satisfactorily implemented, and effective. The CBFO surveillance report is attached.

There was one CBFO Corrective Action Report (CAR-01-081) concerning the procedure for preparation of gas sample aliquots for analysis not being followed. This was provided under separate cover. One recommendation was provided for management consideration, and five conditions adverse to quality were corrected during the surveillance.

If you have any questions or comments concerning this report, please contact me at (505) 234-7423.

Lisa Chism
for Samuel A. Vega
Quality Assurance Manager

Attachment

cc: w/attachment
M. Brown, CBFO
L. Chism, CBFO
D. Winters, DNFSB
S. Monroe, EPA
M. Eagle, EPA
S. Zappe, NMED
B. Walker, EEG
M. Gerle, WTS
T. Bowden, CTAC

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**U.S. DEPARTMENT OF ENERGY
CARLSBAD FIELD OFFICE**

**SURVEILLANCE REPORT
OF THE
ARGONNE NATIONAL LABORATORY
CHEMICAL TECHNOLOGY DIVISION
(ANL-E)**

Chicago, Illinois

SURVEILLANCE NUMBER S-01-15

JULY 10 -11, 2001



**SAMPLE PREPARATION SUPPORT FOR HEADSPACE GAS
PERFORMANCE DEMONSTRATION PROGRAM**

Prepared by: Amelia I. Arceo Date: 7/26/01
Amelia I. Arceo
Surveillance Team Leader

Approved for Issue by: Lea Chism Date: 08/08/01
for Samuel A. Vega
CBFO Quality Assurance Manager

1.0 EXECUTIVE SUMMARY

CBFO Surveillance S-01-15 was conducted to evaluate the adequacy, implementation, and effectiveness of the Argonne National Laboratory (ANL-E) technical support activities for the preparation of samples needed to support the WIPP Headspace Gas (HSG) Performance Demonstration Program (PDP). The organizational structure and compliance with the procedures covering quality assurance records, laboratory notebooks, analytical sample preparation, PDP sample verification analyses, gas standards preparation, preparation and distribution of PDP samples, and canister cleaning activities were assessed. In addition, the organization's Quality Assurance Plan and Work Plan were reviewed to ensure compliance with requirements for employee training and the procurement of gas standards.

The surveillance was conducted at the Argonne National Laboratory (ANL) Chemical Technology Division (CMT) Analytical Chemistry Laboratory (ACL) laboratories and offices in Argonne, Illinois on July 10 - 11, 2001. The surveillance 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 were adequate, satisfactorily implemented and effective. The surveillance team identified six conditions adverse to quality, five of which were corrected during the surveillance (CDS) since they were isolated in nature and required only remedial corrective actions. One condition adverse to quality concerning the procedure for preparation of gas sample aliquots for analysis not being followed required the issuance of a Corrective Action Report (CAR). One recommendation was provided for management consideration. No observations were issued as a result of this surveillance. The CAR, CDSs, and Recommendation are described in Section 6.0 of this report.

2.0 SCOPE

CBFO Surveillance S-01-15 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.

3.0 SURVEILLANCE TEAM, INSPECTOR AND OBSERVER

Amy Arceo
Cliff Watkins

Surveillance Team Leader, CTAC
Technical Specialist, CTAC

4.0 SURVEILLANCE PARTICIPANTS

A list of personnel contacted during the course of the surveillance is provided as Attachment 1 of this report.

5.0 SUMMARY OF SURVEILLANCE RESULTS

5.1 Surveillance Activities

Details of surveillance activities, along with the specific objective evidence reviewed and the results of the reviews are contained within the surveillance checklist. The checklist is maintained as a QA record.

The surveillance team verified ANL CMT ACL compliance with the PDP Plan for Analysis of Simulated Headspace Gas, DOE/CAO-95-1076, Revision 2. The surveillance team determined that the ANL CMT ACL *Quality Assurance Plan* and *Work Plan for HSG PDP Sample Preparation, Verification, and Distribution* adequately defined the current organizational structure of the project. As a result, the surveillance team concluded that the related QA program requirements, procedures, and activities were adequate, satisfactorily implemented, and effective. In addition, the surveillance 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.

5.1.1 Organization

The surveillance team evaluated the adequacy of documents depicting the ANL CMT ACL organization. The ANL CMT ACL *Quality Assurance Plan* and the *Work Plan for HSG PDP Sample Preparation, Verification, and Distribution* adequately depict the current organizational structure and define the responsibilities of the ANL personnel involved in PDP sample preparation efforts.

5.1.2 Work Plan Implementation

The ANL CMT ACL prepared a Work Plan titled *Work Plan for Headspace Gas Performance Demonstration Program (PDP) Sample Preparation, Verification and Distribution* to meet the applicable requirements of the *Quality Assurance Program Document* (CAO-94-1012) Revision 3, and the *PDP Plan for Analysis of Simulated Headspace Gas* (DOE/CAO-95-1076), Revision 2. Activities related to the implementation of the Work Plan were reviewed, including the Standard Operating Procedures, Procurement, Training and Qualification, and QA

Records. The Work Plan activities were determined to be adequate, satisfactorily implemented, and effective.

SOP: ACL-162, *Qualification of Analytical Chemistry Laboratory Analysts* was not referenced in the Training and Qualification Section of the Work Plan. SOP: ACL-159, *Preparation of Samples for Determination of Headspace-Volatile Organic Compounds (VOCs) in SUMMA® Canisters* was not identified in Attachment 1, *Record of Analyst Training* of the Work Plan. During the surveillance, the Work Plan was revised and issued to add SOP: ACL-162 and SOP: ACL-159. It was also verified that the training documentation of analysts included SOP: ACL-159 (CDS #1).

The surveillance team also identified that signed ACL procedures that are listed in the Work Plan are not being maintained in a two-hour rated fire cabinet. During the surveillance, the team verified that signed copies of the procedures were moved to the appropriate storage area (CDS #2).

5.1.3 Disposition of Project-Specific Quality Assurance Records

The surveillance team reviewed the ANL CMT ACL procedure SOP: ACL-228, *Disposition of Project Specific Quality Assurance Records*. This procedure was determined to be adequate. A deficiency was corrected during the surveillance regarding the "Distribution" and "Date Filled" entries not being completed in the "WIPP Headspace Gas Document Log," that serves as the ANL CMT ACL's record index. During the surveillance, the team verified that the document log was completed. SOP: ACL-228 was revised and issued clarifying the record indexing requirements (CDS #3). After correction, this process was concluded to be adequate, satisfactorily implemented, and effective.

5.1.4 Laboratory Notebooks

The surveillance team reviewed SOP: ACL-107, *Review of Scientific Notebooks*. This procedure was determined to be adequate. A deficiency was corrected during the surveillance regarding three notebooks that were not reviewed by the Group Leader every six months. Some corrections to errors were not signed and dated, some pages were not x'ed or z'ed at the bottom of unused pages, and some pages were not signed and dated at the bottom of the page, as required by the procedure. During the surveillance, the team verified that the notebooks were corrected where possible and SOP: ACL-107 was revised and issued allowing the Group Leader to review the notebook at a frequency appropriate for the program for which the notebook is used. HSG PDP samples are prepared once a year, so the review every six months is not critical (CDS #4). After correction, this process was concluded to be adequate, satisfactorily implemented, and effective.

5.1.5 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*. This procedure was determined to be adequate and the process effective. However, the procedure was not being implemented entirely as written. The procedure requires the analysts preparing samples to open a valve ten times to ensure the system is flushed with internal standard gas in order to condition the system prior to adding internal standards to the sample being prepared. The analyst interviewed made it clear that this is typically done less than ten times. Corrective Action Report (CAR 01- 081) was issued to document this procedural deviation.

5.1.6 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*. This procedure was determined to be adequate and the process adequate, satisfactorily implemented, and effective.

5.8 Gas Standards Preparation

The surveillance team reviewed the ANL CMT ACL procedure SOP ACL-169, *Preparation of Volatile Organic Compound Calibration Standards for use with SUMMA® Canisters*. This procedure was determined to be adequate and the process adequate, satisfactorily implemented, and effective. Recommendation #1 was provided for management consideration.

5.9 Preparation and Distribution of PDP Samples

The surveillance 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*. This procedure was determined to be adequate. One concern was noted and resolved. The procedure indicated that the prepared PDP samples were stored in a secured cabinet. The analyst interviewed indicated that due to logistical operations at the time of sample preparation, the prepared samples are actually stored in applicable shipping boxes on the floor of laboratory E102, during the last few

days prior to shipment. The surveillance team agreed that this was an appropriate option to ensure that each shipment contained the correct canister sets. During the surveillance, the team verified that SOP: ACL-211 was revised and issued to allow the prepared HGS PDP samples to be stored in shipping boxes on the floor and to indicate that the laboratory will be secured (locked) when not occupied (CDS #5). After correction, this process was determined to be adequate, satisfactorily implemented, and effective.

5.10 Canister Cleaning

The surveillance team reviewed the ANL CMT ACL procedure SOP ACL-158, "SUMMA[®] canister Cleaning." This procedure was determined to be adequate and the process adequate, satisfactorily implemented, and effective.

6.0 CORRECTIVE ACTIONS, RECOMMENDATION, AND CORRECTED DURING THE SURVEILLANCE

6.1 Corrective Action Reports

Corrective Action Report (CAR) 01- 081 was issued as a result of this surveillance. The preparation of gas aliquots for analysis was not being performed in accordance with SOP: ACL-159, *Preparation of Samples for Determination of Headspace - Volatile Organic Compounds (VOCs) in SUMMA[®] Canisters*. The procedure requires the analysts preparing samples to open a valve ten times to ensure the system is flushed with internal standard gas in order to condition the system prior to adding internal standards to the sample being prepared. The analyst interviewed made it clear that this is typically done less than ten times. This CAR was provided under separate cover.

6.2 Recommendation

Recommendation #1

SOP: ACL-169, Revision 2, Section 4.1.1 states, "The historic primary gas standards (currently available) shall be verified annually by checking against vendor-certified gas mixtures analyzed within six months of preparation and containing at least 20% of the analytes given in Attachment 1. If the recovery shall be within $\pm 30\%$, the historical primary gas mixtures are considered valid for one year."

These two sentences need to be clarified. The first sentence could be misinterpreted to imply that the historic gas standards are being verified using newly obtained, vendor-certified gas standards. It is the old PDP samples (which are vendor-certified gas mixtures) that are used as laboratory control samples and if the results agree with the PDP samples' certified values within $\pm 30\%$, the historic standard is deemed acceptable. The second sentence is not

grammatically correct, and should say, "If the recoveries for all constituents are within $\pm 30\%$, the historical primary gas mixtures are considered valid for one year." It is recommended that this procedure be revised.

6.3 Deficiencies Corrected During the Surveillance (CDS)

CDS #1

The SOP:ACL-162, *Qualification of Analytical Chemistry Laboratory Analysts* was not referenced in the Training and Qualification Section of the Work Plan and SOP:ACL-159, *Preparation of Samples for Determination of Headspace-Volatile Organic Compounds (VOCs) in SUMMA® Canisters* was not identified in Attachment 1, *Record of Analyst Training* of the Work Plan. During the surveillance, the Work Plan was revised and issued to add SOP: ACL-162 and SOP: ACL-159 as appropriate. It was also verified that the training documentation of analysts included SOP: ACL-159.

CDS #2

The signed ACL procedures that are identified in the Work Plan are not being maintained in a two-hour rated fire cabinet. During the surveillance, the team verified that signed copies of the ACL procedures were moved to the appropriate storage area (CDS #2).

CDS #3

The *WIPP PDP Headspace Gas Document Log* that serves as the ANL CMT ACL's record index for HSG PDP was not completely filled-in. The "Distribution" and "Date Filled" entries were not completed. During the surveillance, the team verified that the document log was completed. SOP: ACL-228 was revised and issued clarifying the record indexing requirements.

CDS #4

Three notebooks were not reviewed by the Group Leader every six months. Some corrections to errors were not signed and dated, some pages were not x'ed or z'ed at the bottom of unused pages, and some pages were not signed and dated at the bottom of the page, as required by SOP: ACL-107. During the surveillance, the team verified that the notebooks were corrected where possible and SOP: ACL-107 was revised and issued allowing the Group Leader to review the notebook at a frequency appropriate for the program for which the notebook is used. HSG PDP samples are prepared once a year so the review every six months is not critical (CDS #4).

CDS #5

SOP: ACL-211, *Preparation and Distribution of Simulated Headspace Gas Samples for the WIPP HSGPDP* states that once prepared, HSG PDP sample canisters are stored in a separate cabinet (step 5.6.2). It was identified that prepared samples are stored in the applicable shipping boxes on the floor of laboratory E102, during the last few days prior to shipment. The surveillance team agreed that this was an appropriate option to ensure that each shipment contained the correct canister sets. During the surveillance, the team verified that SOP: ACL-211 was revised and issued to allow the prepared HGS PDP samples to be stored in shipping boxes on the floor and to indicate that the laboratory will be secured (locked) when not occupied.

7.0 ATTACHMENTS

Attachment 1: Personnel Contacted During the Surveillance
Attachment 2: Procedures Reviewed During the Surveillance

PERSONNEL CONTACTED DURING THE SURVEILLANCE

PERSONNEL CONTACTED				
NAME	TITLE/ORG	PRE SURVEILLANCE MEETING	CONTACTED DURING SURVEILLANCE	POST SURVEILLANCE MEETING
Applegate, Daniel V.	Scientific Associate, CMT/ACL	X	X	X
Boparai, Amrit	Project Manager	X	X	X
Green, David W.	Ex ACL Manager	X		X
Kalensky, Mike	Scientific Associate, CMT/ACL	X	X	X
Martino, Fred	QA Coordinator	X	X	X
Tertlus, M.	Clerk		X	

ANL-E CMT ACL PROCEDURES SURVEILLED IN S-01-15

NUMBER	PROCEDURE NUMBER/REVISION	TITLE
1.	SOP: ACL-107, R7	Review of Scientific Notebooks
2.	SOP: ACL-158, R2	SUMMA® Canister Cleaning
3.	SOP: ACL-159, R2	Preparation of Samples for Determination of Headspace-Volatile Organic Compounds (VOCs) in SUMMA® Canisters
4.	SOP: ACL-168, R2	Analytical Method for the Determination of Volatile Organic Compounds in SUMMA® Canisters Sampling Using Gas Chromatographic/Mass Spectrometry
5.	SOP: ACL-169, R2	Preparation of Volatile Organic Compound Calibration Standards for use with SUMMA® Canisters
6.	SOP: ACL-211, R2	Preparation and Distribution of Simulated Headspace Gases Samples for the WIPP Performance Demonstration Program
7.	SOP: ACL-221, R2	Preparation and Distribution of SUMMA® Canisters for the WIPP Performance Demonstration Program for Analysis of Simulated Headspace Gases
8.	SOP: ACL-228, R1	Disposition of Project-Specific Quality Assurance Records
9.	C-0030-0221, R8	Quality Assurance Plan
10.	No Number/R4	Work Plan for Headspace Gas Performance Demonstration Program Sample Preparation, Verification and Distribution