



Department of Energy

Washington, DC 20585

MAR 28 2000

QA: QA

Zell Peterman
U.S. Geological Survey
Denver Federal Center
P.O. Box 25046, M/S 263
Denver, CO 80225

VERIFICATION OF CORRECTIVE ACTIONS AND CLOSURE OF DEFICIENCY REPORT (DR) USGS-00-D-017

The Office of Quality Assurance staff has evaluated the corrective actions of DR USGS-00-D-017 and determined the results to be satisfactory. As a result, the DR is considered closed.

If you have any questions, please contact either James Blaylock at (702) 794-1420 or Donna J. Sinks at (303) 236-5050.

OQA:JB-1061

James Blaylock for
Robert W. Clark, Director
Office of Quality Assurance

Enclosure:
DR USGS-00-D-017



Printed with soy ink on recycled paper

*Public per
Mike Hayden
NMSS07*

MAR 28 2000

cc w/encl:

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8. DEFICIENCY REPORT
 CORRECTIVE ACTION REPORT

NO. USGS-00-D-017

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DEFICIENCY/CORRECTIVE ACTION REPORT

1. Controlling Document:
YMP-USGS-GCP-16, R5-M1, M2, Carbonate Carbon and Oxygen Isotope Analysis

2. Related Report No.:
NA

3. Responsible Organization:
USGS, Environmental Science Team

4. Discussed With:
J. Wheilan, R. Moscati, P. Striffler, T. Oliver, M. Mustard, G. Patterson, USGS; R. Howell, PWT; D. Sinks, OQA/QATSS

5. Requirement:

GCP-16, states, in part, that:

1. Sec. 4.3.5 Operational Check - "An operational check of the mass spectrometer is performed in which the laboratory operational standard is analyzed."
2. Sec. 5.0 Verification and Hold Points - "Results of the mass spectrometer operational check and the system calibration shall be used to demonstrate that the work described in Section 4.0 is performed as required."
3. Sec. 7.0 Calibration - "TS-1 is analyzed approximately each 10 samples and provides the basis for the day-to-day continuity of mass spectrometer calibration."

NOTE: Sec. 6.0 also states that "On a day-to-day basis, the mass spectrometer operating standard may display minor shifts from its accepted value. Such shifts are detected by daily (when in operation) analysis of operational standards; as long as such shifts are less than 0.4 permil, data from unknowns may be corrected day by day based on these operational checks."

6. Description of Condition:

see page 2

7. Initiator

Gary R. Severson
Gary Severson, USGS Date 10/26/99

9. Does a stop work condition exist? (Not required for a DR)

Yes No

If Yes, Check One: A B C D

10. Recommended Actions:

see page 2

11. QA Review:

William J. Glasser for
QAR Donna Sinks Date 11-11-99

12. Response Due Date:

10 working days from issuance

13. DOQA Issuance Approval:

Printed Name Robert W. Clark

Signature

James Blaylock for

Date

11/17/99

22. Corrective Actions Verified:

William J. Glasser for
QAR DONNA SINKS Date 3-23-00

23. Closure Approved by:

DOQA *James Blaylock for*

Date 3/28/00

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DEFICIENCY/CORRECTIVE ACTION REPORT/STOP WORK ORDER CONTINUATION PAGE

Block 6 - Description of Condition (continued):

Operational checks of the mass spectrometer using the TS-1 (calcium carbonate mineral residue from NBS-19 standard) were not conducted approximately every 10 samples, as required. Gas samples analyzed with the mass spectrometer were collected directly from boreholes and not evolved using the extraction techniques performed on solid carbonate mineral samples as described in Sec. 4.2.

NOTE: The lack of operational checks were discovered during completion of the "Data or Technical Information Confirmation Checklist" for a new USGS data package entitled "Gas Chemistry and Isotope Data from Boreholes ESF-LPCA-RBT#1, ESF-NDR-MF#1, and ESF-NDR-MF#2". This data package supports SCP 8.3.1.2.2.4.10, Hydrologic Properties of Major Faults Encountered in the ESF, WBS 1.2.3.3.1.2.4.

Twenty-two samples were collected for analysis with the following SMF SPC numbers: 512088 through 512099 and 501739 through 501748. These twenty-two gas samples (CO₂) were collected directly from boreholes ESF-LPCA-RBT#1, ESF-NDR-MF#1, and ESF-NDR-MF#2 in mylar balloons as whole-gas samples and in silica gel towers using YMP-USGS-HP-56, Gas and Water Vapor Sampling From Unsaturated-Zone Boreholes. The samples were then prepared for analysis using YMP-USGS-GCP-33, Extraction of Soil Gas CO₂ for Stable Isotope Analysis. The samples were subsequently analyzed using YMP-USGS-GCP-16, Carbonate Carbon and Oxygen Isotope Analysis. These gas samples were directly injected manually into the inlet port of the mass spectrometer, serial number SC-16-252. Periodic operational checks of the mass spectrometer, using the TS-1 operational standard, were not conducted as required.

Block 10 - Recommended Actions (continued):

1. Identify other technical product packages that are in-process or were previously submitted to the Records Processing Center and the Technical Data Management System that include gas samples collected in a similar manner to those identified in this deficiency document and that were analyzed using technical procedure GCP-16.
2. Determine the impact on that data as a result of the operational checks with the TS-1 (calcium carbonate mineral residue from NBS-19) not being conducted during analysis of the gas samples.
3. Annotate affected in-process and completed technical product packages with a reference to this deficiency document, regardless of impact on data.
4. Revise applicable technical procedures to accurately reflect current work practices.

TYPE RESPONSE:

- Initial
- Complete
- Amended

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DEFICIENCY/CORRECTIVE ACTION REPORT (RESPONSE)

14. Remedial Actions:

The TDIFs for all data sets identified under the investigative actions will be annotated with reference to this DR and with a statement that there is no negative impact on the data.

15. Extent of Condition:

Extent: This condition is limited to the analysis under GCP-16 of gas mixture samples extracted under GCP-33. These are referred to as "balloon samples." All data sets in the ATDT which relied on GCP-16 will be reviewed to identify the ones containing "balloon samples."

Impact: There is no negative impact from the lack of operational checks during analysis of the "balloon samples." Calibrations of the mass spectrometer were performed before and after analysis. The operational checks in GCP-16 should not have been applicable to the "balloon samples."

16. Cause: (Attach results of root cause determination prepared in accordance with AP-16.4Q for a significant deficiency.)

GCP-16 was written specifically for analysis of carbonate materials and the operational checks were written into the procedure specifically for carbonate materials. When GCP-33 was written to cover gas extraction of the "balloon samples," it pointed to GCP-16 for the analysis. The inappropriateness of the operational checks in GCP-16 for "balloon samples" was overlooked when GCP-33 was written.

17. Action to Preclude Recurrence:

GCP-16 will be modified or revised to incorporate the extraction process from GCP-33, to add a statement that no operational check applies to the "balloon samples," and to reference HP-56 for sample collection. GCP-33 will be rescinded.

Short term actions to preclude recurrence: The Chief, YMPB, has issued an e-mail reminder to YMPB personnel reminding them that work must be done according to approved technical procedures and/or scientific notebooks or it must be stopped until the procedure or notebook can be changed. There are currently no balloon samples to be analyzed and none planned at this time.

Date when activity will be performed in accordance with requirements: 11/29/99 (Date of e-mail reminder)

18. Corrective Action Completion Due Date:
January 31, 2000 (for submittal of complete response)

19. Response by: *Zell C Peterman*
Zell E. Peterman Date 12/3/99 Phone (303) 236-7883

20. Evaluation: Accept Partially Accept Reject

21. Concurrence:
DOQA *James B. ...* Date 12/17/99

QAR *Donna Sinks*
(Donna Sinks) Date 12/14/99

12/16/99 Craig to Clark

TYPE RESPONSE:

- Initial
- Complete
- Amended

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14a. Immediate Action:

The Chief, YMPB, issued an e-mail reminder to YMPB personnel reminding them that work must be done according to approved technical procedures and/or scientific notebooks or it must be stopped until the procedure or notebook can be changed. There are currently no balloon samples to be analyzed and none planned at this time.

Compliance Date: This action was completed on 11/29/99.

14. Remedial Actions:

The investigative actions have been completed for this deficiency and 16 gas sample data sets acquired using GCP-16 (all revisions) have been identified. The Data Tracking Numbers for the associated TDIFs are itemized on Continuation Page 5. All TDIFs have been annotated with the following statement: "It has been determined per deficiency USGS-00-D-017, that lack of operational checks of mass spectrometers during gas sample analysis under GCP-16 (R0 through R5) did not impact the data acquired."

15. Extent of Condition:

Extent: This condition is limited to the analysis under GCP-16 of gas mixture samples extracted under GCP-33. These are referred to as "balloon samples." All data sets in the ATDT which relied on GCP-16 as the analytical method have been reviewed to identify the ones which analyzed the "balloon" or gas samples (see Item #14 above).

Impact: There is no negative impact from the lack of operational checks of the mass spectrometer during analysis of the "balloon samples." Mass spectrometer calibrations were performed before and after analysis. The operational checks identified in GCP-16 should not have been applicable to the "balloon samples."

16. Cause: (Attach results of root cause determination prepared in accordance with AP-16.4Q for a significant deficiency.)

GCP-16 was written specifically for mass spectrometer analysis of carbonate materials and the operational checks were written into the procedure specific to those types of analyses. When GCP-33 was written to cover gas extraction of the "balloon" or gas samples, it pointed to GCP-16 for the analysis. The inappropriateness of the operational checks of the mass spectrometer in GCP-16 for "balloon samples" was overlooked when GCP-33 was written.

17. Action to Preclude Recurrence:

GCP-16,R5 is being revised to incorporate the extraction process for gas samples from GCP-33, R0, to add a statement that no operational check of the mass spectrometer is required for analysis of the "balloon" or gas samples, and to reference HP-56 for gas sample collection. GCP-33, R0 will be rescinded.

18. Due Date: February 16, 2000

- For submittal of complete response
- For completion of corrective action

19. Response by: Zell Peterman

Zell Peterman
Date 1-28-00 Phone 303-236-7883

20. Evaluation: Accept Partially Accept Reject

QAR *(Donna Sinks)*
Donna Sinks Date 1/28/00

21. Concurrence:

DOQA *James Blaylock* Date 2/1/00

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14. Remedial Actions Continued from Page 4:

IDENTIFIED DATA SET DTNs FOR GAS SAMPLES ANALYZED USING GCP-16:

GS931208315215.036	GS950908312271.005
GS940408312271.004	GS960208312261.002
GS940408312271.011	GS961108312261.006
GS940708312261.005	GS961108312271.002
GS941208312261.008	GS970283122410.002
GS950808312261.004	GS970908312271.003
GS950908312271.002	GS970908315213.004
GS950908312271.004	GS981283122410.006

TYPE RESPONSE:

- Initial
- Complete
- Amended

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14a. Immediate Action:

No change

Compliance Date:

14. Remedial Actions:

No change

15. Extent of Condition:

No change

16. Cause: (Attach results of root cause determination prepared in accordance with AP-16.4Q for a significant deficiency.)

No change

17. Action to Preclude Recurrence:

No change

18. Due Date: February 29, 2000

- For submittal of complete response
- For completion of corrective action

19. Response by: Zell E. Peterman

Z. Marshall for ZEP
Date 2/16/00 Phone (303) 236-7883

20. Evaluation: Accept Partially Accept Reject

(Denna Sinks)
QAR *Denna Sinks* Date 2/16/00

21. Concurrence:

DOQA *James Blaylock for* Date 2/18/00

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REQUEST FOR EXTENDED PROCESSING

Actions Not Completed Within Normal Processing and Justification The Completion of Corrective Action for this DR was due February 16, 2000. The actions were to have GCP-16, R5 revised to incorporate a procedural step from GCP-33, R0, and to rescind GCP-33, R0. The draft of technical procedure GCP-16, R6, is currently in technical review comment resolution. The response required for one of the reviewer's mandatory technical comments is taking more time to resolve than the author had anticipated. The process for rescinding GCP-33 has been initiated with the USGS Technical Procedure Coordinator but has not received the required approvals at this time. It is expected that the actions for GCP-16, R6 and GCP-33, R0 will be completed by February 29, 2000.

Impacts: Delay in the rescission of GCP-33, R0 has no impact. There is no work being conducted at the present time that requires the implementation of GCP-16; therefore, there is no negative impact on the resolution of this deficiency due to extended processing.

Zell E. Peterman, USGS, Chief, YMPB Environmental Science Team
Zell E. Peterman, USGS, Chief, YMPB Environmental Science Team

2/16/00
Date

Robert W. Craig, USGS, Chief, Yucca Mountain Project Branch
Robert W. Craig, USGS, Chief, Yucca Mountain Project Branch

02/16/00
Date

TYPE RESPONSE:

- Initial
- Complete
- Amended

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14a. Immediate Action:

No change

Compliance Date:

14. Remedial Actions:

No change

15. Extent of Condition:

No change

16. Cause: (Attach results of root cause determination prepared in accordance with AP-16.4Q for a significant deficiency.)

No change

17. Action to Preclude Recurrence:

GCP-33 will be rescinded. Since there are no more balloon samples planned at this time, there are no actions necessary for GCP-16.

18. Due Date: March 15, 2000

- For submittal of complete response
- For completion of corrective action

19. Response by: Zell E. Peterman

Zell E. Peterman
Date 3-2-00 Phone (303) 236-7883

20. Evaluation: Accept Partially Accept Reject

QAR *Donna Sinks*
(Donna Sinks) Date 3/5/00

21. Concurrence:

DOQA *James Blaylock Jr* Date 3/8/00

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REQUEST FOR EXTENDED PROCESSING:

Actions Not Complete and Justification: The rescission of GCP-33 was originally being coordinated with the revision to GCP-16. Since the decision has been made that no action is required under this DR for GCP-16, the rescission of GCP-33 will proceed independently. The rescission of GCP-33 is now in the approval process. The effective date needs to be coordinated with Document Control and, per AP-6.1Q, should be at least seven business days after submittal to Document Control. The rescission will be complete by March 15, 2000.

Impact: There is no impact because there is no ongoing work nor planned work under GCP-33.

Zell E. Peterman
Zell E. Peterman, USGS, Chief, YMPB Environmental Science Team

3-2-00
Date

Robert W. Craig
Robert W. Craig, USGS, Chief, Yucca Mountain Project Branch

03/03/00
Date

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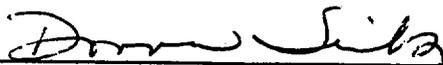
BLOCK 22 – CORRECTIVE ACTIONS VERIFIED (continued from page 1)

Verification of completion of corrective actions has been completed, as described below:

1. TDIFs for the following DTNs were annotated on 1/25/00 with the following statement (checked 3/23/00 using the ATDT Data Base Access on the M&O Intranet page) - "It has been determined per deficiency USGS-00-D-017 that lack of operational checks of mass spectrometers during gas sample analysis under GCP-16 [R0 through R5] did not impact the data acquired."

- GS931208315215.036	GS950908312271.005
- GS940408312271.004	GS960208312261.002
- GS940408312271.011	GS961108312261.006
- GS940708312261.005	GS961108312271.002
- GS941208312261.008	GS970283122410.002
- GS950808312261.004	GS970908312271.003
- GS950908312271.002	GS970908315213.004
- GS950908312271.004	GS981283122410.006
2. YMP-USGS-GCP-33, R0, *Extraction of Soil Gas CO2 for Stable Isotope Analysis*, was rescinded 3/13/00.
3. An e-mail notice was sent on 11/29/99 by R. Craig, USGS YMP Branch Chief, to all USGS YMPB personnel reminding all YMPB personnel to work to approved technical procedures, etc. and that work must be stopped if the procedure cannot be followed. This e-mail was originally sent out in response to USGS-00-D-008.
4. Mass spectrometers (ID # SC-16-251 and SC-16-252) system calibrations were conducted at least annually according to YMP-USGS-GCP-16, as shown in the table in Attachment 2. This table was generated 3/23/00 as a query using the USGS YMPB calibration tracking database.

As a result of the verifications described above, it is recommended that this DR be closed.



Donna Sinks, OQA On-site Representative

3/23/00

Date

US 5-00-D-017
Attachment I
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▶ Martha Mustard
11/29/99 09:08 AM

.....

To: Donna Sinks/YD/RWDOE@CRWMS
cc:

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Subject: Action when work is not covered by approved procedure or scientific notebook.

I can't tell from this acronym if you were included so I'm forwarding this to you just in case.

----- Forwarded by Martha Mustard/YM/RWDOE on 11/29/99 09:04 AM -----

Robert_Craig@notes.ymp.gov on 11/29/99 07:48:55 AM



To: USGS_YMP
cc: Steven Beason/YM/RWDOE, skohn@pwt.com (bcc: Martha Mustard/YM/RWDOE)

Subject: Action when work is not covered by approved procedure or scientific notebook.

This e-mail is to remind all YMPB personnel that work must be done according to approved technical procedures and/or scientific notebooks. When work cannot be accomplished as described in approved procedures and/or scientific notebooks, the work must be stopped until the procedure or scientific notebook is changed to reflect the current work practice. I had previously asked Team Chiefs to insure that staff reviewed technical procedures for consistency with current methods and practices. If you have not done so, please do at this time.

We recently had a DR (USGS-00-D-008) issued because some technical procedures were not kept up-to-date. Let's not have any repeats of this deficient condition.

ID NUMBER	DESCRIPTION	START DATE	END DATE	TEST RESULTS	TESTER
SC-16	Mass Spec System Calibration			GCP-16,R3	J. Whelan
SC-16	Mass Spec System Calibration	11/25/92		GCP-16,R3	Moscato/Whelan
SC-16	Mass Spec System Calibration	8/19/93	8/19/94	GCP-16,R4	R. Moscato/J. Burdett
SC-16	Mass Spec System Calibration	1/21/94	1/21/95	GCP-16,R4	R. Moscato
SC-16-252	Mass Spec System Calibration	1/21/94	1/21/95	GCP-16,R4	R. Moscato
SC-16	Mass Spec System Calibration	8/15/94	8/15/95	GCP-16	J.Burdett/R.Moscato
SC-16-252	Mass Spec System Calibration	1/11/95	1/11/96	GCP-16,R4	R. Moscato
SC-16	Mass Spec System Calibration	8/15/95	8/15/96	GCP-16,R4	J.Burdett/R.Moscato
SC-16-252	Mass Spec System Calibration	1/29/96	1/29/97	GCP-16,R4	R. Moscato
SC-16	Mass Spec System Calibration	8/28/96	8/28/97	GCP-16,R4	Whelan/R.Moscato
SC-16-252	Mass Spec System Calibration	1/23/97	1/23/99	GCP-16	R. Moscato
SC-16-252	Mass Spec System Calibration	1/28/97	1/28/98	GCP-16,R4	R. Moscato
SC-16 251 Kiel	Mass Spec Sys Calib	8/28/97	8/28/98	GCP-16, R5	Moscato/Johnso
SC-16	Mass Spec System Calibration	10/23/97	10/23/98	GCP-16,R5	Whelan
SC-16 251 Kiel	Mass Spec Sys Calib	8/28/98	8/28/99	GCP-16, R5	Whelan
SC-16-252	Mass Spec System Calibration	1/15/99	1/15/00	GCP-16	R. Moscato

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