Radioactive Phosphorus

Principle of Method

Phosphorus carrier and appropriate scavenging carriers are added to the acidified aqueous sample and impurities are removed by a double hydroxide precipitation. The phosphorus is precipitated as $MgNH_4PO_4$ for counting.

Procedure Time

4 samples - 4 hrs.

Reagents

Ammonium hydroxide, NH_4OH: 15 N (conc.), 1 N
Cobalt carrier: 5 mg/ml
Diethyl ether, $(C_2H_5)_2O$: anhydrous
Ethanol, C_2H_5OH: 95%
Hydrochloric acid, HCl: 12 N (conc.), 6 N
Hydrogen peroxide, H_2O_2: 30%
Indicator, methyl red: 0.1%
Magnesia mixture: prepared reagent
Manganese carrier: 5 mg/ml
Nitric acid, HNO_3: 6 N
Phosphorus carrier: 5 mg/ml
Potassium hydroxide, KOH: 6 N
Silver carrier: 20 mg/ml
Zirconium carrier: 10 mg/ml

Procedure

1. To an aqueous sample (200 ml or less), add 1 ml 6 N HNO_3, 1.0 ml phosphorus carrier and 0.5 ml each cobalt, zirconium, silver and manganese carriers.
2. Slowly add at least 2 drops H_2O_2 and 6 N KOH until precipitation of hydrous oxides is complete. Heat to coagulate and filter while hot through Whatman #41 (or equivalent). Discard filter paper.

3. To the filtrate add 2 drops cobalt and zirconium carriers and 2 drops H_2O_2. Heat to coagulate and filter while hot through Whatman #41 (or equivalent). Discard filter paper.

4. Acidify the filtrate with 12 N HCl and boil a few minutes to remove excess H_2O_2. Cool in an ice bath.

5. Add 3 ml magnesia mixture and neutralize to methyl red end point with 15 N NH_4OH to precipitate $MgNH_4PO_4$. Let stand 2 minutes then add 3 ml excess 15 N NH_4OH. Centrifuge and discard supernatant.

6. Wash the precipitate with 10 ml 1 N NH_4OH and discard wash solution.

7. Dissolve precipitate with 1 ml 6 N HCl. Transfer to an ice bath, add 10 ml water and 3 ml magnesia mixture and neutralize to methyl red end point with 15 N NH_4OH. Let stand 2 minutes then add 3 ml excess 15 N NH_4OH. Centrifuge and discard supernatant.

8. Repeat steps 6 and 7 using only 1 ml magnesia mixture.

9. Wash the precipitate with 10 ml 1 N NH_4OH and discard wash solution.

10. Transfer to a tared glass-fiber filter with 1 N NH_4OH. Wash with successive portions of 1 N NH_4OH, ethanol and ether.

11. Dry, cool, weigh, mount and count.

Calculation

Calculate the concentration, D, of phosphorus-32 in picocuries per milliliter as follows:

$$D = \frac{C}{2.22 \times EVR}$$

where:

C = net count rate, counts/min,
E = counter efficiency,
V = milliliters of sample used,
R = fractional chemical yield, and
2.22 = conversion factor from disintegrations/min to picocuries.

Calculate the decay correction for ^{32}P ($t_{1/2}$ = 14.3 d) as follows:

$$A = A_o \, e^{-0.693t/T}$$

where:

A = activity at time t,
A_o = activity at time zero,
e = base of natural logarithms,
t = elapsed time from collection, and
T = half life of ^{32}P, in same units as t.

Confirmation of Purity and Identification of ^{32}P

1. Plot the gamma-ray spectrum of the separated sample to ascertain whether any gamma photopeaks from impurities are present.
2. Beta count the planchet at 1-week intervals to corroborate the 14.3 d half-life of the phosphorus activity.

References:

1. Bowen, H. J. M. and Gibbons, D., Radioactivation Analysis (Oxford University Press, London, 1963) p. 222.
2. Kolthoff, I. M. and Sandell, E. B., Textbook of Quantitative Inorganic Analysis (Macmillan, New York, N. Y., 1952) pp. 316 and 379.

Addendum to Radioactive Phosphorus Procedure

Reagent Preparation

Distilled or deionized water should be used in the preparation of all reagents requiring water as the solvent.

Carrier Solutions – These solutions, prepared as the specific ion, are to be filtered and standardized prior to use. If the carrier is not used for chemical yield they do no have to be standardized.

Ag^{+} - 20 mg/mL. Dissolve 3.150 g $AgNO_3$ in water and dilute to 100 ml. Store in the dark in a brown glass container.

Co^{+2} - 5 mg/mL. Dissolve 2.469 g $Co(NO_3)_2 \cdot 6H_2O$ in water and dilute to 100 mL.

Mn^{+2} - 5 mg/mL. Dissolve 1.801 g $MnCl_2 \cdot 4H_2O$ in water and dilute to 100 mL.

P^{+5} – 5 mg/mL. Dissolve 2.197 g KH_2PO_4 in water and diluted to100 mL (must be standardized).

Zr^{+4} - 10 mg/mL. Dissolve 3.53 g $ZrOCl_2 \cdot 8H_2O$ in 0.1 N HCl and dilute to 100 mL with 0.1 N HCl.

Magnesia Mixture. – Dissolve 20 g NH_4Cl in water, add 10 g $MgCl_2$ and dilute to 100 mL.