

dioxide to a red purple color ( $pH$  6.5–7.0) and brought to a final volume of 20 cc. Inasmuch as the average activity of the gallium as received was 18 millicuries (mc.) this yielded a solution containing approximately 1 mc./cc. which was used in physiological studies. By plotting a decay curve of samples prepared as described above, we have obtained a curve, log activity *vs.* time, for 15 half lives of  $Ga^{72}$ . No significant deviation from the straight line of a half life of 14.3 hours was observed, indicating no significant amounts of long life contaminants.

No attempt has been made to isolate the citrate of  $Ga^{72}$  since this would entail lengthy procedures and provide opportunities for serious radiation exposures. The citrate solutions containing  $Ga^{72}$  as described above contained considerable quantities of other ions, (Cl,  $NO_3$ , Na). The citrate ions in this solution serve to stabilize the mixture, preventing the precipitation of gallium hydroxide at  $pH$  7–8, as would result if the citrate were not present. In a series of toxicological studies with stable gallium using a mixture containing the above mentioned ions plus citric acid or a solution containing only purified gallium citrate we have observed no difference in the effects or toxic properties when a comparison is made on an mg. Ga/kg. basis.

The penetrating  $\beta$ - $\gamma$  spectrum of  $Ga^{72}$  (2.5 mev.  $\gamma$ , 3.1 mev.  $\beta$ ) makes necessary the utmost effort for protection of personnel and especially for those carrying out chemical manipulations. The handling of the ampoules and all containers requires the use of suitable tongs. All glass apparatus, cylinders, beakers, etc., were placed in lead pots having a 25-mm. wall thickness. The chemical operations were carried on behind a wall of lead blocks 5 cm. in thickness. It has been found that when the above precautions were observed, a total body radiation of less than 20 milliroentgens (mr.) was received by personnel carrying out the chemical manipulations. Exposure to the hands was 20 to 40 mr., usually within a space of thirty minutes. Personnel injecting solutions of 0.5 mc./cc. activity in quantities up to 10 mc. per injection received less than 20 mr. total body radiation and less than 40 mr. on the hands, when using lead shielded syringes.<sup>8</sup> Therefore, it is recommended that personnel doing the chemical manipulations not carry out other studies or injections. No more than one unit of  $Ga^{72}$  (10–30 mc.) should be processed by one group in a single 24-hour period.

Studies using  $Ga^{72}$  must be carried out far removed from photographic storage and processing facilities since the hard gamma will penetrate the usual shielding, even that adequate for X-ray.

(6) Dudley, Bronson and Taylor, *Science*, **110**, 16 (1949).

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#### Certain 4,4'-Dinitrodiphenyl Sulfides and Sulfones

**Bis-(2-methoxy-4-nitrophenyl) Sulfide.**—An alcoholic solution of 8 g. (0.0345 mole) of 2-bromo-5-nitroanisole (m. p. 104°)<sup>1</sup> and the potassium ethyl xanthate prepared from 1.96 g. of potassium hydroxide and 2.66 g. of carbon disulfide was refluxed for a total of ninety-six hours. At intervals during this refluxing the mixture was filtered to remove the product. At one point an additional 2 g. of the nitroanisole was added making a total of 10 g. A total of 5.1 g. (70.2%) of bright yellow sulfide (recrystallized from acetic acid) was obtained, m. p. 204–205°.

*Anal.* Calcd. for  $C_{14}H_{12}O_6N_2S$ : S, 9.54. Found: S, 9.61.

**Bis-(2-methoxy-4-nitrophenyl) Sulfone.**—Oxidation of the above sulfide (3.0 g.) was performed with chromic anhydride in acetic acid at reflux. Pouring onto ice and recrystallization from glacial acetic acid afforded 2.4 g. (75%) of fine white crystals melting at 231–232°.

(1) Hanford and Adams, *This Journal*, **57**, 1592 (1935), found m. p. 104°.

*Anal.* Calcd. for  $C_{14}H_{12}O_8N_2S$ : S, 8.70. Found: S, 8.26, 8.39.

**2-Iodo-2'-methoxy-4,4'-dinitrodiphenyl Sulfide.**—An alcohol solution of 4.62 g. (0.025 mole) of 2-methoxy-4-nitrothiophenol (prepared from 2-bromo-5-nitroanisole by the methods of Price and Stacey)<sup>2</sup> 8.45 g. (0.025 mole) of 2-iodo-4-nitrobromobenzene and 2.6 g. (0.02 mole) of suspended potassium carbonate was heated under reflux for about five hours. After isolation in the usual way and recrystallization there was obtained 2.9 g. (33.6%) of light-brown microcrystalline powder melting at 191–192°.

*Anal.* Calcd. for  $C_{13}H_9O_5N_2IS$ : S, 7.42. Found: S, 6.67.

Oxidation of the above sulfide to 2-iodo-2'-methoxy-4,4'-dinitrodiphenyl sulfone was carried out in the customary manner with chromic anhydride in acetic acid. From 2.9 g. of the sulfide there was obtained 3.0 g. (96.5%) of recrystallized light pink colored sulfone melting at 270–271°.

*Anal.* Calcd. for  $C_{13}H_9O_7N_2IS$ : S, 6.89. Found: S, 6.82.

(2) Price and Stacey, *ibid.*, **68**, 498 (1946).

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#### Triphenyl-*o*-methoxymethylphenyltin

Incidental to studies concerning water-soluble organotin compounds, triphenyl-*o*-methoxymethylphenyltin was prepared from the Grignard reagent which was obtained (1) directly from the organic halide, and (2) by means of a halogen-metal interconversion reaction.

**Triphenyl-*o*-methoxymethylphenyltin.**—To *o*-methoxymethylphenylmagnesium bromide prepared by refluxing for six hours 1.95 g. (0.080 gram atom) of magnesium and 15.1 g. (0.075 mole) of *o*-bromobenzyl methyl ether in 150 cc. of ether and 60 cc. of benzene, was added 20.0 g. (0.0518 mole) of triphenyltin chloride. From the mixture, after stirring for several hours, followed by hydrolysis with aqueous ammonium chloride, there was obtained, after two crystallizations from benzene-ethanol, 8.5 g. or a 35% yield of triphenyl-*o*-methoxymethylphenyltin melting at 93–95°. Another recrystallization from the same solvent, subsequent to vacuum drying, raised the melting point to 94.5–95.5°.

*Anal.* Calcd. for  $C_{26}H_{24}OSn$ : Sn, 25.22. Found: Sn, 25.88 and 25.90.

Since direct formation of the Grignard reagent was slow, it was also prepared by means of the halogen-metal interconversion reaction. To 15.1 g. (0.0752 mole) of *o*-bromobenzyl methyl ether in 50 cc. of ether, was added 0.0752 mole of *n*-butyllithium in 145 cc. of ether. After the mixture was stirred for twenty-five minutes, 0.085 mole of anhydrous magnesium bromide in 75 cc. of ether was added. By the procedure outlined above, after the addition of 19.0 g. (0.0493 mole) of triphenyltin chloride, there was obtained 8.8 g. or a 38% yield of triphenyl-*o*-methoxymethylphenyltin melting at 94–95.5°.

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#### *m*-Phenyl-phenylacetic Acid

*m*-Acetylphenyl<sup>1</sup> (3.2 g.) with 1 g. of sulfur and 10 ml. of morpholine was refluxed for seven hours. The mixture was poured into 30 ml. of methanol and water was added until the product precipitated as a gummy

(1) Huber, Renell, Rossow and Mewfy, *This Journal*, **66**, 1109 (1946).