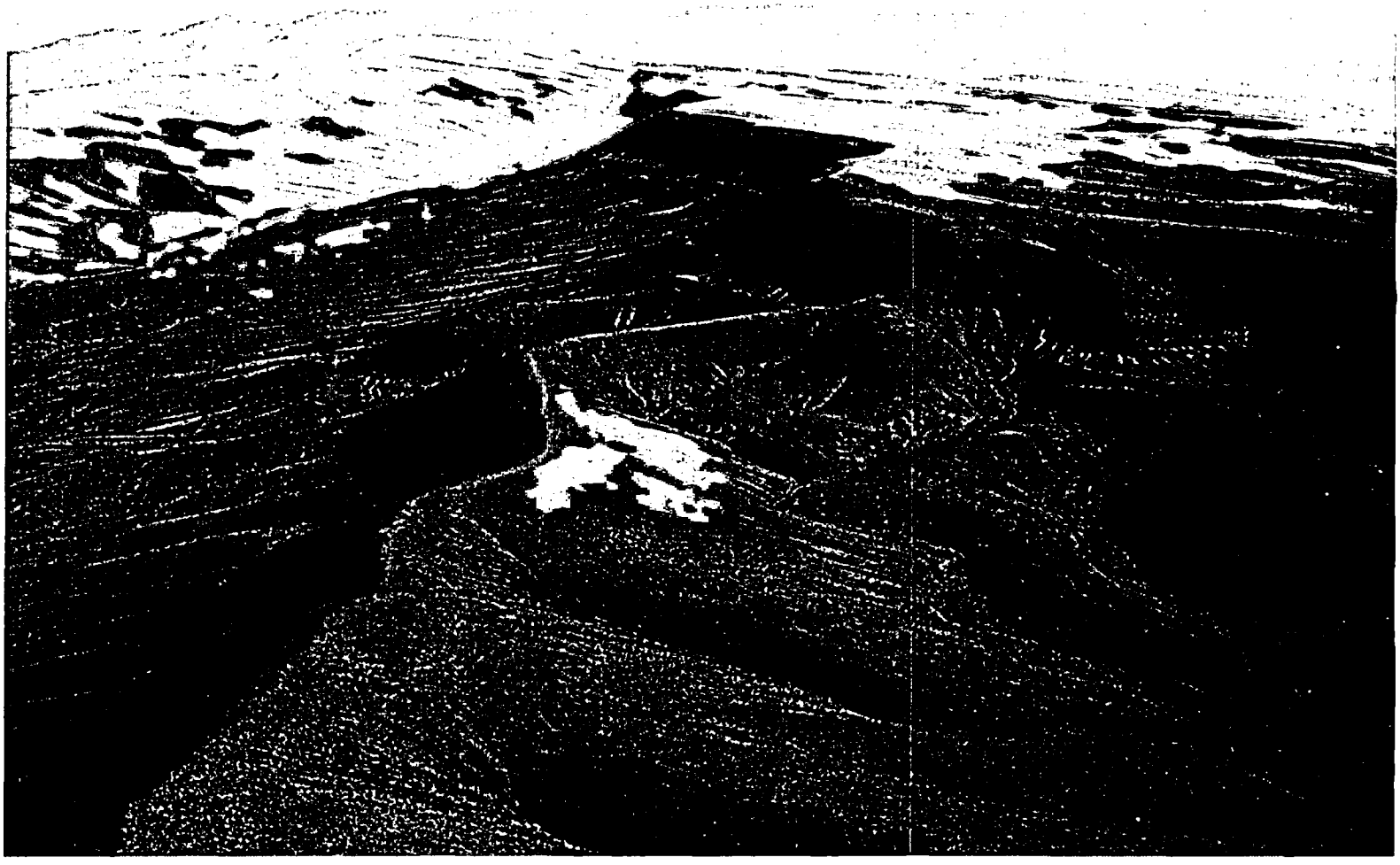
QA:NA

United States Department of Energy

Office of Civilian Radioactive Waste Management

OCRWM Independent Review Team Report of the BSC Quality Assurance Audit BQAP-BSC-05-07

June 2006



APPROVED BY:

Gene E. Runkle — June 20, 2006.

Gene E. Runkle
Program Manager for Independent Evaluations
Office of Civilian Radioactive Waste Management

Date

This page intentionally left blank

TABLE OF CONTENTS

EXECUTIVE SUMMARY v

1.0 INTRODUCTION 1
1.1 Purpose 1
1.2 Audit Background 1
1.3 Summary of NRC Concerns 2

2.0 INDEPENDENT REVIEW TEAM METHODOLOGY 5

3.0 SUMMARY OF INFORMATION GATHERED DURING INDEPENDENT REVIEW 6

3.1 *Were the Vaisala humidity probes calibrated?* 6

3.2 *If the probes were properly calibrated, was the calibration performed in accordance with Yucca Mountain procedures and appropriately documented?* 15

3.3 *Why was the calibration issue not recognized as a quality issue by BSC management and included in the BSC audit findings?* 18

3.4 *Why was line management not included in audit activities at LLNL?* 19

3.5 *With regard to overall organizational culture, what is the ability of the organizations to work to standards and requirements, to stop work as required, and to understand quality assurance requirements and take appropriate actions?* 19

4.0 OTHER FINDINGS 20

A. *LLNL staff did not misrepresent calibration status in scientific notebooks.* 20

B. *No quantifiable requirements are in place before work is performed.* 21

C. *Response to AOIs is not timely* 22

TABLES AND FIGURES

Figure 1: Timeline for Use of Vaisala Probes 8

Table 1: Calibration History of Vaisala Probes 9

APPENDICES

A. National Institute of Standards and Technology Report 23

B. Personnel Contacted by the Independent Review Team at Yucca Mountain Project Offices and LLNL 31

C. Audit Timeline 33

C. History of Changes to the Procedure on Control of Measuring and Test Equipment 35

EXECUTIVE SUMMARY

Bechtel SAIC Company, LLC (BSC) conducted an audit August 15-31, 2005, to evaluate whether the quality of data input into models used to assess the repository waste package and drip shield provided technically sound and defensible results. The Nuclear Regulatory Commission (NRC) conducted an audit observation of the BSC audit team activities.

On January 9, 2006, NRC provided the Office of Civilian Radioactive Waste Management (OCRWM) an Observation Audit Report that expressed concerns about the conduct of the BSC audit and technical issues related to the calibration and use of Vaisala relative humidity/temperature probes by Lawrence Livermore National Laboratory (LLNL) to measure humidity and temperature in experiments. NRC's report observed LLNL's approach to calibration of the probe was not in compliance with the *Quality Assurance and Requirements Description* (QARD) document. NRC "concluded that the BSC audit was not effective in identifying, documenting, and alerting BSC and DOE management" to quality issues.

In February 2006, the OCRWM Acting Director requested that an independent review team investigate the issues identified in the NRC report. The Independent Review Team performed an investigation, including document review and interviews, in an effort to evaluate these issues.

The Independent Review Team found that a number of actions have been undertaken by OCRWM and BSC to address the issues raised by NRC. These actions included two self-assessments, a stop-work order covering LLNL experiments using the relative humidity/temperature probes, two Level A Condition Reports (CRs) with root cause analyses and extent of condition evaluations, and several Level B CRs. These activities are still underway.

The major findings of this review are:

- There is no evidence of intent on the part of LLNL scientists to misrepresent the calibration status of their relative humidity/temperature probes.

- The BSC procedure (LP-12.1Q-BSC) for control of measuring and test equipment does not properly implement the QARD because it allows for use of equipment past its recalibration due date.

- The LLNL technical approach to calibration of Vaisala relative humidity/temperature probes was valid. No national standards exist for calibrating the probes for the high temperatures involved in some experiments. LLNL used the manufacturer's recommended calibration process, and then developed several methods to further confirm probe response at high temperatures.

- The BSC audit team and BSC management did not respond effectively to the concerns expressed by NRC observation team, and did not take timely action to elevate NRC's concerns.

- There were communication failures within OCRWM and BSC which impeded the ability to address the concerns that NRC raised during the audit.

- BSC and LLNL have not defined the requirements for accuracy, at a specified level of confidence, for data inputs to the corrosion models. A more quantitative assessment of the data requirements needed to support modeling efforts is required to determine if the LLNL results on deliquescence testing have appropriately addressed uncertainty.

1.0 INTRODUCTION

1.1 Purpose

This report documents the findings and observations of an Independent Review Team formed in February 2006 to provide information regarding issues raised by the Nuclear Regulatory Commission (NRC) concerning an audit conducted by Bechtel SAIC Company LLC (BSC) and calibration activities performed by scientists at Lawrence Livermore National Laboratory (LLNL). Deficiencies and weaknesses identified by NRC, and Audit Observation Inquiries (AOIs) submitted to DOE by NRC, involved:

- BSC's conduct of the audit
- Calibration work performed by LLNL and related documentation
- The technical content of the Analysis and Model Reports (AMRs) reviewed during the audit.

This report focuses on the concerns related to the LLNL's calibration of probes used in Yucca Mountain Project experiments, and on BSC's audit activities in relation to these concerns. The AOIs and technical matters not related to the calibration issue are being addressed by other ongoing actions.

1.2 Audit Background

On August 15-31, 2005, Bechtel SAIC Company, LLC (BSC) conducted a routine audit[1] to evaluate whether scientific investigations that provide input data into the repository waste package and drip shield models provided technically sound and defensible results. The audit (BQAP-BSC-05-07) was titled *Performance-Based Audit of BSC Licensing and Nuclear Safety: Scientific Investigation, Waste Package and Drip Shield Degradation.* Two AMRs were selected for review: *General and Localized Corrosion of the Waste Package Outer Barrier* (ANL-MD-EBS-000003), and *Engineered Barrier System: Physical and Chemical Environment* (ANL-MD-EBS-000033). In tracing model inputs back to original source data the BSC audit team looked at the scientific approaches and methods used for data collection, including:

- The use and control of measuring and test equipment (M&TE)

[1] The audit was designated as BQAP-BSC-05-07, *Performance-Based Audit of BSC Licensing and Nuclear Safety: Scientific Investigation, Waste Package and Drip Shield Degradation*; the BSC audit report was approved by the BSC Quality Assurance Manager on October 7, 2005.

- Implementation of quality assurance (QA) requirements as defined in the OCRWM *Quality Assurance Requirements and Description* (QARD)[2]

- Collection, documentation, control, and use of data.

BSC quality assurance auditors conducted the audit in accordance with Line Procedure LP-18.6Q-BSC, *QA Internal Audit Program* and Administrative Procedure AP-16.1Q, *Condition Reporting and Resolution.* They also assessed the waste package and drip shield technical work against the NRC's *Yucca Mountain Review Plan* (NUREG-1804). Technical specialists were involved in audit activities in Nevada but not at LLNL.

Since the late 1990s, LLNL has performed experiments to measure the degradation of waste package materials under various conditions in order to provide input to computer models used to predict the performance of waste packages in a geologic repository. Probes to measure humidity and temperature are used during some of this work. In 1998, LLNL purchased relative humidity/temperature probes from Vaisala, a Finnish company that has offices in the United States.

These probes and other experimental measurement equipment must be calibrated in order for the results of experiments in which they are used to be acceptable for use in Yucca Mountain models and analyses. The QARD defines the overarching requirements for calibration, and a procedure, LP-12.1Q-BSC[3], directs implementation of those requirements. Documentation of calibration and testing activities must be included in scientific notebooks or on a form included in LP-12.1Q-BSC. Section 3.1 below provides specific information on the calibration and use of the Vaisala probes.

1.3 Summary of NRC Concerns

NRC observers conducted an audit observation of the BSC audit team activities in accordance with NRC Manual Chapter 2410, "Conduct of Observation Audits." To assess the effectiveness of the audit and to evaluate the implementation of the QA program by BSC and LLNL, NRC observers attended meetings and reviewed documents related to the BSC audit. NRC personnel provided feedback to the BSC audit team during the audit and communicated with BSC and OCRWM after conclusion of the audit. The principal NRC interactions are listed in Table 2 in Section 3.6.

On January 9, 2006, NRC provided its Observation Audit Report OAR-05-05[4] to the OCRWM Director of the Office of License Application and Strategy. NRC concluded that, "Although most of the audit observation results were found to be acceptable, several areas were identified as

[2] OCRWM, Quality Assurance and Requirements Description, DOE/RW-0333P, Rev. 16, August 23, 2004.
[3] BSC, LP-12.1Q-BSC, *Control of Measuring and Test Equipment*, Rev. 0, ICN 1, October 31, 2005. At the time of the audit, Rev. 0, ICN 0 was in effect.
[4] NRC, Observation Audit Report OAR-05-05, *Observation Audit of Bechtel SAIC Company, LLC, Audit of Scientific Investigation, Waste Package, and Drip Shield Degradation, BQAP-BSC-05-07*, January 9, 2006.

deficiencies or weaknesses." The following is a summary of the deficiencies and weaknesses related to the audit process and calibration that were identified in Section 5.1 of the NRC report:

NRC Audit-Related Issues

- BSC reduced the audit scope less than two weeks prior to the beginning of audit activities, and there was a delay in availability of the audit checklists. *(weakness)*

- Technical specialists were not included in audit activities at LLNL. *(weakness)*

- Documents were found to be missing from the records package for corrosion experiments. These were located at LLNL, and upon reviewing them NRC raised several technical issues to the BSC audit team; however, a Condition Report (CR) was not issued. *(deficiency)*

NRC Calibration-Related Issues

- LLNL used Vaisala humidity probes that were not vendor-qualified or calibrated for the required temperature ranges. NRC characterized this matter as an ineffective implementation of the QARD, which requires that test instruments be calibrated before use. *(deficiency)*

- LLNL conducted experiments to calibrate the Vaisala humidity probes at the high temperatures used during experiments. No nationally recognized standard existed, nor was the manufacturer able to provide a standard process for calibration at high temperatures, and LLNL had not adopted a nationally recognized protocol for conducting calibrations. LLNL did not identify these concerns to BSC and OCRWM management as potentially significant issues related to the defensibility of the experiments. *(deficiency)*

- NRC characterized as "potential misrepresentation" LLNL scientists' recording of information regarding calibration in scientific notebooks. *(deficiency)*

- LLNL researchers failed to issue a Condition Report under the Corrective Action Program for issues that could be easily rectified or were minor in nature, which is inconsistent with OCRWM's procedures concerning corrective action reporting. *(deficiency)*

- LLNL had issued two CRs with regard to calibration of the relative humidity/temperature probes, but the corrective actions were ineffective with regard to controls placed on use of the probes in subsequent experiments. This was characterized as an example of ineffective implementation of the QARD as related to the Corrective Action Program. *(deficiency)*

- LLNL did not issue a CR on repetitive non-conformance with the QARD requirements related to instrument calibration, and has not established adequate provisions to prevent recurrence of the problem. This was characterized as an example of ineffective implementation of the QARD as related to the Corrective Action Program. *(deficiency)*

NRC identified five Audit Observation Inquiries (AOI), characterized as "additional areas of technical product weakness," in Section 5.2 of the Observation Audit Report. These AOIs were submitted to BSC in December 2005 for response. One AOI is specifically discussed in this report: AOI-OAR-05-05-05, "Use of the Vaisala Humidity Probes at Temperatures Outside their Calibrated Range."

2.0 INDEPENDENT REVIEW TEAM METHODOLOGY

At the direction of the OCRWM Acting Director the OCRWM Program Manager for Independent Evaluations, Gene Runkle, initiated an independent investigation of the calibration-related issues raised in NRC's report to determine what had occurred and to assess weaknesses that had allowed this situation to develop. This investigation and report provides information to the Acting Director to support management analysis and decision-making.

The Independent Review Team included the following individuals, external to OCRWM, to support the evaluation:

- Dr. Peter Huang, an expert from the National Institutes of Standards and Technology (NIST) with extensive knowledge of humidity monitoring equipment and calibration, including familiarity with the Vaisala brand of probes used at LLNL. Dr. Huang's full report is included as Appendix A.

- Dr. Robert Uhrig, a retired professor of nuclear engineering with executive management experience at a nuclear utility, including responsibility for quality assurance for four nuclear power plants.

The OCRWM Office of the Director developed a number of areas of inquiry, which provided the structure for the independent review.[5] The Independent Review Team conducted interviews of BSC, OCRWM, LLNL, and NRC staff to determine facts and ascertain timelines. The Independent Review Team identified individuals that had direct knowledge of or participated in the audit, as well as key individuals at LLNL that were involved in experiments and calibration activities associated with the relative humidity/temperature probes (see Appendix B). The interviews were conducted over a three-week period in February 2006. Based on these interviews, the team constructed a timeline of audit activities (Appendix C), and a history of changes to the procedure on control of measuring and test equipment (Appendix D). Procedures, technical reports, Condition Reports, and other documents were collected and reviewed as part of this process.[6]

The Independent Review Team's findings in the areas of inquiry appear in Section 3.0.

[5] OCRWM Working Document, "Headquarters Independent Review Research and Evaluation of BSC/LLNL Audit," February 2, 2006.
[6] A list of reviewed documents and a list of relevant Condition Reports are provided on the References disk that accompanies this report.

3.0 SUMMARY OF INDEPENDENT REVIEW TEAM RESULTS AND CONCLUSIONS

The following subsections provide the Independent Review Team's results and conclusions on each of the areas of inquiry identified by OCRWM.

3.1: Were the Vaisala humidity probes calibrated?

The calibration of instruments and equipment against nationally and/or internationally recognized standards applicable to the specific conditions of the experiments is required by the QARD, and is essential to the traceability and defensibility of the technical work related to licensing of the Yucca Mountain repository. When a nationally recognized standard is not available, the QARD requires that the basis for calibration be documented. LP-12.1Q-BSC provides additional requirements concerning implementation of the QARD requirements.

The NRC audit observers were particularly concerned about the calibration of LLNL's Vaisala relative humidity/temperature probes at a low temperature and the use of those probes during experiments at high temperatures (over 100 °C). The calibration processes changed with time, as did the calibration methodology used. The timeline for the use of the Vaisala relative humidity/temperature probes at LLNL is shown in Figure 1.[7] The calibration history of these probes is separated into three time periods on the basis of the calibration methodology used, and is summarized in Table 1.

The Independent Review Team's discussions with LLNL personnel indicated that LLNL's focus was on the scientific adequacy of the guidance given by Vaisala and on continuous improvements of the calibration approach by further defining uncertainty and extending the temperature range.

Definition of Calibration

For the purposes of this report, the Independent Review Team found that the primary calibration of the temperature and humidity probes used in the development of quantitative models was performed by the manufacturer, Vaisala (1998 – 2002), and the qualified vendor, Thunder Scientific Corp (2002). In addition, LLNL developed a salt test (2004) and bi-thermal method (2005) to further confirm experimental results at the higher temperature regions. LLNL used both of these methods to provide a better estimate of the uncertainty of the primary calibration when extrapolated to higher temperatures.

Data for temperatures greater than 120 °C have been labeled "conditional" by LLNL and are not to be used for quantitative model development. To account for probe drift[8], LLNL made some minor adjustments to its data based on the bi-thermal method results and these are recorded in

[7] LLNL, Susan Carroll, "RH Probe and Experimental Timeline," UCRL-AR-218422, March 1, 2006.
[8] Drift is a progressive (continuously upward or continuously downward) change in the number displayed on the readout of a piece of test equipment.

the Data Tracking Number "read me" files that include the adjusted and non-adjusted results. The adjusted probe readings may still be considered to be provisional calibration results, and the resulting data files are marked that they can only be used for qualitative purposes above 120 °C.

Because LLNL and BSC have not formally defined the uncertainty budgets[9] associated with these methods, the confirmatory results have not yet been included in the formal calibration associated with the temperature and humidity probes for data used for quantitative purposes. The ISO Standard 17025:2005 - *General Requirements for the Competence of Calibration and Testing Laboratories* may be useful in defining the uncertainty budget associated with this activity.

Until the uncertainty budgets are fully established, the Independent Review Team recommends that the Vaisala and Thunder Scientific Corp. calibrations be considered the formal calibrations, with the bi-thermal and salt tests providing further confirmation of the high-temperature probe response and uncertainty. Removal of the provisional status for results from the LLNL bi-thermal system should await completion of the uncertainty budget.

[9] An uncertainty budget is a list of components contributing to the uncertainty of a measurement, with a numerical value given for each component. An uncertainty budget may be organized according to the methods used to characterize the components (e.g., statistical analysis of repeat measurements), or it may be organized according to the possible causes of error (e.g., uncertainty of temperature measurement). In either case, the components listed in the budget should cover all significant sources of possible error in the budget.

Figure 1. Timeline for Use of Vaisala Probes

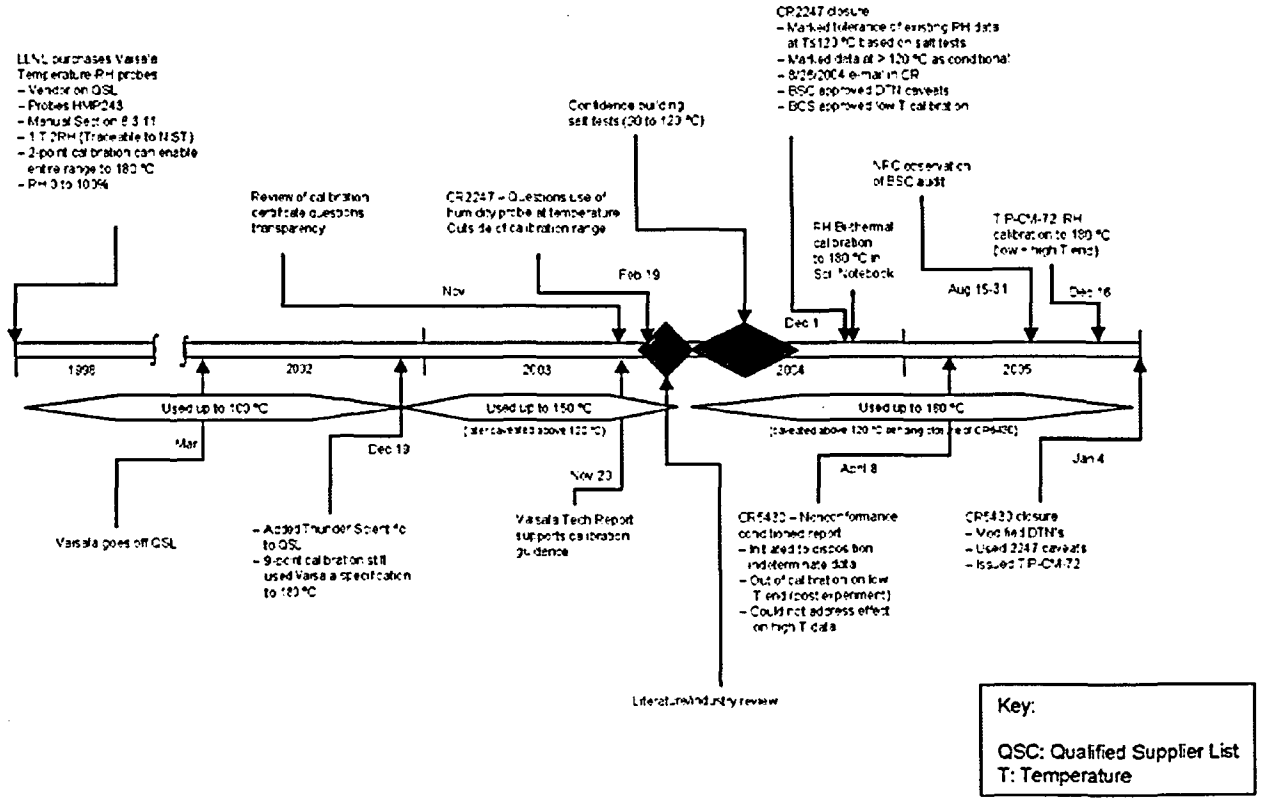


Table 1. Calibration History for Vaisala Probes

Dates	Calibration Approach
Period 1. 1998 to March 2002	
November 1998 – February 2002	• BSC Qualified Supplier's List included Vaisala as manufacturer of relative humidity/temperature probes and provider of associated calibration services. • LLNL purchase order (November 5, 1998) for 3 Vaisala models specified the following: "Transmitter, Digital Display, 0-1 volt outputs (temp & hum), Temp. in degrees C / Humidity in %RH [relative humidity], Temp. Range -40 °C to 180 °C, NIST Certificate. The above equipment shall comply with the attached Buyer's Q.A. Requirements". • Vaisala used NIST-traceable 2-point methodology for calibration (2 relative humidities at least 50% apart at room temperature). • Operating manual for Vaisala's HMP243 probe[10] specified a range of temperatures from -40 °C to 180 °C. Vaisala's guidance was that with annual calibration, probes could be used for experiments up to 180°C and be within a tolerance of 0.025 x % RH + 0.5% RH.[11] • Experiments during this period did not exceed 100°C. LLNL relied on information provided by Vaisala for the calibration process.
February 2002 – March 2002	• BSC's routine supplier audit confirmed Vaisala's effective implementation of quality assurance requirements in prior Yucca Mountain work. • Vaisala changed its quality assurance program to conform to International Standards Organization (ISO) 9000 standard rather than NQA-1 standards specified in QARD. • BSC removed Vaisala from Qualified Supplier List for future work.

[10] Vaisala Oyi, U145en-2.1, HMP243 Transmitter Operating Manual, May 1998.
[11] The tolerance is the manufacturer's assurance that the instrument will read the true value to within the stated limits. From their literature, it is not clear what level of confidence the stated accuracy refers to.

Table 1. Calibration History for Vaisala Probes, continued

Dates	Calibration Approach
	Period 2. Late 2002 to Late 2004
December 2002	• BSC included Thunder Scientific Corp. on Qualified Supplier List for calibration services for relative humidity/temperature probes. • LLNL replaced Vaisala's 2-point (2 humidities at 1 temperature) calibration at room temperature with 9-point (3 humidities at 3 temperatures) NIST-traceable calibration using 3 temperatures (25 °C, 40 °C, and 60 °C) and 3 relative humidities (15%, 50% and 85%). • Thunder Scientific Corp. used 9-point method for calibrating LLNL's probes. • LLNL continued to use the calibrated probes in 2003 to perform experiments at temperatures up to 150 °C, following Vaisala's specification and guidance that its probes calibrated at low temperature accurately measured relative humidity within a specified tolerance up to 180 °C.
November 2003 – February 2004	• LLNL and Thunder Scientific Corp. expressed concerns regarding use of low-temperature calibration techniques at higher temperatures (i.e. >60 °C). • LLNL obtained a Technical Notice, "Product Performance Validation," from Vaisala (November 20) that supported Vaisala's specifications and indicated that probes calibrated at low temperature could be used up to 180 °C. Vaisala would not provide additional, proprietary information.[12] • Both NIST[13] and Thunder Scientific Corp. expressed reservations to LLNL about the Vaisala Technical Notice.

[12] In response to an inquiry from BSC in early 2006, Vaisala released without restrictions three Test Reports dated 11/14/2001, 2/19/2003, and 6/6/2003 on the model HMP233 Humidity & Temperature Transmitter, the pilot model for Vaisala's Model HMP243 probe. The report dated 2/19/2003 indicated that the HMP233 probe was tested over the temperature range from 120 °C to 180 °C.

[13] Dr. Peter Huang provided short consultations by telephone to LLNL staff during development of their calibration process.

Table 1. Calibration History for Vaisala Probes, continued

Dates	Calibration Approach
November 2003 – February 2004, continued	• Without additional information to verify Vaisala specifications and guidance, LLNL initiated CR 2247 (February 19),[14] to identify other calibration options above the 60 °C range. • CR 2247 included recommendations that: 1. LLNL write a report on probe performance at higher temperatures based on simple equilibrium salt tests. 2. BSC review LLNL's report and provide guidance on documenting data collected at higher temperatures.
March 2004 – July 2004	• LLNL conducted equilibrium salt tests in response to CR 2247, which calculated that the probes calibrated to 60 °C were accurate to ±1.6% relative humidity (1σ) from 90 °C to 120 °C. • As part of the corrective action plan for CR 2247, LLNL proposed to: 1. Fabricate a 2-pressure system for confirming probe calibration at higher temperatures 2. Extend calibration confirmation by measuring sensor response at elevated temperatures using the known water vapor pressure of saturated salt solutions.
August 24, 2004 – September 2004	• Corrective actions proposed by LLNL would have required funding beyond what was allocated to LLNL for that Fiscal Year, so BSC notified LLNL that additional funding would need to be requested in the next fiscal year. BSC considered values above 120 °C to be "conditional" because they could not confirm the accuracy of relative humidity measurements at temperatures above 120 °C. • Caveats were required to be added in relevant data tracking number (DTN) "read me" files for any test above 120 °C in the Technical Data Management System (TDMS) where experimental data are recorded. • LLNL conducted a series of deliquescence experiments using resistivity techniques on equilibrium mixed salts to show that brines form from 120 °C to 180 °C.

[14] OCRWM, CR 2247, "M&TE Used Outside the Range of Calibration," February 19, 2004.

Table 1. Calibration History for Vaisala Probes, continued

Dates	Calibration Approach
October 2004 – December 2004	• Based on the equilibrium mixed salt test results, CR 2247 was closed (December 1), with the requirement that relative humidity data at temperatures greater than 120 °C, be recorded as "conditional" with appropriate caveats in Data Tracking Number (DTN) "read-me" files. • The primary method for calibrating the probes was the 9-point method by Thunder Scientific Corp. and application of tolerance limits established in CR 2247. LLNL's results independently supported Vaisala's calibration process and guidance as noted in December 2002 (with some additional small corrections needed because of drift). • LLNL and Thunder Scientific Corp. began development of a bi-thermal method for confirmation of probe response from 115 °C to 180 °C.[15]
	Period 3. 2005 to Present
April – December 2005	• LLNL initiated CR 5430 (April 8) to examine the potential impact of the probe calibration processes on earlier data collected with the probes. • LLNL completed development of the bi-thermal method for confirming response of relative humidity/temperature probes from 115 °C. to 180 °C, pending establishment of formal uncertainty budgets associated with this method. • Both pre- and post-test confirmation measurements were made on relative humidity/temperature probes used in deliquescence and corrosion experiments at elevated temperatures (and documented in scientific notebook LLNL-SCI-487-V.3, as well as in the TDMS). • LLNL reported that uncertainty associated with virtually all earlier relative humidity measurements, up to 120 °C, was ±1.6% relative humidity (one standard deviation).

[15] This activity became part of the work performed under action number 5430-004 in the corrective action plan for CR 5430, "Data Acquired by Vaisala Temperature/Humidity Probes Above 90 Degrees Celsius Is Indeterminate.

Table 1. Calibration History for Vaisala Probes, continued

Dates	Calibration Approach
April – December 2005, continued.	• LLNL decided that the combined bi-thermal confirmation and low-temperature 9-point calibration methods yielded relative humidity measurements from 25 °C to 180 °C that could be used without caveats. • However, BSC required that measurements above 120 °C be treated as conditional. LLNL added caveats to the DTNs for measurements above 120 °C, pending closure of CR 5430. • Technical Implementation Procedure (TIP) CM-72[16] for routine use of bi-thermal calibration method for 115 °C to 180 °C was approved by LLNL (December 16).[17]
January 2006	• CR 5430 was closed (January 4) based on documentation that: 1. Measurements up to 180 °C that included pre- and post-test calibrations no longer needed caveats. 2. Calibration of probes used in earlier tests was within the tolerance established at 120 °C. 3. Values obtained with probes above 120 °C prior to development of bi-thermal calibration method were designated as "conditional" values by caveats in the DTN "read me" files. • LLNL informed BSC (January 20) that it voluntarily suspended use of Vaisala relative humidity/temperature probes pending resolution of issues raised by NRC.
February 2006	• BSC QA Manager issued a stop-work order to BSC on the use of the Vaisala probes (February 7). LLNL acknowledged the stop-work order the same day it was formally issued by BSC to LLNL (February 10).

[16] LLNL, TIP-CM-72, "Calibration of RH Meters Using the Bi-Thermal RH Calibration System."
[17] Dr. Susan Carroll of LLNL indicated that all bi-thermal tests at LLNL were conducted at temperatures up to 180 °C. This TIP refers to an upper temperature of 160°C at one point but refers to 180 °C in all other locations.

NIST Evaluation of the LLNL Relative Humidity and Temperature Probe Calibration Program

As part of this independent review, Dr. Peter Huang of NIST evaluated the approach and technical adequacy of the LLNL calibration processes used since 1998. The evaluation was conducted in accordance with the general guidelines of Sections 5.2 to 5.10 of ISO Standard 17025:2005 - *General Requirements for the Competence of Calibration and Testing Laboratories.* Although LLNL was not required to conform with ISO 17025, for the present evaluation ISO 17025 provides a broadly accepted guide to good practices for calibration laboratories.

Observations from Dr. Huang's evaluation of the calibration methodology currently used by LLNL include:

1. The basic physical principles of LLNL's bi-thermal method are well established, and the choice of methodology was reasonable.

2. The design and operation of the equipment used for the bi-thermal calibration are adequate to the task.

3. Out of the twenty requirements of ISO 17025:2005, sixteen requirements were met (i.e., "In Conformance"), two were not met (i.e., "Out of Conformance"), and with respect to two requirements, NIST had comments noting that minimum standards were met, but recommended improvements to provide a better evaluation of variance or uncertainty.

4. The two "Not in Conformance" requirements were:

   - The determination of the uncertainty budget was not conducted with sufficient rigor, such as described in the *ISO Guide to the Expression of Uncertainty in Measurement.*

   - Quality control procedures for monitoring the validity of tests and calibrations undertaken have not been established.

The amount of additional work needed by LLNL on relative humidity/temperature probe calibration depends on the uncertainty that is acceptable to LLNL and BSC for use in quantitative model development. During February 2006, when the Independent Review Team asked for the allowable uncertainty for use in scientific work, it was reported that LLNL and BSC do not have defined requirements in this area for the quantitative model inputs and analyses. Rather, LLNL had been satisfied when the results fell within the range of ±1.6% relative humidity (one standard deviation). NIST has conservatively estimated the uncertainty of relative humidity measurements using the Vaisala probes under the manufacturer's calibration specifications as ±7% at a level of confidence of 95% (equivalent to approximately ±3.5% at the one standard deviation level of confidence); implementation of the bi-thermal approach brought

this conservative estimate of uncertainty down to 6% (equivalent to approximately ±3% at the one standard deviation level).

On April 4, 2006 the BSC Manager of Post Closure Activities provided a memo identifying the acceptable uncertainty for the LLNL humidity probes. The acceptable uncertainty was based on an analysis of how the data is used in the relevant AMRs. The memo stated that the uncertainty of relative humidity/temperature probes needs to be demonstrated to ±5% RH at 180 °C to be sufficient for their modeling processes. The confidence level used by BSC to define the ±5% was not specified.

Conclusion

Based on discussions with LLNL personnel and Dr. Huang's evaluation, it is the Independent Review Team's conclusion that LLNL's technical approach to calibration of the relative humidity/temperature probes was sound. LLNL's concern about the scientific adequacy of the guidance given by Vaisala motivated them to implement improvements in their calibration approach.

3.2: Was the calibration performed in accordance with Yucca Mountain procedures and appropriately documented?

The QARD establishes requirements for the control, calibration, and maintenance of measuring and test equipment (M&TE) used on the Yucca Mountain Project. With regard to calibration, section 12.2.1A of the QARD requires that M&TE "shall be calibrated, adjusted, and maintained as a unit at prescribed intervals, or prior to use, against reference calibration standards having traceability to nationally recognized standards." The QARD also requires that in cases where no recognized standards exist, "the basis for calibration shall be documented." Additionally, the QARD requires tagging, segregation, and documentation of out-of-calibration M&TE (including equipment where the due date for recalibration has passed), and does not allow M&TE to be used pending recalibration. The QARD does not have provisions for allowance of deviations or exceptions to the requirements for out-of-calibration M&TE. The QARD provides specific requirements for the content of documentation related to calibration. The QARD requirements related to calibration of M&TE are implemented through a BSC procedure, LP-12.1Q-BSC, *Control of Measuring and Test Equipment.*

The Independent Review Team identified several issues related to adherence to procedures and proper documentation. Each of these is discussed below.

Use of Probes Beyond Calibration Effective Date

The Independent Review Team found that prior to 2004, the M&TE control procedure required: (1) an "Out of Service" tag to be placed on M&TE that was out of calibration, (2) the equipment to be segregated, and (3) an Out-of-Calibration Report to be completed. However, BSC revised

the procedure in 2004[18] based on input from LLNL, to allow for use of "other similar tags" as alternatives to the tag specified in the procedure (the other two requirements were unchanged).

Based on interviews with LLNL staff, LLNL followed the revised procedure and placed a limited-use tag on the probes that had exceeded their calibration date, but it is not clear whether this was in place during the time of the BSC audit. The Independent Review Team verified that Out-of-Calibration Reports are routinely issued at LLNL for M&TE that has gone beyond its calibration time limits. Because the wording of the procedure had been changed to allow the use of "other similar tags," and because the rationale for the revision had included an indication that LLNL used M&TE after its recalibration due date in certain circumstances, LLNL staff believed that their practices with regard to M&TE were in compliance with LP-12.1Q-BSC.

The change made in 2004 to LP-12.1Q-BSC does not properly implement requirements in Section 12.2.3 of the QARD. The QARD does not allow for use of M&TE that has exceeded recalibration due dates. BSC, the owner of the procedure, did not recognize during the procedure revision process that the use of M&TE past its recalibration due date is not allowed under the QARD. LP-12.1Q-BSC was again revised in October 2005, but retained the reference to "other similar tags,"[19] and therefore still does not properly implement the QARD. (See Appendix D for the history of changes to LP-12.1Q-BSC.)

NRC's Audit Observation Report also identified a procedural compliance issue involving the use of calibrated probes beyond their calibration effective date:

> "Out of calibration measuring and test equipment were not controlled. The Vaisala humidity probes identified in CR 2247 were not tagged, segregated, nor otherwise controlled to prevent use until they had been calibrated. This is a requirement of section 12.2.3B of the QARD."

The NRC concern is substantiated because LLNL continued to use calibrated probes after their calibration effective date for ongoing experiments that exceeded the annual calibration cycle. There is no provision in the QARD for continued use of M&TE after the calibration due date.

Documentation in Scientific Notebook

In the Observation Audit Report, NRC stated that, "no explanation or notation was included in the scientific notebook identifying the humidity probes as not being calibrated at the high range of temperatures being used in the experiments." NRC specifically referred to Scientific Notebook SCI-484, Volume 3 in making this comment.

In researching this issue, the Independent Review Team discovered that NRC had examined Scientific Notebook SCI-484, Volume 3[20] during the audit, but the information NRC was

[18] BSC, AP-12.1Q, Control of Measuring and Test Equipment, Revision 0, ICN 3, effective date April 30, 2004, approved by the Repository Development Manager.
[19] BSC, LP-12.1Q-BSC, Control of Measuring and Test Equipment, Revision 0, ICN 1, effective date October 25, 2005, approved by the Deputy Manager, Post-Closure Activities.
[20] LLNL, SN-LLNL-SCI Number 484, Volume 3, "Deliquescence of Mixed Salts; Measurements of Deliquescence of Relative Humidity (DRH) and Determination of Boiling Point from DRH Values," notebook initiated August 3, 2004.

seeking was actually in Scientific Notebook SCI-487, Volume 1.[21] However, Scientific Notebook SCI-487, Volume 1 was not provided to NRC at the time of the audit.

Approval of Documentation of the Bi-Thermal Calibration Method

The Independent Review Team reviewed Scientific Notebook SCI-487, Volume 1, and found that it contained full documentation of the bi-thermal process, including pictures of the equipment set-up. LP-12.1Q-BSC provides the requirement for documenting and approving the development of methods to calibrate M&TE where no nationally recognized standards are available. Documentation can be provided either in a scientific notebook, or on an M&TE Justification form that is provided as Attachment 3 to the procedure. If a scientific notebook is used, LP-12.1Q-BSC requires that the documentation provide, at a minimum, the information called for on the M&TE Justification form, including review and approval of the calibration method by a Principal Investigator, Responsible Manager, or Engineering Test Lead. While the requirement for approval is not explicitly stated in the text of LP-12.1Q-BSC Section 5.2, it can be inferred from the information requirements of the M&TE Justification form.

LLNL documented the development of the bi-thermal calibration method in Scientific Notebook SCI-487, Volume 1. However, there is no indication in the notebook of the required approval prior to the process being used. Therefore, this documentation did not meet the requirements of LP-12.1Q-BSC.

When the TIP for the bi-thermal method was approved on December 16, 2005, by the Responsible Manager of the pertinent technical area at LLNL, it met the approval requirement for an alternative calibration process under LP-12.1Q-BSC.

Conclusion

On the basis of discussion with LLNL personnel, the Independent Review Team determined that the LLNL scientific notebooks, along with the data in the TDMS, provide a satisfactory level of documentation of the bi-thermal calibration method. In examining the notebooks, the Independent Review Team found a procedural violation with regard to documenting the required approval for the development of the bi-thermal approach to calibrate the probes. The TIP issued in December 2005, however, was approved by the Responsible Manager.

[21] LLNL, SN-LLNL-SCI Number 487, Volume 1,"Research Supporting Environmental Chemistry Experiments," notebook initiated December 20, 2004.

3.3: Why was the calibration issue not recognized as a quality issue by BSC management and included in the BSC audit findings?

The relative humidity/temperature probe calibration issue was not recognized as a quality assurance issue by BSC management due to multiple technical considerations as well as attitudinal and interpersonal issues.

The technical considerations which led to the lack of recognition of the calibration issue as a quality assurance issue by BSC management include:

- Differences in expectations regarding the scope of the audit and in the definition of terms between NRC representatives and BSC QA personnel began before the audit was initiated. NRC had expected the entire audit to be "performance-based" (i.e., determining the technical adequacy of processes and products), while BSC focused mainly on the compliance issues within the scope of the audit.

- Based on interviews, the Independent Review Team determined that the BSC audit team did not consider the NRC's concern about the use of the relative humidity/temperature probes outside of the designated temperature range to be a problem.

- The BSC audit team thought that they had adequately and successfully evaluated the M&TE area based on questions identified on the audit checklist.

- The BSC audit team did not have expert consultants or other BSC technical staff present at LLNL during the audit to recognize and review issues in greater detail.

- As part of the audit planning process, the BSC QA management and the audit team did not review past CRs for trend information concerning M&TE problems. The Independent Review Team's review of the Quarterly Trend Reports found that they do not provide useful information regarding specific technical areas associated with the CRs.

Conclusion

BSC management clearly missed opportunities to address NRC concerns, which were repeatedly articulated by NRC staff that observed the audit. The relative humidity/temperature probe concerns identified by NRC were not adequately addressed by BSC during the audit period and afterwards. The lack of BSC technical personnel participating in the portion of the audit conducted at LLNL contributed to the lack of recognition of the gravity of the NRC's concerns. The BSC QA audit team assumed that the LLNL technical issues brought to their attention by NRC during the audit were adequately addressed in CRs and did not require further action. Also, BSC's focus on the audit checklist contributed to NRC's concerns not being escalated and addressed.

Both DOE and BSC need to continue to reinforce a nuclear culture that respects the regulatory status of the NRC and improve communications in its audit functions and other interactions with the regulator.

3.4: Why was line management not included in audit activities at LLNL?

BSC line management was not included in audit activities at LLNL due in part to changing practices with regard to participation of line management in QA audits. During previous BSC QA audits, a licensing representative participated in all audits. This changed over time, to where QA would request support from the BSC line organizations only when deemed necessary.

Conclusion

The lack of involvement of line management contributed to the BSC QA staff not recognizing the significance and impact of NRC's concerns during the audit.

The Independent Review Team was informed that, as a result of the issues arising from the August 2005 audit, a representative of the BSC licensing organization will participate in future audits conducted by BSC QA staff.

3.5: With regard to the overall organizational culture, what is the ability of the organizations to work to standards and requirements, to stop work as required, and to understand quality assurance requirements and take appropriate actions?

Based on interviews and a review of documents, it appears that OCRWM and BSC generally work to the implementing procedures and stop work if needed. During the review, it was noted that BSC issued a stop-work order for the LLNL experiments using the suspect humidity probes, and prior to that stop-work order LLNL had voluntarily stopped this work.

From the interviews, it is apparent that staff in OCRWM, BSC, and LLNL believe they have adequate quality assurance training and understand the requirements of quality implementing procedures. However, the issue with allowing "similar" tagging of M&TE past recalibration due dates and the revision of LP-12.1Q-BSC indicates that BSC does not consistently review procedure revisions against the QARD requirements.

Conclusion

OCRWM and BSC have the responsibility to work to standards and the ability to stop work as necessary. LLNL and BSC both took stop-work actions in connection with subjects of the audit addressed in this report.

4.0 OTHER FINDINGS

A. LLNL staff did not misrepresent calibration status in scientific notebooks.

In the NRC Observation Audit report, dated January 9, 2006, the "NRC determined that LLNL had not complied with the requirements of Section 12.0 of the QARD related to the performance of corrosion rate experiments. Specifically, it was concluded that LLNL had initiated these experiments: (i) without properly calibrated instruments; (ii) had not established an adequate basis for calibration when nationally recognized standards were unavailable; and (iii) misrepresented the correct status of the calibration of the humidity probes in the associated scientific notebook."

The Independent Review Team focused closely on the allegation of "misrepresentation," since that term is defined as the intent to mislead or deceive, as distinguished from inaccuracy. The Independent Review Team asked the NRC audit observation team to provide them with the pages in the LLNL Scientific Notebook that indicated LLNL misrepresented the correct status of calibration. NRC indicated that pages 19 and 42 of LLNL-SCI-484-V.1 were used in making that determination. They also indicated that additional entries on pages 58, 59, and 60 were entered after the on-site audit and were of a similar nature. The initial entries are reproduced below.

LLNL-SCI-484-V.1

Page 19 Entry Dated and Signed 8/13/04-
"RH & Temp was measured using a HMP243 S/N Y4750012 Cal due 1/28/05 Probes above cell were from an HMP243 S/N ~~A135733~~ (US110014 signed 8/16/04) Cal due 6/03/05"

Page 42 Entry Dated and Signed 2/16/05-
"Installed Vaisala HMP243 probe (RH) S/N T4610030 Cal due 4/21/05. Connected output of RH probe to Gamry PSTAT cards"

As part of the independent review, the OCRWM Program Manager for Independent Evaluations discussed the issue of misrepresentation with the NRC signatory of the report. This NRC manager stated that the NRC was concerned about the fact that the probes had been calibrated at lower temperatures than the ranges expected in experiments, and that the absence of disclosure of this fact in the scientific notebook meant that the true state of calibration had not been accurately represented. He stated that he recognized the difference between inaccuracy and willful misrepresentation, and that the Observation Audit Report had not intended to suggest willful misrepresentation.

The Independent Review Team has also conducted interviews with managers and staff at LLNL and found no intent to misrepresent or falsify scientific notebook entries and in particular, the calibration of relative humidity/temperature probes. As evidenced in this report, it is clear that

the LLNL staff had performed their calibrations from 1998 to 2002 in accordance with manufacturer's specifications and that thereafter they continued to modify and improve the calibration process through 2006. These modifications included the implementation of a 9-point NIST-traceable calibration performed by Thunder Scientific Corp. from 2002 through 2004 and the addition of the bi-thermal calibration method for calibrating the probes from 115 °C to 180 °C after 2004.

The entries identified by the NRC observers in LLNL-SCI-484-V.1 reference the calibrations by Thunder Scientific Corp. that were performed in 2004 and 2005. As discussed in this report the Independent Review Team and NIST have found the calibration processes performed by LLNL to be sound, since there are no national standards for comparison at high temperature and high relative humidity.

LLNL-SCI-484-V.1 references the calibration processes for Thunder Scientific Corp. and indicates when the calibration was to be performed. The Independent Review Team found no evidence of any intent to mislead a reader.

B. No quantifiable requirements are in place before work is performed.

The Independent Review Team noted in documents and discussions with LLNL staff that statements were made such as "The probes could be used for experiments at temperatures up to 180 °C and be within a tolerance of [0.025 x %RH + 0.5% RH]" and "With some additional small correction for drift, the values of RH were accurate to ±1.6% RH unit at 1-sigma (one standard deviation.)" These tolerances resulted from statistical analyses of experimental results, rather than as design requirements for the experiment.

It appears that experiments were not designed with the specific tolerances in mind. The values for tolerance are dependent upon such factors as the equipment or technology used in the measurement; the variability of the properties of the materials involved in testing; and the care with which the experiment was carried out. Such an analysis may dictate the use of different or more precise measuring equipment. An analysis of the impact of precision (smallest measurable quantity) and accuracy (difference between measured value and true value) on the model results should guide the choice of instrumentation and methodology used.

On April 4, 2006, in response to the Independent Review Team's inquiry, the BSC Manager of Post-Closure Activities provided a memo stating the acceptable variance for the LLNL relative humidity/temperature probes. The acceptable variance was based on an analysis of how the data is used in AMRs. The memo stated that the accuracy of the probes needs to be demonstrated to ±5% relative humidity at 180 °C to be sufficient for the models.

The NIST report indicated that since the end of 2004, the calibrations obtained from Thunder Scientific Corp. and LLNL were internally consistent, and that a conservative estimate of the uncertainty of sensor measurements in the environment used is ±6% relative humidity at 180 °C (95% confidence limit). In addition, NIST defined a ±7% uncertainty estimate for the earlier time period. These values need to be compared on a common basis so LLNL can take steps to

provide the quality of data that is needed as inputs to quantitative models to predict corrosion rates. Work is needed to clarify the level of confidence of the BSC requirement, so that the acceptability of the LLNL results can be determined.

There is a lack of specific requirements defining data needs for the LLNL deliquescence tests. Not having specific data uncertainty requirements may necessitate re-running experiments to appropriately address uncertainty. Without defining acceptable levels of uncertainty in advance, OCRWM and BSC cannot make informed commitments of resources to focus on the most important data needs.

C. Response to AOIs is not timely.

To date, DOE has not provided a response to NRC on the AOIs they issued in August 2005 and revised in December 2005. The State of Nevada and Clark County each submitted an AOI in August 2005. The Department responded to the State of Nevada AOI on June 6, 2006, and has not yet responded to the Clark County AOI.

The BSC audit report does not discuss the AOIs from either NRC or the state and county observers. BSC has begun preparing a response for DOE. The Communications group at OCRWM is now responding to the State and county AOIs, but did not receive them until OLAS-RIS provided them the first week of February. Consideration should be given to reevaluating this process to allow for more expeditious responses to these inquiries and ensure that technical staff are involved in the process.

This page intentionally left blank

Appendix A:
NIST REPORT

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-

May 26, 2006

Mr. Gene E. Runkle
Office of Civilian Radioactive Waste Management
U.S. Department of Energy, RW-1
1000 Independence Ave., SW
Washington, D.C. 20585

Dear Mr. Runkle:

Dr. Peter Huang has completed a technical evaluation of humidity probe tests and calibrations at LLNL, conducted at your request. Enclosed is the final version of the report, which I have reviewed and discussed with Dr. Huang.

Sincerely,

Dr. Dean Ripple
Leader, Thermometry Group
Process Measurements Division
phone: 301-975-4801
fax: 301-548-0206
e-mail: dean.ripple@nist.gov

NIST

Summary of Quality Evaluation of Humidity Probe Tests and/or Calibrations at LLNL

Date of Evaluation: February 15 and 16, 2006
Technical Evaluator: Dr. Peter Huang, NIST

Introduction

I met with the staff members of LLNL and visited their laboratories to assess the technical requirements of the humidity probe calibration activities. Evaluations were made according to the general guidelines of Sections 5.2 to 5.10 (Technical Requirements) of the ISO 17025:2005 Standard "General Requirements for the Competence of Calibration and Testing Laboratories." The LLNL laboratories being evaluated are expected to meet internal LLNL requirements and adhere to LLNL procedures. LLNL has not declared or claimed conformance with ISO 17025. For the present evaluation, 17025 provides a broadly accepted guide of good practices for calibration laboratories.

As a brief technical background, the experiments conducted by LLNL require humidity measurements at temperatures that are not well supported by national measurement standards. I know of no national or commercial laboratories that are presently calibrating humidity probes or providing physical reference standards at temperatures above 100 °C—well below the maximum usage temperature of 180 °C. Additionally, there are no available standardized procedures for producing known moisture concentrations at high temperatures. For example, the ASTM standard E104-02: "Standard Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions" for calibrating humidity sensors has an upper temperature limit of 80 °C. This situation has placed a significant burden on the LLNL staff of calibrating the humidity probes using LLNL-developed equipment and methods. For LLNL to demonstrate calibration of their probes was, and continues to be, a difficult challenge due to the absence of metrological standards and resources relevant for their usage temperature.

During my interview, I assessed the calibration process against Sections 5.2 to 5.10 of the Standard. The results of the evaluation according to the designated number in the ISO 17025:2005 Standard are given below. For an actual 17025 assessment, only the items listed as "Out of Conformance" require mandatory corrective action. If a "Comment" is given in lieu of "In Conformance" or "Out of Conformance," the comment may discuss issues or weaknesses identified by the assessor, but the assessor has judged that the minimum requirements of 17025 have been met.

Understanding that the validity of the humidity sensor calibrations may affect a large body of data that would be expensive to replicate, the section Estimate of Confidence Limits includes additional assessments that exceed the scope of ISO 17025. In particular, I have estimated confidence limits on the calibrated humidity sensors. These estimates are based only on my appraisal of the LLNL experiments and on my best technical judgment. Additional measurements on the sensors in question and additional work on establishing the uncertainty of the LLNL Bi-thermal Calibration System could refine these limits substantially.

Summary

The staff of LLNL working on this project display a good working knowledge of the principles of humidity measurements. The environment and facilities are appropriate for the work they were performing, and documentation of the procedures and equipment was available.

There are two key technical issues:

1. Were the humidity probes appropriate for an operating temperature of 180 °C?
2. Were the humidity probes properly calibrated at these temperatures, given the inability of secondary calibration laboratories or NIST to supply this calibration?

For the first question the LLNL staff produced documentation from the sensor manufacturer, Vaisala, which clearly stated that the sensors were designed for operation up to 180 °C. Overall I found the Vaisala HMP243 humidity/temperature probes were used properly. According to Vaisala's specifications, the probes could be used to 180 °C within a tolerance of 2.5 % of the reading + 0.5 %RH. LLNL therefore used probes in a manner consistent with the manufacturer's recommendations and specifications. Using an HMP233 sensor (serial number 623074), which is identical to the HMP243 sensors used by LLNL except for communications and software features, Vaisala conducted internal tests in 2003 (report dated 2/19/2003) that demonstrated reliable operation of the HMP233/243 series at temperatures up to 180 °C with 6 %RH, but with deviations from the expected values as high as 2.1 %RH.

Regarding the second question, a variety of calibration sources were used. In the period of 1998 to March of 2002 the Vaisala probes were calibrated annually by Vaisala Testing Laboratory located in the United States, along with its in-house reference probes traceable to NIST, for the range from 18 % to 90 % relative humidity values at 23 °C. Corrections were applied for observed drifts in accordance with the manufacturer's recommendations. This calibration protocol was in accordance with the manufacturer's recommendations for calibration of these units.

Beyond the end of 2002, the probes were calibrated by Thunder Scientific at nine points in the range of relative humidity from 15 % to 85 % in the temperature range from 25 °C to 60 °C. Thunder Scientific is a National Voluntary Laboratory Accreditation Program (NVLAP) accredited laboratory. NVLAP adheres to all relevant international standards for accrediting bodies and is one of several U.S. signatories to the Mutual Recognition Arrangement of the International Laboratory Accreditation Cooperation (ILAC). This calibration protocol, covering a larger range in temperature and a nearly equivalent range in relative humidity values, is also consistent with manufacturer's recommendations for calibration of these units.

Since national humidity standards are not available at high temperatures, LLNL decided to develop a bi-thermal method for calibrating humidity probes for 115 °C to 180 °C at the end of the year 2004. The method is based on the diffusion of water vapor produced by boiling water at 100 °C in a glass vessel to another vessel maintained at a higher temperature. In this bi-thermal equilibrium system, the water vapor pressure in the lower temperature vessel is assumed to be equal to that in the higher temperature one. The basic physical principles of their method are well established, and their choice of this method is very reasonable. The construction and operation

of the apparatus are adequate to the task as well. LLNL made required corrections for the drifts over time when the probes were used at high temperature conditions.

Improvements are needed in two areas specifically addressed in ISO 17025. First, the validation of the bi-thermal method is found to be in conformance with the minimum standards of ISO 17025, but establishment of the validity of the method to a high degree of confidence requires some additional work. Second, the calculation of the uncertainty for sensor measurements was found to be out of conformance with ISO 17025, and mandatory corrective action is needed. Ideally, the performance of the LLNL Bi-thermal Calibration System would be evaluated by an interlaboratory comparison of humidity probe calibrations at a full range of temperatures. The absence of other laboratories capable of reaching 180 °C excluded this path to demonstrate validity of the LLNL methods and apparatus. But a number of confirming measurements are possible, even in the absence of other laboratories performing similar work at 180 °C, including:

a. tests of the Vaisala sensors over multiple runs and over an extended time, to assess the repeatability of the test and any possible drift of the sensors;
b. comparison of the sensor probe calibration in the range 25 °C to 60 °C by Thunder Scientific with calibrations conducted using the LLNL Bi-thermal Calibration System;
c. study of the dependence of the sensor probe calibrations on such systematic properties of the Bi-thermal Calibration System as water level, heating power, and rate of gas injection; and
d. determination of the uniformity of temperature in the apparatus.

LLNL staff did implement tests b. and d. above. Mandatory corrective action is needed to establish a rigorously derived uncertainty budget, such as described in the *ISO Guide to Expression of Uncertainty in Measurements*. Relative humidity values measured with probes calibrated by Thunder Scientific were reported to be in agreement with those obtained from the Bi-thermal Calibration System. The thermal uniformity of the apparatus was checked using a NIST-traceable thermometer. To a lesser extent, test a. has been implemented, although the number of runs should have been higher, and the repeatability of multiple runs confirmed. Data already acquired with the Bi-thermal Calibration System could very well be useful in establishing the repeatability of the system without further measurements. Probe drift over several calibration cycles with the Bi-thermal Calibration System ranged from –4 % to 1 % relative humidity at 115 °C and –2 % to 0 % at 180 °C.

To complement the measurements that LLNL has already done, a more rigorous set of repeatability measurements should be made, along with a more rigorous determination of the uncertainty of calibrations with the Bi-thermal Calibration System. Relative humidity values were reported to be accurate to 1 % (one standard deviation) once the required corrections were made. An allowance for probe drift, which is comparable to the 1 % claimed uncertainty, should be added as a component of the uncertainty budget.

I note that ISO 17025 states that sensor drift is not normally included by the calibration laboratory in the uncertainty budget; the calibration uncertainty is only a record of the uncertainty at the time of calibration. If that methodology is adopted, the end user would need to add in the drift component separately. From a technical point of view, it only matters that probe drift be accounted for, by either the calibration laboratory or the end user, and that the users of

the probe have a clear understanding of the uncertainty attainable. We recommend that LLNL procedures identify which party (commercial calibration laboratory, LLNL calibration laboratory, or end user) should be responsible for incorporation of sensor drift allowances into the overall uncertainty of measurement.

Until the uncertainty of the Bi-thermal Calibration System is firmly established, it is recommended that the results from the Bi-thermal Calibration System be treated as provisional.

As an aside, information on the extrapolation of calibration results may prove useful in considering the calibration status of the LLNL probes. In some areas of thermophysical measurements, extrapolation is the norm. Examples are the scaling of viscosity by many orders of magnitude from absolute determinations of the viscosity of water, or the scaling of piston gage response from manometer measurements of a few atmospheres pressure. In other areas, the device being calibrated is never extrapolated, either because extrapolation is not needed or because extrapolation is known to fail. In short, there is no consensus on whether extrapolation is a valid method, and the validity of extrapolation must be considered on a case by case basis. When extrapolation is used, there needs to be firm evidence that the extrapolation process is reproducible from sensor to sensor. In the LLNL case, the situation is not black and white, but gray. On the one hand, extrapolation was favored because a) the manufacturer had proprietary knowledge that their sensor would work with extrapolation, b) the sensor itself is very simple and would be expected to be generally repeatable, and c) the lack of high-temperature standards meant that avoiding extrapolation was technically challenging. On the other hand, extrapolation was discouraged because there was no body of knowledge in the open literature justifying extrapolation.

Conformance to Specific Points of ISO 17025

5.2.1a. Personnel. Staff members are competent in performing humidity tests and/or calibrations, evaluating results. *In conformance.*

5.3.1a. Laboratory facilities. Laboratory facilities are set up to accommodate humidity probe tests and/or calibrations. *In conformance.*

5.4.1a. Appropriate methods and procedures for tests and/or calibrations within its scope are used. Calibration procedures are documented in TIP-CM-72 for the LLNL Bi-thermal Calibration System. Handling, transport, storage and preparation of RH probes to be tested and/or calibrated are documented in Measuring and Test Equipment (M&TE) program requirements and implementation. *In conformance.*
5.4.1b. Instructions on operation of humidity probes are available. *In conformance.*
5.4.1c. Manuals and reference data relevant to the work of the laboratory are readily available. *In conformance.*
5.4.3. The introduction of test and calibration method of Bi-thermal Calibration System developed by the LLNL for its own use is a planned activity. *In conformance.*
5.4.5 Validation of methods. *Comment:* validation has been performed by comparing calibration results between the Bi-thermal Calibration System and Thunder Scientific; however, additional

measurements and analysis are recommended to place the validation on a firmer footing. See discussion in the Summary above.
5.4.6. Estimation of uncertainty of measurement. Uncertainty analysis based on the guidelines of ISO *Guide to the Expression of Uncertainty in Measurement* has not been followed. *Out of conformance.* See discussion in the Summary above.
5.4.7.2c. Computers and automated equipment are maintained properly. *In conformance.*

5.5.1a. Proper sampling, measurement, and test equipment used for calibrations. *In conformance.*
5.5.2a. Humidity probes were used within the accuracy specified by the manufacturers. *In conformance.*
5.5.5. Calibration certificates provided by the manufacture and/or accredited laboratory are maintained. *In conformance.*
5.5.6. Humidity probes were periodically calibrated by the manufacture and/or accredited laboratory based on M&TE program. *In conformance.*
5.5.8. Humidity probes were labeled for calibration status. *In conformance.*

5.6.1a. Thermometers are traceable to NIST standards. *In conformance.*
5.6.2.1.1b. External calibration services were used from the manufacture and/or accredited laboratory that provide traceability to NIST. *In conformance.*
5.6.2.1.1c. Calibration certificates issued by these laboratories contained the results with uncertainty. *In conformance.*
5.6.3.1. A reference standard is not provided for tests and/or calibration of humidity probes. *Comment:* there are no available reference standards. LLNL staff are using the documented vapor pressure and thermodynamic properties of pure water as an intrinsic standard.

5.8. Handling of test and calibration items. Procedures for the transportation, receipt, handling, storage of test and/or calibration items are available from M&TE program requirements. *In conformance.*

5.9.1. Quality control procedures for monitoring the validity of tests and calibrations undertaken have not been established. *Out of conformance.* The basis for a quality control procedure exists in that probes are calibrated by both Thunder Scientific and provisionally calibrated by the LLNL Bi-thermal Calibration System, but a systematic procedure for monitoring the calibrations does not exist.

5.10.1.b. The results of tests and/or calibrations have been reported and documented in scientific notebooks. *In conformance.*

Estimate of Confidence Limits

ISO 17025 mandates corrective action for non-conformances that are identified. The standard does not identify any particular corrective actions, but the corrective action should be designed to prevent recurrence and should be of a "degree appropriate to the magnitude and risk of the problem." In this particular case, where the use of the humidity sensors is well known, the "magnitude and risk of the problem" is best expressed as a question:

> If the claimed uncertainties of the LLNL humidity sensors were in error, would the conclusions of the experiments relying on those sensors be altered appreciably after correction of the sensor uncertainties?

To help address this question, this section, which is outside the scope of a ISO 17025 assessment, has been added.

A simplified definition states that the uncertainty of a measurement is an estimate characterizing the range of values within which the true value lies, at a stated level of confidence. In this section, the goal is not to calculate the uncertainty itself, but to estimate a likely upper limit for the uncertainty. These estimates are the result of our best technical judgment. Determination of the uncertainties would require a careful evaluation of the uncertainty, complete with necessary control experiments and statistical analysis. These estimates are provided as a guide to the likely impact of the sensor calibrations on the body of data obtained with these sensors.

There are three relevant time spans:

Period 1 (1998 to March 2002): single-temperature calibration at 23 °C, extrapolated to 180 °C.

Period 2 (late 2002 to late 2004): calibration over the range 25 °C to 60 °C by Thunder Scientific.

Period 3 (end of 2004 to the present): calibration by Thunder Scientific and provisional calibration by the LLNL Bi-thermal Calibration System.

Data obtained during Period 3 showed that calibrations obtained from Thunder and LLNL were self-consistent. As noted above, the largest contributor to the uncertainty is the sensor drift observed. A conservative estimate of the uncertainty of sensor measurements in the environment of use is 6 %RH (95 % confidence limit) in Period 3, based on combining in quadrature the probable uncertainty of the LLNL Bi-thermal Calibration System (estimated as 3 %RH at 95 % confidence) and the observed sensor drift (estimated at not more than 5 % RH at 95 % confidence).

For Periods 1 and 2, a limit on uncertainty is based on the observation that the sensor drift is significantly larger than the discrepancy between the LLNL Bi-thermal Calibration System and the calibrations performed at much lower temperatures. Thus, the 95 % confidence limit will not be greatly higher than the 6 %RH limit of Period 3. A conservative estimate for this period can be made by combining in quadrature the 6 %RH for Period 3 with an estimate of the uncertainty for the extrapolation process. Using the so-called rectangular probability distribution model, the extrapolation standard uncertainty (at a confidence level of 95%) may be estimated as $(2/\sqrt{3})\ \Delta$, where Δ is the maximum observed discrepancy between the LLNL calibration results and an extrapolated calibration. From Fig. 3 of "RH Probe and Experimental Timeline" by S. Carroll (UCRL-AR-218422, dated 3/1/2006), the extrapolation uncertainty can be calculated to be 3.7 %RH (95 % confidence). Adding this result in quadrature with 6 %RH, one obtains 7 %RH as a conservative 95 % confidence limit for the measurement results using the Vaisala probes during Periods 1 and 2.

In the approximate calculations above, there is some likely over counting of uncertainty components. For example, the observed discrepancy and drift may be partly due to limitations in the repeatability of the LLNL Bi-thermal Calibration System. Since the intent of this section is to provide conservative upper confidence limits on the probe performance, rather than uncertainty estimates, no attempt has been made to avoid over counting.

Appendix B:
Personnel Contacted by the Independent Review Team at Yucca Mountain Project Offices and LLNL

The Independent Review Team interviewed the following staff in Las Vegas, NV and Livermore, CA:

NRC:

Robert Latta	On-Site Representative and Observation Audit Lead for the August 2005 audit, Las Vegas
Fred Brown	NRC Nuclear Material Safeguards and Security Headquarters
D. Blair Spitzberg	Region 4
Vincent Everett	Region 4 Technical Specialist

BSC:

Paul Dixon	Manager – Post Closure Activities
Mike Mason	QA Manager
Robert Hartstern	Quality Verification Manager
Robert Habbe	Audit Section Manager
Michael Carmichael	CAP Manager
Nancy Williams	Licensing
Ted Feigenbaum	General Manager
Charles Warren	QA (former BSC QA LLNL onsite rep)
Donald Beckman	Licensing and Nuclear Safety Manager
Pam Dahl	Licensing and Nuclear Safety

LLNL:

James Blink	LLNL Liaison
Cynthia Atkins-Duffin	Deputy Associate Director
Susan Carroll	Principal Investigator
David McCallen	Nuclear Systems Program Leader
Joe Rard	Investigator
Victor Barish	Engineering Assurance Manager
Leigh Gouveia	VP – PAC (subcontractor to BSC/QA)
Greg Gdouski	Technical area lead
Kirk Staggs	Principal Investigator
Steve Harris	BSC resident QA support
Tanya Reshel	M&TE custodian

OCRWM:

James Blaylock	OQA
Mike Uhlshafer	Acting Manager – OQA
Ram Murthy	OQA
Kerry Grooms	OQA – Quality Assessments Team Lead
Mark Williams (via phone)	OLAS Director
April Gil	OLAS – Regulatory Interactions & Strategy Division (RISD) Director
John Arthur	Deputy Director ORD
Ken Powers	Associate Deputy Director – ORD
Neal Hunemuller	OLAS – RISD
David Haught	OLAS – RISD
Allen Benson	Communications Supervisor

Appendix C: Audit Timeline

Date	*Event*
June - July 2005	Bechtel SAIC Company, LLC (BSC) began scoping and planning of internal audit BQAP-BSC-05-07, "BSC Waste Package Degradation Investigations and Analyses."
July 25, 2005	BSC submitted the original audit plan to the U.S. Nuclear Regulatory Commission (NRC).
August 3, 2005	BSC submitted a revised audit scope, removing reference to the Total System Performance Assessment.
August 15 - 31, 2005 August 15-19 August 22-25 August 29-31	BSC performed internal audit BQAP-BSC-05-07 and conducted initial meeting with NRC Observation Audit Lead and the Office of Civilian Radioactive Waste Management's (OCRWM) Office of Quality Assurance (OQA). - Audit activities in Las Vegas - Audit activities in Las Vegas and at Lawrence Livermore National Laboratory (LLNL) - Audit team returns to Las Vegas to complete audit report.
August 24, 2005	NRC's Observation Audit Lead tried to provide comments to BSC QA Audit staff on deficiencies noted in the audit.
August 31, 2005	Initial debrief in morning, and exit meeting in afternoon between NRC Observation Audit Lead, BSC, and OCRWM OQA and Office of License Application and Strategy's (OLAS) Regulatory Interactions and Strategy Division (RISD). NRC expressed concerns and provided AOIs and weaknesses. The NRC Observation Audit Lead made eight additional attempts to communicate concerns to the BSC Audit Team Lead.
September 28, 2005	NRC's Observation Audit Lead provided clarifications on NRC findings to BSC Licensing and Nuclear Safety (LNS).
October 7, 2005	BSC issued audit report on BQAP-BSC-05-07 to NRC, OCRWM, BSC management and a wide outside distribution.
November 1, 2005	NRC communicated to the OCRWM's OQA and OLAS - RISD that BSC staff were not responsive to NRC concerns (after the issuance of the BSC audit report).
November 11, 2005	LLNL initiated self-assessment MSA-LNS-2006-008, "Management Self-Assessment of LLNL Scientific Notebooks;" final report approved November 30, 2005.
November 16, 2005 (and again on November 25, 2005)	The Office of Repository Development (ORD) Deputy Director was provided with a written description of the issues as part of a set of bulleted potential discussion items for a weekly teleconference with NRC.

Date	***Event***
December 5, 2005	BSC LNS initiated self-assessment MSA-LNS-2006-013, "Effectiveness of BSC Interactions with NRC During Observed Audits" to identify improvements needed in interfaces with NRC during audits. Report approved January 25, 2006. One Level C and 10 Level D CRs were initiated.
December 7, 2005	NRC, BSC, and OCRWM OLAS - RISD and LLNL participated in a second debrief and re-exit of the audit. Five AOIs were identified, including the concern that Scientific Notebook – 484 misrepresented that the Vaisala humidity probes were calibrated.
January 9, 2006	NRC issued Observation Audit Report (OAR-05-05) "*Observation Audit of Bechtel SAIC Company, LLC, Audit of Scientific Investigation, Waste Package, and Drip Shield Degradation.*"
January 30, 2006	BSC initiated a Level A CR (7418) to establish a root cause analysis to address issues related to the use of the Vaisala humidity probes outside their range of calibration.
February 2, 2006	The OCRWM Acting Director initiated an Independent Review of the calibration of the Vaisala probes and BSC interactions.

Appendix D: History of Changes to the Procedure on Control of Measuring and Test Equipment

The Source of the Measuring and Test procedure is OCRWM/RW-0333P, *Quality Assurance Requirements and Description* (the QARD). Specifically, Section 12 of the QARD, entitled "Control of Measuring and Test Equipment," requires M&TE to be properly controlled, calibrated, and maintained.

Although there have been changes to the procedure (see below), the changes have been considered minor, as evidenced by the fact that the changes have been Interim Change Notices (ICNs) rather than Revisions.

The original issuance of the procedure AP-12.1Q (i.e. – Rev 0 ICN 0) *Control of Measuring and Test Equipment* was on 9/15/2000.

AP-12.1Q Rev 0 ICN 1 was effective 03/27/2001.

AP- 12.1Q – Rev 0 ICN 2 has an effective date of 02/06/2002.

AP- 12.1Q – Rev 0 ICN 3 (effective date 04/30/2004). Added the "or similar" tag to the requirement to tag Out-of-Service equipment. LLNL submitted the following comment[22] during the procedure revision process:
"*Section 5.7.2b) –*
Change 1), first sentence to read: " Apply an M&TE Out of Service tag or other similar tag to indicate out of calibration conditions." As stated in the NOTE in comment 1, some M&TE used in ongoing corrosion experiments at LLNL cannot be recalled for calibration at the required intervals because it would disrupt data taking for these experiments. In these instances an OCR is issued to identify that M&TE is in use past its recalibration due date and a tag indicating this condition is affixed to the M&TE . Although tagging is appropriate in these conditions, an out of service tag is not for the following reasons:

a. The equipment is not out of service... it is in use past its calibration due date.
b. Allowing personnel to observe equipment in use with an out of service tag attached might cause them to think that using other equipment with an out of service tag is acceptable.

Change 2) to read: "as soon as possible segregate the out-of-calibration M&TE ... "

The BSC procedure owner accepted change 1 and incorporated the suggested wording into the procedure. LLNL change 2 was not accepted by the procedure owner.

[22] LLNL, Comment Sheet for AP-12.1Q, Rev. 0, ICN 3, dated January 6, 2004.

LP-12.1Q-BSC Rev 0 ICN 0 *Control of Measuring and Test Equipment* (effective date 02/04/2005) – The procedure was approved by the Repository Development Manager and superseded AP-12.1Q. This was the procedure in effect at the time of the August 2005 audit.

LP-12.1Q-BSC Rev 0 ICN 1 has an effective date of 10/31/2005. This clarifies labeling requirements and makes a number of miscellaneous administrative and editorial changes.