# GEOSCIENCES AND ENGINEERING DIVISION
## NONCONFORMANCE REPORT

**Project No.** 20.06002.01.242 **NCR No.** 2006-18

**PART 1: DESCRIPTION OF NONCONFORMANCE**

Analysis of results from a set of sorption experiments (Np-237 sorption on alluvium) revealed that the data generated were not acceptable for use. Several minor procedural and analytical errors appeared to have occurred during the course of the experiments. The combination of errors resulted in unacceptable uncertainties in the data. During discussions of the experiments and results during a routine surveillance (SUR 2006-12) of RTSZ activities, CNWRA QA staff suggested that an NCR be completed to document problems and their corrections.

**Initiated by:** F. Paul Bertetti **Date:** 19 July 2006

**Action Required by:** F. Paul Bertetti **Response Due Date:** 25 July 2006

**PART 2: PROPOSED DISPOSITION AND CORRECTIVE ACTION**

**Disposition:**

Data from the affected experiments will not be used in any evaluation or interpretation of Np-237 sorption behavior. Entries will be made in relevant sections of the affected notebooks (SN 742 and SN 800) to indicate that the data and information associated with this set of experiments are not to be used for evaluation or interpretation of Np-237 sorption behavior.

**Basis of Disposition:**

Analysis of the data from these experiments (19W, 19WO, 22W, 22WO) reveals nonsensical results including calculation of negative sorption coefficients well outside the expected uncertainty range for the experiment parameters. Additionally, several inconsistences in the experiment data reveal procedural errors. These errors are exemplified by measurement of decreased pH values following addition of base to solutions, wide variation in the amounts of solution added for pH adjustment, unreasonable mass calculations for sediment added, variations in measured test tube mass over time, and measurement of Np-237 solution concentrations that are inconsistent with previous measurements.

**Action to Correct Nonconformance:**

See attached sheet.

**Target date for completion:** 25 July 2006

**Proposed by:** F. Paul Bertetti Paul Bertetti **Date:** 19 July 2006

**PART 3: APPROVAL**

**Manager:** E. C. Pearcy **Date:** July 21, 2006

**Director of QA:** R D Brient **Date:** 7/21/2006

**Comments/Instructions:**

**PART 4: CLOSE OUT**

**Comments:** The NCR provides sufficient information for close out. No additional review is necessary.

**Verified by:** R D Brient **Date:** 7/21/2006

**Distribution:**
**Original–QA Records**
**ORIGINATOR** - F.P. Bertetti
**PRINCIPAL INVESTIGATOR** - F.P. Bertetti
**MANAGER** - E. Pearcy
**ASSISTANT DIRECTOR** - G. Wittmeyer

**Nonconformance Report 2006-18 Attachment**

The following summary provides an identification of problems and the corrective actions taken to correct the issues associated with Nonconformance Report 2006-18.

The inability to use the data from experiments 19W, 19WO, 22W, and 22WO was a result of several contributing factors. These contributing factors, when combined, introduced unacceptable uncertainties into the experimental results. Each factor, its proposed root cause, and the corrective action taken is listed here in detail.

**Factor 1. Technician unfamiliarity with the experimental procedure and protocol**
**Root Cause: Inadequate training of the technicians prior to the experiment**

Hands-on experimental work requires not only a basic familiarity with the equipment used in the experiment but also requires experience with experimental protocols and the sequence of procedural steps during the course of the experiment. Based on his experience in training and developing the expertise of new technicians, the principal investigator felt adequate time and instruction had been provided to the technicians involved in conducting these experiments. Each technician had been trained to use each piece of equipment utilized for sorption experiments and had multiple opportunities to use the equipment in practice. Each technician had been employed for several months and had obtained a familiarity with the lab and its arrangement. Both technicians participated as observers and helpers in a similar sorption experiment conducted just prior to initiation of sorption experiments 19W, 19WO, 22W, and 22WO. During the course of the experiments, however, the technicians self-identified several items with which they were unfamiliar. For example, there was some confusion about the exact sequence of measurement of pH and withdrawal of samples from solutions at the end of the experiment. During the course of the experiment, and as questions arose, the principal investigator and other staff reviewed the experimental protocol and discussed the basis of each step in the experiment. In some cases, corrections (e.g., revising the protocol) were made on the spot, and solutions were resampled or steps were redone in an attempt to compensate for any introduced error. In retrospect, the experiments functioned as a hands-on exercise that provided important experience for the technicians. Unfortunately, the price of this training was an introduction of significant data uncertainty and loss of usable data. Contrary to the principal investigator's expectations, hands-on training simulating the entire sequence of actions during the experiment was required. In addition, the technical basis behind each step of the experiment must be better explained so that technicians have an appreciation for the attention to detail required for each step.

**Corrective Actions:**

1. The experimental protocols and procedures were reviewed and revised to make them more clear with respect to the order of actions to be completed during each step. The order of actions and the basis for this order were reviewed with the technicians.

2. The technical basis for each step in the experimental procedure was thoroughly reviewed with all involved staff.

3. Additional training on the sorption experimental protocol was not required because of experience gained during the conduct of these experiments.

4. The principal investigator will review and revise as necessary the manner in which training is provided prior to the conduct of experiments.

**Factor 2: Improper use of experimental equipment.**
**Root Cause: Inadequate training of technicians and inadequate attention to detail during equipment operation**

During the course of the experiment and following discussions with staff after large experimental uncertainties were identified in the results, several errors in the use of equipment were brought to light. These errors and problems included (i) a failure in several instances to properly control the intake and discharge of solutions from pipettes; (ii) the technicians were not familiar with the use of adjustable pipettes; and (iii) there was inadequate care in ensuring that test tubes were weighed under similar conditions.

**Corrective Actions:**

1. Additional training was conducted to develop expertise with the operation of pipettes and pipette tips. A systematic evaluation of the repeatability of solution intake and discharge was conducted.

2. Additional training was conducted to demonstrate the use of adjustable pipettes. Methods for obtaining a solution of specified volume were discussed in detail.

3. A weighing stage was fabricated to facilitate consistent and repeatable conditions for the measurement of mass of test tubes. Training was conducted on the use of this stage.

**Factor 3: Failure to properly follow experimental procedure**
**Root Cause: Inattention to detail**

Analysis of experimental results highlighted values that were inconsistent with results obtained when following proper experimental procedure. For example, measured pH values for some experimental solutions were as low as 2.3 when they were expected to have been near 9. The only cause for this difference would be the inadvertent addition of acid to the experimental solution rather than the addition of base as required by procedure. Additionally, the mass of solid added to one experiment test tube was greater than 1 g instead of the 0.1 g required. Inspection of the tube indicated that 0.1 g was likely added, so the cause must have been a failure to properly record the mass of the tube before or after addition of the solid.

**Corrective Action:**

Additional training and discussions were held between the principal investigator and the technicians to emphasize the importance of accuracy in recording measurements and attention to detail when handling and using solutions.

**Factor 4: Masses of acid added to experimental solutions did not produce the desired pH adjustments**
**Root Cause: Improper use of adjustable pipettes and lack of empirical data for new solid samples**

Adjustment of the pH of experimental solutions to achieve a range of pH values from about 7 to

9 was mostly unsuccessful. The variation of the amount of acid added to solutions due to improper use of the adjustable pipettes combined with the low ionic strength and low buffering capacity of the solutions resulted in many solutions with very low pH values (below 4.5). The amount of acid desired was based on empirical data collected form previous experiments using 19PB sediment. In experiments 19W, 19WO, 22W, and 22WO, sediment from boreholes 19PB and 22PC was used. Moreover, the sediment had been processed to remove calcite (the WO experiments) in some cases. Thus, a contributing factor to the poor pH adjustment was also due to an inappropriate assumption that the sediments would behave similarly.

**Corrective Actions:**

1. A small scoping experiment was designed and conducted to re-evaluate the effects of acid additions on the solutions. A new acid addition protocol was designed using the information derived from this experiment.

2. Training was conducted in the use of adjustable pipettes to ensure a more accurate and repeatable delivery of acid or base to adjust solution pH.

**Factor 5: The Liquid Scintillation Analyzer (LSA) reported concentration values for the original spike solution (#61A-1) that were inconsistent with previous measurements of the spike solution and the calculated concentration of the solution.**

**Root Cause: The spike solution concentration changed during the course of the experiment. The cause of this change is undetermined.**

There were several possible causes of the counting error or inconsistency. Because the LSA is an important component in quantifying the amount of Np-237 in solution, solving this discrepancy was given a high priority. Each potential cause was systematically evaluated.

1. The acid added to each sample vial was investigated for potential ionic strength problems and effects on counting. No problems were found.

2. The scintillation cocktail used was evaluated. No impacts were noted.

3. The LSA counting protocol was examined, along with LSA QA/QC history and LSA performance on other known samples. The LSA appeared to be functioning properly.

4. The spike solution was compared to another known solution. The results of these tests indicated that the LSA reported the proper concentration of the spike and known solution. However, the spike solution value was different than previously measured, indicating a real change in concentration. Possible causes of the change in spike concentration are (i) inadvertent dilution of the spike (not known to have happened), (ii) a change in spike chemistry (not indicated by pH measurement), or (iii) loss of Np from some other cause such as precipitation (not previously observed in our lab).

**Corrective Actions:**

A new spike solution (#61A-2) was prepared for use in the next phase of experiments. Use of

spike solution #61A-1 will be restricted until further tests are completed. If a cause of the change in spike solution #61A-1 concentration cannot be determined, the solution will be removed from use in experiments.

**Overall Corrective Actions and Results:**

The series of experiments impacted was repeated (following the training described previously). Preliminary results of the repeat studies indicate that the results are acceptable for use and contain no obvious errors or deviations from experimental protocol/procedure as described in the scientific notebook. The LSA "problems" were evaluated throughout the repeat studies. The original spike was used, but an understanding of the initial and final spike concentration and evidence that the concentration remained the same throughout the experiment allows for use of these data. The LSA counting "problem" has been resolved and a new spike is used in current experiments. Delays in producing experimental results necessitated delay of a scheduled deliverable until FY 2007. NRC and CNWRA staff and management have concurred with the necessary delay.

All corrective actions complete as of 20 Jul 2006.

Paul Butler