NOTES

The Carrier-free Separation of the Radioactive Isotopes Co^{56}, Co^{57} and Co^{58} from a Manganese Target

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Various organic reagents, by nature of their selectivity, are becoming more and more important in the field of nuclear chemistry. One of the more common complexing reagents, α-nitroso-β-naphthol, is best known for its application in the separation of cobalt from large quantities of nickel. The ability of the reagent to complex cobalt in the presence of manganese is the basis for applying it to the problem of preparing a carrier-free sample of radioactive cobalt from a manganese target. In as much as the sample was to be used for study[2] in the beta spectrometer, it was most important that it be prepared by a carrier-free method of separation. The presence of stable isotopes results in low specific activity and self absorption which hamper spectrometric studies.

Experimental

Reagents.—Manganese, Ajax 99.9%, Battelle Memorial Institute, 505 King Ave., Columbus, Ohio. α-Nitroso-β-naphthol (practical) Eastman Kodak Co., Rochester, N. Y. Benzene (thiophene free) Analytical Reagent, Mallinckrodt Chemical Works, N. Y.

A sample of manganese was bombarded in the cyclotron at the University of California for 3.5 hours with 35 Mev. α-particles. Following a waiting period of several days in which the short lived activities disintegrated, the sample was dissolved in 25 ml. of 6 *N* hydrochloric acid and evaporated to dryness. The residue was then dissolved in 250 ml. of 3 *N* hydrochloric acid and stored in a 250-ml. volumetric flask. This solution became the parent material from which all experimental samples were taken.

The first phase of the study was to determine the length of time the complexing reagent should remain in the sample solution. Two ml. of the target solution was diluted to 12 ml. and the acidity adjusted to 0.5 *N*. To this was added 4 ml. of the complexing reagent (0.25 g. of α-nitroso-β-naphthol dissolved in 100 ml. of 50% acetic acid solution). After an interval of one-quarter hour the solution was extracted with three 25-ml. portions of benzene. The non-aqueous phase containing the complexed cobalt and excess organic reagent was evaporated to dryness in a platinum crucible and the resulting residue ignited in air. This residue was then transferred to a counting dish and the percentage of activity removed from the aqueous target solution was determined using a Potter Scaler and a mica end-window Geiger–Mueller Tube. Successive measurements were made on individual samples following intervals of $^1/_4$, $^1/_2$, 1, 2 and 4 hours. It was found that about 98% of the cobalt was extracted by benzene if the complexing reagent remained in solution one hour or more. Only 37% was extracted in a quarter hour and 69% in a half-hour.

Data were collected from a series of experiments in which the acidity was varied from *p*H 8.7 to 6 *N*. In each test, two ml. of target solution was diluted to 12 ml. with hydrochloric acid or ammonia solution of appropriate strength. The solution was heated to 80° and 4 ml. of the complexing reagent in acetic acid solution was added. After standing one hour the solution was transferred to a separatory funnel and extracted with three 25-ml. portions of benzene. The benzene solution was transferred to a platinum crucible, evaporated to dryness and the crucible ignited in air. There was no visible residue. Following this the material was treated with hot concentrated hydrochloric acid and the acid solution transferred to a counting dish. The percentage extraction was computed from the counting rate of the sample. The optimum acidity for extraction was in the range from *p*H 4 to 0.25 *N* in which range 100% of the cobalt was extracted. As shown in curve A of Fig. 1, 98% or more was extracted in the range from 0.5 *N* acid to *p*H 8.7.

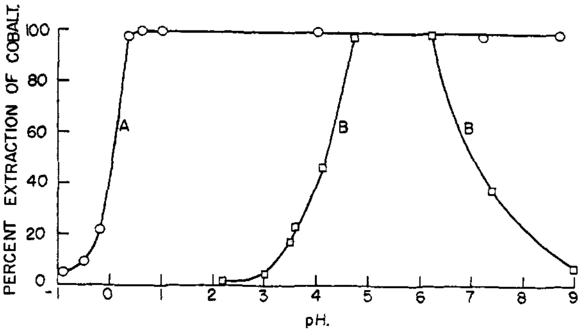

Fig. 1.—Complexing reagent used: A, 4 ml. of 50% acetic acid solution of α-nitroso-β-naphthol containing 250 mg./ml.; B, 0.5 ml. of aqueous solution of α-nitroso-β-naphthol containing 30 mg./100 ml.

A study was made of the effect of *p*H on the percentage of cobalt extracted when a saturated aqueous solution of α-nitroso-β-naphthol was used as complexing agent. The aqueous solution of the complexing reagent contained approximately 30 mg. of α-nitroso-β-naphthol per 100 ml. of water and 0.5 ml. of this solution was used in each sample. The maximum amount of activity was extracted when the acidity of the target solution was maintained between *p*H 4.7 and 6.2. The results of this experiment are shown in curve B of Fig. 1. It is evident that the *p*H of the solution must be maintained in a narrower range when using the lower concentration of complexing agent in aqueous solution than when using the acetic acid solution of complexing agent.

The purpose of using an extraction method rather than filtration for the removal of the cobalt complex of α-nitroso-β-naphthol from the aqueous solution was twofold. The amount of precipitate formed in the reaction of quantities of cobalt of the order of 10^{-9} *M* with α-nitroso-β-naphthol was so small that much of the activity could readily pass through the paper if filtered. Secondly, the ashing of the filter paper, although it is considered ashless by macro standards, contributed a sizeable residue in terms of the cobalt present.

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[2] L. Cheng, J. Dick and J. Kurbatov, *Phys. Rev.*, **88**, 887 (1952).

The Surface Area of Nickel Oxalate Precipitates

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In a recent paper[1] the rates of formation of precipitates of nickel oxalate dihydrate in solutions of nickel sulfate and oxalic acid were related to the concentrations of the reactants. A mechanism for the reaction based, in part, on the formation and

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[1] J. A. Allen, *J. Phys. Chem.*, **57**, 715 (1953).