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SORPTION, DESORPTION RATIO DETERMINATIONS OF GEOLOGIC MATERIALS BY A BATCH METHOD

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SORPTION, DESORPTION RATIO DETERMINATIONS OF GEOLOGIC MATERIALS BY A BATCH METHOD

1. PURPOSE

The purpose of this procedure is to delineate the method for determining sorption and desorption coefficients for the distribution of various radionuclides between geologic materials and natural or synthetic groundwaters.

2. SCOPE

This procedure may be used for sorption and desorption analysis for any task that is applicable.

3. PROCEDURE

3.1 Selection of samples and identification of samples is made by a chemist in CNC-11 and/or a geologist in ESS Division.

3.2 Pretreated groundwaters are prepared by shaking natural or synthetic groundwaters with ground material. The solution-to-solid ratio used is specified in a notebook, and the contact time is at least two weeks. Contacts are made in labeled plastic bottles.

3.3 Groundwaters are centrifuged in a Sorvall Superspeed Centrifuge or equivalent for about one hour, filtered, and stored in plastic bottles identified by sample numbers or names. The centrifuge speed as well as type and pore size of filter will be entered in a notebook.

3.4 Each rock sample may be ground in a Fritsch pulverizer and sieved to desired size using U. S. A. Standard testing sieves, ASTM E-11 specification. The ground fractions are stored in bottles with appropriate identification. Alternate methods of sample preparation may be used. The operation is recorded in a notebook.

3.5 For each type of material, several samples are weighed using the analytical or top-loading balance and placed in labeled centrifuge tubes. The weight of the sample, including the tube, is determined at this time and recorded in a notebook.

3.6 A portion of the proper groundwater (volume to be recorded in a notebook) is added to each sample in its tube and shaken at approximately 200 oscillations/minute in a Lab-line Junior Orbit Shaker or equivalent at room temperature or at approximately 80 osc/min in a controlled temperature bath for at least two weeks.

3.7 Tracer solutions are prepared for each core using pre-treated groundwaters. Radionuclides used for the tracers include ^{85}Sr, ^{137}Cs, ^{133}Ba, ^{141}Ce, ^{152}Eu. Other radionuclides may be used without affecting this procedure. The tracers needed to prepare a traced solution are evaporated to dryness in a plastic tube in a Scientific Products Temp-Block module heater, a steambath or equivalent, or at room temperature. Concentrated HCl may be added, and the mixture again evaporated to dryness.

3.8 The appropriate volume of pre-treated groundwater is added to the tube and transferred to a properly identified plastic bottle and the solution is mixed for about 24 hours or an appropriate period. Whe mixing is completed, the solution if filtered. The type of the filter and pore size is entered in a notebook. The resulting tracer solution is stored in a plastic bottle identified by marking the proper description on the outside of the bottle. The solution is used within a few hours. Aliquots of each tracer solution are taken in labeled counting vessels for gamma counting on the appropriate detector. Documentation of gamma counting is in a notebook. Alternate methods of counting, approved by a staff member, will be used when appropriate.

3.9 The samples prepared in 3.6 above are centrifuged and the solutions are separated from the solids. The weight of solution remaining with the solid is determined by weighing the tube plus its contents after pre-treatment. The weight is recorded in a notebook.

3.10 An aliquot of the prepared tracer solution is added to each sample obtained in 3.8 and shaken at room temperature or at 70°C. Samples are removed at pre-selected time intervals. Once removed from the shaker, the samples are centrifuged three or four times, for about one hour each time. After each centrifuging, solutions are transferred to clean polyethylene tubes and re-labeled. Alternatively, solutions may be separated from solids by filtering through an appropriate filter.

3.11 Aliquots of the solutions are taken for gamma counting and labeled with the proper identification. Counting is done on the appropriate detector. The 4096 channel spectra are recorded in a multichannel analyzer connected to a PDP-9 computer. Results are documented in a notebook. If a multichannel system is not used, data from the counter are recorded directly in a notebook. Alternate counting methods, approved by a staff member, may be used as appropriate.

3.12 The pH of each sample solution is measured at this time, and results are documented in a notebook. The pH meter is standardized according to the manufacturer's instructions with two pH buffers. The pH is not deemed to be a critical measurement.

3.13 A portion of each solid sample, or the entire solid sample, is prepared for gamma counting. Gamma counting is done on the appropriate detector with results recorded in a notebook. Alternate methods of radioassay may be used when appropriate.

3.14 Desorption experiments are conducted following procedure steps 3.10 through 3.13 using untraced pre-treated water added to the solid samples already used for sorption determinations.

Calculations of R_d sorption ratios are performed using the expression

$$R_d = \frac{\text{activity per g of solid}}{\text{activity per ml of solution}}$$

4. CALIBRATION

Balances and gamma counters are included in the calibration program. Other devices used in soprtion-desorption determinations need not be highly accurate and are therefore not included in the calibration program.

5. DOCUMENTATION

Data collected on a day-to-day basis are included in Laboratory notebooks and controlled in accordance with TWS-CMBQA-QP-03, "Document Control." The data will be published in accordance with Los Alamos National Laboratory policy and TWS-CMBQA-QP-07. Staff mambers may direct deviation and modifications of the procedure for specific applications. Such actions are documented in notebooks.

6. PERSONNEL QUALIFICATIONS

Staff members and technicians assigned to this work shall be qualified in accordance with section 10. of TWS-CMBQA-QP-03, "Document Control."