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Radiochemical Separation by Solvent Extraction

In this experiment radiochemical separation by solvent extraction is illustrated through the use of the readily available radionuclide ^{32}P. Phosphorus is extracted from aqueous solution into an organic solvent as molybdophosphoric acid[1]. This type of separation from nonextractable radioactive substances has been used in the determination of ^{32}P in the effluent cooling water from the Hanford reactors[2].

Apparatus and Materials

Radiation-measurement equipment
- Two glass-stoppered 10-ml graduated separatory funnels or graduated cylinders
- Support stands for separatory funnels
- Micropipette with control

Four volumetric pipettes, 0.50 or 1.00 ml, with control
- Source-mounting materials, e.g., glass-cupped planchets
- ^{32}P tracer

Waste container for ^{32}P

Reagent solutions
- Sulfuric acid, 6 *N*
- Ammonium molybdate, 10 percent

Phosphate carrier, e.g., H_3PO_4, 50 mg/ml
- Ethyl acetate
- Ammonium hydroxide, 6 *N*

- Two 5-ml pipettes with control
- Two 15-ml centrifuge tubes with rack or block support
- Heat lamp

Procedure

To a 10-ml graduated separatory funnel or cylinder add 3.0 ml of distilled water, 3 drops of H_2SO_4, phosphate carrier (one of the following amounts: 0.5, 1.0, 5.0, or 10 mg), and ^{32}P in an amount sufficient to provide satisfactory counting rates. To a second funnel add exactly the same materials except for the carrier, which should be replaced by distilled water. Mix the solution and add 4 drops of ammonium molybdate solution. Mix the solution again. Add 3.0 ml of ethyl acetate and shake the funnel vigorously for 1 to 2 min. Be certain the stopper is tightly seated, and use gloves. Allow the layers to separate, and record the volume of each.

Carefully transfer the contents of the funnel to a 15-ml centrifuge tube and separate the phases by centrifugation. Using separate pipettes withdraw aliquots, e.g., 0.5 ml, of the organic and aqueous phases from each tube. Take the sample from the organic phase first, avoiding contact with the lower aqueous layer. Next remove the remainder of the organic layer plus a little of the aqueous layer and finally take an aliquot of the aqueous layer with a

clean pipette. The unused portions of each layer should be transferred to the waste container. The aliquots for counting should be dried carefully in labeled glass-cupped planchets. It is important that the organic layers be dried very slowly under the heat lamp to avoid creepage of the liquid to the top of the planchet. The latter behavior may be detected visually for the sample with added carrier, since the molybdophosphoric acid is yellow. When the sources appear to be about dry, add 4 drops of ammonium hydroxide to neutralize the acid and dry the sources completely. Measure the counting rates of all four samples in a like manner.

Analysis of Data

Calculate the distribution ratio and the percent extraction for each extraction system. When this experiment is performed by several laboratory groups, the results for the carrier-free system should be compared and the results for various carrier concentrations collected and presented graphically to show the dependence on initial phosphate concentration (moles per liter) in the aqueous phase.

1 G.H. Morrison and H. Freiser, "Solvent Extraction in Analytial Chemistry," Wiley, New York, 1957.

2 W.B. Silker, Radiochemical Determination of Phosphorus-32, *Anal. Chem.*, vol. 28, pp. 1782-1783, 1956.