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AN ANION EXCHANGE-PRECIPITATION PROCEDURE FOR
THE DETERMINATION OF Na²⁴ IN REACTOR EFFLUENT WATER

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AN ANION EXCHANGE-PRECIPITATION PROCEDURE FOR
THE DETERMINATION OF Na^{24} IN REACTOR EFFLUENT WATER

INTRODUCTION

The isotope Na^{24} is one of the major short half-life constituents of reactor effluent water. It has a half-life of 15 hours⁽¹⁾ and decays of emission of a 1.39 Mev maximum beta particle. Other radioisotopes present in reactor effluent water from which Na^{24} must be separated are Si^{31} , P^{32} , Mn^{56} , Cu^{64} , As^{76} , Sr^{91-92} , $\text{Ba}^{139-140}$ and rare earths. This report describes a rapid and very simple method for the quantitative determination of Na^{24} in a radiochemically pure form.

THEORY

Recent work on precipitation separations with anion exchange resins⁽²⁾ has demonstrated that cations can be precipitated from solution by passing the solution through an anion exchange bed saturated with the precipitating anion. It was demonstrated that these precipitates form around the resin particle as adhering films and a clear effluent is obtained from the column. The efficiency of this type of precipitation is not dependent on the concentration of the cation (if column saturation is not approached) and is thus equally efficient for tracer or macro concentrations.

The radioisotopes in reactor effluent water are believed to exist in the ionic forms listed below.

Cations
 Cu^{++}
 Mn^{++}
 Na^{+}
 Ba^{++}
 Sr^{++}
 RE^{+3}

Anions
Silicates
Phosphates
Arsenates °

If a solution containing these ions were allowed to pass slowly through an anion exchange column in the carbonate form, it would be expected that the cations, with the exception of Na^+ , would be precipitated as carbonates on the resin, and that the anions would be retained on the column by the normal anion exchange reaction. The only radioisotope carried through in the effluent would then be Na^{24} .

EQUIPMENT

The columns were 200 mm by 10 mm ID West Condensers modified by removing the male standard taper joints and replacing them with 2 mm stopcocks. Dowex 1, 8% crosslinkage (CO_3^- , 100 - 200 mesh, Lot No. 3562-18), was the anion exchange resin employed. The column reservoirs were 6" x 35 mm ID tubes connected to a 24/40 standard taper joint through a 2 mm stopcock.

A photograph of a packed column and column reservoir is shown in Figure 1.

ANALYTICAL PROCEDURE

The column preparation and analytical procedure are described in detail in Appendices A and B respectively.

The anion exchange column (carbonate form) is maintained at $60^\circ - 65^\circ \text{C}$ by circulating hot water through its condenser jacket. A 25 ml aliquot of reactor effluent water containing 1 ml of sodium carrier (10 mg NaNO_3/ml) is placed in the column reservoir and allowed to percolate through the column. This is followed with 50 mm of distilled water. The combined effluent is acidified with HNO_3 , evaporated to a small volume, then evaporated to dryness on a standard 1" counting dish for beta counting.

FIGURE 1
RESIN COLUMN AND COLUMN RESERVOIR FOR Na²⁴ ANALYSIS
Photograph unclassified.

RADIOCHEMICAL YIELD AND PURITY

^{24}Na radioisotope for making radiochemical yield measurements was obtained from reactor effluent water by separation on an anion $[\text{CO}_3^-]$ exchange column. Eight twenty-five ml aliquots of the solution were then analyzed for ^{24}Na by the analytical procedure. Two standard plates were prepared by evaporating 25 ml aliquots of the solution with 1 ml of Na carrier on 1" counting dishes. The radiochemical yield for the procedure was calculated by comparing the counting rates of the analyzed aliquots with that of the standard plates. The yields obtained are shown in Table I.

TABLE I
RADIOCHEMICAL YIELD DETERMINATION

<u>Observed c/m</u>	<u>Time Counted</u>	<u>Corrected*₃ c/m, x 10⁻³</u>	<u>Yield, %</u>
2590.7	1520	2.61	95.2
2513.5	1720	2.78	101.5
2294.7	1730	2.56	93.5
2373.5	1745	2.68	97.8
2483.0	1800	2.83	103.3
2465.9	1810	2.83	103.3
2431.6	1820	2.82	103.0
2362.3	1835	2.76	100.7
Standard plates, Average		2.74	
Average Yield			99.6%
Standard Deviation			3.6%

*Corrected for decay

The above data indicate a high and fairly reproducible radiochemical yield.

Decay measurements were made on the ^{24}Na separated from approximately fifty reactor effluent water samples and all decay measurements showed a high radiochemical purity. A typical decay curve for the ^{24}Na separated from a reactor effluent water sample is shown in Figure 2.

FIGURE - 2
TYPICAL DECAY CURVE FOR No^{24}
SEPARATED FROM REACTOR EFFLUENT WATER

COLUMN LIFE

⊙ The number of analyses for which a column can be used before regeneration is required is limited by the exchange capacity of the ion exchange column and the salt content of the reactor effluent water. A packed column contains about 15 meq of anion exchange resin and the total salt content of reactor effluent water is about 1 meq/liter as measured by ion exchange.⁽³⁾ The sodium carrier added to the reactor effluent water samples is at a concentration of about 5 meq/liter so the theoretical exchange and/or precipitation capacity of the column is about 2500 ml, or 100-25 ml samples.

Exhaustive column tests have not been made for the procedure, but breakthrough of other radioisotopes was not observed after using a column for 29 analyses. After this number of analyses a column is about 30% exhausted, and further usage may risk the breakthrough of other radioisotopes. Probably 20 analyses per column before regeneration would be a practical and safe number.

DISCUSSION

Heated columns are used for the analysis mainly because faster flow rates can be obtained; however, tests have shown that ion exchange and precipitation reactions also occur faster in heated column, thus allowing a shorter column to be used. The analysis can be performed satisfactorily using slightly longer columns (8" columns) without heating but much slower flow rates are realized.

The Na²⁴ analysis can be performed without carrier; however, the yield is erratic and somewhat lower than when carrier is present. Studies with various amounts of carrier indicated that 1 ml (1 mg NaNO₃/ml) was sufficient to make the procedure quantitative. If it were desired to check the yield gravimetrically, larger quantities of carrier could be used; however, this would reduce the column capacity correspondingly.

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APPENDIX A
COLUMN PREPARATION

1. Activate a suitable quantity of anion resin (Dowex 1, 8% crosslinkage, 100-200 mesh) by washing it alternately two or three times with 1 M HCl and 1 M NaOH.
2. Place a glass wool plug in the bottom of the column and fill the column with resin by pouring a water slurry of the resin into the column.
3. Place the column reservoir on the column and allow 50 ml of about 2 M Na_2CO_3 to percolate through the column.
4. Allow distilled water to percolate through the column until the effluent is neutral to pH indicator paper.

APPENDIX B
ANALYTICAL PROCEDURE

1. Circulate hot water ($60^\circ - 65^\circ\text{C}$) through the condenser jacket of the column during the filtration.
2. To 25 ml of reactor effluent water add 1 ml of Na carrier (10 mg NaNO_3/ml) and allow the solution to filter through the column.
3. Add 50 ml of degassed water to the column reservoir and allow it to flow through the column. (Note 1)
4. Acidify the combined effluents with HNO_3 and evaporate to about 3 ml.

Note 1. Degassed water is used to prevent formation of gas bubbles in the column. Sufficient degassing of the water is obtained by bringing it to a boil.

5. Transfer the solution to a standard 1" counting dish and evaporate to dryness under a heat lamp (Note 2). Wash the beaker with two 2 ml portions of H_2O and evaporate these on the counting dish also.
6. Measure the beta activity in a standard beta counter.

Note 2. A thin layer of silicon grease placed around the rim of the counting dish will prevent the salts from creeping onto the rim during drying.

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ABSTRACT

An analytical procedure for the determination of Na^{24} in reactor effluent water was developed. Na^{24} is separated from the other radioisotopes by passing a reactor effluent water sample through an anion $[\text{CO}_3^-]$ column where the radioanions are removed by anion exchange and the radiocations, with the exception of Na^{24} , by precipitation on the resin as insoluble carbonates. The Na^{24} is collected in a radiochemically pure form in the effluent and is evaporated on a standard 1" dish for beta counting.

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