Isolation of Carrier-free Tellurium from Antimony Target

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(Received November 10, 1962)

On the separation of tellurium from antimony, several different procedures were described in the literature, including coprecipitation method,[1] ion-exchange method[2] and so forth. These methods, however, do not give satisfactory results for the separation of trace amounts of tellurium from several grams of antimony. In a study of the energy level schemes of tin nuclei, it became necessary to separate trace amounts of radioactive tellurium in the state of carrier-free from proton-irradiated antimony targets. The radioactive tellurium thus separated is to be electroplated on a metal foil and to be measured with a β-ray spectrometer.

As reported by one of the present authors,[3] tellurium(IV) is extracted with a carbon tetrachloride solution of dithizone. As to antimony, dithizonate of trivalent antimony was reported,[4] but no definite description on the pentavalent state was made. A simple test showed that antimony(V) is not extracted into a carbon tetrachloride solution of dithizone from acidic solution. Considering such properties, the authors devised the following separation procedure and applied it to bombarded samples.

Separation Procedure

Dissolution of Antimony Target and Extraction of Tellurium.—Several grams of antimony target (metal powder) are dissolved in 30 ml. of aqua regia and the solution is slowly evaporated to dryness. To the residue, another 30 ml. of aqua regia are added and again evaporated to dryness. Such treatment should be repeated at least three times for completion of the oxidation of antimony to pentavalent state. After the treatment with aqua regia, the resulting white residue is dissolved in 20 ml. of 6 M hydrochloric acid and the solution is warmed for a while to ensure the tetravalency of tellurium. Then the solution is diluted with distilled water so as to be 3 M in hydrochloric acid. At lower acidity than 3 M in hydrochloric acid, antimony is hydrolyzed and gives a white precipitate which prevents tellurium from being extracted with dithizone. Extraction of tellurium from the hydrochloric acid solution is carried out in a separatory funnel with an equal volume of concentrated dithizone solution. With a volume ratio of 1 : 1, extractabilities of tellurium under the given condition are 30% with a saturated dithizone-carbon tetrachloride solution and 75% with a saturated dithizone-chloroform solution[*1]. Therefore, extraction process repeated three times may be enough for essentially complete removal of tellurium from the aqueous layer, when a saturated dithizone-chloroform solution is used.

Back-Extraction of Tellurium.—After combined dithizone extracts are washed with dilute hydrochloric acid, they are transferred into another separatory funnel, together with a suitable amount of distilled water. To this separatory funnel, the aqueous solution of cupric chloride is added until the amount of cupric ion becomes a little in excess to the equivalent amounts combining with dithizone, and then the funnel is thoroughly shaken. When dithizone is saturated with copper, tellurium is liberated from dithizone chelate into the aqueous layer. This is because copper has a greater affinity than tellurium does for dithizone.

Then, the aqueous phase is separated. A small amount of copper contained in it can be removed by repeated extractions with a very dilute carbon tetrachloride solution of dithizone ($\sim 10^{-6}$ M/l.). The loss of tellurium during this operation is negligible, if a sufficiently dilute solution is used for the extraction. By this procedure, more than 90% of radioactive tellurium is brought into the aqueous phase.

Results and Discussion

A preliminary experiment showed that, when trace amouts of $^{129\text{m}}$Te artificially mixed with 1 g. of antimony were separated by the separation procedure described above, the amount of antimony contaminated in tellurium fraction was less than 60 μg.[*2] (0.006% of initial antimony). On the other hand, when carbon tetrachloride was used as a solvent of dithizone instead of chloroform, antimony contamination was less than 30 μg. (0.003%).

As good results were obtained in the preliminary experiment, this method was applied to the separation of a bombarded sample. About 5 g. of antimony were irradiated with 56 MeV. proton (beam current 0.4 μamp.) for 24 hr. After the bombardment, the target was dissolved in aqua regia and treated with a chloroform solution of dithizone as described above. Tellurium fraction thus obtained was again purified with a carbon tetrachloride solution of dithizone in a similar way. Figure 1 shows γ-ray spectra of tellurium and antimony fractions. They are clearly different

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[1] N. Goldberg and S. Frankel, *Phys. Rev.*, **100**, 1350 (1955).

[2] Y. Sasaki, This Bulletin, **28**, 615 (1955).

[3] H. Mabuchi, ibid., **29**, 842 (1956).

[4] J. K. Carton, W. C. Bradbury and R. Kruh, *Anal. Chim. Acta*, **12**, 101 (1955).

[*1] The solubilities of dithizone in carbon tetrachloride and chloroform are 0.64 g. and 17.8 g. per liter at room temperature, respectively.

[*2] The amout of antimony was determined by rhodamine B method.

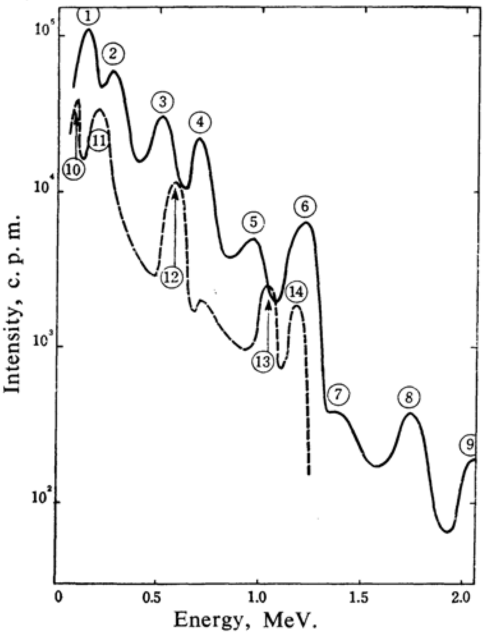

Fig. 1. γ-Ray spectra of tellurium and antimony fractions.
Full line: tellurium fraction one day after the bombardment.
Broken line: antimony fraction two days after the bombardment.

Assignment of the peaks

No. of Peaks	Energy Mev.	Nuclides
1	0.15	$^{119}\mathrm{Sb}(\leftarrow{}^{119}\mathrm{Te})$
2	0.27	$^{119}\mathrm{Sb}(\leftarrow{}^{119}\mathrm{Te})$
3	0.51	$^{118}\mathrm{Sn}(\leftarrow{}^{118}\mathrm{Sb}\leftarrow{}^{118}\mathrm{Te})$
4	0.66, 0.71	$^{119}\mathrm{Sb}(\leftarrow{}^{119\mathrm{m}}\mathrm{Te})$
5	0.92, 0.95, 0.98	$^{119}\mathrm{Sb}(\leftarrow{}^{119}\mathrm{Te})$
6	1.22	$^{118}\mathrm{Sn}(\leftarrow{}^{118}\mathrm{Sb}\leftarrow{}^{118}\mathrm{Te})$, $^{119}\mathrm{Sb}(\leftarrow{}^{119}\mathrm{Te})$
7	1.37	$^{119}\mathrm{Sb}(\leftarrow{}^{119\mathrm{m}}\mathrm{Te})$
8	1.74	$^{119}\mathrm{Sb}(\leftarrow{}^{119\mathrm{m}}\mathrm{Te})$
9	2.06	$^{119}\mathrm{Sb}(\leftarrow{}^{119}\mathrm{Te})$
10	0.09	$^{120}\mathrm{Sn}(\leftarrow{}^{120\mathrm{m}}\mathrm{Sb})$
11	0.20	$^{120}\mathrm{Sn}(\leftarrow{}^{120\mathrm{m}}\mathrm{Sb})$
12	0.58	$^{122}\mathrm{Sn}(\leftarrow{}^{122}\mathrm{Sb})$
13	1.03	$^{120}\mathrm{Sn}(\leftarrow{}^{120\mathrm{m}}\mathrm{Sb})$
14	1.17	$^{120}\mathrm{Sn}(\leftarrow{}^{120\mathrm{m}}\mathrm{Sb})$

from each other, and the peaks in the spectra of antimony fraction were all assigned to those of antimony isotopes.

The important part of this separation method is the oxidation of antimony with aqua regia, because complete oxidation of gram-scale antimony is fairly difficult. Clear-cut separation depends on the completeness of oxidation.

The authors wish to express their hearty thanks to Dr. Hidetsugu Ikegami of the Institute for Nuclear Study of the University of Tokyo for his cordial guidance to physical problems and careful discussions throughout this research.

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