

Reactions of 2-(5-methyl-2-phenyl-2*H*-1,2,3-diazaphosphol-4-yl)-4*H*-1,3,2-benzodioxaphosphinin-4-one with chloral and hexafluoroacetone

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*2-Chlorocarbonylphenyl 2,2-dichlorovinyl (5-methyl-2-phenyl-2*H*-1,2,3-diazaphosphol-4-yl)phosphonate 2*. MS, *m/z*: 488 (C₁₈H₁₃Cl₃N₂O₄P₂ 488) [M]⁺, 453 [M – Cl], 418 [M – 2 Cl], 406 [M – CCl₂], 377 [M – OCH=CCl₂], 358 [M – OCH=CCl₂ – Cl], 330 [M – OCH=CCl₂ – C(O)Cl], 176 [C₉H₈N₂P + H]⁺, 92 [C₆H₅O]. IR, ν/cm^{-1} : 3107, 3076, 1774, 1708, 1599, 1496, 1478, 1450, 1384, 1342, 1282, 1214, 1195, 1151, 1133, 1073, 1025, 977, 926, 848, 815, 754, 737, 690, 661, 646, 613, 513. ¹H NMR, δ : 8.15 [br.d, H⁶, 1H, ³J_{H⁷CCH⁶ 8.0, *X*-part of *ABMX*-system], 7.77 [br.d, H^{7b}, 2H, ³J_{H^{8b}CCH^{7b} 7.9-8.0 Hz, *XX'*-part of *ABB'XX'*-system], 7.71 [br.dd, H⁸, 1H, ³J_{H⁹CCH⁸ 8.4 Hz, ³J_{H⁷CCH⁸ 7.7 Hz, *B*-part of *ABMX*-system], 7.62 [br.d, H⁹, 1H, ³J_{H⁸CCH⁹ 8.3 Hz, *A*-part *ABMX*-system], 7.44 [br.dd, H^{8b}, 2H, ³J_{H^{9b}CCH^{8b} 8.1 Hz, ³J_{H^{7b}CCH^{8b} 7.9 Hz], 7.36 [br.dd, H⁷, 1H, ³J_{H⁸CCH⁷ 7.6 Hz, ³J_{H⁶CCH⁷ 8.0 Hz, *M*-part *ABMX*-system], 7.35 [br.t, H^{9b}, 1H, ³J_{H^{8b}CCH^{9b} 8.1 Hz, *A*-part of *ABB'XX'*-system], 2.75 [s, H^{10b}, 3H]. ¹³C NMR, δ_{C} : 148.34 [dddd (d), C^{9a}, ³J_{HC⁸CC^{9a} 10.7 Hz, ³J_{HC⁶CC^{9a} 9.0 Hz, ²J_{POC^{9a} 8.3 Hz, ²J_{HC⁹C^{9a} 3.2 Hz, ⁴J_{HC⁷CCC^{9a} 1.7 Hz]; 126.11 [br.ddd (d), C^{5a}, ³J_{HC⁷CC^{5a} 8.0 Hz, ³J_{POCC^{5a} 5.7 Hz, ³J_{HC⁹CC^{5a} 5.7-6.0 Hz]; 134.34 [dddd (s), C⁶, ¹J_{HC⁶ 165.5 Hz, ³J_{HC⁸CC⁶ 8.5 Hz, ²J_{HC⁷C⁶ 2.2 Hz]; 126.00 [dddd (d), C⁷, ¹J_{HC⁷ 165.3 Hz, ³J_{HC⁹CC⁷ 7.8 Hz, ⁵J_{POCCCC⁷ 1.1 Hz]; 136.11 [ddd (d), C⁸, ¹J_{HC⁸ 164.0 Hz, ³J_{HC⁶CC⁸ 8.9 Hz, ⁴J_{POCCC⁸ 1.4 Hz]; 122.25 [ddd (d), C⁹, ¹J_{HC⁹ 159.4 Hz, ³J_{HC⁷CC⁹ 8.0 Hz, ³J_{POCC⁹ 3.3 Hz]; 164.02 [br.dd (s), C⁵, ³J_{HC⁶CC⁵ 6.7 Hz, ⁴J_{HCCCC⁵ 1.2 Hz]; 132.88 [dd (d), C⁴, ¹J_{HC⁴ 201.1 Hz, ²J_{POC⁴ 4.5 Hz]; 114.65 [dd (d), C¹⁰, ²J_{HCC¹⁰ 13.8 Hz, ³J_{POCC¹⁰ 13.8 Hz]; 134.69 [ddq (dd), C^{4b}, ¹J_{P^{II}C^{4b} 201.8 Hz, ¹J_{P^{IV}C^{4b} 48.7 Hz, ³J_{HC^{10b}CC^{4b} 2.8 Hz]; 158.68 [ddq (dd), C^{5b}, ²J_{P^{II}CC^{5b} 9.7 Hz, ²J_{P^{IV}CC^{5b} 4.8 Hz, ²J_{HC^{10b}C^{5b} 4.7 Hz]; 142.70 [br.d.m (d), C^{6b}, ²J_{P^{II}NC^{6b} 11.1 Hz, ³J_{HC^{8b}CC^{6b} 8.5 Hz]; 120.72 [ddm (d), C^{7b}, ¹J_{HC^{7b} 162.2 Hz, ³J_{P^{II}NCC^{7b} 9.7 Hz, ³J_{HC^{7b}CC^{7b} 6.0-8.0 Hz, ³J_{HC^{9b}CC^{7b} 6.0-8.0 Hz]; 129.63 [dd (s), C^{8b}, ¹J_{HC^{8b} 162.1 Hz, ³J_{HC^{8b}CC^{8b} 8.1 Hz]; 128.27 [br.dtd (d), C^{9b}, ¹J_{HC^{9b} 162.2 Hz, ³J_{HC^{7b}CC^{9b} 7.5 Hz, ⁵J_{P^{II}NCCCC^{9b} 1.1 Hz]; [15.56 q (s), C^{10b}, ¹J_{HC^{10b} 129.7]. ³¹P NMR, δ_{P} : 251.9 (br.d, P^{II}, ²J_{P^{IV}CP^{II} 84.5 Hz), 11.3 (d, P^{IV}, ²J_{P^{II}CP^{IV} 84.5 Hz).}}