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A Novel Method for the Preparation of Carrier-Free Lead-212[1]

by

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Carrier-free lead-212, a member of the naturally occurring thorium-232 series, has been prepared by collecting the active deposit from highly emanating thorium oxide samples[2] and by the sweeping of thoron from

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(2) E. M. Morimoto and Milton Kahn, J. Chem. Educ. 36, 296 (1959).

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thorium nitrate solutions with a stream of air.[3] In the procedure

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(3) E. Broda, H. Fabitschowitz and T. Schonfeld, Monatsh. 83, 482 (1952).

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reported here a nitric acid solution of thorium nitrate is refluxed in a Soxhlet extractor in which the usual extraction thimble is replaced by a small (22 x 55 mm.) cation-exchange resin column. The thoron which escapes from the boiling solution is carried along with the vapors into the upper part of the apparatus where the water vapor condenses; it is in this region that the thoron collects and decays to polonium-216 which in turn decays to lead-212; the lead-212 is subsequently washed onto and retained by the resin. In order to prevent the escape of thoron from the apparatus into the laboratory, the condenser is sealed with Parafilm

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after most of the air in the apparatus has been displaced by water vapor (about 2 minutes after refluxing has begun). The lead activity is eluted from the resin and removed from the walls of the condenser with 1 N hydrochloric acid; the resulting solution may be used directly or evaporated to dryness in a quartz crucible under a heat lamp and subsequently dissolved in a dilute strong acid or acetic acid.

The results of several experiments are summarized in Tables 1, 2 and 3. The yield was essentially independent of reflux times less than 15 hours (Table 1), decreased slightly with increase in volume of the solution (Table 3), and was only slightly dependent on the nitric acid concentration; the yield was highest (~45%) for a nitric acid concentration of 1 molar (Tables 2 and 3). It is noteworthy that when a pure thorium nitrate solution was used most of the activity was found on the walls of the condenser (Table 1); when the acidity of the thorium nitrate solution was increased by addition of nitric acid, a greater percentage of the activity was found on the resin. This behavior may be correlated with the relatively high pH (~ 5) of the condensate of a pure thorium nitrate solution compared with that (pH = ~2) of thorium nitrate solutions, 1 molar in nitric acid.

Solutions of lead-212, in equilibrium with its decay products, were counted with a scintillation detector employing a sodium iodide (thallium activated) crystal and a #5819 R. C. A. phototube. The per cent recovery was based on the lead-212 activity separated from a known amount of thorium nitrate in equilibrium with its decay products; the growth and decay of lead-212 in the apparatus was taken into account in these calculations. This separation was effected by the precipitation of lead carrier from a thorium nitrate solution with hydrogen sulfide; the sulfide was washed with 0.1 N nitric acid, dissolved in 2.5 N nitric acid, diluted to 0.1 N nitric acid with water and again precipitated with hydrogen sulfide. The final precipitate was dissolved in 2.5 N nitric acid and diluted for counting. Dowex-50 cation-exchange resin, acid form, 8% cross linkage, 100-200 mesh, obtained from Bio Rad Laboratory, was further purified by treatment with 6 N hydrochloric acid and subsequently washing with water. The thorium content of a thorium nitrate solution was checked by employing a gravimetric procedure based on the precipitation of thorium oxide.[4]

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(4) C. J. Roddin, Editor, Analytical Chemistry of the Manhattan Project, National Nuclear Energy Series, VIII-1, p. 183. McGraw-Hill, New York (1950).

Table 1

Dependence of Recovery of Lead-212 from
Aqueous Thorium Nitrate Solutions on Time of Reflux[a]

Time of reflux, hrs.	Activity on resin, %	Activity in condenser, %
3.7	1	24
6.1	2	29
15.1	3	21
42.4	21	12

a) 575 ml. of 0.065 M $Th(NO_3)_4$ was refluxed in a 1-liter flask.

Table 2

Effect of Nitric Acid Concentration on Recovery of Lead-212 from Thorium Nitrate Solutions[a]

Volume of soln., ml.	$Th(NO_3)_4$ conc., M	HNO_3 conc., M	Activity on resin, %	Activity in condenser, %
575	0.065	0.014	22	14
575	0.065	0.014	10	17
580	0.065	0.14	26	11
580	0.065	0.14	37	6
625	0.060	1.3	40	5
625	0.060	1.3	41	5
675	0.055	2.38	33	6
675	0.055	2.38	34	5

a) Average time of reflux = 10.8 $\pm$ 0.2 hours.

Table 3

Dependence of Recovery of Lead-212 from Aqueous Thorium Nitrate Solutions on Volume of Solution[a]

Volume of soln., ml.	Volume of boiling flask, ml.	Activity on resin, %	Activity in condenser, %
50	100	36	15
50	100	37	11
250	500	32	8
500	1000	28	3

a) Average time of reflux = 12.0 ± .5 hours; $Th(NO_3)_4$ concentration = 0.75 M; HNO_3 concentration = 1 M.