which was alongside the 800-mg. samples during each irradiation.

Since the success of the Szilard–Chalmers reaction is known to be dependent on the level of the γ-radiation in the pile it was desirable that all samples be irradiated under the same conditions.[2] The tetraphenyltin samples used in experiments 1 and 2 were alongside each other in the pile during the irradiation as were the samples used in experiments 3 and 4. It is believed that the γ-fluxes during these two irradiations were approximately the same because the beta activity of the samples used in experiments 3 and 4 was only 15% higher than that of the samples used in experiments 1 and 2.

The β-activity of the 1.1-day Sn^{121} was detected in all experiments. During the two-hour irradiation the activities of Sn^{113} and Sn^{123} produced are negligible compared to that of Sn^{121}. The 2.7-year Sb^{125} activity that is produced through decay of the 10-minute Sn^{125} is also negligible compared to the Sn^{121} activity. The tin was precipitated as stannic phenylarsonate,[3] suspended in 5 ml. of 95% alcohol and then prepared for counting by filtration on a tared, 24 mm. diameter, Whatman #50 filter paper, dried for 30 minutes at 110°, and weighed. The counting samples measured 14 mm. in diameter and were mounted on 2 × 2.5 in. cardboard cards with scotch tape placed directly over the sample. These cards were placed in a holder which fixed the position of the sample relative to an Amperex #100 C Geiger–Muller tube. All counting samples had activities within the range from 600 to 6000 counts per minute, and in this range the response of the counter was linear. The thickness of the samples, which was of the order of 28 mg./cm.^{2}, varied as much as 2 mg./cm.^{2}, necessitating corrections for self-absorption of the β-particles. The self-absorption correction amounted to a 1.15% increase in the total counting rate of a sample for each milligram increase in sample weight.

In order to determine the tin activity in the aqueous phase a 5-ml. aliquot was warmed slightly to remove traces of benzene, and then treated with a drop of liquid bromine so that all the tin present would be in the stannic state. The excess bromine was removed by gentle heating, and 1 ml. of a solution of stannic chloride containing 10 mg. of tin per ml. was added. The tin was precipitated as stannic hydroxide which was then dissolved in a minimum of 6 *f* hydrochloric acid. One-half ml. of 6 *f* hydrochloric acid was added in excess, and the sample diluted to 5 ml. with water. The sample was heated to 90° and then 5 ml. of a saturated aqueous solution of phenylarsonic acid was added. After a digestion period of ten minutes at 90°, the sample was cooled, allowed to stand at room temperature for 15 minutes, and then mounted for counting. The percentage of tin in the precipitate formed under these conditions was equal to that calculated from the formula of stannic phenylarsonate within a probable error of 1%.

The tin activity in the benzene phase was determined by evaporating an aliquot to dryness, ashing the tetraphenyltin residue with concentrated sulfuric acid and 30% hydrogen peroxide, and finally precipitating the tin as stannic phenylarsonate which was then mounted for counting. The tin activity in the 36-mg. sample of tetraphenyltin was determined in the same way.

In all the experiments only about 80% of the total tin activity was recovered. It turned out that approximately 80% of the activity lost was adsorbed on the walls of the beaker in which the irradiated solid tetraphenyltin was originally dissolved. In one experiment[4] an irradiated sample of tetraphenyltin was dissolved directly in a separatory funnel prior to extraction with 3 *f* hydrochloric acid and accordingly a larger enrichment factor (3400) and a higher yield (61%) were obtained. These results suggest the formation of tin radiocolloids in benzene.

The total tin present in the aqueous phase was determined colorimetrically by observing the molybdenum-blue color produced by the action of stannous tin on a molybdate reagent.[5] After reduction to the stannous state the color produced by the tin present in an aliquot of the aqueous phase was compared, visually, with that produced in several standard solutions of tin.

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[2] R. R. Williams, *J. Phys. Colloid Chem.*, **52**, 603 (1948).

[3] E. G. Meyer, Ph.D. Thesis, University of New Mexico, 1950.

[4] On the basis of the β-activity of this irradiated sample, the γ-flux during the irradiation of this sample was the same as that during the irradiation of the samples used in experiments 3 and 4.

[5] F. D. Snell and C. T. Snell, "Colorimetric Methods of Analysis," 3rd. ed., D. Van Nostrand Co., New York, N. Y., 1949, p. 217.

COMMUNICATIONS TO THE EDITOR

THE SYNTHESIS OF BUTATRIENE[1]

Sir:

Butatriene has been synthesized in high yield by the action of powdered zinc on 1,4-dibromobutyne in purified diethylene glycol diethyl ether at 70°. The reaction product was collected as a low boiling liquid in a Dry Ice–acetone trap, or as a solid in a liquid nitrogen trap. The material polymerized upon being warmed to room temperature in the absence of air and with hydroquinone or other inhibitors present. The monomer showed negative tests with acetylenic hydrogen reagents such as alkaline mercuric iodide. With bromine in carbon tetrachloride it gave 1,2,3,4-tetrabromobutene-2 (80% yield), m.p. 68–69.5°, identical with an authentic sample prepared by bromination[2] of 1,4-dibromobutyne-2.

The butatriene exhibited the following physical properties: C/H ratio on two different samples, 92.29/7.71 and 91.82/8.18; mol. wt. (Dumas method at 250 mm. pressure), 52.6; mass spectrum (relative intensities): mass 52 (100), mass 51 (72), mass 50 (54); u.v. max. (95% ethanol), 241 mμ (20,300), apparent max. 310 mμ (250). In the infrared[3] (gas, 100 mm., 10 cm. cell), strong bands were shown at 2990, 1708, 1610, and 860 cm.$^{-1}$ (broad); medium bands at 2030, 1358, 1206, and 1065 cm.$^{-1}$ (broad).

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[1] Supported in part by the Research Corporation.

[2] A. Valette, *Ann. chim.*, [12] **3**, 644 (1948).

[3] For aiding in obtaining the data we wish to thank Dr. David Eggers, who is now working on a more detailed interpretation of the infrared spectrum.