



Department of Energy
Carlsbad Field Office
P. O. Box 3090
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August 26, 2003

ENTRIFIED



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

RE: Evaluation of Corrective Action Plans Argonne National Laboratory-East (ANL-E)
CARS 03-065 and 03-066 for Audit A-03-18

Dear Dr. Boparai:

The Carlsbad Field Office (CBFO) has evaluated the Corrective Action Plans for CARS 03-065 and 03-066. The remedial actions, investigative action, root cause and actions to prevent recurrence for CAR 03-065 were not acceptable. The remedial action for CAR-03-066 was acceptable; however, verification of the remedial action based on the review of documentation submitted with the CAP on August 7, 2003 was not acceptable. The evaluation results are documented on the enclosed CAR Continuation Sheets.

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
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CBFO QA File
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CBFO CORRECTIVE ACTION REPORT

(Continuation Sheet)

1. CAR No. 03-065

2. Activity Report No. A-03-18

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Block # 17 Evaluation of Corrective Actions Proposed by the Responsible Organization:

The response submitted by Argonne National Laboratory on August 7, 2003 is rejected. The response is rejected based on the following:

1. a. SW-846, Chapter One, section 4.6, Calibration Records & Traceability of Standards/Reagents states that "Calibration is a reproducible reference point to which all sample measurements can be correlated.....The accuracy of the calibration standards is important because all data will be in reference to the standards used".
- b. SW-846, Method 8000B, section 7.4 states that "Initial calibration of an analytical instrument involves the delineation of the relationship between the response of the instrument and the amount or concentration of an analyte introduced into the instrument.In order to perform quantitative measurements, this relationship must be established prior to the analysis of any samples..."

Conclusion: The analytical scheme ANL-E is proposing does not comply with the above requirements. The site proposes to use calibration standards as the "unknown" in the scheme and the PDP samples as the reference point. This is not acceptable.

2. a. SW-846, Method 8260B, section 5.7 states that stock standards "may be prepared from pure standard materials or purchased as certified solutions". A standard that has expired can no longer be regarded as a "certified" standard.
- b. SW-846, Method 8260B, section 5.12 states "Calibration standards - There are two types of calibration standards used for this method; initial calibration standards and calibration verification standards. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations".

Conclusion: Expired standards cannot be used for calibration as they are no longer "certified".

The remainder of ANL-E's response was not assessed as it is based on the premise of calibration using expired standards. This practice is not acceptable.

Part of the mission of ANL-E, with regard to the PDP program, is to verify that the concentration of analytes in the PDP samples is correct. It is unclear how the laboratory would do this when they propose using expired standards for calibration and the PDP samples as the only "knowns" in the analytical scheme.

It is acceptable for the laboratory to utilize a documented protocol to verify expired standards so that they may be used beyond the original expiration date. However, this analytical scheme must be based on valid instrument calibration using unexpired, certified standards.

Evaluated By: Dorothy Gill

Name

8/26/03

Date

CBFO CORRECTIVE ACTION REPORT

(Continuation Sheet)

1. CAR No. 03-066

2. Activity Report No. A-03-18

3. Page 1 Of 1

Block # 17 Evaluation of Corrective Actions Proposed by the Responsible Organization and Verification of Corrective Action Documentation:

The response submitted by Argonne National Laboratory on August 7, 2003 was to revise SOP, ACL 168, Rev. 5 to address the adequacy issues identified in this CAR; however, the documentation submitted, SOP, ACL 168, Rev. 6, did not address the issues identified. Verification of corrective action is not acceptable based on the following:

CAR item A:

The initial calibration section (8.4.1) does not describe how retention time and retention time windows are determined.

Response: "Section 8.8.1 of the revised SOP, ACL 168, Rev. 6, addresses item A given above".

Verification of Corrective Action: Section 8.8.1 of the revised SOP, ACL 168, Rev. 6 only requires determination of retention time windows on an annual basis. Other circumstances, such as major instrument maintenance or installation of a new analytical column, that should initiate RTW determination are not addressed.

CAR item B:

The procedure does not require generation of method precision and accuracy data.

Response: "Section 10 of the revised SOP, ACL 168, Rev. 6, addresses item B given above".

Verification of Corrective Action: Section 10 of the revised SOP, ACL 168, Rev. 6 reads: "Method Performance The laboratory has demonstrated it's ability to meet the program required MDLs". This CAR item deals with precision and accuracy data, not MDLs.

CAR item C:

The procedure does not describe what records are generated by this procedure, nor how these records are dispositioned.

Response: "Section 9.6 of the revised SOP, ACL 168, Rev. 6, addresses item C given above".

Verification of Corrective Action: Section 9.6 of the revised SOP, ACL 168, Rev. 6 reads: "Raw data will be stored in a 1 hour fire safe. Refer to Work Plan for Headspace Gas Performance Demonstration Program Sample Preparation, Verification, and Distribution". Section 9.6 appears to only address raw data rather than including records generated during the work process. The response does not state what information, associated with the CAR item, will be found in the PDP Plan, nor does it provide an exact reference for that information.

CAR item D:

The procedure does not address the holding temperature requirement for VOCs SUMMA canister, hence there was no documentation for monitoring the temperature in the area for storage of PDP and calibration canisters.

Response: "Section 7 of the revised SOP, ACL 168, Rev. 6, addresses item D given above".

Verification of Corrective Action: Section 7 of the revised SOP, ACL 168, Rev. 6 does not address the holding temperature requirement for headspace gas samples. Additionally, the response did not specify if temperature monitoring of the storage area will be required.

Evaluated By:

Dorothy Gill
Name

8/26/03
Date

Name

Date