PREPARATION AND CONDUCTION OF DISSOLUTION RATE EXPERIMENTS

LOS ALAMOS
QUALITY PROGRAM

APPROVAL FOR RELEASE	
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Los Alamos
Yucca Mountain Site Characterization Project

HISTORY OF REVISION

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE
R0	03/20/96	N/A	Initial procedure.
R1		3,4, & 8	Revised to comply with LANL-YMP-QP-06.3 requirements.

PREPARATION AND CONDUCTION OF DISSOLUTION RATE EXPERIMENTS

1.0 PURPOSE

This procedure is designed to experimentally measure changes in aqueous chemical concentrations that result from dissolution of rocks from Yucca Mountain.

2.0 SCOPE

This detailed procedure (DP) applies to all YMP personnel who prepare and conduct experiments within the Water Chemistry Task of the Los Alamos National Laboratory (Los Alamos) YMP.

3.0 REFERENCES

LANL-YMP-QP-02.7, Personnel Training
LANL-YMP-QP-03.5, Documenting Scientific Investigation
LANL-YMP-QP-08.1, Identification and Control of Samples
LANL-YMP-QP-12.3, Control of Measuring and Test Equipment and Standards
LANL-YMP-QP-17.6, Records Management
LANL-CST-DP-35, pH Measurement
LANL-CST-DP-63, Preparation of Core Samples for Crushed Rock Experiments
LANL-CST-DP-89, Procedure for Sieving Soil and Rock Samples
LANL-CST-DP-107, Use of ICP-Atomic Emission Spectrometry in Analyte Determinations
LANL-EES-4-DP-806, Ion-Chromatographic Determination of Constituent Concentrations in Solution

4.0 DEFINITIONS

NA

5.0 RESPONSIBILITIES

The following personnel are responsible for activities identified in Section 6.0 of this procedure.

- Principal Investigator (PI)
- User

6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this DP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified, replaced by a new document, or current work practice is documented in accordance with QP-03.5, Section 6.1.6.

- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.

- When this procedure becomes obsolete or superseded, it must be destroyed or marked "superseded" to ensure that this document is not used to perform work.

6.1 Principle

This procedure describes the establishment and proper conduction of dissolution experiments. The results are used to evaluate the dissolution rates of Yucca Mountain rocks in contact with various artificial groundwaters. By comparing the measured chemical parameters of the artificial groundwaters as they equilibrate with various rocks over time, one can calculate the rock dissolution rates.

6.2 Equipment and Hardware/Software

The equipment needed is listed below. Items equivalent to those listed below may be used provided they perform the same function with an acceptable level of performance as judged by the PI.

- Calibrated balance

- Reagent-grade Chemicals for artificial groundwater preparation as specified by PI in step 6.5.1.1

- Deionized water with a maximum resistivity of ≥ 17.5 Megohms

6.2.1 Equipment Malfunctions

The equipment used to implement this procedure shall be in good repair before the experiment begins.

6.2.2 Safety Considerations

The equipment required to carry out this procedure shall be used in accordance with any applicable Los Alamos safety procedure.

6.2.3 Special Handling

Samples are stored in the refrigerator after filtration in order to minimize the potential for evaporative loss. The exact temperature of storage is not relevant.

6.3 Preparatory Verification

6.3.1 Hold Points

Specific PI determined activities (such as particle size) are recorded in a laboratory notebook.

6.3.2 Calibration

The analytical balance is controlled pursuant to QP-12.3. When data are collected from a balance, the unique identifier of that balance is recorded in the user's laboratory notebook along with the data collected. Proper procedure for documenting this data is provided in QP-03.5.

6.3.3 Environmental Conditions

Work is conducted under normal laboratory conditions of cleanliness at room temperature.

6.4 Control of Samples

Sample code numbers are referenced in a YMP controlled notebook. It is imperative that sample identification and control be sufficient to trace a sample and its derivatives from its original field location to the point of analysis and that the integrity of the sample be safeguarded during the entire analytical process. Consequently, users must follow the guidelines of QP-08.1.

6.5 Implementing Procedure

6.5.1 Preparation of Artificial Groundwaters

The user conducts the following steps:

6.5.1.1 Consult with the PI to specify the desired composition and concentrations for artificial groundwaters, the amount of artificial groundwater to be prepared, and the containers to be used; and record this guidance in the laboratory notebook.

6.5.1.2 Calculate the quantities of each chemical reagent required to achieve the desired compositions, and record these results in the laboratory notebook.

6.5.1.3 Transfer from each of the chemical containers approximately twice the weight of the chemical needed for use, and dry in an oven at about 100 degrees Celsius for a time period specified by the PI.

6.5.1.4 Cool dried chemicals in a desiccator.

6.5.1.5 Weigh with a precision of 1% and record the weight, manufacturer, chemical formula and lot number of individual chemical constituents. Weights should be within 10% of the target values calculated in Step 6.5.1.2.

6.5.1.6 Use deionized water to rinse the container specified by the PI to hold the artificial groundwater.

6.5.1.7 Add all but 2 L of the PI specified quantity of deionized water to the large container.

6.5.1.8 Add the weighed chemicals to 2 L of deionized water and stir until dissolved. Add this solution to the large container and stir for 15 minutes.

6.5.2 Baseline Analysis of Artificial Groundwaters

6.5.2.1 Pass a PI specified quantity of the artificial groundwater through a 0.1 µm nylon filter. Discard the first 2 mL to flush the filter of impurities and particles.

6.5.2.2 Analyze sample by inductively coupled plasma atomic emmision (ICP / AE) spectrometry for cations in solution as directed in DP-107.

6.5.2.3 Analyze sample by Ion Chromatography for anions in solution as directed by DP-806.

6.5.3.4 Analyze sample for alkalinity using a HACH Alkalinity test kit following guidance in QP-03.5 to document this as a notebook procedure.

6.5.3.5 Analyze sample for pH per DP-35.

6.5.2.6 Analyze sample for dissolved oxygen following guidance in QP-03.5 to document this as a notebook procedure.

6.5.2.7 Analyze sample for redox potential following guidance in QP-03.5 to document this as a notebook procedure.

6.5.2.8 Record all data required by cited DPs and QP-03.5 in a laboratory notebook.

6.5.3 Preparation of Crushed Rock

6.5.3.1 Consult with PI to determine origin, quantity, and range of particle sizes to be used in this experiment. Record results of this discussion in a laboratory notebook.

6.5.3.2 Prepare solid rock samples to meet PI specifications following DP-63 and DP-89.

6.5.4 Equilibration of Crushed Rock with Artificial Groundwater

6.5.4.1 Appropriately label and weigh a PI specified polyethylene amber container, and record its weight with a precision of 1%.

6.5.4.2 Weigh a selected amount of crushed rock prepared in 6.5.3.2, and record its weight with a precision of 1%.

6.5.4.3 Add the crushed rock to the container, place on a balance, and completely fill the container with the appropriate artificial groundwater. Record the weight with a precision of 1%.

6.5.4.4 A control sample is produced by adding only artificial groundwater to a container and recording the weight with a precision of 1%.

6.5.4.5 Place equilibrating rock:water mixtures and control samples at room temperature; weekly invert containers to facilitate mixing.

6.5.4.6 Following the frequency specified by the PI, pass a PI specified amount of the equilibrating sample through a 0.1 µm nylon filter. Analyze cation, anion, dissolved oxygen, pH and alkalinity concentrations and oxidation-reduction potentials following the procedures specified in 6.5.2.

6.5.4.7 Repeat Step 6.5.4.6 following the analytical schedule specified by the PI until the PI determines that the experiment can be ended.

6.6 Potential Sources of Error and Uncertainty

Potential sources of error and uncertainty for preparing and conducting experiments include the following:

- Improperly calibrated balances - The balance calibration due date is to be recorded with each data set.

- Contamination of crushed rock - DP-63 should be followed to avoid contamination of crushed rock samples.

- Mislabeling containers - Containers should be carefully labeled before samples are transferred to them.

- Reuse of filters - Sample filters should be disposed of after a single use.

- Inconsistent mixing - Samples must be inverted weekly.

6.7 Data Acquisition and Reduction

Throughout this procedure, it is mandatory to maintain data in a laboratory notebook and record identifiers of the balance(s) utilized. Analytical data are evaluated separately for each of the analyses employed (see DP-35, DP-107, DP-806 and QP-03.5).

7.0 RECORDS

Records generated as a result of this DP are entries in laboratory notebooks or attachments to laboratory notebooks. The documentation should consist of any applicable items identified in Section 6.0 of this procedure. Laboratory notebooks should be kept in accordance with QP-03.5.

All records should be submitted to the Records Processing Center in accordance with QP-17.6.

8.0 ACCEPTANCE CRITERIA

The criteria that show the correct implementation of this procedure are the records identified in Section 7.0. The acceptability and precision of the final reported analytical data are evaluated by the PI, taking into account the sources of error listed in Section 6.7.

9.0 TRAINING REQUIREMENTS

9.1 Training to this procedure is accomplished by “read only”. Training will be documented per QP-02.7.

10.0 ATTACHMENTS

NA