REACTION OF 3,5-BIS(4-METHOXY-PHENYL)-1,2-OXATHIOLANE-2-OXIDE WITH PHOSPHORUS TRICHLORIDE

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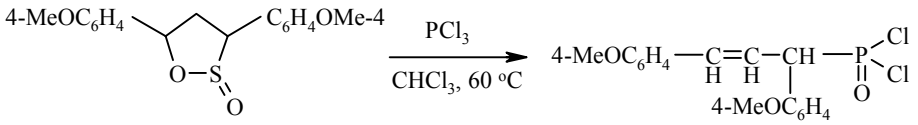
While studying the reactivity of 3,5-diaryl-1,2-oxathiolane-2-oxides (γ-sultines), we have shown for the first time that heating under reflux 3,5-bis(4-methoxyphenyl)-1,2-oxathiolane-2-oxide (**1**) [1] with excess of phosphorus trichloride (1:4) in chloroform for 8 h leads to formation of 1,3-bis(4-methoxyphenyl)allylphosphonic acid dichloride (**2**) in 80% yield.



1 **2**

The reaction was carried out by adding a solution of phosphorus trichloride (0.38 g, 2.7 mmol) in dry chloroform to a solution of sultine **1** (0.22 g, 0.68 mmol) in chloroform and refluxing for 8 h. The mixture was cooled, poured into water, extracted with chloroform, washed with a saturated solution of sodium bicarbonate and then with water, dried with calcium chloride, and evaporated. Obtained: 0.2 g (80%) of compound **2** as a rose-red oil, which was recrystallized from a chloroform–hexane mixture; 0.17 g compound **2** was isolated as light pink crystals with mp 147°C. IR spectrum (vaseline oil), ν, cm^{-1}: 1617 (C=O), 1255 (P=O). ^{1}H NMR spectrum (400 MHz, $CDCl_3$, 30°C), δ, ppm, *J* (Hz): 3.81, 3.82 (6H, 2s, CH_3O); 4.46 (1H, dd, $^3J_{HH}$ = 9.4; $^2J_{HP}$ = 18.2, CHP); 6.39 (1H, dt, $^3J_{HH}$ = 15.6; $^3J_{HH}$= 9.4; $^3J_{HP}$ = 9.4; CH=); 6.72 (1H, dd, $^3J_{HH}$ = 15.6; $^4J_{HP}$ = 7.6; ArCH=); 6.88, 6.96 (4H, 2d, $^3J_{HH}$ = 8.8; CHAr); 7.37 (2H, d, $^3J_{HH}$ = 8.8, CHAr); 7.43 (2H, dd, $^3J_{HH}$ = 8.8, $^4J_{HP}$ = 3.4, CHAr). ^{13}C NMR spectrum (100 MHz, $CDCl_3$, 30°C), δ, ppm *J*(Hz); 55.42 (CH_3O), 63.20 (d, $^1J_{CP}$ = 89, CHP); 114.22 (CHAr); 114.73 (d, $^4J_{CP}$ = 3, CHAr); 117.93 (d, $^2J_{CP}$ = 9.8, CH=); 124.43 (d, $^2J_{CP}$ = 7.5; CAr); 128.20 (d, $^5J_{CP}$ = 3, CHAr), 128.58 (d, $^4J_{CP}$ = 4.5, CAr); 130.63 (d, $^3J_{CP}$ = 9, CHAr); 137.26 (d, $^3J_{CP}$ = 19.5, CH=); 160.03, 160.08 (CAr). ^{31}P NMR spectrum (161.9 MHz, $CDCl_3$, 30°C), δ, ppm: 47.1. Found, %: C 54.93; H 4.54. $C_{17}H_{17}Cl_2O_3P$. Calculated, %: C 55.01; H 4.62.

REFERENCES

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