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A MODIFIED RADIOCHEMICAL STRONTIUM PROCEDURE

by

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SUMMARY

A modified procedure for radiochemical strontium analysis has been developed. Data are presented to show that comparable results and radiochemical purity are obtained for strontium samples prepared by the modified procedure compared to samples prepared by the conventional fuming nitric acid procedure.

The modified procedure consists of adding strontium carrier to the sample, removing three ferric hydroxide scavenging precipitates, and isolating the strontium as strontium carbonate.

INTRODUCTION

The conventional procedure used for the radiochemical strontium analysis of fission product solutions has been that of Glendenin¹ which uses fuming nitric acid to separate the strontium and barium activities from the bulk of the fission products. The strontium and barium activities are further purified by ferric hydroxide scavenging precipitations, and the barium activity separated by a barium chromate precipitation at pH 4 to 5. The purified strontium is finally precipitated as strontium oxalate.

The above procedure has been hazardous for routine laboratory analysis because of the use of fuming nitric acid as a reagent. This reagent causes very severe acid burns when in contact with the skin, and its use presents a definite hazard.

The fuming nitric acid precipitation step of the regular strontium procedure was used to get a bulk separation of the barium and strontium activities from the rest of the fission products. In the modified procedure this same purification is accomplished by three ferric hydroxide scavenging precipitations. The fission activities, such as cesium, which are commonly encountered and which are not removed by the ferric hydroxide, are separated by the final carbonate precipitation of the strontium and barium carriers.

¹Coryell and Sugarman, Radiochemical Studies: The Fission Products (McGraw-Hill Book Co., New York, 1951), N.N.E.S. Div. IV, Vol. 9, Book 3, Paper 236.

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A short radiochemical strontium procedure may be useful for waste disposal work since all wastes must be surveyed for strontium activity before discharge from the Laboratory. This is necessary since strontium activities present a health hazard, and precautions must be taken to prevent accidental discharge of this activity in any quantity. The modified procedure can be used to survey a waste in about a half hour if one omits the chemical yield determination as this value is fairly constant at about 60 per cent. Thus, the modified procedure will expedite the discharge of wastes in which the absence of the bone-seeker strontium can be easily demonstrated.

EXPERIMENTAL

A modified radiochemical procedure has been developed to fulfill the above needs. The detailed procedure is given in the Appendix.

The results of the analysis of four mixed fission product solutions which had decayed various lengths of time are given in Table 1. The solutions were analyzed by both the fuming nitric acid procedure and the modified procedure. The values listed give the average of four samples and the standard deviation.

Aluminum absorption curves were made on the beta activity of two strontium samples isolated from Solution B; Sample 1 was prepared by the fuming nitric acid procedure, and Sample 2 was prepared by the modified procedure. The aluminum absorption curve data are presented in Figure 1. The beta activities of the two samples were identical within experimental error. The samples show two betas whose energies, by visual range determination, are 0.5 Mev and 1.6 Mev with no associated gamma activity. These results agree well with the published values of 1.50 Mev for the ^{89}Sr beta and 0.54 Mev for the ^{90}Sr beta² with no associated gammas.

DISCUSSION

Radiochemical strontium analysis done by the modified procedure gives results comparable with those obtained by using the fuming nitric acid procedure. Comparison of data obtained by both procedures shows no bias. The precision of the modified procedure appears to be slightly better than that of the fuming nitric acid procedure. The radiochemical purity of samples prepared by either procedure are identical, based on aluminum absorption curve studies of the beta radiation.

The modified procedure requires 1.5 hours to run a sample in quadruplicate, which is about 0.5 hour shorter than the time required to carry out the fuming nitric acid procedure.

²National Bureau of Standards Circular 499.

If one were to omit the chemical yield determination part of the procedure, answers accurate to $\pm 30\%$ could be obtained in about 0.5 hour. This type of analysis would be very useful since a quick, semi-quantitative analysis is often necessary before waste disposal.

Table 1

COMPARISON OF RADIOCHEMICAL STRONTIUM ANALYSIS OF
MIXED FISSION PRODUCT SOLUTIONS BY THE FUMING
NITRIC ACID AND MODIFIED PROCEDURES

Values given are the averages of four separate
determinations with the standard deviations.

Solution	Days Since End of Irradiation	Strontium, ct./((min.)(ml.))	
		Fuming HNO ₃ Procedure	Modified Procedure
A	120	16,280 $\pm$ 210	16,860 $\pm$ 440
B	1898	37,850 $\pm$ 220	36,890 $\pm$ 170
C	1898	15,010 $\pm$ 340	15,120 $\pm$ 220
D	438	1,980 $\pm$ 70	1,990 $\pm$ 50

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FIGURE 1.

ALUMINUM ABSORPTION CURVE ON Sr^{89} – Sr^{90}
ACTIVITY ISOLATED FROM MIXED FISSION ACTIVITY.

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APPENDIX

Modified Procedure for Radiochemical Strontium Analysis

The modified procedure is as follows:

1. Place the sample (1 to 15 ml volume) in a 50-ml centrifuge tube and add strontium carrier (Note 1). Add 5 mg ferric ion (10 mg Fe^{+3} /ml, 1 N nitric acid) (Note 2), heat almost to boiling, and add 6 M ammonium hydroxide dropwise until the ferric ion is precipitated. Centrifuge and decant supernate into a clean 50-ml centrifuge tube.
2. Add 5 mg ferric ion (10 mg Fe^{+3} /ml, 1 N nitric acid) (Note 2) to supernate from Step 1, heat almost to boiling, and add 6 M ammonium hydroxide dropwise until ferric ion is precipitated. Centrifuge and decant supernate into a clean 50-ml centrifuge tube.
3. Repeat Step 2. In the decantation step decant the supernate through a piece of Whatman 41 H filter paper into the clean 50-ml centrifuge tube (Note 3).
4. Neutralize supernate with 6 N nitric acid and add 1 ml 3 M acetic acid and 4 ml 6 M ammonium acetate. Add 10 mg barium carrier (10 mg Ba^{+2} /ml prepared from barium nitrate and water), heat to boiling, and add 1 ml 1.5 M sodium chromate dropwise with stirring. Let tube stand for about 2 minutes, then centrifuge and decant supernate into a clean test tube. Add 10 mg barium carrier to solution to make a second barium chromate scavenging precipitation. Let stand 2 minutes, centrifuge, and decant supernate through a piece of Whatman 41 H filter paper (Note 3) into a clean 50-ml centrifuge tube (Note 4).
5. Heat the supernate almost to boiling and add 15 ml 3 M ammonium hydroxide - 3 M ammonium carbonate solution. Let stand for about 5 minutes with occasional stirring. Centrifuge and discard supernate. Wash precipitate in test tube with 3 to 10 ml water washes followed by 3 to 10 ml absolute ethanol washes. After third alcohol wash collect strontium carbonate on a pretreated piece of 7/8-in. diameter Whatman No. 42 filter paper (Note 5) and dry by the rare earth vacuum drying technique (Note 6).
6. Mount the sample and count. Be sure to count sample not later than 4 hours after the last ferric hydroxide scavenging (Note 6).

Notes7

1. Add sufficient calibrated strontium carrier so that after the chemistry has been completed a final precipitate of 15 to 20 mg strontium carbonate will be collected. The strontium carrier is calibrated by precipitating and collecting the strontium as strontium carbonate using gravimetric techniques followed by washing, drying, and weighing as used in the procedure.
2. The ferric ion solution should be made up of about 10 mg Fe^{+3} /ml in 1 N nitric acid using ferric chloride. The nitric acid prevents hydrolysis and also prevents the ferric ion from precipitating immediately when added in Steps 2 and 3, since the added nitric acid serves to neutralize any residual ammonium hydroxide.
3. Whatman 41 H filter paper is a fast filtering paper and serves to retain some suspended particles which would be decanted with the supernate. The paper recommended for use is 7-cm diameter and is folded first into half and the resulting half folded into thirds to give a cone which is placed in the top of the tube and through which the supernate is decanted.
4. Step 4 may be omitted if barium activity is known to be absent.
5. Pretreat the filter paper before use exactly as done in the sample preparation and dry in the same manner before weighing.
6. The rare earth vacuum drying technique is as follows: evacuate for 2 minutes, release vacuum, evacuate for 15 seconds, release vacuum, evacuate for 2 minutes, release vacuum, evacuate for 15 seconds, release vacuum, evacuate for 2 minutes, release vacuum and weigh. It has been found that in this drying process the filter paper becomes dehydrated and requires about 5 minutes to reach moisture equilibrium with the surrounding air; this equilibrium can be checked by determining that there is no further gain in weight.
7. Routine fission product samples contain two strontium activities, 54d Sr^{89} and 20y Sr^{90} . The Sr^{89} decays to stable Y^{89} but the Sr^{90} decays to Y^{90} which decays with a 61h half life by a 2.18 Mev beta to stable Zr^{90} . The sample as received is in secular equilibrium with respect to Sr^{90} - Y^{90} , but this equilibrium is destroyed with the ferric hydroxide scavengings since the Y^{90} is removed and the Sr^{90} remains in solution. Thus, the final separated strontium will exhibit beta growth due to the Y^{90} , so it is important to limit the time after separation to less than 4 hours to prevent counting difficulties from this beta growth.