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Synthesis of Alkyl(diphenyl)phosphines by Hydrophosphination of Vinylarenes Catalyzed by Transition Metal Complexes*

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Abstract—Hydrophosphination of styrenes and their heteroanalogs with diphenylphosphine in the presence of nickel or palladium complexes was accomplished for the first time. The reaction ensures high yields and regioselectivity: the corresponding anti-Markownikoff adducts are exclusively formed.

Until recently, addition of compounds containing a hydrogen–heteroelement bond to unsaturated compounds, primarily to alkynes and alkenes, catalyzed by transition metal complexes, included mainly hydrosilylation reactions [1], which are widely used in industry. Later on, catalysis by metal complexes was applied to hydroboration with weakly reactive boranes like catecholborane [2]. Transition metal complex-catalyzed addition of dialkyl hydrogen phosphites [3] and phosphine oxides [4] to nonactivated alkynes and alkenes was discovered relatively recently. Interest in these reactions originates not only from their great synthetic potential but also from the fact that synthesis of many products by addition rather than substitution reactions is the most efficient from the viewpoint of "green chemistry": ideally, such syntheses may be effected without formation of by-products, i.e., with a 100% atom efficiency.

The above stated explains our interest in the synthesis of tertiary phosphines by addition reactions. Tertiary phosphines constitute an important class of organophosphorus compounds which are widely used as ligands in metal-complex catalysis. It is not an overstatement to say that success in one or another transition metal-catalyzed reaction, which should give a new C–C or C–E bond, is determined mainly by the nature of the ligand, primarily of the phosphine ligand. Usually, triarylphosphines are used as ligands in these processes, but in some cases the presence of an alkyl group at the phosphorus atom is necessary to enhance electron-donor power of the phosphine and its ability to affect oxidative addition to a metal atom. Just the use of $(t\text{-Bu})_3P$ made it possible to replace expensive aryl bromides by cheap aryl chlorides in the oxidative addition to Pd(0) [5]. Phosphines like Ph_2PAlk are commonly prepared by reactions of organolithium or organomagnesium compounds with Ph_2PHlg or by reactions of alkyl halides with phosphide anion [6]. Alkyldiarylphosphines can also be obtained by radical addition to olefins or by acid- or base-catalyzed addition to activated alkenes [7]. Phosphine addition to Michael acceptors like acrylonitrile or alkyl acrylates was effected under catalysis by transition metal complexes [8]. While performing the present work, addition of diphenylphosphine to alkenes and alkynes in the presence of lanthanide complexes was reported [9].

We were the first to accomplish addition of diphenylphosphine to vinylarenes(heteroarenes) (which can be regarded as weakly activated olefins) in the presence of palladium or nickel complexes. Such addition is more difficult to occur than analogous reaction with alkynes [10]; it is successful only with substrates having an aryl or hetaryl group. As catalytic precursors we used $NiCl_2$, $NiBr_2$, $Ni(PPh_3)_2Cl_2$, $Ni(PPh_3)_2Br_2$, $Ni(acac)_2$, $Ni(cod)_2$, $Ni[P(OEt)_3]_4$, $Ni[P(OPh)_3]_4$, $Pd(CH_3CN)_2Cl_2$, $Pd(PPh_3)_2Cl_2$, and $Pd(PPh_3)_4$. These complexes are capable of promoting the reaction of diphenylphosphine with styrene in benzene at 25–130°C in a sealed ampule (Table 1). The reaction gives two products: β-adduct **I** and tetraphenyldiphosphine (**II**) (Scheme 1). The latter is

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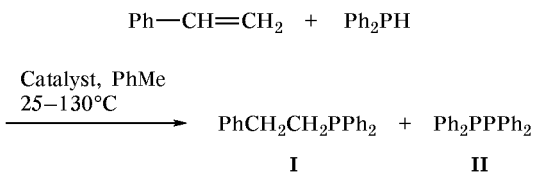
formed via oxidative dimerization of Ph_2PH [11], and the product ratio depends on the catalyst.

Scheme 1.



In the presence of Ni(II), Pd(II), and Pd(0) complexes, oxidative dimerization of diphenylphosphine to tetraphenyldiphosphine occurs very readily and is the main reaction pathway (Table 1, run no. 1). The process is strongly accelerated in the presence of styrene which (as was shown by special experiments) is converted into ethylbenzene (Scheme 2). In the absence of styrene, oxidative dimerization of diphenylphosphine to diphosphine **II** is promoted by $Ni(PPh_3)_2Br_2$, but under considerably more severe conditions (Table 1, run no. 2); no reaction occurs at room temperature.

Taking into account that oxidation of diphenylphosphine to tetraphenyldiphosphine by the action of Ni(II) could lead to formation of HBr which in turn is capable of converting Ni(0) into Ni(II) via oxidative addition (Scheme 1), we performed the reaction in the presence of triethylamine to neutralize liberated hydrogen bormide (Table 1, run nos. 3, 4). In the absence of styrene about 5% of tetraphenyldiphosphine was formed (run no. 3), whereas in the presence of styrene a mixture of the addition product and tetraphenyldiphosphine was obtained (run no. 4). Probably, in this case tetraphenyldiphosphine is formed by a different mechanism.

Table 1. Transformation of diphenylphosphine in the presence of $Ni(PPh_3)_2Br_2$[a]

Run no.	Temperature, °C (time, h)	Conversion of Ph_2PH, %	Ratio **I**:**II**, %
1[b]	20 (48) or 50 (5)	100	0:100
2	130 (30)	100	0:100
3[c]	130 (30)	5	0:5
4[d]	130 (28)	100	50:50

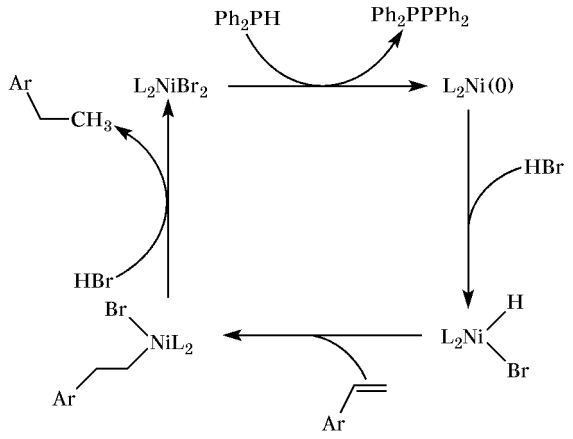
[a] Amounts of the reactants: diphenylphosphine, 1.5 mmol; benzene, 2 ml; $Ni(PPh_3)_2Br_2$, 5 mol %.
[b] Styrene, 3 mmol, was added.
[c] Triethylamine, 0.15 ml, was added.
[d] Styrene, 3 mmol, and triethylamine, 0.15 ml, were added.

Table 2. Addition of diphenylphosphine to styrene in the presence of Ni(0) complexes[a]

Run no.	Catalyst, 5 mol%	Temp., °C (time, h)	Conversion, %	Ratio **I**:**II**, %
1	$Ni[P(OEt)_3]_4$	90 (40)	85	95:5
2	$Ni[P(OEt)_3]_4$	130 (40)	95	90:10
3[b]	$Ni[P(OEt)_3]_4$	130 (40)	95	100:0
4[b,c]	$Ni[P(OEt)_3]_4$	130 (20)	100	100:0
5[b,c]	$Ni[P(OPh)_3]_4$	130 (30)	100	100:0

[a] Amounts of the reactants: styrene, 1.5 mmol; diphenylphosphine, 1.5 mmol; benzene, 2 ml.
[b] Triethylamine, 0.15 ml, was added.
[c] The amount of styrene was 3 mmol.

Scheme 2.

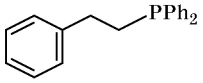
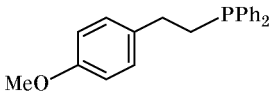
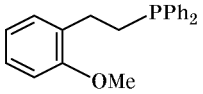
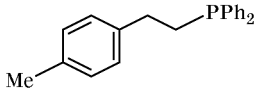
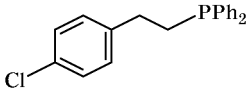
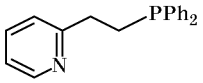
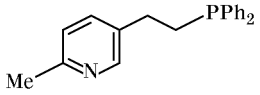
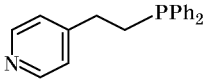


The pattern dramatically changed when Ni(0) complexes were used as catalysts (Table 2, run nos. 1, 2). Here, adduct **I** was the major product, and it was formed as the only one in the presence of triethylamine (Table 2, run nos. 3, 4). The reaction can be accelerated to a considerable extent through the use of excess styrene (Table 2, run no. 4).

The reaction under study is regioselective, and it yields exclusively the corresponding β-adduct. We also performed reactions with substituted styrenes under the optimal conditions (Table 3). According to the NMR data, in all cases the products were formed in almost quantitative yield, and the yield of the isolated products was 85–95%.

The absence of α-substituted products in the addition of Ph_2PH to styrene and vinylpyridines allows us to exclude formation of a π-allyl intermediate like

Table 3. Addition of diphenylphosphine to substituted phenyl- and pyridylethenes[a]

Run no.	Initial compound	Adduct	Time, h	Yield,[b] %
1	$C_6H_5CH=CH_2$	 (**I**)	20	>99 (95)
2	4-$MeOC_6H_4CH=CH_2$	 (**Ia**)	25	>99 (85)
3	2-$MeOC_6H_4CH=CH_2$	 (**Ib**)	25	>99 (95)
4	4-$MeC_6H_4CH=CH_2$	 (**Ic**)	20	>99 (90)
5[c]	4-$ClC_6H_4CH=CH_2$	 (**Id**)	20	>99 (85)
6	(C_5H_4N-2)$CH=CH_2$	 (**Ie**)	20	>99 (85)
7	(2-MeC_5H_3N-5)$CH=CH_2$	 (**If**)	10	>95 (90)
8	(C_5H_4N-4)$CH=CH_2$	 (**Ig**)	20	>99 (87)

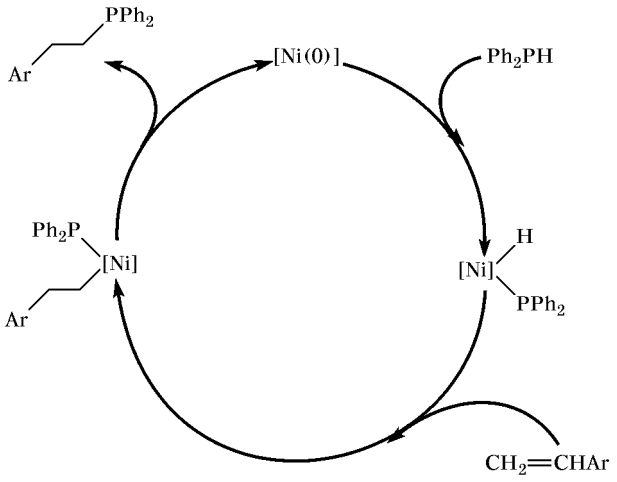
[a] A typical experiment was carried out with 3 mmol of alkene, 1.5 mmol of diphenylphosphine, 5 mol % of $Ni[P(OEt)_3]_4$, 2 ml of benzene, and 0.152 ml of triethylamine; 130°C.

[b] According to the ^{31}P NMR data; the yield of the isolated product is given in parentheses.

[c] The reaction was carried out with 1 equiv. of 4-chlorostyrene.

that assumed for the Pd-catalyzed hydroamination of styrene [12]. Scheme 3 shows a probable catalytic cycle which includes oxidative addition of phosphine to Ni(0) with formation of a hydridophosphide complex, olefin insertion into the Ni–H bond, and reductive elimination.

Scheme 3.



It was interesting to perform hydrophosphination of α-methylstyrene, which should give rise to an asymmetric center. Here, the possibility for stereoselective synthesis exists. The reaction with α-methylstyrene occurred at a considerably lower rate (Table 4, run nos. 1–5). Increase in the catalyst amount did not affect the reaction rate (Table 4, run nos. 2, 3). The

Scheme 4.

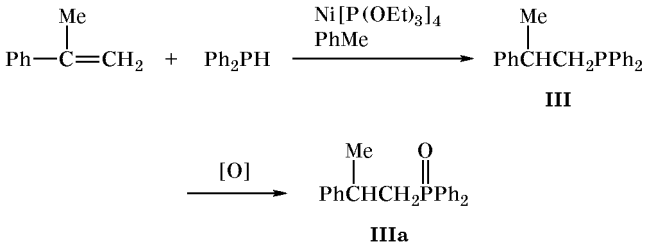


Table 4. Addition of diphenylphosphine to α-methylstyrene[a]

Run no.	Temperature, °C (time, h)	Conversion of Ph_2PH, %	Ratio **III**:**II**, %
1	130 (20)	35	100:0
2	130 (40)	46	98:2
3[b]	130 (40)	46	98:2
4[c]	130 (40)	49	99:1
5[c]	130 (100)	82	98:2

[a] Amounts of the reactants: α-methylstyrene, 3 mmol; diphenylphosphine, 1.5 mmol; $Ni[P(OEt)_3]_4$, 5 mol %; toluene, 2 ml; triethylamine, 0.152 ml.

[b] The catalyst was added in 5% portions every 20 h.

[c] After 20 h, an additional 1.5 mmol of α-methylstyrene was added.

addition was accelerated by the use of 3 equiv of α-methylstyrene (Table 4, run nos. 2, 4), but in this case the complete conversion cannot be attained (Table 4, run no. 5). The resulting phosphine readily undergoes oxidation during isolation, and the product was characterized as phosphine oxide **IIIa** (Scheme 4).

Thus we have developed a new almost wasteless procedure for synthesizing alkyl(diphenyl)phosphines. The procedure is applicable to sterically hindered styrenes. We are now studying the possibility for asymmetric hydrophosphination of alkenes.

EXPERIMENTAL

All operations, including preparation, isolation, and generation of phosphorus(III) compounds, were performed under dry argon. The solvents were purified by standard procedures: hexane, benzene, toluene, and THF were refluxed over metallic sodium and then distilled over it.

The progress of reactions was monitored, and the newly synthesized compounds were identified, by ^1H and ^{31}P NMR spectroscopy. The ^1H NMR spectra were recorded on a Varian VXR-400 spectrometer at 400 MHz using TMS or HMDS as internal reference. The ^{31}P NMR spectra were measured on the same instrument at 161.9 MHz using 85% H_3PO_4 as external reference. The chemical shifts are given relative to TMS (^1H) or 85% H_3PO_4 (^{31}P).

Dichlorobis(triphenylphosphine)nickel(II) [13], tetrakis(triethyl phosphite)nickel(0) [14], dibromobis(triphenylphosphine)nickel(II) [13], tetrakis(triphenyl phosphite)nickel(0) [15], diphenylphosphine [16], 2-methoxystyrene [17], and 4-methoxystyrene [17] were synthesized by known methods. Styrene, 2-vinylpyridine, 4-vinylpyridine, 2-methyl-5-vinylpyridine, 4-chlorostyrene, 4-methylstyrene, and α-methylstyrene were commercial products.

General procedure for hydrophosphination of aryl- and hetarylethenes with diphenylphosphine. A 8-mm (i.d.) ampule was charged with 279 mg (1.5 mmol) of diphenylphosphine, 2 ml of benzene or toluene, and 54 mg (5 mol %) of tetrakis(triethyl phosphite)nickel(0). Alkene, 3 mmol, and triethylamine, 152 mg (1.5 mmol), were then added, and the ampule was sealed and heated at 130°C until the reaction was complete (according to the ^{31}P NMR data). When the signal of initial diphenylphosphine, δ_P –40.6 ppm, disappeared from the spectrum, the ampule was cooled and opened, and the solvent and excess alkene were distilled off under reduced pressure. The residue was either recrystallized from THF–hexane (on cooling to –18°C) or purified by chromatography on silica gel using gradient elution with hexane to 3:1 hexane–benzene.

Diphenyl(2-phenylethyl)phosphine (I) [18] was obtained from 312 mg (3 mmol) of styrene; reaction time 20 h. The product was purified by chromatography. Yield 560 mg (92%), colorless oil. ^1H NMR spectrum (C_6D_6), δ, ppm: 2.31–2.36 m (2H), 2.74–2.80 m (2H), 7.06–7.21 m (11H), 7.48–7.52 m (4H). $^{31}P-\{^1H\}$ NMR spectrum (C_6D_6): δ_P 16.1 ppm.

[2-(4-Methoxyphenyl)ethyl]diphenylphosphine (Ia) was synthesized from 402 mg (3 mmol) of 4-methoxystyrene; reaction time 25 h. After chromatographic purification, 410 mg (95%) of compound **Ia** was isolated as a colorless oil. ^1H NMR spectrum (C_6D_6), δ, ppm: 2.33–2.37 m (2H), 2.74–2.80 m (2H), 3.43 s (3H), 6.79–6.86 m (4H), 7.15–7.21 m (6H), 7.50–7.54 m (4H). $^{31}P-\{^1H\}$ NMR spectrum (C_6D_6): δ_P 16.3 ppm. Found, %: C 79.01; H 6.52. $C_{21}H_{21}PO$. Calculated, %: C 78.75; H 6.56.

[2-(2-Methoxyphenyl)ethyl]diphenylphosphine (Ib) was obtained from 402 mg (3 mmol) of 2-methoxystyrene; reaction time 25 h. The product was purified by recrystallization. Yield 455 mg (95%). ^1H NMR spectrum (C_6D_6), δ, ppm: 2.45–2.49 m (2H), 2.97–3.03 m (2H), 3.36 s (3H), 6.61 d (1H), 6.91 t (1H), 7.08–7.20 m (8H), 7.54–7.58 m (4H). $^{31}P-\{^1H\}$ NMR spectrum (C_6D_6): δ_P 15.6 ppm. Found, %: C 78.11; H 7.03. $C_{21}H_{21}PO$. Calculated, %: C 78.75; H 6.56.

[2-(4-Methylphenyl)ethyl]diphenylphosphine (Ic) was obtained from 354 mg (3 mmol) of 4-methylstyrene; reaction time 20 h. The product was purified

by chromatography. Yield 460 mg (90%), colorless oil. ^{1}H NMR spectrum (C_6D_6), δ, ppm: 2.28 s (3H), 2.40–2.44 m (2H), 2.80–2.90 m (2H), 7.09–7.25 m (10H), 7.55–7.63 m (4H). ^{31}P–{^{1}H} NMR spectrum (C_6D_6): δ_P 16.5 ppm. Found, %: C 82.46; H 6.85. $C_{21}H_{21}P$. Calculated, %: C 82.89; H 6.90.

[2-(4-Chlorophenyl)ethyl]diphenylphosphine (Id) was obtained from 229 mg (1.5 mmol) of 4-chlorostyrene; reaction time 10 h. The product was purified by chromatography. Yield 430 mg (85%), colorless oil. ^{1}H NMR spectrum (C_6D_6), δ, ppm: 2.17–2.21 m (2H), 2.56–3.62 m (2H), 7.13–7.20 m (10H), 7.45–7.51 m (4H). ^{31}P–{^{1}H} NMR spectrum (C_6D_6): δ_P 16.7 ppm. Found, %: C 74.44; H 5.53. $C_{21}H_{20}PCl$. Calculated, %: C 74.44; H 5.90.

2-[2-(Diphenylphosphino)ethyl]pyridine (Ie) [19] was obtained from 314 mg (3 mmol) of 2-vinylpyridine; reaction time 20 h. The product was purified by chromatography. Yield 370 mg (85%), colorless oil. ^{1}H NMR spectrum (C_6D_6), δ, ppm: 2.65–2.69 m (2H), 2.99–3.05 m (2H), 6.68–6.71 m (2H), 7.06–7.14 m (7H), 7.53–7.57 m (4H), 8.56–8.58 m (1H). ^{31}P–{^{1}H} NMR spectrum (C_6D_6): δ_P 15.8 ppm.

4-[2-(Diphenylphosphino)ethyl]pyridine (If) was obtained from 314 mg (3 mmol) of 4-vinylpyridine. Reaction time 20 h. The product was purified by chromatography. Yield 380 mg (87%), colorless noncrystallizable oil. ^{1}H NMR spectrum (C_6D_6), δ, ppm: 2.65–2.69 m (2H), 2.99–3.05 m (2H), 6.69–6.71 m (2H), 7.09–7.18 m (6H), 7.53–7.57 m (4H), 8.56–8.58 m (2H). ^{31}P–{^{1}H} NMR spectrum (C_6D_6): δ_P 16.1 ppm.

5-[2-(Diphenylphosphino)ethyl]-2-methylpyridine (Ig) was obtained from 357 mg (3 mmol) of 2-methyl-5-vinylpyridine. Reaction time 20 h. The product was purified by chromatography. Yield 410 mg (90%), colorless oil. ^{1}H NMR spectrum (C_6D_6), δ, ppm: 2.27–2.31 m (2H), 2.46 s (3H), 2.60–2.67 m (2H), 6.97–7.01 m (2H), 7.25–7.35 m (6H), 7.39–7.45 m (4H), 8.27 s (1H). ^{31}P–{^{1}H} NMR spectrum (C_6D_6): δ_P 16.5 ppm. Found, %: C 78.33; H 6.48. $C_{20}H_{20}NP$. Calculated, %: C 78.68; H 6.55.

Diphenyl(2-phenylpropyl)phosphine oxide (IIIa) was obtained from 531 mg (4.5 mmol) of α-methylstyrene. Reaction time 100 h. Recrystallization gave 144 mg (30%) of product **IIIa**. ^{1}H NMR spectrum (C_6D_6), δ, ppm: 1.51–1.53 d (3H), 2.31–2.40 m (1H), 2.43–2.50 m (1H), 3.35–3.50 m (1H), 7.03–7.15 m (12H), 7.75–7.83 m (3H). ^{31}P–{^{1}H} NMR spectrum (C_6D_6): δ_P 23.5 ppm. Found, %: C 78.21; H 6.54. $C_{21}H_{21}PO$. Calculated, %: C 78.75; H 6.56.

Detection of ethylbenzene in the reaction of diphenylphosphine with styrene. A 5-mm NMR ampule was purged with argon and charged with 0.1395 g of diphenylphosphine, 0.117 g of styrene, and 11 mg (5 mol %) of $Ni(PPh_3)_2Br_2$, and 0.6 ml of benzene-d_6 was added. The mixture was kept for 2 days at room temperature. The ^{31}P–{^{1}H} NMR spectrum contained only one signal at δ_P –17.6 ppm (Ph_2PPPh_2). In the ^{1}H NMR spectrum we observed signals belonging to ethylbenzene, δ, ppm: 1.19–1.23 t (3H), 2.55–2.61 q (2H).

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