Some *n*-Butyl and Phenyl Phosphonium Salts

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DURING THE COURSE of a study of the infrared spectra of phosphonium structures (*3*), a number of unreported *n*-butyl and phenyl phosphonium salts (Table I) were prepared. These salts were obtained in acceptable yields by simple quaternization (*2*) of the appropriate phosphine by an alkyl halide in refluxing benzene or in the absence of solvent. In two instances, the halides obtained from the reaction mixture could not be crystallized satisfactorily; conversion to the corresponding iodide led to the formation of crystalline material.

EXPERIMENTAL

The starting materials di-(*n*-butyl)phenylphosphine and *n*-butyldiphenylphosphine, used in the preparation of compounds I and V, VI, respectively, were prepared by the method of Davies and Jones (*1*). Compounds II-IV were prepared from tri-*n*-butylphosphine and compounds VII and VIII from triphenylphosphine. Compound VII, (*p*-methylbenzyl)triphenylphosphonium chloride, was prepared by the following procedure (other compounds listed were prepared by similar procedures with the exceptions noted in Table I).

A solution of 52.4 grams (0.2 mole) of triphenylphosphine and 28.0 gram (0.2) mole of *p*-methylbenzyl chloride in 100 ml. of anhydrous benzene was refluxed for five hours; the product crystallized on cooling to room temperature. Filtration gave 28.0 grams of crude product; an additional 23.0 grams of product was obtained on concentration of the mother liquor. The product washed with ether and recrystallized from a mixture of chloroform and hexane to a constant melting point of 252–254°.

Compounds II and III were prepared by the reaction of β-chlorohydrin and phenacyl bromide with tri-*n*-butylphosphine. In the former case, a viscous oil which could not be crystallized separated from the reaction mixture; in the latter case, the bromide separated as a crystalline solid which was somewhat hygroscopic. Both of these materials were converted to the corresponding iodide by the addition of aqueous potassium iodide to a solution of the salt in boiling water; the iodides could be recrystallized satisfactorily from water and were not hygroscopic.

Analyses were performed by Galbraith Microanalytical Laboratories. All melting points were uncorrected.

LITERATURE CITED

(1) Davies, W.C., Jones, W.J., *J. Chem. Soc.*, **1929**, p. 33.
(2) Kosolapoff, G.M., "Organophosphorus Compounds", p. 78, John Wiley, New York, 1950.
(3) Witschard, G., Griffiin, C.E., *Spectrochim. Acta* **19**, 1905 (1963).

RECEIVED for review May 15, 1963. Accepted December 26, 1963. This work was supported in part by the National Science Foundation and by the Directorate of Chemical Sciences, Air Force Office of Scientific Research under Grant No. AF-AFOSR-62-48.

Table I. Phosphonium Salts $[R_1R_2R_3R_4P]^+ X^-$

	R_1	R_2	R_3	R_4	X	Reaction Time (hr.)	Yield	M.P.	Recrystallization Solvent
I	C_6H_5	n-C_4H_9	n-C_4H_9	n-C_4H_9	Br	3.0	53	162.5–163.0	C_6H_6-C_6H_{14}
II	$HOCH_2CH_2$	n-C_4H_9	n-C_4H_9	n-C_4H_9	I	0.5	20[a]	113–116	H_2O
III	$C_6H_5COCH_2$	n-C_4H_9	n-C_4H_9	n-C_4H_9	I	1.0	34	135.0–137.5	H_2O
IV	$C_6H_5CH_2$	n-C_4H_9	n-C_4H_9	n-C_4H_9	Cl	2.0	40	160–162	...[b]
V	C_6H_5	C_6H_5	n-C_4H_9	n-C_4H_9	Br	3.0	53	162–163	C_6H_6-C_6H_{14}
VI	C_6H_5	C_6H_5	CH_3	n-C_4H_9	I	0.5	100	151.5–152.5	C_6H_6
VII	p-$CH_3C_6H_4CH_2$	C_6H_5	C_6H_5	C_6H_5	Cl	5.0	64	252–254	$CHCl_3$-C_6H_{14}
VIII	p-$ClC_6H_4CH_2$	C_6H_5	C_6H_5	C_6H_5	Cl	2.0	55	279–280	$(CH_3)_2CO$

	Analysis					
	Calcd.			Found		
	C	H	P	C	H	P
I	60.16	8.91	...	60.26	8.86	...
II	44.92	8.62	8.28	44.78	8.48	8.52
III	53.57	7.64	6.91	53.47	7.68	6.84
IV	69.58	10.45	...	69.66	10.56	...
V	63.33	7.44	8.17	63.61	7.30	8.00
VI	53.14	5.77	8.06	52.97	5.88	8.29
VII	77.51	6.00	7.69	77.38	5.91	7.60
VIII	70.93	5.00	...	70.71	5.09	...

[a] Reaction carried out in absence of solvent. [b] Compound obtained in analytical purity by washing with ether without recrystallization.