TWS-INC-DP-15, R2

CRUSHED ROCK COLUMN STUDIES

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CRUSHED ROCK COLUMN STUDIES

1. PROCEDURE IDENTIFIER: TWS-INC-DP-15

INTRODUCTION

2. PURPOSE

The purpose of this procedure is to describe dynamic transport tests on crushed rock columns by studying the flow of a known tracer with a known flow rate through the column.

3. SCOPE

This procedure applies to the assembly, preparation and sampling of LANL YMP crushed rock columns.

4. APPLICABLE DOCUMENTS

a. TWS-QAS-QP-05.2 "Preparation of a Detailed Technical Procedure"

b. TWS-QAS-QP-07 "Procedure for Technical Review of Publications"

c. TWS-QAS-QP-12.1 " NNWSI Instrument Calibrations"

d. TWS-MSTQA-QP-14 "Research and Development (Experimental) Procedure"

e. TWS-INC-DP-62, "Bulk NTS Well Water Samples"

f. TWS-INC-DP-63 "Preparation of NTS Core Samples for Crushed Rock Experiments"

5. RESPONSIBILITIES

The PI has the responsibility of organizing and overseeing all operations. He may assign appropriate tasks to personnel trained to this DP. It is the responsibility of the

users of this DP to adhere to the procedure. Investigators may direct deviations from the procedure upon approval of the responsible PI. It is the responsibility of the user to document such deviations in accordance with TWS-MSTQA-QP-14. If change requests are in process it is the responsibility of the user to document the procedure change in the laboratory notebook. It is the responsibility of the users of this DP to report unplanned deviations from this procedure to the responsible PI.

6. PRINCIPLE

This DP can be used to study crushed rock columns in order to verify the results of batch sorption measurements under flowing conditions. These studies by measuring differences from batch measurements will be most sensitive to multiple species formation, colloid formation, and other geochemical reactions.

7. DEFINITIONS

tracer solution = solution to be loaded onto the column containing the substance(s) whose transport and sorption behavior is to be studied.

eluate = the solution resulting form an elution process.

collection time = time interval between collection of eluate samples.

flow rate = the amount of liquid eluted through the column per unit of time.

$C_0(t)$ = tracer concentration in initial tracer solution at time t (i.e., the aliquot of the initial tracer solution taken at the beginning of the experiment corresponds to $C_0(t = 0)$).

$C_i(t)$ = tracer concentration in solution i at time t (solution i represents the eluate collected during time t_i).

PROCEDURE

8. ADEQUATE AND APPROPRIATE EQUIPMENT, INSTRUMENTATION, AND SOFTWARE

A pump is required to maintain a constant flow rate through the crushed rock

column. The column is made from acrylic tubing, cut to the length desired, with ends machined to fit polypropylene Luer fittings containing polyethylene frits. Commercially available hand tools are used to measure the length of the column. The outlet from the column to the collection container may be Teflon tubing. Samples from the column may be collected using a fraction collector. The volume of eluate collected depends on the sensitivity of the analytical technique to be used for quantitative analysis of the tracer in the eluate. Consequently, the volume of the collected samples varies depending on the experiment. A calibrated balance is used to determine the flow rate through the column.

9. EXPERIMENTAL PREPARATORY STEPS AND VERIFICATION

Before initiation of the experiment:

a. Prepare the crushed rock according to directions in TWS-INC-DP-63.

b. Prepare the Column

b.1 The column is packed with either dry or wet crushed rock. In either case, a Luer fitting with frit is attached to one end of the column and a funnel to the other.

b.2 If the column is to be dry packed, the rock is poured in and the column is tapped or vibrated, if necessary, to make the rock reach the bottom of the column. As a result of differences in particle densities some banding will appear. If the banding is too severe, the rock is removed from the column and the packing begun again. After the packing, the funnel is removed and replaced with a Luer fitting and frit. When water is pumped through the column, some settling may occur. If this happens, the top Luer fitting and frit are removed and more crushed rock is added.

b.3 If the column is to be wet packed, a slurry of crushed rock and water is poured into the funnel. Once the column is full, the funnel is removed and a Luer fitting and frit are attached. As with the dry packed column, if settling occurs, the top Luer fitting and frit are removed and more crushed rock added.

b.4 After either method of filling the column, the flow rate through the column is determined. Ten or fifteen test tubes are weighed and the weights recorded.

A definite volume of water is pumped through the column for a fixed period of time and collected in each of the tubes. The tubes are weighed again and the weights recorded. The weight of the water in each tube and the time of collection permit the determination of the flow rate.

c. measure the length of the column to within 0.25 in.

d. prepare the tracer solution.

e. place the vials to be used for eluate collection in the fraction collector.

Notebook Entries

Verify that the following entries have been made in the laboratory notebook:

a. unique identifier of the sample used to prepare the column (carried from the procedure TWS-INC-DP-63). If more than one column has been prepared from the same NTS sample and additional alphanumeric character will be added to the identifier in order to maintain uniqueness.

b. the length of the crushed rock column.

c. unique identifier of the water used (carried from TWS-INC-DP-62).

d. the flow rate through the crushed rock column.

e. tracer solution to be used and analytical method of preparation (e.g., the experimental steps used for preparation, notebook TWS number and pages used specifying these steps, or a detailed procedure number).

f. collection time to be used on fraction collector.

10. SUITABLE AND CONTROLLED ENVIRONMENTAL CONDITIONS

No special conditions are required for this DP. If any special conditions are utilized, record them in the laboratory notebook.

11. EXPERIMENTAL STEPS AND ACCEPTANCE OR REJECTION CRITERIA OF

DATA COLLECTED

a. Place the traced solution into the pump and remove all air bubbles from the system.

b. Turn collector on, start the pump, and record the Julian time; also place an aliquot of the tracer solution in a container (the tracer concentration of this aliquot will be used to determine C_0 at an arbitrary time t).

c. Determine the amount of tracer in the initial solution and in the eluates collected using the appropriate analytical method.

Notebook Entries

The following entries must be made in the laboratory notebook:

a. start time of eluate collection and amount of initial tracer solution to be analyzed, specifying the units of volume or weight used for measuring amount.

b. analytical technique used and reference to the detailed procedure for this technique.

The most common source of error that results in data rejection is leakage of the column. Leakage can be caused by loose Luer connectors. Leakage is detected by periodic visual inspection. If leakage is detected, record this problem in the laboratory notebook and reject the data.

12. POTENTIAL SOURCES OF UNCERTAINTY AND ERROR

Evaporation of the tracer in the collection vials can cause errors in the results. Consequently, keep the collection vials capped after eluate collection.

13. METHOD OF DATA REDUCTION

The tare and the final weight of eluate vials to be used for flow rate determination are recorded. These data are later reduced by subtracting the tare weights from final weights of the collection vials and dividing by the elapsed time in order to obtain the flow rate of the column. The relative concentration of the tracer, $C_i(t)/C_0(t)$, will be calculated for each eluate collected.

14. METHODS OF RECORDING AND STORING DATA AND RESULTS

The information recorded during preparatory verification has been specified in section 9. If any special conditions are used during the experiment, they should be recorded (see section 10). The information recorded during the experiment has been specified in section 11. The reduced data specified in section 13 will be stored on an optical disk cartridge using LOTUS 1-2-3 on an IBM PC.

15. SAMPLE/SITE TRACEABILITY

The unique identifier of the sample used to prepare the column will be carried from TWS-INC-DP-63. If more than one column has been prepared from the same NTS sample an additional alphanumeric character will be added to the identifier in order to maintain uniqueness. This identifier will be recorded in the laboratory notebook.

QUALITY ASSURANCE REQUIREMENTS

16. QUANTITATIVE OR QUALITATIVE ACCEPTANCE CRITERIA FOR DETERMINING THAT ACTIVITIES HAVE BEEN SATISFACTORILY ACCOMPLISHED

Upon completion of the experiment, verify that no leakage has occurred since the beginning of the experiment (see section 11). Verify that the information specified in sections 9-11 has been recorded in the laboratory notebook.

17. HANDLING, SHIPPING, AND STORAGE REQUIREMENT

No special requirements are necessary for samples used in this procedure.

18. IDENTIFICATION OF QA RECORDS TO BE GENERATED AND THEIR CONTROL

The records produced by this procedure will be the laboratory notebooks and a LOTUS 1-2-3 file on an optical disk cartridge having a TWS number. The data will be published in accordance with LANL Policy and TWS-QAS-QP-07. Investigators may direct deviations and modifications of the procedure for specific applications. Such actions are documented in notebooks.

19. TRAINING REQUIREMENTS AND METHODS

The PI or his designee will train the investigator assigned to use this DP. The training will require observation and evaluation of the performance of the trainee as he follows this DP.

20. CALIBRATED INSTRUMENTATION INFORMATION

The balance(s) used to determine the flow rate must be calibrated according to TWS-QAS-QP-12.1.

21. PROVISION FOR DOCUMENTATION, REPORTING, AND EVALUATION OF PROCEDURAL DEVIATION

Unplanned deviations from this procedure will be documented in the laboratory notebook. The responsible PI or his designee will make a determination as to whether to accept or reject data affected by the deviation. If a decision to accept the data is made, the justification for the acceptance must also be entered in the laboratory notebook.

22. SUBJECTS REQUIRING VERIFICATION

Verify that the entries specified in section 9 have been made in the laboratory notebook before starting the experiment.

23. APPENDIX AND/OR ATTACHMENTS

None