TABLE II

THERMODYNAMIC FUNCTIONS FOR $KReO_4(c)$

T, °K.	C_p	$S°$	$\left(\frac{H°-H_0°}{T}\right)$	$-\left(\frac{F\ -H_0°}{T}\right)$
10	0.400	0.137	0.104	0.033
15	1.290	.441	.329	.112
20	2.320	.950	.689	.261
30	4.596	2.298	1.609	.689
40	7.032	3.968	2.662	1.306
50	9.225	5.781	3.763	2.019
60	11.154	7.636	4.836	2.800
70	12.842	9.481	5.858	3.623
80	14.344	11.294	6.888	4.406
90	15.694	13.068	7.793	5.275
100	16.921	14.786	8.645	6.141
120	19.078	18.067	10.209	7.858
140	20.906	21.150	11.611	9.539
160	22.412	24.044	12.871	11.173
180	23.670	26.758	14.002	12.756
200	24.840	29.312	15.028	14.284
220	25.887	31.730	15.968	15.762
240	26.830	34.027	16.838	17.189
260	27.778	36.211	17.642	18.569
280	28.660	38.303	18.388	19.915
298.16	29.303	40.123	19.032	21.091
300	29.364	40.305	19.097	21.208

calorie equal to 4.1840 joules (absolute). The mean deviation was observed to be about ±0.1%, but the over-all accuracy of the data was estimated to be ±0.2% except at the very low temperatures. There was no indication of a transition in potassium perrhenate between 16 and 300°K. The heat capacity is approximately a linear function of temperature above 230°K.

Roth and Becker[4] have reported the calorimetrically determined heat of solution of potassium perrhenate to give a dilute solution (0.04 M) as 13,800 cal. mole^{-1} at 18°. Neglecting any further heat change on dilution from that concentration to infinite dilution, and considering the heat of solution to be constant in this range of temperature as observed by Smith,[3] the value of 13,800 cal. mole^{-1} for the heat of solution at 25° was assumed.

From the data of Smith[3] the calculated value for the solubility at 25° is 0.0406 mole liter^{-1}. The activity coefficient of potassium perchlorate at this concentration[8] is $\gamma_{\pm} = 0.81$, while the value calculated for $KReO_4$ from the Debye–Hückel limiting law is 0.791. Using $\gamma_{\pm} = 0.81$ and $K_{sp} = 1.082 \times 10^{-3}$, the free energy of solution is then 4047 cal. mole^{-1} and the entropy of solution, 32.71 cal. mole^{-1} deg.$^{-1}$. Using the value of 24.5 ± 0.2 e.u. for the entropy of K^+, aq,[9] and the value given above for the entropy of $KReO_4$(c) (40.12 e.u.) the entropy of ReO_4^-, aq, was calculated to be 48.3 ± 0.3 e.u. Using 8.89 for the entropy of Re(c),[5] the entropy of formation of the ReO_4^-, aq, ion from its elements at 25° becomes[10] -74.2 e.u. Using the revised heat of formation[11] of the perrehenate ion as $-189{,}200 \pm 800$ cal. mole^{-1}, the free energy of formation of ReO_4^-, aq, then becomes $-167{,}100 \pm 800$ cal. mole^{-1}.

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(8) G. Scatchard, S. S. Prentiss and P. T. Jones, THIS JOURNAL, **56**, 805 (1934).

(9) W. M. Latimer, "Oxidation Potentials," Prentice–Hall, Inc., New York, N. Y., 1952.

(10) Auxiliary thermodynamic data from U. S. Bureau of Standards, "Selected Values of Chemical Thermodynamic Properties," 1949.

(11) J. W. Cobble, W. T. Smith, Jr., and G. E. Boyd, THIS JOURNAL, **75**, 5783 (1953); paper III of this sequence.

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The Separation of Technetium from Molybdenum, Cobalt and Silver[1]

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RECEIVED FEBRUARY 2, 1953

In order to study the chemistry of technetium formed by the neutron bombardment of molybdenum the technetium had to be separated from its molybdenum matrix. This also involved the separation of radioactive trace impurities. The separation was accomplished by means of the Amberlite ion-exchange resins IRA 400 and IR 120. Cobalt and silver were identified as the trace impurities by determining approximately the energies of their beta particles and by carrying the radioactivity on inert cobalt and silver compounds to constant specific activity. The technetium was obtained as ammonium pertechnetate free from radioactive impurities. Quantitative analysis by counting techniques showed that the concentration of technetium in this sample of molybdenum was about ten micrograms per gram. This method is an example of the separation and determination of a radioelement formed in trace amounts by bombardment of large amounts of another element.

One of the methods available for preparing technetium is the neutron–gamma reaction on molybdenum.[2a]

$$Mo^{98}(n,\gamma)\ Mo^{99} \xrightarrow{\beta^-} Tc^{99m} \xrightarrow{\gamma} Tc^{99}$$

In order to obtain pure technetium, this would, ideally, demand only a separation of technetium from molybdenum. Such a separation can be accomplished to a relatively high degree by successive distillations of ditechnetium heptoxide from concentrated sulfuric acid slurries of oxidized molybdenum.[2b] However, for microscopic and ultramicroscopic concentrations of technetium, as might occur naturally, such distillative techniques become impracticable. Also, metallic molybdenum targets for thermal neutrons are not free from impurities; hence, a procedure was needed for the separation of various radioactive species which might co-occur with technetium.

Recently, Fisher and Meloche[3] reported a separa-

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(1) Supported in part by funds supplied by the Wisconsin Alumni Research Foundation.

(2a) E. Segrè and G. T. Seaborg, *Phys. Rev.*, **54**, 772 (1938).

(2b) G. W. Parker, J. Reed and J. W. Ruch, AECD-2043, Jan. 9, 1948.

(3) S. A. Fisher and V. W. Meloche, *Anal. Chem.*, **24**, 1100 (1952).

tion of rhenium from molybdenum using Amberlite IRA 400, a strong base anion-exchange resin. As technetium is chemically similar to rhenium, an ion-exchange technique for the required separations seemed to be promising. Atteberry and Boyd[4] have developed a chromatographic separation of rhenium from technetium using Dowex-2 with a solution of ammonium sulfate and ammonium thiocyanate as the eluting agent.

Reagents.—Neutron irradiated metallic molybdenum:[5] the sample was allowed a "cooling off" period of about three years. Ammonium pertechnetate, approximately 1×10^{-4} *M*.[6] Potassium hydroxide, J. T. Baker Analyzed and potassium oxalate, Mallinckrodt Analytical Reagent. Solution 0.5 *M* in each. Molybdenum metal, 200 mesh 99.8%, Eimer and Amend. Ammonium thiocyanate solutions, J. T. Baker Analyzed, 0.5–3.0 *M*. Chloroplatinic acid solution, Mallinckrodt, 1 mg. Pt per ml. Perchloric acid, Mallinckrodt, 100 ml. 60% diluted to 1 liter. Hydrogen peroxide, 30%, Merck. Amberlite IRA 400, 40–60 mesh, Rohm and Haas (Dowex-2 is a similar resin manufactured by the Dow Chemical Co.); Amberlite IR 120. Rohm and Haas (Dowex-50, Dow Chemical Co.). Stannous sulfate, E. H. Sargent, 1 g. dissolved in 100 ml. of 5% by vol. sulfuric acid, stored over granulated tin. Helium, Matheson. Ethyl alcohol, absolute.

Apparatus.—Nuclear Instrument Laboratory Scaling Unit with Higinbotham scale of 128. North American Philips Geiger tube with thin mica window. Nancy Wood preflush windowless counter, Type K-3. Glass columns, over-all length 55 cm., the top 20 cm. made from 25 mm. i.d. Pyrex tubing, the next 23 cm. from 12 mm. i.d. Pyrex tubing, and the bottom 12 cm. from 6 mm. Pyrex tubing with a #2 Ŧ stopcock 3 cm. from the bottom. Aluminum discs, 3.8 cm. diameter and 0.5 mm. thick.

Experimental

Preparation of the Resins.—Ten grams of IR 120 in the hydrogen form was placed in one of the columns on top of a small tuft of glass wool. The column was backwashed with distilled water until thoroughly wet, and then 100 ml. 1:1 hydrochloric acid was added. Due to an iron impurity, it was necessary to wash the resin with four or five 100-ml. portions of acid. After a negative thiocyanate test for iron was obtained, the column was allowed to drain. It was then backwashed with water until the effluent was basic to methyl orange. During the backwashing, the rate of flow of water was such so as to maintain most of the resin in the upper part of the column. This assured efficient washing of the resin and prevented channeling.

Five grams of the anion exchanger, IRA 400, in the chloride form was transferred to a second column and treated with four successive 100-ml. portions of the perchloric acid solution. The last portion was allowed to remain in contact with the resin for about one half hour. It was then drained and backwashed as in the case of the cation-exchange resin. The direction of flow of all solutions was vertically downwards with the exception of the distilled water used as a backwash during the preparation of the resins.

Elution of Technetium with Ammonium Thiocyanate Solutions.—The effect of the concentration of ammonium thiocyanate on the elution of technetium is shown in Table I. An aliquot of the ammonium pertechnetate solution containing 24.6 micrograms of technetium was treated with alkaline hydrogen peroxide in order to simulate the conditions for solution of the irradiated molybdenum, and the excess peroxide removed by boiling the solution for a few minutes. The sample was transferred to a 30-ml. separatory funnel and put onto the perchlorate form of the anion-exchange resin at a flow rate of 1.9 ml./min. The funnel was washed thoroughly with water, and then the thiocyanate solution was added from a 500-ml. separatory funnel at the same flow rate. The effluent was collected in 25-ml. volumetric flasks, and a $^1/_{50}$ or $^1/_{100}$ aliquot of each cut evaporated on an aluminum disc under a 250-watt heat lamp. The drops were placed as concentrically as possible and as near the center as possible. The micropipet was then washed twice with water, and the washings also evaporated. One of the reasons ammonium thiocyanate was chosen as the eluting agent was because it decomposes upon heating to 150° leaving only a small amount of decomposition product. This eliminated some of the self absorption of the 0.3 mev. beta particles by the sample. The sample was then counted using the mica window Geiger tube. Later counting of technetium samples was done with the windowless preflush counter which was only recently acquired. The counting gas was helium which was bubbled through alcohol at 0°, the alcohol acting as a quench. No correction for decay was necessary due to the long half-life of Tc^{99}, 2.12×10^5 years.[7] The counting rate was compared to a standard technetium sample.

It can be seen by inspection of Table I that as the concentration of ammonium thiocyanate is increased, the rate of elution of technetium is also increased, and that a further increase in concentration of ammonium thiocyanate would not have an appreciable effect on the rate of elution. The relatively large deviation from 100% is probably due to a combination of factors. When such low activities are counted, the relative error is great. This error is then magnified by multiplying the counting rate by the aliquot factor. As the concentration of thiocyanate is increased, the amount of solid material left on the aluminum disc as decomposition product is increased thus accounting for increased self absorption.

TABLE I

EFFECT OF AMMONIUM THIOCYANATE CONCENTRATION ON TECHNETIUM ELUTION (24.6 MICROGRAMS Tc ADDED)

Eluent vol. cumulative, ml.	0.53 *M*		1.07 *M*		2.0 *M*		3.0 *M*	
	Tc, μg.	Tc cumulative, μg.	Tc, μg.	Tc cumulative, μg.	Tc, μg.	Tc cumulative, μg.	Tc μg.	Tc cumulative, μg.
25	0	0	0.14	0.14	0.28	0.28	2.13	2.13
50	0.14	0.14	2.49	2.63	10.93	11.21	14.37	16.50
75	0.34	0.48	8.17	10.80	7.26	18.47	4.0	20.90
100	2.41	2.89	6.36	17.16	2.20	20.67	0.72	21.62
125	5.17	8.06	3.53	20.69	0.52	21.19	.14	21.76
150	6.07	14.13	1.59	22.28	.10	21.29	.14	21.90
175	4.76	18.89	0.48	22.76	.07	21.36	.11	22.01
200	3.10	21.99	.21	22.97	.03	21.39	.27	22.28
225	1.70	23.69	.14	23.11			.11	22.39
250	0.83	24.52	.14	23.25			.03	22.42
275	.48	25.00	.28	23.53				
300	.34	25.34	.14	23.67				
325	.28	25.62	.07	23.74				
350	.21	25.83						
375	.14	25.97						
400	.07	26.04						
Total		26.0		23.7		21.4		22.4
Tc recovered, %		105.7		96.4		87.0		91.2

Recovery of Technetium from Ammonium Thiocyanate Solutions.—If only the volume of solution used to elute the technetium is used as a guide, the 3 *M* ammonium thiocyanate would seem to be the most efficient. However, if the actual number of millimoles of ammonium thiocyanate is considered, elution with the 0.5 *M* solution requires about 185 millimoles, and the 3 *M* solution requires about 450 millimoles. Therefore, as the ammonium thiocyanate must be decomposed in order to recover the technetium from it, the 0.5 *M* solution was used in subsequent work. It was necessary to recover the technetium because only a limited supply was available.

The decomposition of the ammonium thiocyanate solution was carried out in the following manner. Twenty-two grams of sodium hydroxide was added and the solution boiled until free from ammonia. Then 30% hydrogen peroxide was added dropwise. There is an induction period for the peroxide–thiocyanate reaction so care must be taken that the hydrogen peroxide is not added too rapidly. Completeness of decomposition was ascertained when a drop of an iron(III) chloride solution failed to give a red color when it was added to a small portion of the sample acidulated

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(4) R. W. Atteberry and G. E. Boyd, THIS JOURNAL, **72**, 4805 (1950).

(5) Obtained through the courtesy of the Argonne National Laboratory, Chemical Division.

(6) Oak Ridge National Laboratory, Isotopes Division.

(7) S. Fried, *et al.*, *Phys. Rev.*, **81**, 741 (1951).

with sulfuric acid. When the decomposition was complete, the test solutions were added to the main body of solution, and it was boiled for about 10 min. to destroy the excess of peroxide. The solution was then neutralized to the phenolphthalein end-point with concentrated sulfuric acid, and a sufficient excess of acid was added to make the solution 6–8 *N*. There was usually a small amount of a white gelatinous precipitate in the solution. This was filtered on a fine porosity sintered porcelain crucible and washed with dilute sulfuric acid. No activity was held on the precipitate. One milliliter of the platinum solution was added to the filtrate, the solution heated to 90°, and hydrogen sulfide passed in for two hours. Technetium precipitates as the heptasulfide,[8] and it is quantitatively carried on platinum sulfide. The mixed sulfides were filtered on a fine porosity porcelain filter crucible and washed with 1:100 sulfuric acid. After the crucible was sucked dry, carbon disulfide was used as a further wash. Air was pulled through the crucible until the carbon disulfide was volatilized. The sulfides were next dissolved in a solution of ammonium hydroxide–hydrogen peroxide, equal volumes of each being a suitable mixture. The filtrate contained the platinum carrier and the technetium, the latter of which was probably present as ammonium pertechnetate. The filtrate was then heated to decompose excess peroxide, diluted to a known volume, and an aliquot evaporated on an aluminum disc and counted.

Elution of Molybdenum.—Molybdenum has been quantitatively removed by Preuss from the anion-exchange resin IRA 400 by elution with a 1 *M* solution of potassium oxalate.[9] This also has been accomplished by Fisher and Meloche using a sodium hydroxide solution.[3] The oxalate method is the more rapid of the two.

No attempt was made to determine the molybdenum quantitatively. The point of interest lay in a complete separation of molybdenum from technetium. This was done using the method of Preuss with the modification that 0.5 *M* potassium oxalate and 0.5 *M* potassium hydroxide replaced the 1 *M* oxalate solution. Completeness of elution of molybdenum was determined by the absence of color when a portion of the eluent was made acid with sulfuric acid (180 ml. of sulfuric acid plus 640 ml. of water), and a few milliliters of a 20% solution of potassium thiocyanate and 3–4 drops of stannous sulfate solution were added. This is the method of Larson for micro amounts of molybdenum.[10]

Separation of Molybdenum from Technetium in Synthetic Mixtures.—Several samples were prepared using inactive metallic molybdenum and radioactively pure technetium. The molybdenum was put in solution with 10% sodium hydroxide and enough 30% hydrogen peroxide to effect solution after adding the technetium as a solution of ammonium pertechnetate. The excess peroxide was expelled by boiling, and the sample transferred to a 30-ml. separatory funnel. The sample was put on the perchlorate form of the anion-exchange resin at a flow rate of 1.9 ml./min., and, in this case, 10% sodium hydroxide was used to elute the molybdenum. Either sodium hydroxide or potassium oxalate–potassium hydroxide could have been used. The technetium was not removed from the resin by either one. Following removal of the molybdenum, the technetium was removed with ammonium thiocyanate solution as previously explained, and aliquot portions of each cut counted. Results are shown in Table II. It can be seen that the quantity of molybdenum used had no obvious effect on the recovery of technetium.

TABLE II

SEPARATION OF TECHNETIUM FROM VARYING AMOUNTS OF MOLYBDENUM

Mo added, g.	Tc added, μg.	Tc recovered, μg.	Recovery, %
0.3[a]	10.0	10.0	100
.5	10.0	10.2	102
.7	20.1	19.3	96.1
1.0	10.1	9.2	91.1
50.0	10.1	10.5	104

[a] Added as ammonium molybdate.

Identification of Unknown Radioactive Species and Determination of Technetium Concentration in the Molybdenum.—A sample of irradiated molybdenum was dissolved in ammonium hydroxide and hydrogen peroxide, the latter of which was added dropwise. It was then evaporated to dryness on a water-bath to remove excess peroxide and ammonium hydroxide. The solids were dissolved in water, and the sample was transferred to a 30-ml. separatory funnel. The sample was then put on the hydrogen form of the cation-exchange resin at a flow rate of 1.9 ml./min. The column was washed with 400 ml. of water, and the effluent put directly on the perchlorate form of the anion-exchange resin. After the addition of the sample, the anionic resin was washed with 400 ml. of water. For the sake of identification, this cut is called the "sample" cut. The quantity of water used to wash both resins was determined by washing until a 1–2 ml. sample showed no activity. The molybdenum was next eluted from the anion resin with potassium oxalate–potassium hydroxide solution at a flow rate of 1.9 ml./min. Following a 100-ml. water wash, the technetium was eluted with 0.5 *M* ammonium thiocyanate solution. After counting an aliquot from each technetium cut, and determining the amount of technetium eluted, the remainder of the thiocyanate solution was decomposed and the technetium recovered. This amount was then compared to the amount determined by counting the individual cuts. Results are given in Table III.

TABLE III

TECHNETIUM RECOVERED FROM IRRADIATED MOLYBDENUM

Tc calcd. from each cut, μg./g. Mo	Tc recovered from NH_4SCN, μg./g. Mo	Recovery, %
10.8	10.0	92.6
11.3	9.0[a]	79.6

[a] More solid material on aluminum disc than other cases. This probably caused a low result due to increased self-absorption.

The "hard" activity (gamma ray emitters) present was found to be in two places, (1) in the "sample" cut, and (2) on the upper part of the cation-exchange resin. The activity in the sample cut was determined in the following manner. About 0.1 g. of silver was added as silver nitrate, and silver chloride precipitated by adding dilute hydrochloric acid. The precipitate carried essentially no activity. A survey meter indicated that the activity was in the filtrate. The filtrate was made more strongly acid with sulfuric acid, and the molybdenum (which was present from the sample) was precipitated by passing hydrogen sulfide gas through the hot solution. There was a copious quantity of precipitate. This was counted, and it was also practically void of activity. After boiling to free the solution of hydrogen sulfide, 0.05 g. of iron was added as ferric ammonium sulfate, and hydrous ferric oxide precipitated with ammonium hydroxide. The filtrate still contained most of the activity although the hydrous ferric oxide contained more than a negligible amount. These were treated individually. About 0.05 g. of cobalt as cobalt chloride was added to the filtrate from the hydrous ferric oxide precipitation, and the ammonium sulfide group precipitated. The cobalt sulfide was extremely active. Considering the members of the ammonium sulfide group, cobalt, nickel, zinc, manganese and thallium, the only one, that had both a satisfactory half-life and beta particle and gamma rays with energies comparable to those determined by aluminum and lead absorption curves, was Co^{60}. It was not considered necessary to attempt to determine the half-life of the activity as it was known to be long due to the fact that it persisted over the long "cooling" period. Aluminum and lead absorption curves were obtained on a portion of the sample and are shown in Figs. 1 and 2, respectively. From these curves it was possible to obtain an approximate value of the energy of the beta particle and at least a semi-quantitative value of the energy of the gamma ray or rays. As shown on the graphs, a beta particle of about 0.27 mev. and a gamma ray of about 1.3 mev. were found. These results compared more favorably to Co^{60} than to the other members of the ammonium sulfide group. Therefore, the cobalt was precipitated as potassium hexanitrocobaltate[11] and specific ac-

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[8] C. L. Rulfs and W. W. Meinke, THIS JOURNAL, **74**, 235 (1952).

[9] A. F. Preuss, Ph.D. thesis, University of Wisconsin, 1952.

[10] W. J. Larson, Ph.D. thesis. University of Wisconsin, 1950.

[11] W. F. Hillebrand and G. E. F. Lundell, "Applied Inorganic Analysis," John Wiley and Sons, New York, N. Y., 1950, p. 324.

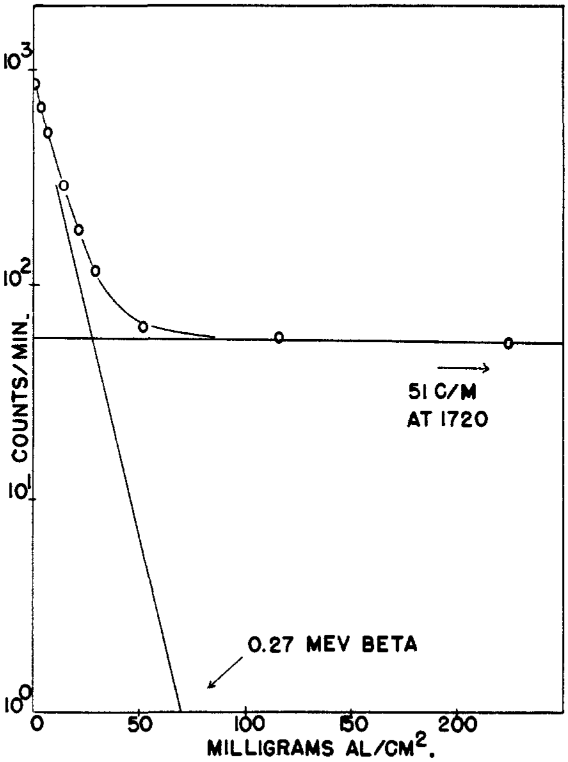

Fig. 1.—Aluminum absorption curve of the activity on the cobalt carrier.

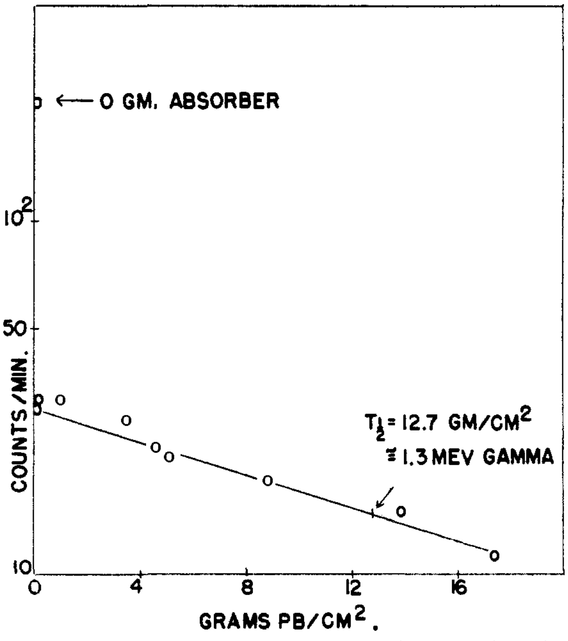

Fig. 2.—Lead absorption curve of the activity on the cobalt carrier.

tivities determined. Table IV shows the results. From these data, it was concluded that Co^{60} was one of the contaminants in the molybdenum.

TABLE IV

DETERMINATION OF SPECIFIC ACTIVITY OF COBALT

	Determination First	Determination Second
Wt. $K_3Co(NO_2)_6$, g.	0.4075	0.3982
Counts/min.	23420	24040
Specific activity, c./m./mg.	57.4	60.3

Concerning the activity on the hydrous ferric oxide, it is well known that the freshly precipitated oxide has strong adsorptive properties. Therefore, cobalt was added to the dissolved iron and the hexanitrocobaltate procedure repeated. As can be seen from Table V, constant specific activity was obtained in two determinations, and it was concluded that a small amount of the cobalt activity was adsorbed on the hydroxide precipitate.

The "hard" activity on the upper part of the cation-exchange resin was removed from the resin using approximately 300 ml. of concentrated hydrochloric acid at a flow rate of about 2 ml./min. Figure 3 is an aluminum absorption curve of this activity and Fig. 4 is a lead absorption curve. Figure 3 indicates two beta particles of about 0.28

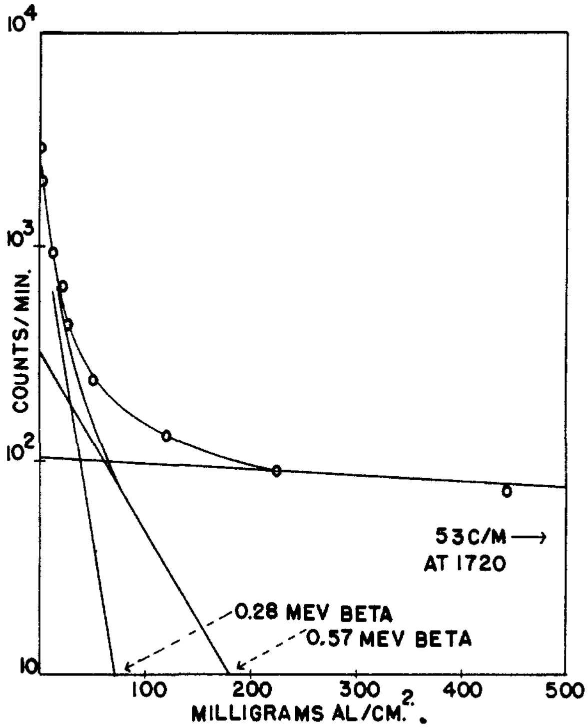

Fig. 3.—Aluminum absorption curve of the hard activity on the cation resin.

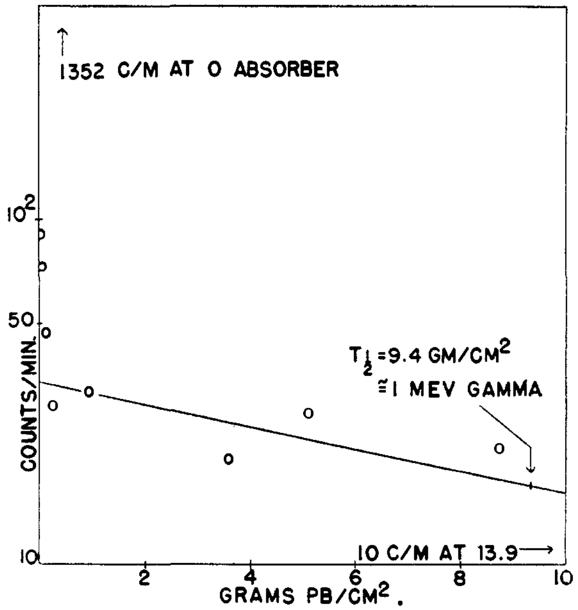

Fig. 4.—Lead absorption curve of the hard activity on the cation resin.

Table V

Determination of Cobalt Specific Activity Adsorbed on Ferric Hydroxide

	Determination First	Second
Wt. $K_3Co(NO_2)_6$, g.	0.0373	0.0286
Counts/min.	1050	905
Specific activity, c./m./mg.	29.2	31.6

and 0.57 mev., and a gamma ray or rays. Figure 4 permits a very rough estimate of the gamma ray energy (or the resultant energy from several gammas). This is approximately 1 mev. It is to be emphasized that energies obtained from absorption curves similar to those shown are at best only approximate and that logarithmic subtractions tend to enhance the uncertainties. The beta energies shown above correspond approximately to the betas given off by Co^{60} and Ag^{110}, respectively. The National Bureau of Standards Circular 499 gives, for Co^{60} —0.31 mev. beta plus 1.17 and 1.31 mev. gamma rays, and for Ag^{110} —0.09 mev. beta, 0.53 mev. beta, and several gamma rays.[12]

The concentrated hydrochloric acid eluent from the cationic resin was evaporated to dryness and dissolved in water and ammonium hydroxide. About 0.1 g. of silver was added as silver nitrate and silver chloride was precipitated by the addition of dilute hydrochloric acid. The precipitate was allowed to stand overnight in the dark before filtration. Results of specific activity measurements are given in Table VI.

Table VI

Specific Activity of Silver Chloride

	First	Determination Second	Third
Wt. AgCl, g.	0.1463	0.1234	0.0837
Counts/min.	4168	2489	1854
Specific activity, c./m./mg.	28.5	20.2	22.1

The agreement between the second and third determinations plus the energy noticed for one of the beta particles supports the statement that one of the radioactive species in the sample is silver. The first determination gave a higher specific activity probably because of coprecipitated cobalt (as shown below).

The filtrate from the silver chloride precipitation was evaporated to dryness after adding about 0.05 g. of cobalt as cobalt chloride. This was dissolved in water and precipitated by adding potassium nitrite in 10% acetic acid. Table VII gives the data. Therefore, cobalt is also one of the radioactive species on the cation-exchange resin. No explanation is given as to why all of the cobalt is not held on the cation-exchange resin. To check the validity of the statement that some of the cobalt goes through the cation resin, 0.05 g. of cobalt as cobalt chloride was dissolved in water and treated in the same manner as the neutron irradiated molybdenum sample. Ammonium hydroxide and hydrogen peroxide were added, and the solution evaporated to dryness. The residue was only sparingly soluble in water so it was filtered. The majority of the cobalt remained as insoluble residue, but the filtrate did contain some cobalt. This was put through the cation-exchange column, and a small portion of the effluent was tested for cobalt. It proved to be present, which confirmed the fact that all of the cobalt was not held by the cation-exchange resin.

Table VII

Specific Activity of Cobalt

	Determination First	Second
Wt. $K_3Co(NO_2)_6$, g.	0.2251	0.2220
Counts/min.	7835	7598
Specific activity, c./m./mg.	34.8	34.3

In order to see if all the gamma activity in the sample could be related to cobalt and silver, a sample of molybdenum was dissolved in ammonium hydroxide–hydrogen peroxide, and the excess peroxide removed by boiling. Silver and cobalt were added as silver nitrate and cobalt nitrate to the ammoniacal molybdenum solution. An aliquot was then evaporated in a stainless steel dish and the activity determined by counting through an aluminum absorber. The remainder of the solution was treated with sodium hydroxide and boiled to remove the ammonia. The hydroxides of cobalt and silver precipitated during this treatment. They were filtered on paper and the precipitate and paper decomposed with sulfuric and nitric acids. After taking to dryness, the residue was dissolved in water. Silver chloride was precipitated and the filtrate evaporated to dryness, dissolved in water and potassium hexanitrocobaltate precipitated. The silver and cobalt precipitates were dissolved and combined. An aliquot was counted as with the original sample. Table VIII gives counting results. This was considered to be a satisfactory comparison. It shows that no gamma activity is present other than that originating from silver and cobalt. As beta activity other than that from technetium, cobalt and silver was not found during the investigation, it was concluded that silver and cobalt were the only two contaminating activities. However, when the molybdenum sample was originally removed from the neutron flux, other activities were most probably present, but due to the relatively long "cooling" period, they would have decayed to an insignificant amount.

Table VIII

Gamma Activity through Aluminum Absorber

	Initial	Final
Counts/min. (aliquot)	30	28
Counts/min. (total)	7500	7000

Madison, Wisconsin

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[12] K. Way, *et al.*, "Nuclear Data," Circular of the National Bureau of Standards 499, U. S. Dept. of Commerce, U. S. Government Printing Office, 1950, *et sequel.*