

The reaction of red phosphorus with KOH and hydrazine hydrate in aqueous HMPTA under 15 gauge atmospheres acetylene in an autoclave at 105-115°C for 5 h led to trivinylphosphine in 10% yield, which was identified by chromato-mass spectrometry. Trivinylphosphine could not be isolated by distillation. The mass spectrum of trivinylphosphine,  $m/z$  (relative intensity, %): 112 (32), 98 (28), 97 (25), 86 (15), 84 (12), 83 (19), 72 (100), 58 (91), 57 (40), 56 (63). To our knowledge, this is the first example of the synthesis of trivinylphosphine by the addition of phosphide ions to acetylene.

#### LITERATURE CITED

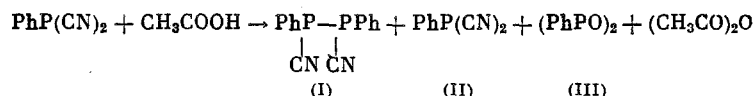
1. É. E. Nifant'ev, The Chemistry of Organophosphorus Compounds [in Russian], Izd. Mosk. Gos. Univ., Moscow (1971), p. 347.
2. G. M. Bogolyubov and A. A. Petrov, Dokl. Akad. Nauk SSSR, 173, 1076 (1967).
3. G. M. Bogolyubov and A. A. Petrov, Zh. Obshch. Khim., 36, 1505 (1966).

#### SYNTHESIS OF DIPHENYLDICYNODIPHOSPHINE

A. N. Pudovik, V. N. Nazmutdinova, L. P. Chirkova,  
Yu. Ya. Efremov, and R. Z. Musin

UDC 542.91:547.1'118

We have found that the reaction of  $\text{PhP}(\text{CN})_2$  with acetic acid at 20°C in the presence of catalytic amounts of sodium acetate leads to the formation of diphenyldicyanodiphosphine (I). The  $^{31}\text{P}$  NMR spectrum has two signals with  $\delta_{\text{P}} = -47$  ppm, which indicates the formation of two diastereomers. In addition, acetic anhydride was isolated in 87% yield. The  $^{31}\text{P}$  NMR spectrum of the reaction mixture also has signals for  $\text{PhP}(\text{CN})_2$  (II) ( $\delta_{\text{P}} = -76$  ppm) and a compound with a tetracoordinated phosphorus atom ( $\delta_{\text{P}} = 2$  ppm), which corresponds, according to mass spectrometry, to metaphenylphosphonate (III):  $\text{M}^+$ ,  $m/z_{\text{obs}}$  420.0063,  $m/z_{\text{calc}}$  420.00816 ( $\text{C}_{18}\text{H}_{15}\text{O}_6\text{P}_3$ ). The reaction proceeds through the following scheme:



The structure of (I) was supported by hydrolytic cleavage, which gives a mixture of phenylphosphonic ( $\delta_{\text{P}} = -18$  ppm) and phenylphosphorous acids ( $\delta_{\text{P}} = -20$  ppm,  $J_{\text{HP}} = 580$  Hz).

Diphenyldicyanodiphosphine (I) was obtained in 84% yield, mp 104-106°C. Mass spectrum,  $m/z$  (relative intensity, %): 269 (7.7),  $\text{M}^+$  268 (60), 135 (9.6),  $[\text{M} - \text{C}_6\text{H}_5\text{PC}]^+$  134 (100), 110 (3.8),  $[\text{C}_6\text{H}_5\text{P}]^+$  108 (15),  $[\text{C}_6\text{H}_4\text{P}]^+$  107 (34).