CarrierFree Radioisotopes from Cyclotron Targets. I. Preparation and Isolation of Sn113 and In114 from Cadmium

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a determination than with the conventional method and without sacrifice of accuracy. The proposed assembly is adaptable to a portable piece of equipment for surface area measurements.

The apparatus includes a U-shaped adsorption tube, one arm of which is capillary tubing and the other contains the sample. A gas entering the tube has intimate access to all parts of the sample before leaving. Calibrated gas burets of approximately equal volume are connected to each of the two arms of the adsorption tube. Both burets are equipped with hand-operated mercury leveling bulbs.

The sample contained in the adsorption tube is freed from conflicting impurities, such as air and moisture, by heating in a continuous stream of helium. Before making the dead-space-calibration, the exit connection is closed and the furnace surrounding the adsorption tube replaced by a Dewar flask containing liquid oxygen. A quantity of helium is next drawn into the system filling the sample tube and only one of the burets. The resultant pressure is designated as p_1. When the helium is expanded so as to include the second buret, as well, its pressure becomes p_2. The "dead-space" volume may be calculated according to the easily derivable relationship:

$$V/T=[p_2/(p_1-p_2)](V_2/T_0)-V_1/T_0,$$

where V = geometrical volume of dead space, T = temperature of liquid O_2 or N_2 bath, V_2 = volume of buret No. 2, V_1 = volume of buret No. 1, p_2 = pressure of helium in the system including both burets and dead space, p_1 = pressure of same quantity of helium when compressed into the volume of No. 1 buret and the dead space, and T_0 = room temperature.

A measured amount of nitrogen is then introduced and the helium-nitrogen mixture is passed from one gas buret through the sample to the other buret. This process is repeated until a constant pressure reading is obtained. The liquid oxygen bath is not removed between the time of calibration and nitrogen adsorption. An additional quantity of nitrogen is added for each subsequent adsorption point. In the calculation it is necessary to subtract the partial pressure of the helium from the total pressure to obtain that due to the nitrogen. The surface area may be then calculated from the adsorption data in the well-known procedure of Brunauer, Emmet, and Teller.

It has been shown for a number of adsorbents, such as silica gel, coconut-shell charcoal, an activated clay, titania, and bone char, that the pressure of helium has very little, if any, influence on the adsorption of nitrogen at liquid oxygen temperature. For example, values for surface area of 60, 61, 61 m^2/g were obtained for a sample of bone char with the respective partial pressure of helium of 80, 79, and 405 mm Hg. The value obtained for the same sample in the absence of helium in a conventional system was 59 m^2/g.

Carrier-Free Radioisotopes from Cyclotron Targets. I. Preparation and Isolation of Sn^{113} and In^{114} from Cadmium*

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THE cyclotron is the only practical source of many carrier-free[1] radioisotopes. The preparation and radiochemical isolation of a number of these activities, produced in the 60-in. cyclotron of Crocker Laboratory, will be presented in this paper and in subsequent papers of this series. In most cases the carrier-free radioisotopes were prepared for use in biological systems and the final preparations were in the form of isotonic saline solutions at a range of pH from 5 to 8.

The present paper reports the radiochemical isolation of carrier-free Sn^{113} and In^{114} produced by bombarding cadmium with 38-Mev alpha-particles. At this energy, Sn^{113} and In^{114} are produced in a thick target by the nuclear reactions:[2] $Cd^{110}(\alpha, n)Sn^{113}$, $Cd^{111}(\alpha, 2n)Sn^{113}$, $Cd^{112}(\alpha, 3n)Sn^{113}$, $Cd^{111}(\alpha, p)In^{114}$, $Cd^{112}(\alpha, pn)In^{114}$. The shorter-lived tin and indium activities, together with the possible radioisotopes of silver produced by (n, p) reactions, were allowed to decay out prior to the chemical separations.

The target, a block of C.P. cadmium metal, soft-soldered to a water-cooled copper plate, was bombarded with 38-Mev alpha-particles for a total of 450 μa-hr. at an average beam intensity of 3.4 μa. After aging for one week, the bombarded surface was milled off and dissolved in a minimum volume of 16N HNO_3.

A 0.2 g of target cadmium nitrate was dissolved in 25 ml of water, and the tin and indium activities were carried quantitatively on 10 mg of $Fe(OH)_3$ precipitated with NH_4OH. The $Fe(OH)_3$ was dissolved in 15 ml of 36N H_2SO_4 and transferred to an all-glass distilling flask.[3,4] The 9N HBr was added dropwise while a stream of CO_2 was bubbled through the solution at 220°C. The distillate containing the carrier-free Sn^{113}, HBr, Br_2, and traces of H_2SO_4 was caught in a series of traps filled with 12N HCl; the indium activity remained in the residue. Carrier-free radio-tin collected in HNO_3 or H_2SO_4 forms a radiocolloid[5,6] and is adsorbed onto the walls of the containing vessel. The 12N HCl keeps the radio-tin in solution, presumably as the chlorostannate complex. The trap contents were treated with 5 ml of 16N HNO_3 to destroy HBr, 15 mg of citric acid were added and the solution was evaporated on a steam bath to the 1- to 2-ml volume of H_2SO_4 carried over in the distillation. Citric acid prevents the formation of radiocolloid after removal of HCl.

The H_2SO_4 solution was diluted with 25 ml of water and the radio-tin was carried down on $Fe(OH)_3$ precipitated with NH_4OH. The $Fe(OH)_3$ was dissolved in 8N HCl and iron was extracted with isopropyl ether. The aqueous phase, containing HCl, Sn^{113}, and equilibrium amounts of the In^{113} daughter, was evaporated to dryness on 10 mg of sodium citrate. The activity dissolved quantitatively in distilled water.

The carrier-free Sn^{113} was identified by its 105-day half-life and by the 0.39-Mev conversion electron of the In^{113} daughter.[7] The indium fraction from a chemical separation of an equilibrium mixture using tin and indium carriers, showed the 105-min. period of In^{113}.

The residue from the tin distillation, containing Fe^{+++} and In^{114}, was neutralized with NH_4OH. The $Fe(OH)_3$ plus indium activity was dissolved in 8N HCl, and extracted with isopropyl ether. The HCl solution of In^{114} was evaporated to dryness on 10 mg of NaCl. The activity dissolved quantitatively with the addition of distilled water. The In^{114} was identified by the assigned 48-day half-life and by the 0.19-Mev conversion electron.[7,8]

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[1] This term is used to indicate that no stable isotopic carriers have been intentionally added. In a "carrier-free" separation the specific activity is determined by the chemical purity of the reagents.

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