LETTERS TO THE EDITOR

REACTION OF BIS(α -HYDROXYETHYL)PHENYLPHOSPHINE WITH PHENYLARSINE OXIDE AND DIMETHYL ESTER OF PHENYLARSENOUS ACID

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Replacement of the hydroalkyl group in phosphines by an arsenic-containing radical has not been described in the literature. We found that in the reaction between bis(α -hydroxy-ethyl)phenylphosphine and phenylarsine oxide or dimethyl ester of phenylarsenous acid, a crystalline product is obtained, with an elemental composition corresponding to that of phospharsobenzene (PhP=AsPh). From the values of the molecular weight and the chemical shift of the P atom signal in the ³¹P NMR spectrum, the structure of hexaphenyl-1,3,5-triphospha-2,4,6-triarsacyclohexane (I) was ascribed to the product

$$\begin{array}{c} \text{CH}_{3} \\ \text{PhP(CH-OH)}_{2} - \boxed{\begin{array}{c} + \text{(CH}_{5}\text{O})_{2}\text{AsPh} \\ + \text{PhAs=O} \end{array}} \xrightarrow{Ph-As} \begin{array}{c} \text{As-Ph} \\ \text{Ph-P} \\ \text{Ph-P} \\ \text{Ph-Ph} \\ \text{Ph} \\ \text{(I)} \end{array}$$

A 3.57-g portion (0.02 mole) of phenylarsine oxide was added in an argon atmosphere to 4.21 g (0.02 mole) of bis(α -hydroethyl)phenylphosphine, A precipitate separated after standing for 12 h. The mixture was diluted with C_6H_6 , the precipitate was filtered, washed with CH_3CN and CH_3OH and crystallized from C_5H_5N . The yield of (I) was 3.13 g (57%), mp 190°C, δ^{3} P — 21 ppm (PhCN, high fields). Found 55.18; H 4.00; P 11.63; As 28.05%. $C_{36}H_{30}P_{3}As_{3}$. Calculated: C 55.38; H 3.84; P 11.92; As 28.84%. The reaction with the dimethyl ester of phenylarsenous acid was carried out in a similar way and gave (I) in a yield of 35%. The mol. wt. of (I), determined cryoscopically in naphthalene, is 848 (a 0.0566-g sample) and 890 (a 0.1151-g sample), theor. 780.

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