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Separation of Iron and Cobalt on Synthetic Resin

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

August 5, 1953

Berkeley, California

SEPARATION OF IRON AND COBALT ON SYNTHETIC RESIN

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Donner Laboratory of Medical Physics and Radiation Laboratory
University of California, Berkeley, California

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In the separation of mixtures of radioactive iron and cobalt from neutron irradiated targets and in the removal of iron from cobalt targets for cyclotron production of radioiron, it is desirable to have a method superior to extraction from isopropyl ether (1, 2) in efficiency and ease of manipulation. Recent studies by Moore and Kraus on the separation of cobalt and nickel (3) and on the behavior of iron (4) in hydrochloric acid on Dowex-1 resin in relation to the selective adsorption of chloride complexes of these elements have suggested the use of resin for the separation of iron and cobalt.

PROCEDURE AND MATERIALS

Preparation of resin columns. Dowex-1, 200-400 mesh, a quaternary amine, polystyrene divinyl benzene anion exchange resin, was washed free of iron with three liter aliquots of 0.5 M hydrochloric acid and was then washed three times with liter aliquots of 5 M hydrochloric acid per 500 gm. of resin in a column 9 cm. in diameter. Washed resin was stored in 5.0 M hydrochloric acid and was poured as a slurry upon a pad of glass wool in a Pyrex column 1.13 cm² in cross section. The resin bed was 13 cm. long.

Iron and cobalt. Tracer iron and cobalt were obtained by neutron bombardment of the metals. Carrier iron and cobalt were prepared by dissolving the metals in 5 M hydrochloric acid. The tracer and carrier salts were heated to dryness with concentrated nitric acid and hydrogen peroxide three times. The oxides were converted to the chlorides by heating to dryness at 80° C with concentrated hydrochloric acid. The dry residue was dissolved in 5 M hydrochloric acid and was transferred to the resin bed with this medium.

Elution conditions. The cobalt and iron were eluted successively from the resin column with 5 M followed by 0.5 M hydrochloric acid supplied by a syringe pump at a constant flow rate of 10 ml. per hr. per cm². Aliquots of 1.67 ml. of eluate were collected continuously with an automatic sample collector, dried at 60° in an air oven and counted.

Resin capacity. The capacity of Dowex-1 for adsorption of iron were determined by measuring the breakthrough volume of a solution of 5 M hydrochloric acid containing 8 mg. of labeled iron per ml. on a standard column, 1.13 cm² x 13 cm., at a flow rate of 10 ml. per cm² per hr.

Distribution coefficient, K_D. The distribution coefficient for iron K_D, where $K_D = \frac{\text{mg. Fe/gm. resin}}{\text{mg. Fe/ml. solution}}$ was determined by continuous shaking of 1.0 gm. of Dowex-1 in the chloride form with 5 ml. of a solution containing 8.0 mg. of labeled iron per ml. in 5 M hydrochloric acid at 18°C. Samples of the supernatant were taken at intervals for scintillation counting.

Radioactivity measurement. The gamma activity of iron and cobalt was determined by counting liquid samples in 1 dram vials with a sodium iodide crystal counter and commercial scaler.

RESULTS

The results of elution experiments with 5 mg. of labeled iron and cobalt on our columns with 5 M and 0.5 M hydrochloric acid are shown in Fig. 1. Carrier and tracer cobalt was eluted in the first 35 ml. of 5 M hydrochloric acid. There was no visual indication that the band of adsorbed iron had migrated appreciably during washing with 135 ml. of 5 M hydrochloric acid. After changing to 0.5 M hydrochloric acid as elutant, iron was contained in 45 ml. of eluate. Recovery of added radioactivity from the resin was 95 to 100 percent for iron and for cobalt. In triplicate experiments in which a mixture of 5 mg. each of iron and cobalt, with added radioactive iron or cobalt, was fractionated on a column, a maximum cross contamination of 0.6 percent was found by radioactive measurements. Mixtures of radioactive iron and cobalt in 8 M hydrochloric acid that had been separated by triple extraction with isopropyl ether could be further fractionated on Dowex-1 in 5 M hydrochloric acid. Thus the resin is superior to isopropyl ether in efficiency of separation of iron and cobalt. Failure to convert iron to its

higher oxidation state by treatment with heat, nitric acid and hydrogen peroxide before application to the resin resulted in the formation of two bands of iron, one of which migrated in 5 M hydrochloric acid and a second which was adsorbed on the resin. This fractionation of iron was apparently due to the presence of ferrous and ferric ions with differing tendencies to form chloride complexes. Ferric iron was adsorbed by the resin in 5 M acid.

The capacity of Dowex-1 for iron in 5 M hydrochloric acid at breakthrough of radioiron was 1.04 gm. of iron on a resin bed $1.13 \text{ cm}^2 \times 13 \text{ cm}$, or 154 mg. of iron per gm. of dry resin in the chloride form.

Figure 2 shows the change of distribution coefficient of iron with time of contact of Dowex-1 with labeled iron in 0.5 M and 5 M hydrochloric acid at 18°C . Equilibrium was approached at 3 hr. and had not been attained at the end of 30 hr. of equilibration. The K_D at 3 hrs. was 0.19 in 0.5 M and 77 in 5 M hydrochloric acid. At 30 hrs. the K_D was 0.32 in 0.5 M and 82 in 5 M hydrochloric acid. The rapid increase of K_D in the first hour of contact of iron with a fixed amount of resin is in agreement with the experimentally rapid adsorption of iron on a resin column with a moving boundary.

We have been able to remove adsorbed iron from Dowex-1 columns with 0.5 M hydrochloric acid and to equilibrate the resin with 5 M hydrochloric acid for reuse with no impairment of efficiency of the resin.

ACKNOWLEDGMENT

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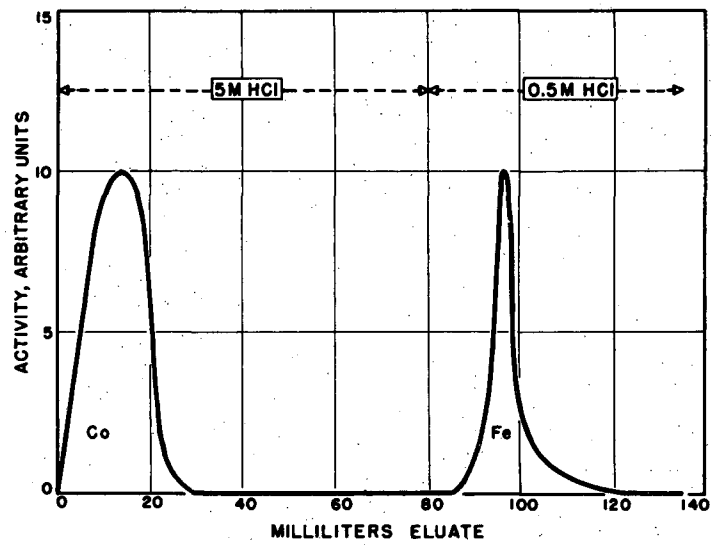


Fig. 1

Elution curve for 5 mg. of labeled iron and cobalt on Dowex-1 resin with 5 M and 0.5 M hydrochloric acid.

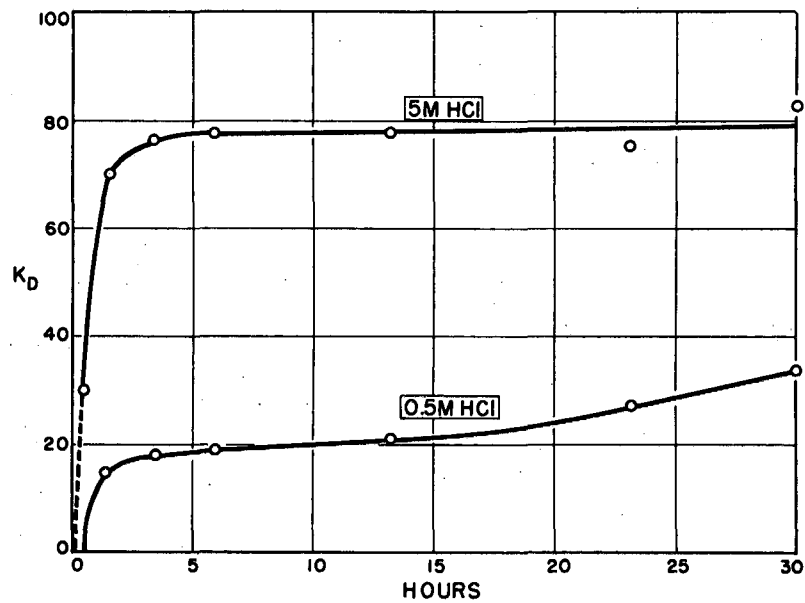


Fig. 2

Change of distribution coefficient of labeled iron with time in contact with Dowex-1 resin in 0.5 and 5 M hydrochloric acid.