Tetrahedron Letters 50 (2009) 624–626

Contents lists available at ScienceDirect

Tetrahedron Letters

journal homepage: www.elsevier.com/locate/tetlet

Photoinduced hydrophosphinylation of alkenes with diphenylphosphine oxide

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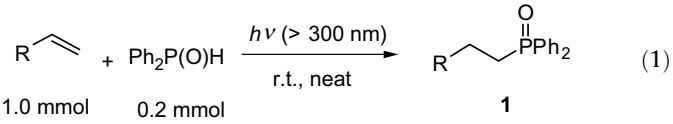
ARTICLE INFO

Article history:
Received 22 September 2008
Revised 11 November 2008
Accepted 17 November 2008
Available online 27 November 2008

ABSTRACT

Photoinduced hydrophosphinylation of alkenes with diphenylphosphine oxide took place regioselectively affording the corresponding phosphine oxide in good yields. This hydrophosphinylation is of simple operation and widely tolerant to a variety of the functionalities.

Because of the wide applications of organophosphorus compounds[1] in coordination chemistry, agrochemicals, and polymer science, the development of highly selective and environment-friendly synthetic methods of phosphorus compounds has been of current interest. In this Letter, we wish to report a solvent-free, atom-economical, and highly efficient synthesis of organophosphorus compounds by the photoinduced hydrophosphinylation of alkenes with diphenylphosphine oxide. Thus, for example, the hydrophosphinylation of 1-octene with $Ph_2P(O)H$ can be conducted at room temperature upon irradiating a mixture of 1-octene and $Ph_2P(O)H$ with a xenon lamp through Pyrex (Eq. 1):



(1)

The addition of P(X)–H bonds (X = lone pair, O, or S) to carbon–carbon unsaturated bonds is one of the most straightforward methods for the synthesis of organophosphorus compounds. This hydrophosphinylation is known as 'Pudovik reaction'.[2] The Pudovik reaction can proceed via a radical and/or ionic mechanism. In the cases of carbon–carbon unsaturated compounds activated by an electron-withdrawing group, the reaction proceeds via ionic mechanism.[3] When inactive olefins and organophosphorus compounds such as $R_2P(O)$–H are employed, this addition prefers a radical pathway.[2b] Recently, the hydrophosphinylations of alkenes with $R_2P(O)$–H using radical initiator[4–6] or microwave[7] were reported. Photoirradiation[8,9] is thought to be one of the simplest methods; however, such a photoinduced hydrophosphinylation of alkenes with diphenylphosphine oxide has not been investigated in detail.[10,5b]

When a mixture of 1-octene (1.0 mmol) and diphenylphosphine oxide (0.2 mmol) in a sealed NMR tube (φ = 4 mm, Pyrex) under N_2 atmosphere was irradiated with a xenon lamp at room temperature, a regioselective hydrophosphinylation took place to give diphenyloctylphosphine oxide (**1a**) in excellent yield (Table 1, entry 1).[11] This reaction also proceeded efficiently in a larger scale (entry 2). The hydrophosphinylation did not take place without photoirradiation (entry 3). When the hydrophosphinylation reactions were performed using various olefins bearing chloro, cyano, hydroxyl, phenyl, phenoxy, or amino groups, the corresponding products **1b–g** were obtained in good to excellent yields without affecting these functional groups (entries 4–9). Moreover, internal alkenes were also found to be good substrates for the reaction (entries 10 and 11). 3,4-Dihydro-2*H*-pyrane reacted with diphenylphosphine oxide regioselectively affording the corresponding hydrophosphinylation product **1i**. The hydrophosphinylation also took place successfully even with a bulky vinyltrimethylsilane (entry 12).

A similar hydrophosphinylation, however, proceeded slowly in solvents such as THF, benzene, chloroform, and ethanol, affording the adducts in low yields. Interestingly, we found that the addition of pyridine to the mixture can accelerate the reaction (Table 2). Thus, when a mixture of 1-octene (0.1 mmol), diphenyphosphine oxide (0.3 mmol), and pyridine (0.3 mmol) in chloroform-*d* (0.6 mL) was irradiated, **1a** was obtained in 91% yield (entry 1). Noted that the yield of **1f** and **1j** from allyl phenyl ether and vinylsilane, respectively, were dramatically improved compared to those in the absence of pyridine (entries 2 and 4).

To clarify the reaction mechanism, a mixture of 1,6-heptadiene and diphenylphosphine oxide was irradiated under solvent-free condition (condition A) or in $CDCl_3$ in the presence of pyridine as an additive (condition B) (Scheme 1). Under both conditions, the reaction proceeded via cyclization, affording the five-membered ring compound[5a] in good yields. These result indicates that this hydrophosphinylation in the presence or absence of pyridine proceeds via a radical mechanism.

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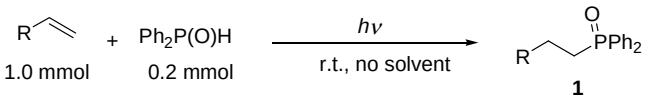
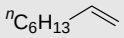
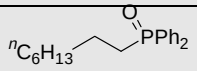
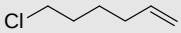
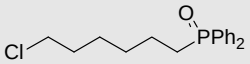
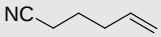
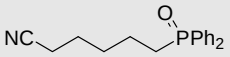
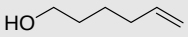
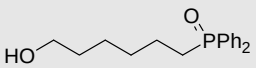
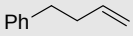
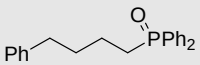
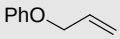
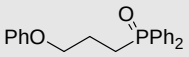
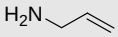
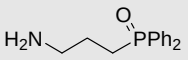
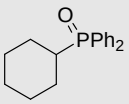
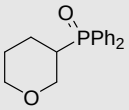
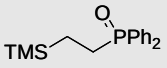
doi:10.1016/j.tetlet.2008.11.079

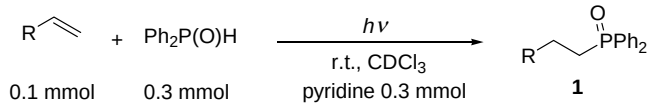
Table 1
Photoinduced hydrophosphinylation of alkenes with $Ph_2P(O)H$

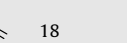
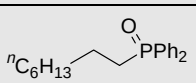
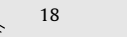
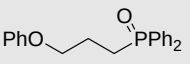
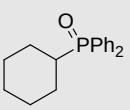
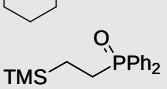


Entry	Alkene	Time (h)	Product		Yield[a] (%)
1		18		**1a**[12]	99
2[b]		18			93
3[c]		18			0
4		16		**1b**[13]	99
5		15		**1c**[14]	96
6		18		**1d**[15]	92
7		16		**1e**[16]	90
8		18		**1f**[17]	70
9		16		**1g**[18]	57 (81)
10		16		**1h**[19]	(71)
11		45		**1i**[20]	99
12		27		**1j**[21]	48 (58)

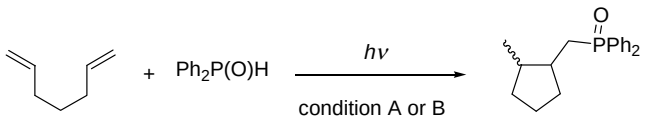
[a] Isolated yield (^1H NMR yield).
[b] Reactions were performed using alkene 7.0 mmol, $Ph_2P(O)H$ 1.4 mmol.
[c] Without photoirradiation.

Table 2
Photoinduced hydrophosphinylation of alkenes with $Ph_2P(O)H$ in the presence of pyridine



Entry	Alkene	Time (h)	Product		Yield[a] (%)
1		18		**1a**	91
2		18		**1f**	93
3		35		**1h**	(66)
4		12		**1j**	(74)

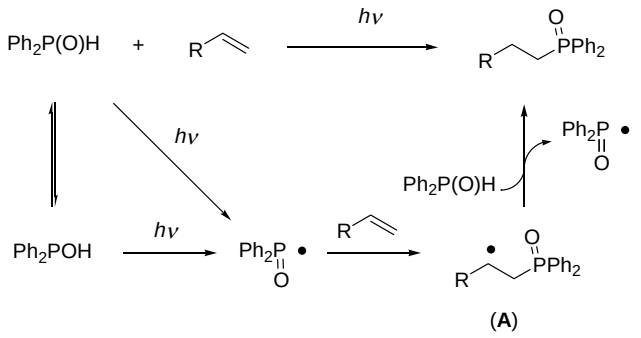
[a] Isolated yield (^1H NMR yield).



Scheme 1. Condition A: 1,6-heptadiene (0.1 mmol), $Ph_2P(O)H$ (0.2 mmol), C_6D_6 (0.6 mL); B: 1,6-heptadiene (0.1 mmol), $Ph_2P(O)H$ (0.3 mmol), pyridine (0.3 mmol), $CDCl_3$ (0.6 mL).

A possible pathway for this hydrophosphinylation is shown in Scheme 2. Upon photoirradiation, diphenylphosphine oxide generates a phosphinoyl radical. A phosphinoyl radical selectively attacks the terminal carbon of an alkene, affording the corresponding radical intermediate (**A**). This intermediate (**A**) abstracts a hydrogen of diphenylphosphine oxide, affording the hydrophosphinylation product with generation of another phosphinoyl radical.

In summary, we have developed a photoinduced hydrophosphinylation of alkenes with diphenylphosphine oxide, which is widely tolerant to a variety of functionalities, giving high yields of the corresponding adducts regioselectively.



Scheme 2. A possible pathway of photoinduced hydrophosphinylation.

Acknowledgments

This work is supported by Grant-in-Aid for Scientific Research on Priority Areas (Area 444, No. 19020061) and Scientific Research (B, 19350095), from the Ministry of Education, Culture, Sports, Science and Technology, Japan.

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10. For the transition metal-catalyzed hydrophosphinylation and hydrophosphination of alkene, see: (a) Shulyupin, M. O.; Kazankova, M. A.; Beletskaya, I. P. *Org. Lett.* **2002**, *4*, 761; (b) Allen, A., Jr.; Ma, L.; Lin, W. *Tetrahedron Lett.* **2002**, *43*, 3707; (c) Takaki, K.; Koshoji, G.; Komeyama, K.; Takeda, M.; Shishido, T.; Kitani, A.; Takehira, K. *J. Org. Chem.* **2003**, *68*, 6554; (d) Dobashi, N.; Fuse, K.; Hoshino, T.; Kanada, J.; Kashiwabara, T.; Kobata, C.; Nune, S. K.; Tanaka, M. *Tetrahedron Lett.* **2007**, *48*, 4669.
11. When $^{t}Bu_2P(O)H$ was also employed as a dialkylphosphine oxide, the corresponding hydrophosphinylation product was obtained in 37% yield by the irradiation through quartz tube with high pressure mercury lamp ($h\nu$ >200 nm).
12. For the spectral and analytical data of diphenyloctylphosphine oxide (**1a**): white solid; mp 48–50 °C; IR (KBr) 2928, 2855, 1437, 1182, 1121, 718, 694, 548, 511 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.85 (t, J = 6.9 Hz, 3H), 1.12–1.44 (m, 10H) 1.54–1.68 (m, 2H) 2.19–2.30 (m, 2H), 7.40–7.54 (m, 6H), 7.70–7.85 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 14.0, 21.4, 21.6, 22.6, 29.0, 29.7 (d, J_{C-P} = 71.7 Hz), 30.9 (d, J_{C-P} = 15.1 Hz), 31.7, 128.6 (d, J_{C-P} = 11.3 Hz), 130.8 (d, J_{C-P} = 9.0 Hz), 131.6, 133.2 (d, J_{C-P} = 96.6 Hz); ^{31}P NMR ($CDCl_3$) δ 33.2; HRMS (EI) calcd for $C_{20}H_{27}OP$: 314.1800, found: 314.1804; Anal. Calcd for $C_{20}H_{27}OP$: C, 76.40; H, 8.66. Found: C, 76.48; H, 8.58.
13. For the spectral and analytical data of (6-chlorohexyl)diphenylphosphine oxide (**1b**): colorless oil; IR (NaCl) 3418, 3055, 2933, 2862, 1636, 1591, 1485, 1437, 1309, 1180, 1121, 1072, 1028, 997, 945, 748, 721, 696, 648, 550, 515 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.41–1.72 (m, 8H), 2.23–2.29 (m, 2H) 3.48 (t, J = 6.4 Hz, 2H), 7.45–7.53 (m, 6H), 7.71–7.75 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 21.2, 26.2, 29.6 (d, J_{C-P} = 98.4 Hz), 29.9 (d, J_{C-P} = 13.0 Hz), 32.1, 44.8, 128.6 (d, J_{C-P} = 11.0 Hz), 130.6 (d, J_{C-P} = 8.0 Hz), 131.6, 132.9 (d, J_{C-P} = 98.4 Hz); ^{31}P NMR ($CDCl_3$) δ 33.0; HRMS (EI) calcd for $C_{18}H_{22}ClOP$: 320.1097, found: 320.1095.
14. For the spectral and analytical data of (5-cyanopentyl)diphenylphosphine oxide (**1c**): pale yellow oil; IR (NaCl) 3418, 3057, 2934, 2868, 2245, 1734, 1645, 1437, 1177, 1121, 1072, 1028, 997, 748, 721, 698, 544, 513 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.51–1.67 (m, 6H), 2.21–2.32 (m, 4H), 7.44–7.60 (m, 6H), 7.66–7.81 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 16.7, 20.7, 24.8, 29.2 (d, J_{C-P} = 66.3 Hz), 29.7 (d, J_{C-P} = 10.0 Hz), 119.3, 128.6 (d, J_{C-P} = 11.0 Hz), 130.6 (d, J_{C-P} = 8.0 Hz), 131.7, 132.7 (d, J_{C-P} = 97.4 Hz); ^{31}P NMR ($CDCl_3$) δ 32.7; HRMS (EI) calcd for $C_{18}H_{20}NOP$: 297.1283, found: 297.1284.
15. For the spectral and analytical data of diphenyl-(6-hydroxyhexyl)phosphine oxide (**1d**): colorless oil; IR (NaCl) 3366, 3057, 2930, 2858, 1437, 1173, 1121, 1057, 1028, 997, 748, 721, 696, 548, 511 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.31–1.65 (m, 8H), 2.25 (dt, J = 11.0, 8.2 Hz, 2H) 3.07 (s, 1H) 3.54–3.58 (m, 2H), 7.43–7.52 (m, 6H), 7.70–7.78 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 21.3, 25.1, 29.4 (d, J_{C-P} = 72.3 Hz), 30.4 (d, J_{C-P} = 15.1 Hz), 32.3, 62.2, 128.5 (d, J_{C-P} = 11.0 Hz), 130.6 (d, J_{C-P} = 8.0 Hz), 131.6, 132.9 (d, J_{C-P} = 97.4 Hz); ^{31}P NMR ($CDCl_3$) δ 33.8; HRMS (EI) calcd for $[M+H^+]$ $C_{18}H_{24}O_2P$: 303.1514, found: 303.1512.
16. For the spectral and analytical data of diphenyl-(4-phenylbutyl)phosphine oxide (**1e**): pale yellow oil; IR (NaCl) 3413, 3026, 2934, 2860, 1489, 1456, 1435, 1184, 1121, 1028, 997, 932, 744, 696, 517 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.67–1.77 (m, 4H), 2.24–2.32 (m, 2H), 2.59 (t, J = 7.6 Hz, 2H), 7.10–7.26 (m, 5H), 7.44–7.50 (m, 6H), 7.69–7.74 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 21.3, 29.7 (d, J_{C-P} = 71.0 Hz), 32.8 (d, J_{C-P} = 14.4 Hz), 35.5, 128.4, 128.7 (d, J_{C-P} = 11.5 Hz), 128.8, 130.8, 130.9 (d, J_{C-P} = 8.6 Hz), 132.3 (d, J_{C-P} = 122.8 Hz), 133.7, 142.0; ^{31}P NMR ($CDCl_3$) δ 33.1; HRMS (EI) calcd for $C_{22}H_{23}OP$: 334.1487, found: 334.1489.
17. For the spectral and analytical data of diphenyl-(3-phenoxypropyl)phosphine oxide (**1f**): colorless oil; IR (NaCl) 3420, 3057, 2932, 1585, 1497, 1437, 1389, 1290, 1244, 1182, 1121, 997, 908, 808, 754, 719, 694, 544, 513 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.05–2.15 (m, 2H), 2.41–2.51 (m, 2H), 4.00 (t, J = 6.0 Hz, 2H), 6.81–6.88 (m, 2H), 6.90–6.95 (m, 1H), 7.21–7.28 (m, 2H) 7.40–7.55 (m, 6H), 7.71–7.79 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 21.8, 26.4 (d, J_{C-P} = 72.7 Hz), 67.4 (d, J_{C-P} = 14.1 Hz), 114.4, 120.8, 128.7 (d, J_{C-P} = 11.5 Hz), 129.4, 130.7 (d, J_{C-P} = 9.5 Hz), 131.8, 132.8 (d, J_{C-P} = 98.7 Hz), 158.6; ^{31}P NMR ($CDCl_3$) δ 33.4; HRMS (EI) calcd for $C_{21}H_{21}O_2P$: 336.1279, found: 336.1275; Anal. Calcd for $C_{21}H_{21}O_2P$: C, 74.99; H, 6.29. Found: C, 74.13; H, 6.10.
18. For the spectral and analytical data of (3-aminopropyl)diphenylphosphine oxide (**1g**): yellow oil; IR (NaCl) 3389, 2359, 2340, 1437, 1317, 1159, 1123, 719, 696, 667, 548 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.72–1.79 (m, 2H), 2.00 (br s, 2H), 2.30–2.37 (m, 2H), 2.77 (t, J = 6.5 Hz, 2H), 7.40–7.55 (m, 6H), 7.72–7.79 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 25.1, 27.0 (d, J_{C-P} = 72.3 Hz), 42.6 (d, J_{C-P} = 15.1 Hz), 128.6 (d, J_{C-P} = 11.0 Hz), 130.7 (d, J_{C-P} = 10.0 Hz), 131.7, 132.8 (d, J_{C-P} = 97.4 Hz); ^{31}P NMR ($CDCl_3$) δ 33.4; HRMS (EI) calcd for $[M+H^+]$ $C_{15}H_{18}NOP$: 260.1204, found: 260.1209.
19. For the spectral and analytical data of cyclohexyldiphenylphosphine oxide (**1h**): white solid; mp 146–148 °C; IR (KBr) 2930, 2853, 2363, 1437, 1178, 1119, 721, 693, 559, 543 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.20–1.81 (m, 10H), 2.19–2.28 (m, 1H), 7.28–7.50 (m, 6H), 7.73–7.85 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 24.7, 25.7, 26.3 (d, J_{C-P} = 13.1 Hz), 37.1 (d, J_{C-P} = 73.3 Hz), 128.5 (d, J_{C-P} = 10.0 Hz), 131.0 (d, J_{C-P} = 10.0 Hz), 131.4, 131.9 (d, J_{C-P} = 102.4 Hz); ^{31}P NMR ($CDCl_3$) δ 35.0; HRMS (EI) calcd for $C_{18}H_{21}OP$: 284.1330, found: 284.1331.
20. For the spectral and analytical data of diphenyl(tetrahydropyran-3-yl)phosphine oxide (**1i**): white solid; mp 148–151 °C; IR (KBr) 3053, 2947, 2845, 1439, 1178, 1119, 1097, 1074, 1028, 995, 951, 904, 883, 856, 839, 752, 721, 700, 569, 534 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.64–1.97 (m, 4H), 2.59–2.66 (m, 1H), 3.39 (td, J = 11.5, 3.7 Hz, 1H), 3.67 (td, J = 11.5, 2.7 Hz, 1H), 3.91–3.95 (m, 2H), 7.48–7.56 (m, 6H), 7.75–7.86 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 21.8, 25.8 (d, J_{C-P} = 10.6 Hz), 36.6 (d, J_{C-P} = 71.0 Hz), 66.9, 68.1, 128.8 (d, J_{C-P} = 8.6 Hz), 126.9 (d, J_{C-P} = 10.6 Hz), 130.9 (d, J_{C-P} = 7.7 Hz), 130.9 (d, J_{C-P} = 7.7 Hz), 131.7, 131.8, 131.9, 132.0; ^{31}P NMR ($CDCl_3$) δ 30.9; HRMS (EI) calcd for $C_{17}H_{19}O_2P$: 286.1123, found: 286.1127.
21. For the spectral and analytical data of diphenyl-[2-(trimethylsilyl)ethyl]-phosphine oxide (**1j**): colorless oil; IR (NaCl) 3433, 3055, 2951, 2897, 1437, 1412, 1250, 1184, 1155, 1121, 841, 721, 698, 532, 511 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.00 (s, 9H), 0.72–0.75 (m, 2H), 2.14–2.16 (m, 2H), 7.45–7.55 (m, 6H), 7.68–7.73 (m, 4H); ^{13}C NMR ($CDCl_3$) δ −2.1, 6.9 (d, J_{C-P} = 7.7 Hz), 23.9 (d, J_{C-P} = 70.1 Hz), 128.7 (d, J_{C-P} = 11.5 Hz), 131.0 (d, J_{C-P} = 9.6 Hz), 131.7, 132.8 (d, J_{C-P} = 97.4 Hz); ^{31}P NMR ($CDCl_3$) δ 35.0; HRMS (EI) calcd for $C_{22}H_{23}OP$: 334.1487, found: 334.1489.