BIS-α-HYDROXYCYCLOHEXYLPHOSPHINIC ACID CHLORIDE

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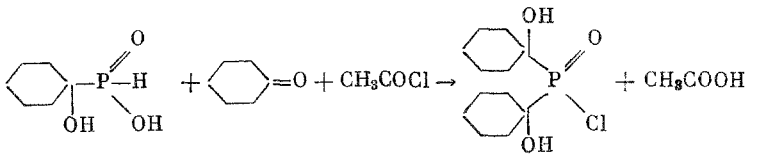
Bis-α-hydroxyalkylphosphinic acid chlorides have not been described in the literature. We have shown that the reaction of α-hydroxycyclohexylphosphorous acid (I) with cyclohexane and acetyl chloride gives bis-α-hydroxycyclohexylphosphinic acid chloride (II).



(I) (II)

Acid (I) was prepared from cyclohexanone and H_3PO_2 by analogy with the work of Ville [1] and Marie [2] in 64% yield, mp 96-98°C (from benzene). Found, %: C 44.0, H 8.0, P 18.4. $C_6H_{13}O_3P$. Calculated, %: C 43.9, H 8.0, P 18.9. ^{31}P NMR spectrum: δ 40.0 ppm, J_{PH} = 552.3 Hz (in acetone).

A sample of 1.1 g (0.014 mole) acetyl chloride was added to a suspension of 1.64 g (0.01 mole) (I) in 10 ml cyclohexanone and 10 ml absolute benzene and stirred at 20-25°C until the acid fully dissolved over 10-15 min. The crystalline precipitate formed upon standing overnight in a refrigerator was filtered off and washed with ether to give 2.25 g (80%) (II), mp 103-105°C (from acetone). Found, %: C 51.4, H 7.9, P 11.2, Cl 12.7. $C_{12}H_{22}ClO_3P$. Calculated, %: C 51.3, H 7.9, P 11.0, Cl, 12.7. IR spectrum in Vaseline mull (ν, cm^{-1}): 550 (P—Cl), 1155 (P O), 3410, 3335 (C—OH). ^{31}P NMR spectrum: 78.1 ppm (in $EtCO_2H$).

Heating acid chloride (II) with 10% aqeuous KOH at reflux gives bis-α-hydroxycyclohexylphosphinic acid, $[C_6H_{10}(OH)]_2P(O)OH$ in 90% yield, mp 198-200°C (from ethanol). Found, %: C 55.3, H 8.9, P 12. $C_{12}H_{23}O_4P$. Calculated, %: C 55.0, H 8.8, P 11.8. IR spectrum in vaseline mull (ν, cm^{-1}): 1090 (P=O), 2600-1660 (PO_2H), 3470, 3335 (C—OH). ^{31}P NMR spectrum: δ 44.7 ppm (in DMSO).

LITERATURE CITED

1. M. J. Ville, Comptes Rendus Acad. Sci., 109, 71 (1889).
2. M. C. Marie, Comptes Rendus Acad. Sci., 133, 219 (1901).

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