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SEPARATION OF IRON, COBALT, ZINC, AND PHOSPHORUS ON SYNTHETIC RESIN

Harold L. Helwig, James K. Ashikawa, and Elmer R. Smith

July 19, 1954

Berkeley, California

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ABSTRACT

This paper describes an ion-exchange column technique, employing Dowex-I anion-exchange resin, for the separation of radioactive iron, cobalt, and zinc in neutron-irradiated biological tissue ash. The method described is particularly useful as a radiochemical technique because phosphorus, a common contaminant with cation exchangers, is easily separated from the cations mentioned.

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INTRODUCTION

In activation analysis the separation of trace elements from neutron- or cyclotron-irradiated biological tissue ashes by standard radiochemical methods often becomes unwieldly owing to the large amount of phosphorus which constitutes the major portion of the total radioactivity of the sample shortly after irradiation. Furthermore, chemical methods of separation are often laborious and time-consuming, requiring the addition of precipitating agents, heating or cooling, centrifugation, filtration, extraction, etc. In addition, the experimenter is unnecessarily exposed to radiation. In view of these difficulties, a method that is efficient and easy to execute is desired. Recent investigations by Kraus and Moore on the behavior of the transition elements, Mn to Zn, in hydrochloric acid on the anion-exchange resin Dowex-I[1,2,3] have suggested the use of this resin as a possible method of separating the radioisotopes produced by neutron activation.

PROCEDURE AND MATERIAL

Preparation of the Ion-Exchange Columns

To remove trace impurities, 500 grams of the commercially available anion-exchange resin was placed in a column 9 cm in diameter and washed

by passing several liters of 0.5 M HCl through the resin followed with several liters of 5.0 M HCl. The washed resin was stored in 5.0 M HCl until used in packing the ion-exchange column. This washed resin was then pipetted and packed down by air pressure (fed through a syringe) into pyrex ion-exchange columns[4], and packed with a thin layer of glass wool at the bottom of the column to prevent the resin from passing through. The columns of about 1.4 cm^2 cross-sectional area were packed with 12 to 24 cm of resin and equilibrated with 12.0 M HCl.

Iron, Cobalt, Zinc, and Phosphorus.

The radiopurity of the Fe^{99}, Co^{60}, Zn^{65}, and P^{32} samples obtained by neutron bombardment was determined with the gamma-ray spectrometer and by measuring their rate of decay. The carrier salts were prepared by dissolving 0.625 g of iron, 0.625 g of zinc, and 0.125 g of cobalt in moderately strong hydrochloric acid. Radioactive iron, phosphorus, zinc, and cobalt were then added to these salts and the solution heated to near dryness. After a minute of cooling, 2 to 3 drops of concentrated nitric acid were added to insure complete oxidation of the metals[5] before taking the solution down to dryness. The oxides were converted to the chlorides with a few drops of concentrated hydrochloric acid, and gently heated to dryness. The salts were then taken up in a 25-ml volumetric flask and made up to volume with 12 M hydrochloric acid.

Method of Elution

A 2-ml aliquot of this solution was pipetted onto the resin bed and the phosphorus, cobalt, iron, and zinc successively eluted from the column with 12 M, 4 M, 0.5 M and 0.005 M hydrochloric acid[3]. The acid was fed by a mechanically operated syringe pump at a constant flow rate

of 10 ml per cm^2 per hour. Elution curves for phosphorus, cobalt, iron, and zinc were obtained by collecting 1.67-ml aliquots of eluate continuously with an automatic sample collector, dried at 60° C in an oven, and counted.

Radioactivity Measurement

The recovery of the eluted components was based on radioactivity measurements. The gamma activity of the iron, cobalt, and zinc was determined by counting aliquots of the liquid samples in one-dram vials with a sodium iodide crystal counter. The beta activity of the phosphorus fraction was determined with a G-M counter in polyethylene counting cups.

RESULTS AND DISCUSSION

Duplicate sets of columns containing 12 and 24 cm of Dowex-I resin were used to obtain the elution curves for phosphorus, cobalt, iron, and zinc, and to determine the percent recovery of the added radioactivity and the possible cross-contamination of the active components. Two ml of the carrier salts containing 7.50×10^5 cpm of P^{32}, 8.00×10^4 cpm of Co^{60}, 1.60×10^5 cpm of Fe^{59}, and 1.05×10^5 cpm of Zn^{65} of high specific activities were placed on each column and eluted with hydrochloric acid. As shown in Fig. 1, under these conditions, P^{32} is very slightly adsorbed on the resin and was readily removed with 12 M HCl. No activity was detected in the fraction eluted with 60 ml of 6 M HCl. In our work with copper, gallium, and zinc (unpublished), copper has been found to appear in this fraction and gallium in the iron fraction. Cobalt, iron, and zinc were readily removed with 50 ml each of 4 M HCl,

0.5 M HCl and 0.005 M HCl respectively. As suggested by Moore and Kraus[3], the flow rate was increased in one set of columns to 20 ml per cm^2 per hour with no apparent change in the elution curves.

On the basis of decay rate, gamma-ray spectroanalysis, and activity measurements, the recovery of the radioactive components from the resin was better than 98 percent, with no indication of cross-contamination in the cobalt, iron, and zinc fraction (see Figs. 3, 4, and 5). For comparison, controls of these trace metals were also prepared, measured, and plotted on the figures above. The phosphorus fraction (Fig. 2) appeared very slightly contaminated by some long-life component. However, colorimetric analysis of this fraction for cobalt, iron, and zinc was negative.

ACKNOWLEDGMENT

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5. H. L. Helwig, J. K. Ashikawa, H. Clokie, and E. R. Smith, Separation of Iron and Cobalt on Synthetic Resin, University of California Radiation Laboratory Report No. 2296, Aug. 1953.

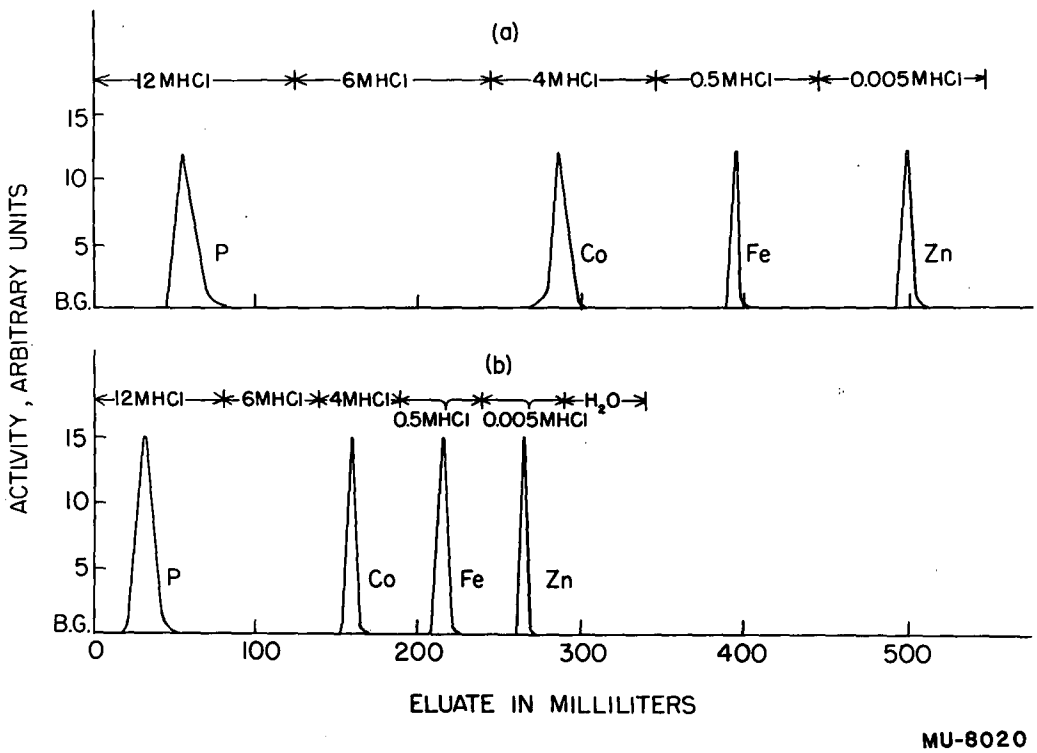
(a)
12 M HCl
6 M HCl
4 M HCl
0.5 M HCl
0.005 M HCl
15
10
5
B.G.
P
Co
Fe
Zn
(b)
12 M HCl
6 M HCl
4 M HCl
0.5 M HCl
0.005 M HCl
H_2O
15
10
5
B.G.
P
Co
Fe
Zn
0
100
200
300
400
500
ACTIVITY, ARBITRARY UNITS
ELUATE IN MILLILITERS
MU-8020

Fig. 1

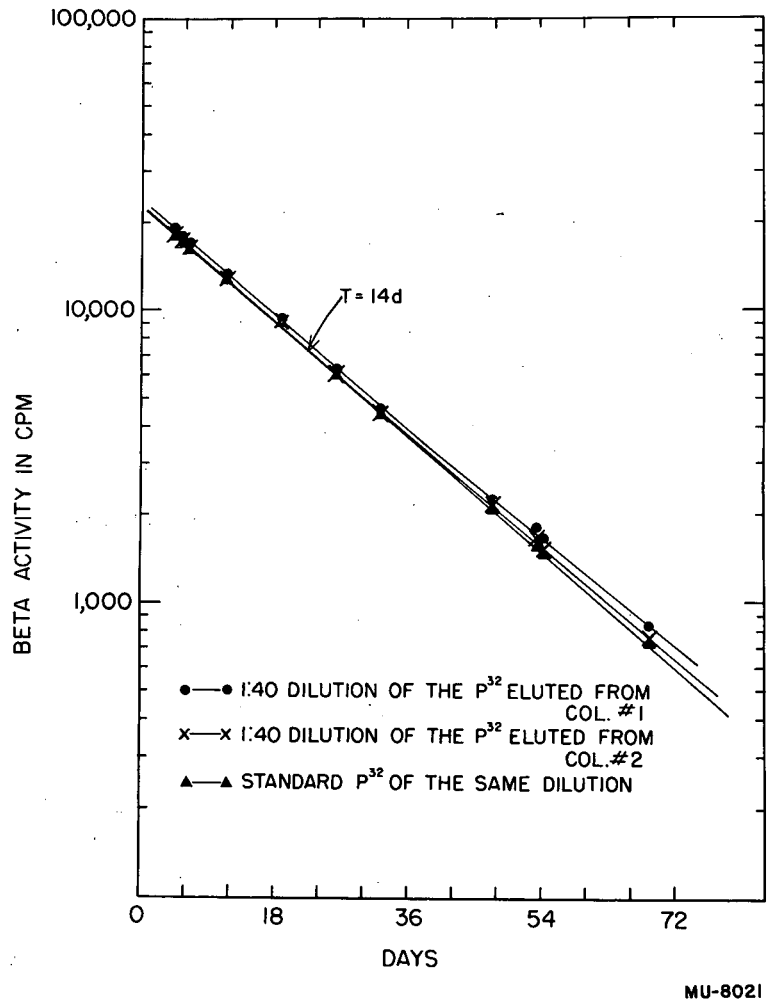
100,000
10,000
1,000
BETA ACTIVITY IN CPM
T = 14d
0
18
36
54
72
DAYS
●—● 1:40 DILUTION OF THE P32 ELUTED FROM COL. #1
x—x 1:40 DILUTION OF THE P32 ELUTED FROM COL. #2
▲—▲ STANDARD P32 OF THE SAME DILUTION
MU-8021

Fig. 2

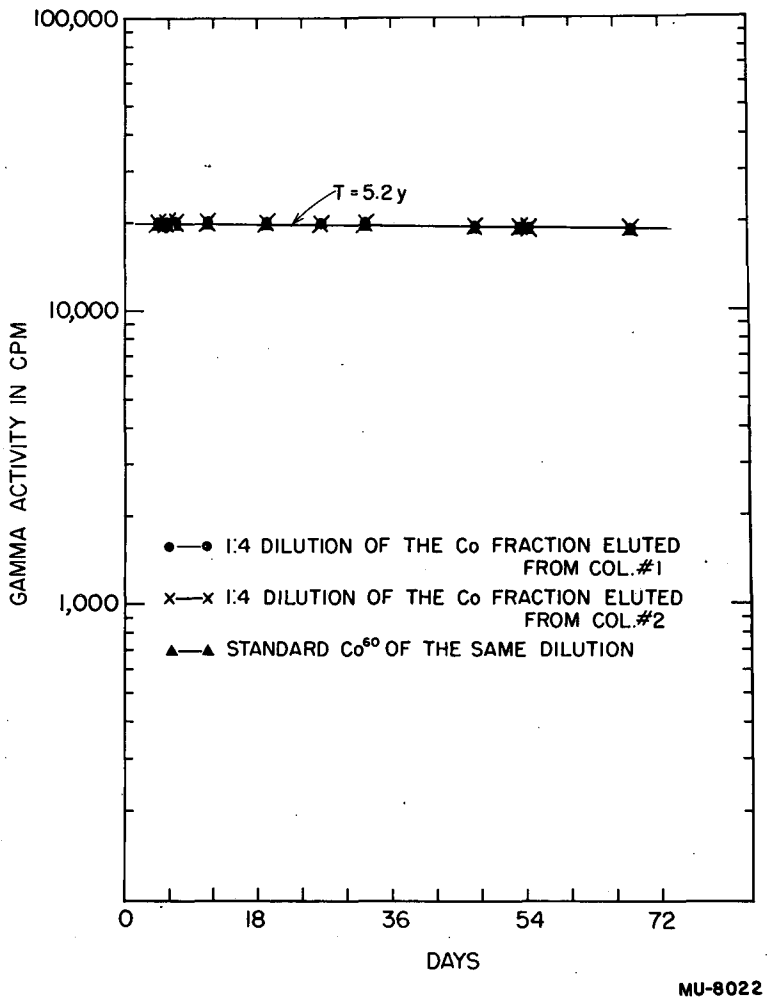
100,000
10,000
1,000
GAMMA ACTIVITY IN CPM
T = 5.2 y
●—● 1:4 DILUTION OF THE Co FRACTION ELUTED FROM COL.#1
x—x 1:4 DILUTION OF THE Co FRACTION ELUTED FROM COL.#2
▲—▲ STANDARD Co60 OF THE SAME DILUTION
0
18
36
54
72
DAYS
MU-8022

Fig. 3

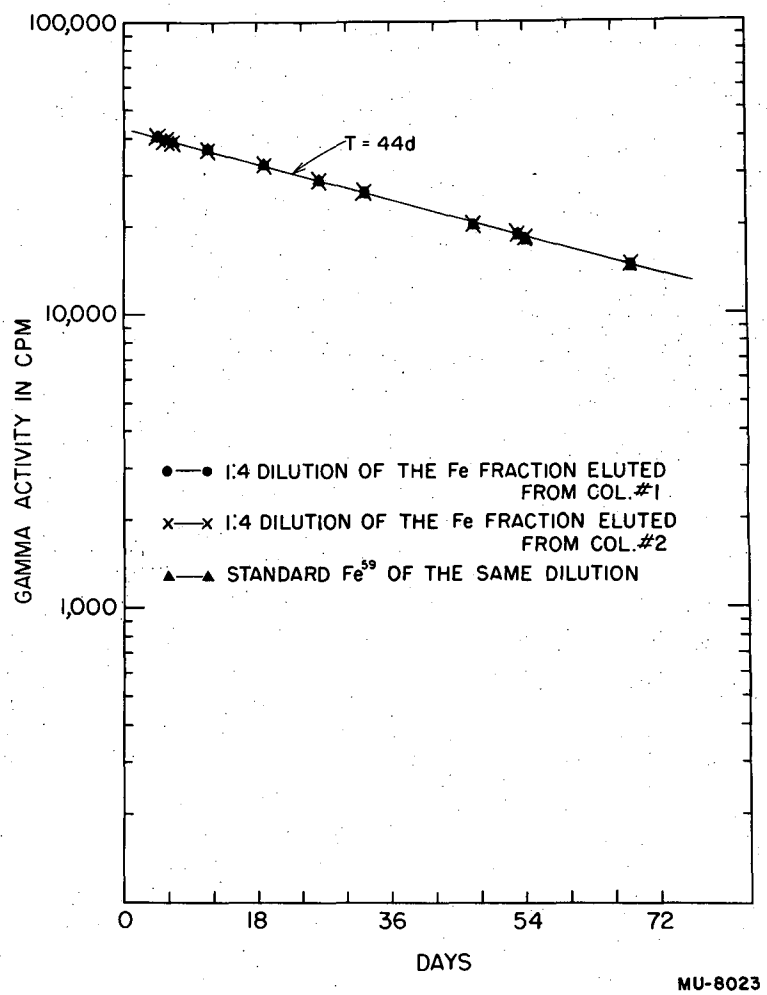
100,000
10,000
1,000
GAMMA ACTIVITY IN CPM
T = 44d
●—● 1:4 DILUTION OF THE Fe FRACTION ELUTED FROM COL.#1
x—x 1:4 DILUTION OF THE Fe FRACTION ELUTED FROM COL.#2
▲—▲ STANDARD Fe59 OF THE SAME DILUTION
0
18
36
54
72
DAYS
MU-8023

Fig. 4

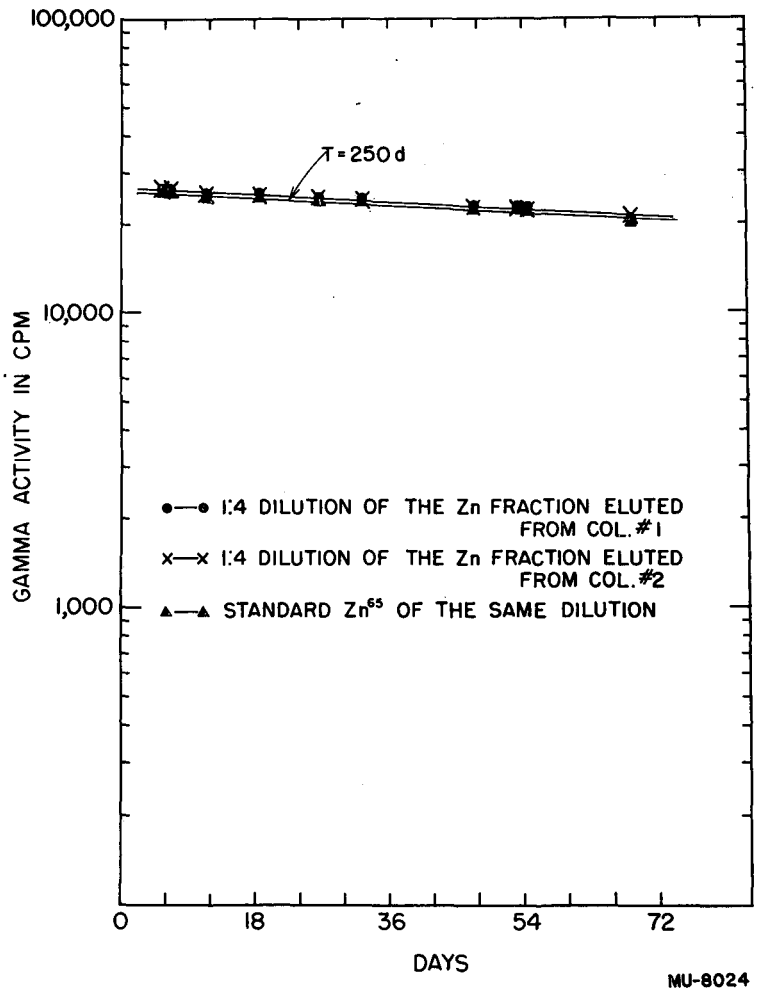
100,000
10,000
1,000
GAMMA ACTIVITY IN CPM
T = 250 d
●—● 1:4 DILUTION OF THE Zn FRACTION ELUTED FROM COL. #1
×—× 1:4 DILUTION OF THE Zn FRACTION ELUTED FROM COL. #2
▲—▲ STANDARD Zn65 OF THE SAME DILUTION
0
18
36
54
72
DAYS
MU-8024

Fig. 5