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# Chemical Analyses for Alcove 8/Niche 3 Tracer Studies

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**University and Community College System of Nevada (UCCSN)  
Scientific Investigation Plan (SIP)**

**Task Title:** Chemical Analyses for Alcove 8/Niche 3 Tracer Studies

**Task Number:** ORD-FY04-011

**Document Number:** SIP-UNLV-035

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Date

**REVISION HISTORY**

<b><u>Revision Number</u></b>	<b><u>Effective Date</u></b>	<b><u>Description of Change</u></b>
0	05/06/2004	This task is a continuation of Cooperative Agreement DE-FC28-98NV12081, Task 35, "Chemical Analyses For Alcove 8/Niche 3 Tracer Studies". Task 35 planning documentation is described in SIP-UNLV-025.

## **1.0 SCOPE AND OBJECTIVES**

The objective of this task “Analytical Support For Tracer Studies in Alcove 8 / Niche 3” is to provide chemical analyses for the tracer studies at Alcove 8 / Niche 3 in the Exploratory Studies Facilities (ESF). This analytical support includes developing procedures for measuring tracer concentrations and then performing the tracer (and other background constituent) analyses for the water samples generated from each Alcove 8 / Niche 3 tracer test. These tracer tests are part of the Flow and Seepage Testing in Alcove 8/Niche 3 (TWP-NBS-HS-000004 REV 00). The overall goal of this study is to quantify large scale infiltration and seepage processes in the unsaturated zone at the ESF at Yucca Mountain.

Appendix I lists a set of tracers that have been approved for usage for the ESF filtration studies along with the maximum allowed quantity and concentrations of the injected tracers. The specific tracers used for testing will be selected from this list (Appendix I) by the Lawrence Berkeley National Laboratories (LBNL) Principal Investigator. Once the specific suite of tracers has been selected, analytical procedures will be developed to optimize the analyses. Samples will then be analyzed for these tracers throughout the testing period. In addition, a set of water samples (input water as well as seepage water) will be analyzed for additional constituent concentrations. The concentrated initial tracer solutions (injectate solutions) will also be prepared under this task.

This work is subject to University and Community College System of Nevada (UCCSN) Quality Assurance (QA) Program requirements. Details of this SIP that are not available at this time will be added in future revisions. This Scientific Investigation Plan presents an independent confirmatory study supporting previously gathered information. Previous analytical work in support of the tracer studies at Alcove 8 / Niche 3 was performed under the UCCSN QA Program in Task 35, “Chemical Analyses for Alcove 8 / Niche 3 Tracer Studies”. Task ORD-FY04-011 a continuation of that work.

## **2.0 APPROACH**

LBNL and USGS researchers will perform all field work including injection of the tracer solutions as well as sample collection. A portion of the final tracer solution used for the injection and the seepage samples collected from Niche 3 will be transferred to the Harry Reid Center for Environmental Studies (HRC). Once samples are received at the HRC, implementing procedures (IPLVs), approved by the UCCSN QA Program, will be followed for all sample control and analysis.

The instrumentation used to quantify the tracers will depend on the particular tracer. Fluorinated benzoate tracers will be analyzed using a high performance liquid chromatograph (HPLC) with an ultraviolet (UV) detector. Bromide will be analyzed using an ion chromatograph (IC) with a conductivity detector. Iodide will be analyzed using an ion chromatograph (IC) with either a UV detector or a conductivity detector. Lithium will be analyzed using an inductively coupled plasma mass spectrometer (ICP-MS). Instrumentation may be changed during the course of the project and will be documented in the scientific notebook. Method development work will be performed to determine optimal conditions for the analysis of tracers selected for testing. The mobile phase solvents used for the HPLC and IC are not quality affecting. This includes the pH adjustment of the buffer solutions. The quality of the mobile phase solvents is determined through the quality control requirements for calibrations described in the IPLVs.

Any special environmental conditions are described in the corresponding IPLV or scientific notebook. Any processes not addressed in the IPLV will be documented in the scientific notebook. Special controls are specified in the corresponding IPLV. The skills of the chemists needed to perform these measurements are outlined in their position descriptions. There are no special training requirements for this work beyond the education and experience requirements of each employee’s position description.

### **3.0 SCHEDULE OF WORK**

All IPLVs anticipated for this task have been previously approved by the UCCSN QA Program and have been in use at the HRC. The IPLV's for this task are in revision and will be completed by 5/30/04. Samples will be analyzed at a minimum of 30 days following their transfer to the HRC. Reports summarizing the progress of this study will be submitted quarterly. In accordance with QAP-3.6, Q data will be submitted to the UCCSN Technical and Electronic Data specialist for entry into the UCCSN Technical Data Archive (TDA) quarterly and within 30 days following the completion of each phase of tracer testing. Q data consists of the concentration data for the field samples that have been verified using the check lists included in the appropriate instrument IPLV and for which the associated scientific notebook has been reviewed both for QA and technical content. QA records (described in the SIP and implementing procedures) will be submitted to the UCCSN Records Office within 90 days following the completion of each phase of tracer testing and also successful entry of tracer concentration data into the TDA.

### **4.0 INTERFACE CONTROLS/SAMPLES**

LBNL and USGS researchers will select the tracers used for each test and perform all field work. Field samples will be collected by LBNL or the USGS and transported to the HRC in accordance with LBNL sample collection, transportation, and chain of custody procedures. Preliminary data will be labeled as such and sent via email as a spreadsheet file to the external Principal Investigators associated with the field tests. Access to the final concentration data in the TDA can be obtained through the UCCSN website ([hrcweb.nevada.edu/data/tda](http://hrcweb.nevada.edu/data/tda)).

All sample collection activity will be performed by LBNL and the USGS. Samples will be controlled at HRC in accordance with QAP-8.0, "Identification and Control of Items and Samples". The Chain of Custody Section of IPLV-8.3 will be followed once custody is transferred to the HRC.

#### **External Interfaces**

**Yucca Mountain Cooperative Agreement Technical Contact:** Institutional Affairs Office (DOE)

**DOE Technical Task Representative:** Drew Coleman

**Principal Investigators Associated with Field Test:** Rohit Salve (LBNL) and David Hudson (USGS)

#### **Internal Interfaces**

**Principal Investigator:** Klaus Stetzenbach                      **Investigator:** Jeanette Daniels

**Analysts:** Tatjana Jankovic    Julie Bertoia    Mike Tabriz    James Cizdziel

**Students:** Roselynn Gentles    Jennifer Petchsaiprasert    Danae Cummings

### **5.0 STANDARDS/ PROCUREMENTS/SUBCONTRACTS**

Standards used to quantify the tracers in the field samples will be prepared directly from the tracer stock used for field testing rather than from NIST-traceable standards or other high-quality standard that is intended to allow absolute concentration measurement. Most of the chemicals used as tracers are not available from NIST or qualified suppliers. This is not important, however, considering the nature of a tracer test. When tracer transport experiments are interpreted, it is not necessary to know the absolute concentration of tracers in the samples collected, but rather it is important to know tracer concentrations relative to the amount that was injected. Using material from the bulk stock to prepare standards will also

inherently contain any interferences that are present in the samples to be analyzed. Standards prepared from this same stock will therefore more accurately represent the condition of the samples than if a high purity standard is used. Results of the analyses are quality-affecting. Calibration items and services are procured in accordance with QAP-7.0. Balances are calibrated annually by a qualified supplier. The reference mass set used to check working mass sets is calibrated every two years by an organization on the Qualified Supplier List (QSL). Calibrations of Pipettors are checked annually. Calibrations and calibration checks will be performed by HRC staff or by an organization on the QSL. No subcontractors will be used on this task.

## **6.0 IMPLEMENTING PROCEDURES**

1. Analytical and Top Loading Balance Use, IPLV-003
2. High Performance Liquid Chromatograph Operation, IPLV-004
3. Groundwater Sample Collection and Control, IPLV-8.3 (Chain of Custody Section only)
4. Measurement of Trace Elements in Water Samples by Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS), IPLV-009
5. Measurement of Inorganic Anions in Water Samples by the Ion Chromatography System, IPLV-008
6. Field Measurement of Conductivity, Alkalinity, and pH, IPLV-012
7. Pipettor Calibration Check, IPLV-017
8. Measurement of Major Cations in Water Samples by the Flame Absorption Spectrometry System, IPLV-011

## **7.0 EQUIPMENT AND INSTRUMENTATION**

The specific equipment used for each measurement, described in 2.0, will be documented in the scientific notebook. Documentation will include the instrument manufacturer, model, and serial number as well as all applicable instrument manuals. Relative calibration, that is, establishment of an analytical instrument response curve is performed prior to use end of sample batch analysis. The manufacturer and lot number of each chemical used as a tracer and used to calibrate, in relative terms, the instrument systems will be recorded in the applicable scientific notebook. All Measurement and Test Equipment (MT&E) will be stored in a locked laboratory to prevent loss and tampering.

## **8.0 SOFTWARE**

Software will be qualified in accordance with QAP-3.2 as applicable. It is not anticipated that this will be necessary for other than routines or macros. The software packages used in this study include 1) The analytical instrumentation software used for data acquisition, and 2) Spreadsheet software such as Quattro Pro or Excel for data reduction. Use of the analytical instrumentation data acquisition and spreadsheet software will be documented or referenced, along with the specific version used, in the instrument scientific notebook. Control of electronic data is addressed in each IPLV that involves electronic data management, primarily instrument system IPLVs. No models are used in this work.

## **9.0 HOLD POINTS**

Submittals and documents must complete technical and QA reviews prior to approval. Decision points associated with the analytical measurements are addressed by use of quality controls to indicate when there is an analytical or other problem which needs action described in the IPLVs.

## **10.0 QUALITY CONTROL**

Objectives for precision and accuracy, for the analytical measurement and how results are evaluated are described in each corresponding IPLV. Precision will be determined using laboratory duplicates. Accuracy of standards preparation will be determined using initial calibration checks (ICC) prepared by someone other than the person who prepared the calibration standards. The calibration standard and the ICC must agree within 10% in order to proceed with sample analysis. Accuracy of the calibration curve is verified by use of the ICCs. Maintenance of that accuracy, or a lack of instrument drift, is verified by the use of continuing calibration checks.

Potential sources of error and uncertainty are addressed in the associated IPLV. In a study of this nature, the majority of error is generally attributed to field sample collection and handling rather than to laboratory analysis.

## **11.0 DATA RECORDING, REDUCTION, AND REPORTING**

Data packages consisting of the hard copies of raw data generated from each instrument will be referenced by the analysis date and will be attachments to the scientific notebook. Data recording requirements for each scientific notebook are described in the corresponding IPLV. A summary of the data generated from each instrument (described in section 2.0) is exported to a spreadsheet (Microsoft Excel) where final data reduction is performed. Reduction of data involves calculating the concentration of the tracers in each sample based on the slope and intercept of an instrument response curve and also calculating averages when multiple injections are made from the same autosampler vial. A hard copy of the spreadsheet containing the reduced data will be included in the data package. The concentration of the tracer is generally calculated using the analytical instrumentation software but may be calculated using a spreadsheet software package (Microsoft Excel). The calculation of averages is performed using the spreadsheet software. Following data reduction, the spreadsheet file will be labeled as preliminary and sent to the external PIs. The reported results will include sample name, tracer concentration, bar code ID, and analysis dates. Data will be submitted to the TDA following verification. Raw and reduced data will be controlled in accordance with QAP-3.1. by being stored on password protected computers in locked rooms. The data will be backed up to the HRC server on a regular basis. The final verified spreadsheet of reduced data for submittal to the TDA will also be controlled in accordance with QAP-3.1.

## **12.0 REVIEWS AND VERIFICATIONS**

Internal verification of all data will be performed by someone other than the originator to check compliance to the procedures and to verify the accuracy of data reduction. Internal technical review will be performed and documented on the data, scientific notebooks, all reports, and all journal articles (non-deliverables) generated in this task. Any report of data generated without full internal verification will be labeled as “preliminary” data. Data review and verification will include the following:

- 1) Check for compliance with the QA/QC criteria described in each IPLV using the associated checklist. The completed checklists will be included with the data packages.
- 2) Compare tracer concentrations printed in the data packages to the reduced data.
- 3) Final data submitted for entry into the TDA will be visually compared to the reduced data spreadsheets contained within the data packages to ensure that the reported concentration data are accurate.

Data will be acceptable when the data review and verification steps 1 – 3 above are successfully completed. Scientific notebooks as well as data packages and attachments will be reviewed in accordance with QAP-3.0 when complete or prior to submittal of data to the TDA. Technical reports will be reviewed in accordance with QAP-3.4.

### **13.0 RECORDS AND DELIVERABLES (SUBMITTALS)**

#### **QA Records**

QA records are handled in accordance with QAP 17.0, “Quality Assurance Records.” Records designated as QA records in the UCCSN QAPs and IPLVs listed include but are not limited to:

1. Hard copies and/or electronic media containing raw and reduced concentration data including calibrations and QC results.
2. Scientific Notebooks including attachments.
3. Calibration and checks for each balance and pipettor used to collect or produce quality affecting data for this study.
4. Data Review Check Sheets
5. Chain of Custody Forms.
6. Copies of quality-affecting submittals.
7. Records will be stored for protection in a locked fire-proof cabinet until they are completed.

#### **Deliverables (Submittals)**

Submittals are submitted to the HRC Administrative Task PI for submittal to DOE in accordance with the cooperative agreement.

1. Quarterly progress reports and the final technical report.

### **14.0 REFERENCES**

1. IPLV-003, “Analytical and Top Loading Balance Use”
2. IPLV-004, “High Performance Liquid Chromatograph Operation”
3. IPLV-8.3, “Groundwater Sample Collection and Control”
4. IPLV-008, “Measurement of Inorganic Anions in Water Samples by the Ion Chromatography System”
5. IPLV-009, “Measurement of Trace Elements in Water Samples by Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS)”
6. IPLV-012, “Field Measurement of Conductivity, Alkalinity, and pH”
7. IPLV-017, “Pipettor use and Calibration Check”
8. QAP-3.1, “Control of Electronic Data”
9. QAP-3.2, “Software Management”
10. QAP-3.4, “Technical Reports”
11. QAP-3.6, “Submittal of Data”
12. QAP-8.0, “Identification and Control of Items and Samples”



13. QAP-7.0, "Control of Quality-affecting Procurement and Receipt"  
14. QAP 17.0, "Quality Assurance Records"

<b>Tracer</b>	<b>Maximum Quantity Injected (grams or liters)</b>	<b>Maximum Concentration (ppm)</b>
Lithium Bromide	1,000 g	500
Potassium Fluoride	100 g	50
Potassium Iodide	50 g	10
2,3-Difluorobenzoic Acid	50 g	50
2,4-Difluorobenzoic Acid	50 g	50
2,5-Difluorobenzoic Acid	50 g	50
2,6-Difluorobenzoic Acid	50 g	50
3,4-Difluorobenzoic Acid	50 g	50
3,5-Difluorobenzoic Acid	50 g	50
2,3,4-Trifluorobenzoic Acid	50 g	50
2,3,6-Trifluorobenzoic Acid	50 g	50
2,4,5-Trifluorobenzoic Acid	50 g	50
2,4,6-Trifluorobenzoic Acid	50 g	50
3,4,5-Trifluorobenzoic Acid	50 g	50
2,3,4,5-Tetrafluorobenzoic Acid	50 g	50
Pentafluorobenzoic Acid	50 g	50
FD&C Blue No. 1	20 g	20
Sulpho Rhodamine B	10 g	10
Fluorescein	10 g	10
Pyranine	10 g	10
Rhodamine WT	10 g	10
Fluorescent Microspheres	1 L	---

**APPENDIX 1**