

## RADIOCHEMICAL SEPARATION OF COBALT\*

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## INTRODUCTION

The separation of cobalt-60 from other radio-isotopes emitting hard  $\gamma$ -rays is of great importance, *e.g.* in activation analysis\*\*. In many irradiated samples (blood, steel, etc.) especially iron-59 and zinc-65 are present in large excess over the cobalt-60.

The methods suggested for the separation of cobalt-60 include paper chromatographic, ion-exchange, precipitation and extraction techniques<sup>2-6</sup>; but with a large excess of iron-59 or zinc-65 we found that none of these methods was quite satisfactory.

PŘIBIL *et al.*<sup>7</sup> made use of the extraordinary stability of the cobalt diethyldithiocarbamate complex for the selective colorimetric determination of microgram amounts of cobalt. The complex was extracted into chloroform and all other colored diethyldithiocarbamate complexes were decomposed by shaking with an aqueous solution of mercury(II).

The same reactions were applied by the present author for the radiometric determination of submicrogram amounts of cobalt, using either diethyldithiocarbamate-<sup>35</sup>S or mercury-203 (in contradistinction to microgram amounts of cobalt, extraction with diethyldithiocarbamate at pH 14 was incomplete)<sup>8</sup>.

In the present work cobalt and other carriers were added to the irradiated sample and extraction of the cobalt complex was carried out at pH 14. The treatment with mercury(II) was unnecessary, because iron-59 and zinc-65 were co-extracted only to the extents of 0.0005 and 0.04% respectively.

## PROCEDURE

After the irradiation add metal-carriers in 100  $\mu$ g\*\*\* amounts, ash and/or dissolve in mineral acid and, if necessary, separate the heavy metals by precipitation as the hydroxides.

Dissolve in a few ml of 1 *N* hydrochloric acid and place the solution in a tightly lidded P.V.C. pill-capsule of diameter 25 mm and height 76 mm.

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\*\* KAISER AND MEINKE<sup>1</sup> carried out the neutron activation analysis of cobalt, using the soft  $\gamma$ -radiation of cobalt-60m ( $\tau_{1/2} = 10.5$  min).

\*\*\* Alternatively 1 mg of each metal is added as a carrier; in this case 0.05 *M* tartrate and 10 mg of diethyldithiocarbamate are used.

Dilute to 5 ml, add some phenolphthalein and add potassium sodium tartrate up to 0.01 *M*\*. Neutralise with alkali, add an excess of 0.6 ml of 10 *N* sodium hydroxide and then 1 mg of diethyldithiocarbamate\*.

Shake mechanically\*\* with 5 ml of carbon tetrachloride (AnalaR), centrifuge lightly to remove drops from the upper walls and remove the upper layer with a pipette. Rinse by shaking with alkaline tartrate, dry the carbon-tetrachloride layer by filtration (*e.g.* through a SS-595 paper) and evaporate by a stream of nitrogen to the volume of the well-type counter. Measure the radio-activity of cobalt-60 by scintillation counting\*\*\* and the amount of the cobalt complex with a spectrophotometer (4000 Å).

The time required for a determination in duplicate is 40 min; the efficiency for cobalt is about 50%.

## DISCUSSION

The results (Table I) show that less than 0.0005 and 0.04% of iron and zinc respectively are extracted. About 0.05% of the activity of both pile- and cyclotron-produced

TABLE I  
EXTRACTION OF IRON-59 AND ZINC-65 BY DIETHYLDITHIOCARBAMATE AT pH 14<sup>a</sup>

<sup>59</sup> Fe	Pile <sup>b</sup>	Cyclotron <sup>c</sup>
1 Freshly irradiated	40	48
2 After 70 days (1.5 times $\tau_1$ )	105	185
3 As no. 2; purified <sup>d</sup>	<0.5	<3
4 As no. 2; no Co-carrier <sup>e</sup>	<0.5	<0.5
<sup>65</sup> Zn	Pile	
1 Non-purified	35	
2 Purified <sup>f</sup>	40	

\* Tracer is added to give an original count-rate of 10<sup>6</sup> C/min; mean values of 4 experiments are given in C/min found in the final organic layer.

<sup>b</sup> <sup>59</sup>Fe was produced by the <sup>58</sup>Fe(*n*, $\gamma$ ) <sup>59</sup>Fe reaction; some <sup>60</sup>Co was formed (by (*n*, $\gamma$ ) reactions) from the small amounts of cobalt in the iron target. (Amersham - UK)

<sup>c</sup> <sup>59</sup>Fe was produced by the <sup>59</sup>Co (*d*, 2p)–<sup>59</sup>Fe reaction (in concurrence with the <sup>59</sup>Co (*d*,p)–<sup>60</sup>Co reaction) from a cobalt-target. Most of the <sup>60</sup>Co was removed by the manufacturer (Philips-Duphar, Amsterdam).

<sup>d</sup> Purified — after the addition of Fe- and Co-carriers — by *N* successive extractions with isopropylether from 7 *N* HCl, evaporation of the ether, addition of new carriers and HCl up to 7 *N*, and repetition (*M*-times) of the process. In column 2: *N* = 2 and *M* = 3, the efficiency for Fe and Co was 80 and 0.02% respectively. In column 3: *N* = 3 and *M* = 2, and the respective efficiencies were 95 and 1%.

<sup>e</sup> Carrier-free cobalt is extracted very incompletely (see introduction).

<sup>f</sup> <sup>65</sup>Zn was purified by descending paper chromatography. The solvent (acetone–4 *N* HCl = 75 : 25) was allowed to drip off.

\* Alternatively 1 mg of each metal is added as a carrier; in this case 0.05 *M* tartrate and 10 mg of diethyldithiocarbamate are used.

\*\* Shaking was carried out in a vertical position in the "Microid flask shaker" at 900 periods per min for 3 min.

\*\*\* A Philips detector (PW 4111 W) and scaler (PW 4022-4032-4052), combined with an E.K.A.F. single channel analyser (30301–20102), were used.

iron-59 is due to cobalt-60. (For the pile-produced iron-59 the manufacturer gave the value:  $<0.1\%$ ).

When the alkaline back-extraction is omitted or when it is replaced by a simple rinsing, results with iron-59 are 5 to 30 times higher. When the procedure is carried out with 1 ml of both layers, evaporation of the organic layer is not required but results with iron-59 are doubled.

Addition of the diethyldithiocarbamate reagent at lower pH values results in higher extraction percentages of iron-59. The addition of ammonium tartrate, sodium fluoride, pyrophosphate, "tiron", or ascorbic acid does not have any favourable influence on the results. The use of 10 mg of each carrier in the 5-ml volume resulted in precipitation. The addition of mercury(II) or potassium cyanide to the alkaline tartrate used for the back-extraction did not improve the results.

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#### SUMMARY

A method is described for the radiochemical separation of cobalt based on the extraordinary stability of cobalt diethyldithiocarbamate. Interferences are few; only very small amounts of zinc and iron accompany cobalt, which is important in neutron-activation analysis.

#### RÉSUMÉ

Une méthode est proposée pour la séparation radiochimique du cobalt, sous forme de diéthyl-dithiocarbamate; ce composé présente une stabilité remarquable.

#### ZUSAMMENFASSUNG

Es wird eine radiochemische Abtrennungsmethode für Kobalt beschrieben. Sie beruht auf der aussergewöhnlichen Stabilität des Kobalt-Diäthyl-dithiocarbamat Komplexes.

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