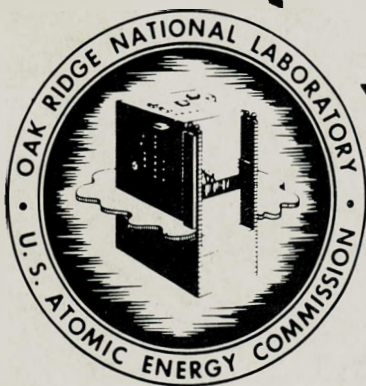


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ORNL-2330  
Chemistry-General  
TID-4500 (13th ed.)

ISOLATION OF CARRIER-FREE  $\text{Mn}^{54}$  AND  $\text{I}^{125}$   
FROM CYCLOTRON TARGETS

H. T. Russell



OAK RIDGE NATIONAL LABORATORY  
OPERATED BY

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ORNL-2330

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OPERATIONS DIVISION

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DATE ISSUED

JUL 31 1957

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## ISOLATION OF CARRIER-FREE $\text{Mn}^{54}$ AND $\text{I}^{125}$ FROM CYCLOTRON TARGETS

H. T. Russell

### ABSTRACT

Chemical processing for the recovery and purification of carrier-free  $\text{I}^{125}$  and  $\text{Mn}^{54}$  produced by bombardment of isotopically enriched  $\text{Te}^{125}$  and  $\text{Cr}^{54}$  with protons in the ORNL 86-Inch Cyclotron is discussed. The enriched  $\text{Te}^{125}$  and  $\text{Cr}^{54}$  were recovered for re-use.

### INTRODUCTION

Carrier-free radioisotopes of  $\text{Mn}^{54}$  and  $\text{I}^{125}$  were isolated from enriched target materials which were irradiated with protons in the ORNL 86-Inch Cyclotron.<sup>1</sup>

Manganese-54 was produced by the  $(p,n)$  reaction on  $\text{Cr}^{54}$  which had been enriched to 65%. Five hundred milligrams of enriched  $\text{Cr}^{54}$  was electroplated<sup>2</sup> on a  $2 \times 3$  in. area of the  $6 \times 7 \times 0.5$  in. silver backing and bombarded with 2.0 ma of 18-Mev protons for a period of 10.7 hr.<sup>1</sup> The yield was 36 mc of  $\text{Mn}^{54}$  activity.

The  $\text{I}^{125}$  activity was produced by the  $(p,n)$  reaction on  $\text{Te}^{125}$ . To prevent loss of the iodine activity during bombardment and prior to chemical separation, the  $\text{TeO}_2$  target was sealed in an aluminum capsule.<sup>3</sup> The 310-mg sample of 88%  $\text{Te}^{125}$  as  $\text{TeO}_2$  was irradiated in the sealed capsule with 22-Mev protons from the ORNL 86-Inch Cyclotron<sup>1</sup> for a period of 8.3 hr to produce 14 mc of  $\text{I}^{125}$ .

### $\text{Mn}^{54}$ Purification

The chemical separation method described here was developed by the author in 1951 for use in the recovery and purification of  $\text{Mn}^{54}$  activity from cyclotron targets.

Prior to production of  $\text{Mn}^{54}$  by the ORNL 86-Inch Cyclotron in March 1954, targets were obtained from other cyclotrons<sup>4</sup> to provide the  $\text{Mn}^{54}$  activity required for the ORNL radioisotope distribution program.

Isolation of the carrier-free  $\text{Mn}^{54}$  from the chromium target was accomplished by an ion-exchange method as described below.

The chromium was dissolved from the silver backing in a minimum amount of 12 N HCl. The  $\text{CrCl}_3$  solution was evaporated to dryness, and the  $\text{CrCl}_3$  cake containing the  $\text{Mn}^{54}$  activity was dissolved in 5% oxalic acid solution. This solution was evaporated to dryness, and the cake was redissolved in 50 ml of 5% oxalic acid solution.

From this oxalic acid solution, the  $\text{Mn}^{54}$  activity was adsorbed in a narrow band at the top of an IR-120 resin column approximately 2 cm long and 5 mm in inside diameter. The chromium, which was not adsorbed on the column, was collected in the effluent and recovered for re-use.

The column was washed with 5% oxalic acid solution to remove trace amounts of chromium from the  $\text{Mn}^{54}$  activity. Distilled water was then passed through the column to remove oxalic acid solution. The carrier-free  $\text{Mn}^{54}$  activity was eluted from the column in 1 N HCl.

The effluent containing the  $\text{Mn}^{54}$  activity was fumed with 16 N  $\text{HNO}_3$  to destroy organic compounds and then fumed with 12 N HCl to destroy the nitrates. The solution was evaporated to dryness and the activity was taken up quantitatively as the chloride in 0.1 N HCl.

<sup>1</sup>F. L. Green and J. A. Martin, "Increased Yields of  $\text{Mn}^{54}$ ,  $\text{I}^{125}$ , and  $\text{I}^{130}$  with a 22-Mev Proton Cyclotron," to be submitted for publication to *Nuclear Science and Engineering*.

<sup>2</sup>P. S. Baker, F. R. Duncan, and L. O. Love, "Cyclotron Targets Using Enriched Stable Isotopes," to be submitted for publication to *Nuclear Science and Engineering*.

<sup>3</sup>J. A. Martin and F. L. Green, "Cyclotron Target for the Irradiation of Chemical Compounds," *Nuclear Science and Engineering* 1, 185-190 (1956).

<sup>4</sup>Washington University, St. Louis, Missouri; University of California, Radiation Laboratory, Berkeley, California.

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Analysis of the  $\text{Mn}^{54}$  product solution was as follows:

Volume (ml)	100
$\text{Mn}^{54}$ activity (mc/ml)	0.36
$\text{Mn}^{52}$ activity (mc/ml)	$< 4 \times 10^{-4}$
Radiocontaminants	None detected
Heavy metals (ppm)	$< 10$
Acidity (HCl normality)	0.10
Total solids (mg/ml)	$< 1$
Nonvolatile materials (mg/ml)	$< 1$

The  $\text{Mn}^{54}$  activity was identified by gamma-ray spectroscopy and by the 310-day half life reported<sup>5</sup> for  $\text{Mn}^{54}$  in the literature.

#### $\text{I}^{125}$ Purification

The aluminum capsule containing the bombarded  $\text{TeO}_2$  was dissolved in a closed distillation system, to prevent loss of iodine, in a minimum amount of 8% NaOH and 16%  $\text{NaNO}_3$  solution. The dissolution progressed at a satisfactory rate without the application of heat until all the aluminum capsule was dissolved. Nitric acid (70%) was added dropwise to the boiling solution to make the solution acidic and dissolve the  $\text{TeO}_2$ . The iodine, nitrogen oxides, and water were then distilled under slight negative pressure at  $95^\circ\text{C}$  into 1 N NaOH solution.

Further purification and concentration of the iodine was necessary to remove the activity from the NaOH solution and purify from traces of nitrate

and nitrite ions. This was accomplished by oxidizing the iodine to the nonvolatile iodate and distilling the nitric acid. The iodate was then reduced to iodine, which was distilled into 0.15 N NaOH solution. The chemical steps involved are described below.

A 1 N  $\text{KMnO}_4$  solution was added to the sodium hydroxide solution to oxidize the iodine to the iodate. Then 18 N  $\text{H}_2\text{SO}_4$  was added to neutralize the sodium hydroxide and to raise the sulfuric acid concentration in the solution to 6 N. During the acidification 1 N  $\text{KMnO}_4$  was added to maintain the purple permanganate color. Twenty per cent of the solution was distilled and discarded to remove traces of nitric acid from the nonvolatile iodate. The sulfuric acid concentration in the distillation flask was re-adjusted to 6 N by the addition of water. A saturated  $\text{H}_3\text{PO}_3$  solution was added dropwise to the boiling solution to reduce the excess potassium permanganate and to reduce the iodate to iodine. Elemental iodine was then distilled and was trapped in weak sodium hydroxide solution. The rate of distillation was accelerated by the addition of a few drops of 30%  $\text{H}_2\text{O}_2$  to the distillation flask.

Fourteen millicuries of  $\text{I}^{125}$  was recovered in 100 ml of 0.15 N NaOH solution. The radionuclides of  $\text{I}^{124}$  and  $\text{I}^{126}$  were identified in the solution; however, the amounts were not determined.

The  $\text{I}^{125}$  activity was identified by examination on the gamma-ray spectrometer and by the 60-day half life reported<sup>6</sup> for  $\text{I}^{125}$ .

The isotopically enriched  $\text{Te}^{125}$  was recovered for re-use.

<sup>5</sup>J. M. Hollander, I. Perlman, and G. T. Seaborg, "Table of Isotopes," reprinted from *Revs. Mod. Phys.* 25(2), 493 (1953).

<sup>6</sup>*Ibid.*, p 546.