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Palladium-catalyzed New Carbon-Phosphorus Bond Formation

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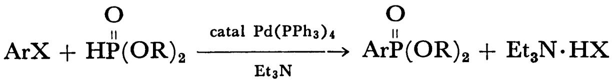
Dialkyl arylphosphonates are prepared by the palladium-catalyzed reaction of aryl bromides with dialkyl phosphite in the presence of triethylamine. The similar treatment of vinyl bromides gives dialkyl vinylphosphonates stereoselectively.

Arbuzov and Michaelis-Becker reactions provide facile procedures for the formation of carbon-phosphorus bonds.[1] These methods, however, are not applicable to the formation of *sp*^{2} hybridized carbon-phosphorus bonds. Only few methods have been reported for the syntheses of arylphosphonates[1,2] and vinylphosphonates.[1,3] For example, the photoinitiated reaction of iodobenzene with trialkyl phosphite[2f,g] or dialkyl phosphite ion[2e] affords dialkyl phenylphosphonate. Direct reaction of aryl[2i] or vinyl halides[3g] with trialkyl phosphite in the presence of nickel halide requires severe reaction conditions, and the stereochemistry of vinylphosphonates has not been clarified. In our preceding reports, we found new and versatile syntheses of dialkyl arylphosphonates[4] and vinylphosphonates[5] using palladium catalyst. The stereoselective synthesis of dialkyl vinylphosphonates has been accomplished by this method. Herein, we wish to report further details of the palladium-catalyzed carbon-phosphorus bond formation.

Results and Discussion

Preparation of Dialkyl Arylphosphonates. Dialkyl arylphosphonates (**1**) were prepared in good yields by the reaction of aryl bromides or iodide with *O,O*-dialkyl phosphonate in the presence of triethylamine and a catalytic amount of tetrakis(triphenylphosphine)palladium. The results are summarized in Table 1.



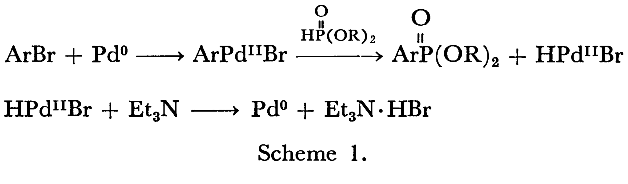
$$\underset{X=Br,\ I}{ArX} + H\overset{O}{\overset{\|}{P}}(OR)_2 \xrightarrow[Et_3N]{catal\ Pd(PPh_3)_4} \underset{\mathbf{1}}{Ar\overset{O}{\overset{\|}{P}}(OR)_2} + Et_3N\cdot HX$$

Use of palladium acetate instead of tetrakis(triphenylphosphine)palladium resulted a moderate yield of **1a**. But palladium chloride was less effective. Triethylamine was superior to tri-*n*-butylamine and pyridine as base. Although bromobenzene readily underwent phosphonation with *O,O*-diethyl phosphonate, chlorobenzene did not react under the same reaction conditions. On treatment of *p*-bromochlorobenzene with *O,O*-diethyl phosphonate, diethyl *p*-chlorophenylphosphonate was obtained in 85% yield as a sole product. *O,O*-Diisopropyl and *O,O*-dibutyl phosphonates were also allowed to react with bromobenzene to give the corresponding phosphonates. Diethyl arylphosphonates, bearing a variety of electron-withdrawing and electron-donating substituents, such as *p*-NO$_2$, *p*-CN, *p*-MeCO, *o*-COOMe, *p*-MeCONH, *p*-MeO, *p*-Me, *o*-Me, were prepared in high yields. *o*-Bromobenzoic acid was subjected to esterification with diethyl phosphite as well as phosphonation to give diethyl *o*-ethoxycarbonylphenylphosphonate in 24% yield. Attempts to prepare *p*-hydroxy- and *p*-aminophenylphosphonates were unsuccessful. These observations are consistent with the results reported by Heck[6] that aryl bromides with strongly electron-donating substituents do not undergo the palladium-catalyzed vinylic substitution reaction in acceptable yields. The palladium-catalyzed phosphonation of 1-bromonaphthalene and 3-bromopyridine gave the corresponding phosphonates in good yields.

It should be noted that *o*-dibromobenzene reacted with 2 equiv. of *O,O*-diethyl phosphonate to yield *o*-bromophenylphosphonate (34%) and phenylphosphonate (6%), whereas substitution of both bromine atoms of *p*-dibromobenzene was observed. Though the mechanism for the formation of diethyl phenylphosphonate from *o*-dibromobenzene has not been clarified, it is conceivable that diethyl phosphite plays an important role in the present reduction reaction. Previously, we reported a facile reduction of *gem*-dibromo derivatives with diethyl phosphite and triethylamine.[7] In this connection, the dark reactions of *o*-fluoroiodobenzene and *o*-chloroiodobenzene with (EtO)$_2$PO$^-$ in liquid ammonia were reported to give fluorobenzene and chlorobenzene, respectively, and such a reduction reaction has not been observed for *m*- and *p*-iodohalobenzenes.[8] *o*-Bromobenzyl alcohol was also subjected to reduction of hydroxyl group to produce diethyl *o*-tolylphosphonate in 18% yield.

The formation path of dialkyl arylphosphonates is outlined in Scheme 1. Palladium(0) species undergoes oxidative addition with aryl bromide to give the arylpalladium complex, which may be considered to be a key intermediate. The attack of dialkyl phosphite to the arylpalladium complex leads to dialkyl arylphosphonate. Triethylamine regenerates palladium(0) species with the deposition of Et$_3$N·HBr. Thus obtained palladium(0) species is available to another reaction cycle. In the nickel-catalyzed Arbuzov reaction, arylnickel complexes have also been suggested.[9]

Diethyl sodiophosphonate was able to be employed in the palladium-catalyzed reaction. For prevention of the photoinitiated phosphonation,[2e] the reaction



$$ArBr + Pd^0 \longrightarrow ArPd^{II}Br \xrightarrow{H\overset{O}{\overset{\|}{P}}(OR)_2} Ar\overset{O}{\overset{\|}{P}}(OR)_2 + HPd^{II}Br$$

$$HPd^{II}Br + Et_3N \longrightarrow Pd^0 + Et_3N\cdot HBr$$

Scheme 1.

Table 1. Preparation of dialkyl arylphosphonates (**1**)

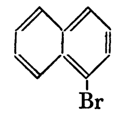
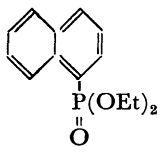
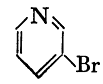
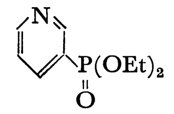
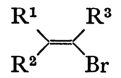
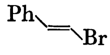
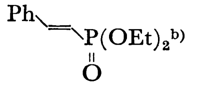
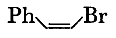
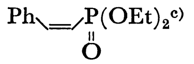
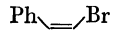
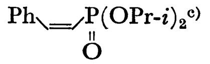
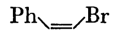
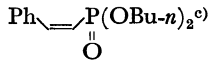
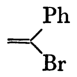
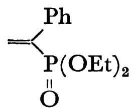
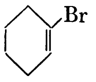
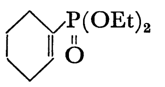
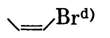
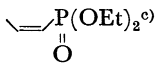
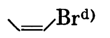
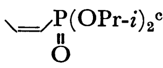
ArX	$HP(O)(OR)_2$ R	Base	Catalyst	Reaction a) time/h	Product **1**	Yield/%
PhBr	Et	Et_3N	$Pd(PPh_3)_4$	2.5	$Ph\underset{\overset{\|}{O}}{P}(OEt)_2$ **1a**	92
PhBr	Et	n-Bu_3N	$Pd(PPh_3)_4$	2.5	**1a**	51
PhBr	Et	pyridine	$Pd(PPh_3)_4$	16	**1a**	trace
PhBr	Et	Et_3N	$Pd(OAc)_2$	12	**1a**	58
PhBr	Et	Et_3N	$PdCl_2$	12	**1a**	7
PhBr	i-Pr	Et_3N	$Pd(PPh_3)_4$	2.5	$Ph\underset{\overset{\|}{O}}{P}(OPr\text{-}i)_2$ **1b**	90
PhBr	n-Bu	Et_3N	$Pd(PPh_3)_4$	2.5	$Ph\underset{\overset{\|}{O}}{P}(OBu\text{-}n)_2$ **1c**	94
PhI	Et	Et_3N	$Pd(PPh_3)_4$	2.5	**1a**	96
p-Me-C_6H_4Br	Et	Et_3N	$Pd(PPh_3)_4$	17	p-Me-$C_6H_4\underset{\overset{\|}{O}}{P}(OEt)_2$ **1d**	94
o-Me-C_6H_4Br	Et	Et_3N	$Pd(PPh_3)_4$	30	o-Me-$C_6H_4\underset{\overset{\|}{O}}{P}(OEt)_2$ **1e**	91
p-Cl-C_6H_4Br b)	Et	Et_3N	$Pd(PPh_3)_4$	8	p-Cl-$C_6H_4\underset{\overset{\|}{O}}{P}(OEt)_2$ **1f**	85
p-MeO-C_6H_4Br	Et	Et_3N	$Pd(PPh_3)_4$	17	p-MeO-$C_6H_4\underset{\overset{\|}{O}}{P}(OEt)_2$ **1g**	95
p-O_2N-C_6H_4Br b)	Et	Et_3N	$Pd(PPh_3)_4$	8	p-O_2N-$C_6H_4\underset{\overset{\|}{O}}{P}(OEt)_2$ **1h**	73
p-$Me\overset{\overset{O}{\|}}{C}$-$C_6H_4Br$ b)	Et	Et_3N	$Pd(PPh_3)_4$	8	p-$Me\overset{\overset{O}{\|}}{C}$-$C_6H_4\underset{\overset{\|}{O}}{P}(OEt)_2$ **1i**	90
p-$Me\overset{\overset{O}{\|}}{C}NH$-$C_6H_4Br$ b)	Et	Et_3N	$Pd(PPh_3)_4$	28	p-$Me\overset{\overset{O}{\|}}{C}NH$-$C_6H_4\underset{\overset{\|}{O}}{P}(OEt)_2$ **1j**	64
p-NC-C_6H_4Br b)	Et	Et_3N	$Pd(PPh_3)_4$	2	p-NC-$C_6H_4\underset{\overset{\|}{O}}{P}(OEt)_2$ **1k**	95
o-$MeO\overset{\overset{O}{\|}}{C}$-$C_6H_4Br$	Et	Et_3N	$Pd(PPh_3)_4$	35	o-$MeO\overset{\overset{O}{\|}}{C}$-$C_6H_4\underset{\overset{\|}{O}}{P}(OEt)_2$ **1l**	43
o-HOOC-C_6H_4Br b)	Et	Et_3N	$Pd(PPh_3)_4$	10	o-$EtO\overset{\overset{O}{\|}}{C}$-$C_6H_4\underset{\overset{\|}{O}}{P}(OEt)_2$ **1m** f)	24
p-H_2N-C_6H_4Br b,c)	Et	Et_3N	$Pd(PPh_3)_4$	35	——	
p-HO-C_6H_4Br b,c)	Et	Et_3N	$Pd(PPh_3)_4$	24	p-HO-$C_6H_4\underset{\overset{\|}{O}}{P}(OEt)_2$ **1n**	3
o-$HOCH_2$-C_6H_4Br b,d)	Et	Et_3N	$Pd(PPh_3)_4$	19	**1e**	18
p-Br-C_6H_4Br b,e)	Et	Et_3N	$Pd(PPh_3)_4$	8	p-$(EtO)_2\underset{\overset{\|}{O}}{P}$-$C_6H_4\underset{\overset{\|}{O}}{P}(OEt)_2$ **1o**	81
o-Br-C_6H_4Br e)	Et	Et_3N	$Pd(PPh_3)_4$	64	o-Br-$C_6H_4\underset{\overset{\|}{O}}{P}(OEt)_2$ **1p**	34
					1a	6
	Et	Et_3N	$Pd(PPh_3)_4$	24	 **1q**	96

Table 1. (continued)

ArX	HP(O)(OR)$_2$ R	Base	Catalyst	Reaction$^{a)}$ time/h	Product **1**	Yield/%
	Et	Et$_3$N	Pd(PPh$_3$)$_4$	3	 **1r**	77

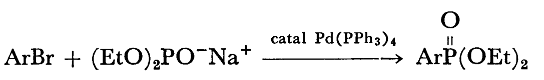
a) Solvent was not used unless otherwise stated; reactions at 90 °C. b) Toluene was added as a solvent. c) 0.25 equiv of tri-*o*-tolylphosphine was added. d) Reaction temperature, 100 °C. e) 2 equiv. of diethyl phosphite and triethylamine were used. f) Carboxyl group of the product was esterified by ethoxy group of *O*,*O*-diethyl phosphonate *in situ*.

Table 2. Preparation of dialkyl vinylphosphonates (**2**)

	HP(O)(OR^4)$_2$ R^4	Reaction$^{a)}$ time/h	Product **2**	Yield/%
	Et	0.2	 **2a**	93
	Et	1.5	 **2b**	91
	*i*-Pr	12	 **2c**	81
	*n*-Bu	1.5	 **2d**	92
	Et	30	 **2e**	66
	Et	9	 **2f**	69
	Et	1.5	 **2g**	98
	*i*-Pr	6	 **2h**	92

a) Solvent was not used unless otherwise stated; reactions at 90 °C. b) Formation of no *Z*-isomer was observed in the ^{1}H NMR spectrum. c) No *E*-isomer was detected in the ^{1}H NNR spectrum. d) Toluene was added as a solvent. Yields were based on the starting dialkyl phosphite.

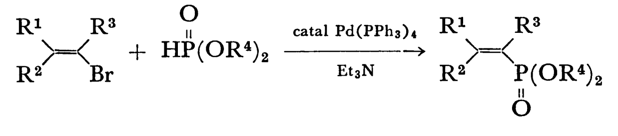
was carried out in the dark, but gave diethyl arylphosphonates in moderate yields.



$$\mathrm{ArBr} + (\mathrm{EtO})_2\mathrm{PO}^-\mathrm{Na}^+ \xrightarrow{\text{catal Pd(PPh}_3)_4} \mathrm{Ar}\overset{\mathrm{O}}{\overset{\|}{\mathrm{P}}}(\mathrm{OEt})_2$$

Ar=Ph 54%
=*p*-Me-C_6H_4 36%

Preparation of Dialkyl Alkenylphosphonates. The palladium-catalyzed phosphonation was successfully applied to stereoselective syntheses of dialkyl vinylphosphonates (**2**) from vinyl bromides. The results are summarized in Table 2.



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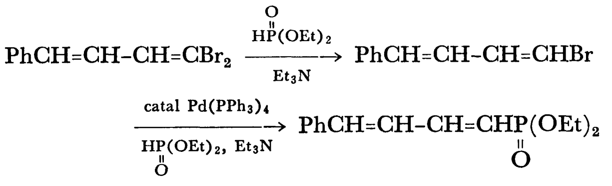
Stereoselectivity is illustrated as follows. Treatment of (*E*)- and (*Z*)-*β*-bromostyrenes with diethyl phosphite in the presence of palladium catalyst gave diethyl (*E*)- and (*Z*)-styrylphosphonates, respectively, without any contamination of the isomer. Phosphonomycin, (−)-(1*R*,2*S*)-1,2-epoxypropylphosphonic acid, is a new antibiotic.[10] Diisopropyl (*Z*)-1-propenylphosphonate, its important synthetic intermediate, was obtained in a high yield by this phosphonation.

1-Monobromodiene was easily prepared by the reaction of *gem*-dibromodiene with *O*,*O*-diethyl phosphonate and triethylamine according to our method.[8] 1-Bromo-4-phenylbutadiene was converted to diethyl 4-phenylbutadienylphosphonate (**3**) by the palladium-catalyzed phosphonation. One-step synthesis of the dienylphosphonate **3** was attempted by the reaction of 1,1-dibromo-4-phenylbutadiene with *O*,*O*-diethyl phosphonate in the presence of triethylamine and a

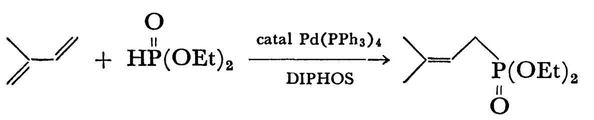


78% **3**

catalytic amount of tetrakis(triphenylphosphine)palladium, but only 1-bromo-4-phenylbutadiene was obtained. The substitution with diethyl phosphite was not observed.

Vinylpalladium complexes seem to be involved as key intermediates,[11] and the same mechanism as given for dialkyl arylphosphonates may be operative.

It is well known that the reaction of diene with nucleophiles is catalyzed by palladium complex.[12] But the reaction of dienes with dialkyl phosphite has not been studied. Isoprene was treated with *O,O*-diethyl phosphonate in the presence of a catalytic amount of tetrakis(triphenylphosphine)palladium and 1,2-bis(diphenylphosphino)ethane (DIPHOS) to give diethyl 3-methyl-2-butenylphosphonate (**4**) in 10% yield selectively. Although there are two possible routes of nucleophilic attack of *O,O*-diethyl phosphonate to isoprene, formation of diethyl 1-methyl-2-butenylphosphonate was not observed in the ^1H NMR spectrum.



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The results described here thus show the palladium-catalyzed phosphonation to be a useful method for organophosphorus syntheses, especially in the synthesis of dialkyl aryl- and vinylphosphonates.

Experimental

^1H NMR spectra were obtained on a JEOL JNM-PMX-60 with tetramethylsilane as an internal standard. IR spectra were recorded with a JASCO IRA-1 spectrometer. The mass spectra were taken with a Hitachi RMU-6E spectrometer.

Materials. (*Z*)-β-bromostyrene,[13] bromocyclohexene,[13] (*Z*)-1-bromo-1-propene,[14] and 1-bromo-4-phenylbutadiene[8] were prepared by the reported methods. Tetrakis(triphenylphosphine)palladium was easily prepared from palladium chloride.[15] Other reagents and solvents are commercial ones and were purified by distillation under nitrogen atmosphere or recrystallization.

Dialkyl Arylphosphonate (1). General Procedure: To a stirred mixture of *O,O*-dialkyl phosphonate (4.4 mmol), triethylamine (0.61 ml, 4.4 mmol), and tetrakis(triphenylphosphine)palladium (0.23 g, 0.2 mmol), aryl bromide (4.0 mmol) was added under nitrogen atmosphere, and the resultant mixture was stirred at 90 °C for 2—64 h. When aryl bromide is solid, toluene (1 ml) was added as a solvent. After the addition of ether (50 ml), triethylamine hydrobromide was removed by filtration. The residue obtained upon evaporation was purified by Kugelrohr distillation or column chromatography on silica gel to give dialkyl arylphosphonate (Table 1). Dialkyl arylphosphonates (**1a**,[21] **1b**,[21] **1c**,[1b] **1d**,[21] **1e**,[21] **1f**,[21] **1g**,[21] **1h**,[16] **1i**,[21] **1j**,[2j] **1k**,[17] **1l**,[21] **1m**,[21] **1n**,[21] **1o**,[21] **1q**,[1b] and **1r**[18]) were identified by comparison of their IR and ^1H NMR spectra with those of authentic samples. **1p**: IR (neat) 1245, 1050—1020, and 965 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.37 (t, 6H, *J*=7.0 Hz), 4.20 (dq, 4H, *J*=7.0 and 7.0 Hz), 7.30—8.30 (m, 4H); MS *m/e* 293 (M^+); Found: C, 40.96; H, 5.05; Br, 27.48; P, 10.32%. Calcd for $C_{10}H_{14}BrO_3P$: C, 40.98; H, 4.81; Br, 27.26; P, 10.57%.

Palladium-catalyzed Reaction of Aryl Bromide with Diethyl Sodiophosphonate. To a stirred mixture of bromobenzene (0.63 g, 4.0 mmol) and tetrakis(triphenylphosphine)palladium (0.23 g, 0.2 mmol) in toluene (3 ml), diethyl sodiophosphonate (4.4 mmol) in toluene (4 ml) was added. The resultant mixture was stirred at 90 °C for 30 h under nitrogen atmosphere. After the addition of ether (20 ml), sodium bromide was removed by filtration. The residue obtained upon evaporation was distilled by Kugelrohr to give diethyl phenylphosphonate (**1a**, 0.46 g, 54%). Diethyl *p*-tolylphosphonate (**1d**) was prepared in 36% yield similarly (reaction time, 24 h).

Dialkyl Vinylphosphonate (2). General Procedure: The vinylphosphonate **2** was prepared in the similar manner as that of **1** (Table 2). In the case of (*Z*)-1-bromo-1-propene as a starting bromide, reactions were carried out by mixing (*Z*)-1-bromo-1-propene (0.58 g, 4.8 mmol), *O,O*-dialkyl phosphonate (4.0 mmol), triethylamine (0.55 ml, 4.0 mmol), and tetrakis(triphenylphosphine)palladium (0.23 g, 0.2 mmol) in toluene (1 ml) at 90 °C. Yields of **2g** and **2h** were based on the starting *O,O*-dialkyl phosphonate. Dialkyl vinylphosphonates (**2a**,[3g] **2e**,[3g] **2f**,[1b] **2g**,[3f] and **2h**[10a]) were identified by comparison of their IR and ^1H NMR spectra with those of authentic samples. **2b**: IR (neat) 1600, 1235, 1050—1020, and 955 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.12 (t, 6H, *J*=7.0 Hz), 3.98 (dq, 4H, *J*=7.0 and 7.0 Hz), 5.78 (dd, 1H, *J*=14.0 and 14.9 Hz), 6.70—7.90 (m, 5+1H); MS *m/e* 240 (M^+). **2c**: IR (neat) 1605, 1240, and 1030—960 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.15 (d, 6H, *J*=6.5 Hz), 1.25 (d, 6H, *J*=6.5 Hz), 4.25—5.08 (m, 2H), 5.69 (dd, 1H, *J*=14.0 and 14.5 Hz), 6.53—7.83 (m, 5+1H); MS *m/e* 268 (M^+). **2d**: IR (neat) 1605, 1245, and 1080—950 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.62—1.75 (m, 14H), 3.19 (dt, 4H, *J*=6.5 and 6.5 Hz), 5.70 (dd, 1H, *J*=14.0 and 15.0 Hz), 6.69—7.83 (m, 5+1H); MS *m/e* 296 (M^+).

Diethyl 4-Phenylbutadienylphosphonate (3). The butadienylphosphonate **3** was similarly prepared by the palladium-catalyzed reaction of 1-bromo-4-phenylbutadiene with *O,O*-diethyl phosphonate in the presence of triethylamine. The *E*:*Z* ratio could not be determined.[19]

Diethyl 3-Methyl-2-butenylphosphonate (4). The mixture of isoprene (1.70 g, 25 mmol), *O,O*-diethyl phosphonate (3.45 g, 25 mmol), 1,2-bis(diphenylphosphino)ethane (0.10 g, 0.25 mmol), and tetrakis(triphenylphosphine)palladium (0.29 g, 0.25 mmol) in a sealed tube was heated at 150 °C for 20 h. After evaporation of unreacted isoprene, **4** was isolated in 10% yield by preparative GLC. The structure of **4** was confirmed by the ^1H NMR spectrum.[20]

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