EXPERIMENTELLES

Die experimentelle Durchführung unserer Versuche auf der Grundlage der Mitführungsmethode ist bereits an anderer Stelle beschrieben.[6] Die Strömungsgeschwindigkeit des als Trägergas benutzten Stickstoffs betrug im Mittel 20 ml/min, was zur Einstellung eines quasistatischen Gleichgewichtes ausreicht.

In_2S_3 wurde durch Fällung dargestellt und bei 500°C im H_2S-Strom getrocknet; InS wurde durch Synthese aus den Elementen im Quarzrohr[7] erhalten; das verwandte Indium war 99·999 Prozent. Die Analysen der Verbindungen entsprachen den berechneten Werten.

Das aus der Gasphase kondensierte In_2S schied sich in Form eines feinen grauen bis schwarzen Pulvers ab, das ausserordentlich fest an den Quarzrohren der Apparatur haftete. Dieses Pulver erleidet beim Aufbewahren an der Luft keine merkliche Veränderung; es löst sich leicht in oxidierenden Säuren. Das Pulverdiagramm (CuK_α-Strahlung) zeigt nur die Linien des InS. Nach diesem Befund und dem Zustandsdiagramm[1] sollte man daher annehmen, dass In_2S_{fest} instabil ist und gemäss dem Schema

$$In_2S_{gas} \rightarrow In_2S_{fest} \rightarrow InS_{fest} + In_{fl.} \qquad (4)$$

disproportioniert. Wir konnten jedoch im Debye-Scherrer-Diagramm des aus der Gasphase erhaltenen Produktes der Bruttozusammensetzung In_2S auch nach längerem Aufbewahren keine Linien des metallischen Indiums entdecken. Worauf dies zurückzuführen ist, ist noch nicht geklärt.

Anerkennungen—Wir danken den Unterharzer Berg- und Hüttenwerken, Oker am Harz, für die Überlassung reinsten Indiums.

F. KLANBERG
H. SPANDAU

Eduard-Zintl-Institut fur anorganische und physikalische Chemie der Technischen Hochschule, Darmstadt, Germany.
Anorganisches-chemisches Institut der Technischen Hochschule, Braunschweig, Germany.

[6] H. SPANDAU und F. KLANBERG, *Z. anorg. allg. Chem.* **295**, 291 (1958)
[7] W. KLEMM und H. U. VON VOGEL, *Z. anorg. allg. Chem.* **219**, 45 (1934)

Search for an antimony isotope with a 30 days half-life

(*Received* 20 *January* 1961)

A fission product isotope of antimony which decays with a half-life around 30 days has been reported by several authors.[1,2,3] As in previous work at our laboratory with the heavy isotopes of antimony we had never observed such a half-life, some experiments were performed to determine its existence and nuclear properties.

EXPERIMENTAL

0·5 grams of UO_2 were irradiated for 8 hours in the RA1 reactor. 30 days after the end of the irradiation the antimony fraction was separated chemically and purified by reduction with zinc to form volatile stibine.[4]

Radioactivity measurements were performed with a thin end-window G.M. counter, over approximately 8 months. Because of the low intensity of the source available, the gamma spectrum could not be measured.

The analysis of the curve showed a half-life of approximately 4 days corresponding to Sb^{127}, which had not completely decayed, and a much longer half-life, of over one year, which may be due to Sb^{125}.

In another experiment UO_2 was irradiated for 8 hours with 28 MeV deuterons from the Buenos Aires synchrocyclotron. The antimony fraction was separated 31 days after the end of irradiation. A series of gamma spectra of the source obtained were taken at from 32 to 200 days after the end of irradiation with a single channel scintillation spectrometer with a 2″ × 2″ NaI(Tl) crystal. These spectra show the more important rays of Sb^{124} (60 days), Sb^{125} (2 years), Sb^{126} (11 days) and Sb^{127}

[1] E. W. GRUMMITT and G. WILKINSON, *Nature, Lond.* **158**, 163 (1946).
[2] J. W. BARNES and A. J. FREEDMAN, *Phys. Rev.* **84**, 365 (1951).
[3] T. T. SUGIHARA P. J. DREVINSKY E. J. TROIANELLO and J. M. ALEXANDER, *Phys. Rev.* **108**, 1264 (1957).
[4] H. BOSCH and H. MUNCZEK, *Phys. Rev.* **106**, 983 (1957).

(4 days). The peaks of the Sb^{127}, as was expected, were only found in the first spectrum taken. In the spectra taken after 190 days only the peaks belonging to Sb^{125} were left.

The same source was beta-counted without absorber and with a 270 mg/cm² aluminium foil absorber. The decay curve without absorber showed four half-lives: 4 days (Sb^{127}), 12 days (Sb^{126}), 60 days (Sb^{124}) and a residual activity of half-life over one year which may be due to Sb^{125}. The analysis of the curve taken with aluminium absorbers shows only the 12 days and 60 days half-lives.

In earlier work at our laboratories and in other centres[5] no 30 day activity was found on irradiating the enriched heavy isotopes of tellurium with fast neutrons. In order to confirm these results enriched Te^{126} (80 per cent), Te^{128} (85 per cent) and Te^{130} (78 per cent) were irradiated once more with fast neutrons produced by 28 MeV deuterons on beryllium in the Buenos Aires synchro cyclotron. Some gamma spectra were taken at different times from the end of the irradiation and the decay curves were followed in a gamma-ray spectrometer. In no case could a 30 day activity be seen.

DISCUSSION

In the antimony fraction obtained from thermal fission 30 days from the end of the irradiation we only observed the half-lives of Sb^{127} and Sb^{125}, which were known already. The Sb^{124} and Sb^{126} half-lives were not seen because both isotopes are shielded and their independent yields in thermal fission are small.

In fission induced by 28 MeV deuterons, as was expected under our working conditions, all the isotopes with half-lives longer than 30 days were found. As the energy of the bombarding particles increases, Z_p (most probable charge in a fission chain) shifts towards Z_A (most stable charge), so that the independent fission yield of isotopes close to Z_A increases rapidly.

Both disintegration curves show no sign of a 30 days half-life.

The series of gamma spectra show only peaks belonging to isotopes already known, even though a complete analysis of the spectra was not possible because there were too many isotopes present with complex disintegration schemes.

The decay curves of the gamma activity were obtained by integration of several parts of the spectra taken at different times and no 30 day half-life was observed.

From our experiments we may conclude that in thermal fission and fission induced by 28 MeV deuterons on natural uranium no antimony isotope of 30 days half-life is formed. If such nuclide should exist, its mass number would be over 125. The only mass numbers possible would then be 126, 128 or 130, because the isotopes with odd A are known and do not admit isomers. Two isomers are known for each mass number for masses 126, 128 and 130, but a third isomer could exist as in the case for Sb^{124}. However, the experiments with enriched isotopes of tellurium of masses 126, 128 and 130 did not show any half-life of approximately 30 days.

Acknowledgement—The authors wish to express their appreciation to the crew of the synchro-cyclotron and the RA1 for the irradiations performed and to the Electronics Department for maintenance of the equipment.

M. C. PALCOS
R. RADICELLA
J. RODRIGUEZ

Radiochemistry Division National Atomic Energy Commission
Buenos Aires, Argentine Republic

[5] H. BOSCH. Private communication (1960).

Formation of antimony orthophosphate, $SbPO_4$

(*Received* 24 *December* 1960; *in revised form* 4 *February* 1961)

THERE are very few references in the literature to the preparation of antimony phosphates: standard reference sources[1,2,3] all quote identical early original sources and include only one or two more recent references.

[1] GMELIN, "*Handbuch der anorganische chemie*", System nummer 18, Teil B, Lieferung 3 (1949).
[2] P. PASCAL, "*Nouveau traité de chimie minéral*", Tome XI, p. 641, Masson et Cie, Paris (1958).
[3] J. MELLOR, "*Inorganic and Theoretical Chemistry*", Vol. IX, p. 585, Longmans Green and Co., London (1933).