CYCLIZATION OF DIPHENYLBIS(β-HYDROXYETHYL)PHOSPHONIUM CHLORIDE

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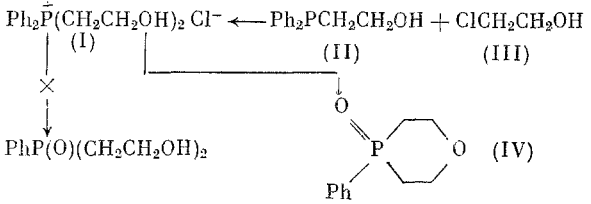
We have found that the acid cleavage of diphenylbis(β-hydroxyethyl)phosphonium chloride (I), obtained by the reaction of diphenyl-β-hydroxyethylphosphine (II) with ethylenechlorohydrin (III), gave its dehydration product, namely, 4-phenyl-4-oxo-1,4-oxaphosphorinane (IV) instead of the expected bis(β-hydroxyethyl)phenylphosphine.



Diphenylbis(β-hydroxyethyl)phosphonium Chloride (I). A mixture of 5.0 g (20 mmoles) (II) [1] and 6.4 g (80 mmoles) (III) was heated for 3 h at 140°C and evaporated in vacuum to give 6.2 g (80%) (I), mp 149-150°C (from 2-butanone–ethanol). Found: C 61.0; H 6.8; Cl 10.9%. Calculated for $C_{16}H_{20}ClO_2P$: C 61.8; H 6.5; Cl 11.40%. ^{31}P NMR spectrum in ethanol (δ, ppm): 24.8. IR spectrum in vaseline mull (ν, cm^{-1}): 3300 (OH).

4-Phenyl-4-oxo-1,4-oxaphosphorinane (IV). A sample of 3.2 g (80 mmoles) 10% aq. NaOH was added dropwise with stirring to a solution of 15.4 g (50 mmoles) (I) in 20 ml water at 20°C. The mixture was heated for 1.5 h at 70°C and extracted with chloroform. The extract was washed with 1:10 H_2SO_4–H_2O and aqueous $NaHCO_3$ followed by drying over Na_2SO_4 and evaporation in vacuum to give 7.4 g (75%) (IV), mp 119-120°C (from ethyl acetate) [2]. Found: C 61.1; H 6.9; P 15.8%. Calculated for $C_{10}H_{13}O_2P$: C 61.2; H 6.7; P 15.8%. ^{31}P NMR spectrum in ethanol (δ, ppm): 22.9. ^{13}C NMR spectrum in $C_2Br_2F_4$ (δ, ppm, J, Hz): 30.0 (CH_2P), $^2J_{PC}$ = 32.4, 65.1 (CH_2O), $^3J_{PCC}$ = 2.5. IR spectrum in CCl_4 (ν, cm^{-1}): 1220 (P=O).

LITERATURE CITED

1. E. N. Tsvetkov, N. A. Bondarenko, I. G. Malakhova, and M. I. Kabachnik, Zh. Obshch. Khim., 55, 11 (1985).
2. D. L. Collins, L. E. Rowley, and J. M. Swan, Austr. J. Chem., 27, 841 (1974).

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