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Reactions of Triarylphosphines with Organolithium Reagents. Formation of Biaryls

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ABSTRACT

Triarylphosphines bearing 2-pyridyl or 2-benzothiazyl groups react with organolithium reagents at −78°C in THF to afford both coupling products and ligand exchanged triarylphosphines. The ligand coupling and ligand exchange are considered to take place via an intermediate formed by the nucleophilic attack of a lithium reagent on the phosphorus atom.

INTRODUCTION

A variety of tertiary phosphines has been synthesized by the reactions of phosphorus trichloride with organometallic reagents. Moore and Whitesides reported that tris(2-benzothiazyl)phosphine was not obtained by treatment of 2-benzothiazyllithium with phosphorus trichloride [1]. However, they did not mention anything about the reaction products actually formed. In the previous article, we have reported the reaction of phosphorus trichloride with 2-benzothiazyllithium in THF, in which tris(2-benzothiazyl)phosphine, initially formed, further reacts with the lithium reagent to give a coupling product, 2,2′-bibenzothiazyl, as the major product [2]. A similar coupling reaction had accidentally been found earlier by Parks et al. in the reaction of phosphorus trichloride with 2-pyridyllithium, in which a small amount of 2,2′-bipyridyl was formed, together with tris(2-pyridyl)phosphine (**1**) [3]. In our active search for ligand coupling reactions, we have examined the reaction of **1** with 2-pyridyllithium and found the reaction to give 2,2′-bipyridyl in a good yield [2].

We have also found that ligand coupling took place in the reactions of phosphine oxides bearing two or three 2-pyridyl groups with organometallic reagents [4]. Even by the treatment of phosphine oxides or phosphonium salts [5,6] bearing two 2-pyridyl groups with dilute hydrochloric acid, 2,2′-bipyridyl was formed in good yields.

These coupling reactions have been considered to proceed by attack of a nucleophile, i.e., an organometallic reagent or even water, on the phosphorus atom to form initially a pentacovalant intermediate. Within the intermediate, an equatorial ligand couples with an axial group to yield the coupling product. In this article, we report the reactions of triarylphosphines which contain 2-pyridyl or 2-benzothiazyl groups with some organometallic reagents.

RESULTS AND DISCUSSION

Triarylphosphines Bearing the 2-Pyridyl Group(s)

When tris(2-pyridyl)phosphine (**1**) was treated with an equimolar amount of phenyllithium in THF at 5°C, ligand exchanged products, phenylbis(2-pyridyl)phosphine (**2a**) and pyridine, and two coupling products, 2,2′-bipyridyl and 2-phenylpyridine, were formed. Upon treatment of **1** with two equivalents of phenyllithium at 5°C, the exchanged phosphine

This article is dedicated to Prof. Antonino Fava on the occasion of his seventieth birthday.

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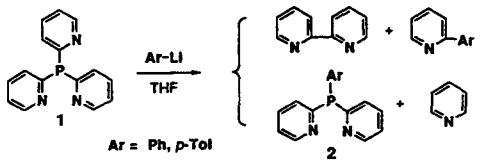
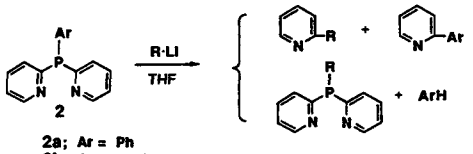
(**2a**) was not obtained, but the yield of 2-phenylpyridine increased markedly, while the yield of 2,2'-bipyridyl decreased. The reaction with phenyllithium proceeded even at −78°C and afforded the exchanged phosphine **2a** and 2,2'-bipyridyl plus a trace of 2-phenylpyridine (Equation 1). A similar result was obtained from the reaction of **1** with *p*-tolyllithium. The results are summarized in Table 1.



(1)

These results suggest that the exchanged phosphine (**2a**) also would undergo a similar coupling reaction with the lithium reagents. Therefore, we have examined the reactions of **2a**, bis(2-pyridyl)-*p*-tolylphosphine (**2b**), and *p*-chlorophenylbis(2-pyridyl)phosphine (**2c**) with 0.8 equivalent of alkyl or aryllithium reagents in THF at −78°C. The results are summarized in Table 2.

The reaction of **2a** with methyllithium proceeded rapidly and was complete within 30 minutes to give 2-phenylpyridine in a good yield; however, 2,2'-bipyridyl was not found in the reaction mixture. In the reaction of **2a** with phenyllithium, the only coupling product detected was 2-phenylpyridine. The reaction with *p*-tolyllithium gave 2-phenylpyridine, together with a small amount of 2-(*p*-tolyl)pyridine, as the coupling product. A ligand exchanged phosphine **2b** was formed, but no phenyl(2-pyridyl)(*p*-tolyl)phosphine, which would have resulted from exchange of the 2-pyridyl group of **2a** with a *p*-tolyl group, was formed in this reaction (Equation 2).



(2)

The reaction of **2a** with *p*-chlorophenyllithium afforded mainly 2-(*p*-chlorophenyl)pyridine as the

TABLE 1 Reaction of Tris(2-pyridyl)phosphine (**1**) with Phenyllithium or *p*-Tolyllithium

ArLi *Ar*	*Ratio* *ArLi/Py_3P*	*Reaction Temperature* (°C)	*Product Yield* (%)[a]				*Recovery* (%)
			Py_2PAr	*Py-Py*	*Py-Ar*	*Py-H*	*Py_3P*
Ph	1.0	5	33	46	9	52	5
Ph	2.0	5	0	7	52	62	0
Ph	0.5	−78	57[b]	9[b]	trace	31[b]	79
Ph	1.0	−78	77	21	trace	91	trace
p-Tol	0.8	−78	43[b]	38[b]	trace	85[b]	9

[a]Based on **1**.
[b]Based on the lithium reagent.

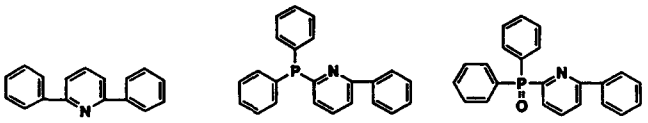
TABLE 2 Reactions of Arylbis(2-pyridyl)phosphines (**2**) with Alkyl- or Aryllithium Reagents at −78°C

Py_2PAr *Ar*	*RLi* *R*	*Reaction Time*	*Product Yield* (%)[a]				*Recovery* (%)
			Py_2PR	*Py-Ar*	*Py-R*	*Ar-H*	*Py_2PAr*
Ph	Me	30 min	[b]	80	0	trace	4
Ph	Ph	1 h	—	76	—	—	43
Ph	*p*-Tol	5 min	41	48	15	13	14
Ph	*p*-Tol	1 h	39	53	15	10	14
Ph	*p*-ClC_6H_4	2 h	trace	8	65	trace	40
p-Tol	Me	30 min	[b]	65	0	[b]	21
p-Tol	Ph	5 min	9	30	39	16	35
p-Tol	Ph	1 h	8	33	43	16	32
p-Tol	*p*-Tol	1 h	—	59	—	—	37
p-ClC_6H_4	Me	30 s	[b]	56	0	trace	28
p-ClC_6H_4	Me	20 min	[b]	80	0	trace	6
p-ClC_6H_4	Ph	1 h	45	70	3	21	trace
p-ClC_6H_4	Ph	2 h[c]	11	38	18	24	3
p-ClC_6H_4	*p*-Tol	1 h	64	54	trace	68	trace
p-ClC_6H_4	*p*-Tol	2 h[c]	15	46	14	23	2
p-ClC_6H_4	*p*-ClC_6H_4	2 h	—	86	—	—	38

[a]Based on the lithium reagent.
[b]Not determined.
[c]Reaction quenched after the mixture had been warmed to room temperature.

coupling product. A similar result was obtained in the reaction of **2b** with the other lithium reagents. For the reactions of **2c** with the various lithium reagents, the major coupling product was 2-(*p*-chlorophenyl)pyridine. For the reactions of **2** with lithium reagents, the results given in Table 2 show that the pyridyl group of **2** does not couple with another pyridyl group or with a methyl group but does couple with a phenyl group; furthermore, a phenyl group which has an electron-withdrawing substituent, e.g., a *p*-chlorophenyl group, preferentially couples with the 2-pyridyl group.

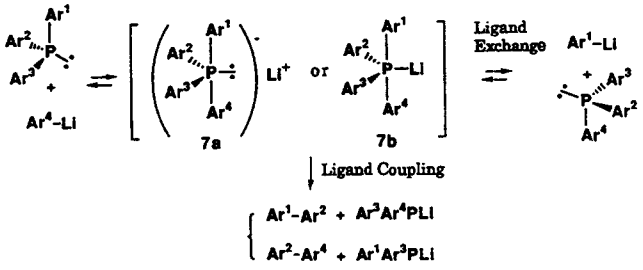
When the reaction of diphenyl(2-pyridyl)phosphine (**3**) with phenyllithium was carried out at −78°C, the starting phosphine was recovered quantitatively. However, when the reaction was carried out with an equivalent of phenyllithium at room temperature, reaction proceeded to the extent of 46% after 15 hours and diphenyl(6-phenylpyrid-2-yl)phosphine (**5**) (14%) was formed, together with 2-phenylpyridine (7%) and trace amounts of 2,6-diphenylpyridine (**4**) and triphenylphosphine, but the amount of diphenylphosphine was not determined. The phenylated phosphine (**5**) was sensitive to oxygen, and it was partially oxidized to the corresponding phosphine oxide during workup. Therefore, **5** was isolated as diphenyl(6-phenylpyrid-2-yl)phosphine oxide (**6**) by treatment of the initial product with hydrogen peroxide in acetone. The structure of **6** was confirmed by comparing its physical properties with those of an authentic sample prepared by the reaction of 6-phenylpyrid-2-yllithium with chlorodiphenylphosphine and subsequent oxidation.



4 **5** **6**

There are two possible routes for the formation of the phenylated pyridine **4**. Thus, **4** could conceivably be formed by the ligand coupling reaction of **5** with phenyllithium. However, this possibility was ruled out because the reaction of **5** with phenyllithium did not proceed under the same conditions. An alternative route leading to **4** could involve the reaction of initially formed 2-phenylpyridine with phenyllithium. We examined independently the reaction of 2-phenylpyridine with an equimolar amount of phenyllithium in THF at room temperature, and we found that 2-phenylpyridine easily reacts with phenyllithium, yielding **4**.

Similar ligand coupling and ligand exchange reactions have been considered to proceed through hypervalent intermediates in the reaction of sulfoxides with organometallic reagents [7–11]. In the recent ^{1}H and ^{13}C NMR studies by Ogawa et al., direct evidence for the formation of tetraphenylsulfurane [12] or tetraarylselenuranes [13] was obtained in the reactions of triphenylsulfonium hexafluorophosphate and diphenyl sulfoxide with phenyllithium at −105°C and also in the reactions of a triphenylselenonium salt or a 2,2′-biphenylenephenylselenonium salt, respectively, with phenyllithium at −100°C. Ogawa et al. showed that the tetraphenylsulfurane and the tetraarylselenuranes, respectively, decomposed to the ligand coupling products in good yields when warmed to room temperature.



SCHEME 1

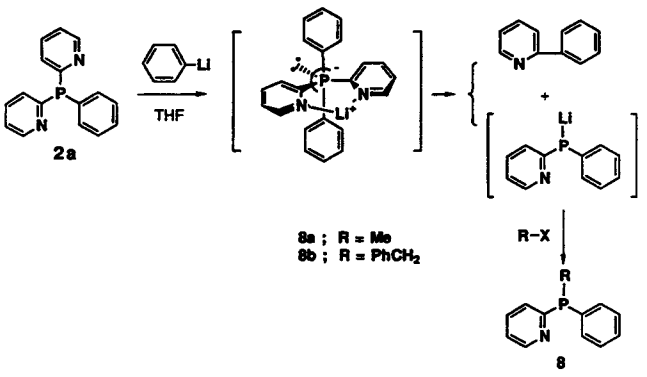
Recently, 2-thienyllithium was found to react with thionyl chloride [14], forming 2,2′-bithienyl in an excellent yield. Even phenylethynyllithium was found to react with thionyl chloride, similarly giving the corresponding coupling product [15]. All these reactions can be classified as ligand couplings.

In the reactions of triarylphosphines with aryl or alkyl lithium reagents, both the ligand coupling and ligand exchange reactions are considered to proceed through an ate complex (**7a**) or a pentacoordinated phosphorus intermediate (**7b**) formed by nucleophilic attack of the lithium reagent on the phosphorus atom, as shown in Scheme 1.

Within the appropriate intermediate, the equatorial ligand would couple with an axial group to yield a 2-substituted pyridine and the lithium diarylphosphide. On the other hand, a ligand exchange product would be formed by the departure of an axial group from the intermediate.

When the reaction of **2a** with phenyllithium was carried out and subsequently the reaction mixture was treated with methyl iodide or benzyl bromide, methylphenyl(2-pyridyl)phosphine (**8a**) and benzylphenyl(2-pyridyl)phosphine (**8b**), respectively, were formed in addition to the coupling and ligand exchange products. These phosphines were very sensitive to oxygen and underwent oxidation during the workup to give the corresponding phosphine oxides. These results obviously show that lithium phenyl(2-pyridyl)phosphide is formed as the phosphorus residue in the coupling reaction which then reacts with the halides to yield the corresponding phosphines, as shown in Scheme 2.

In our previous articles, we reported that a 2-pyridyl group easily couples with another 2-pyridyl group to form 2,2′-bipyridyl by similar ligand



SCHEME 2

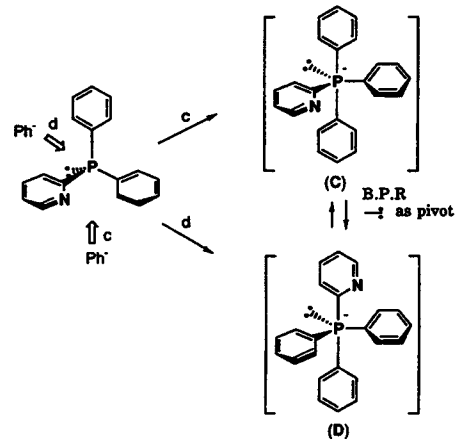


FIGURE 2

coupling reactions on either sulfur [7] or phosphorus atoms [4–6] in appropriate substrates. In the reaction of **1** with an equimolar amount of phenyllithium, the main coupling product was 2,2′-bipyridyl, as mentioned earlier.

It is very interesting that no 2,2′-bipyridyl was formed in the reaction of phosphines (**2**) bearing two pyridyl groups with either an aryl or alkyl lithium reagent. For the reaction of **2** with an aryl or alkyl lithium reagent, attack of the nucleophile on the phosphorus atom would give two different possible intermediates having trigonal bipyramidal (TBP) geometry, depending on the direction of approach of the nucleophile, as shown in Figure 1. If one assumes that, in the reaction of **2a** with phenyllithium, the phenyl group approaches and enters from the side opposite another phenyl group of the phosphine (attack a), a TBP intermediate (**A**) with two phenyl groups at the axial positions and two pyridyl groups at the equatorial positions would be formed. The intermediate (**A**) could then give only 2-phenylpyridine as the coupling product, provided that the coupling proceeds much faster than pseudorotation.

Another TBP intermediate (**B**), with one phenyl group and one pyridyl group in the axial positions, may be formed by attack of the nucleophile at the side opposite either pyridyl group (attack b). The intermediate (**B**) could give two coupling products, i.e., 2,2′-bipyridyl and 2-phenylpyridine, if one assumes that the coupling proceeds much faster than pseudorotation.

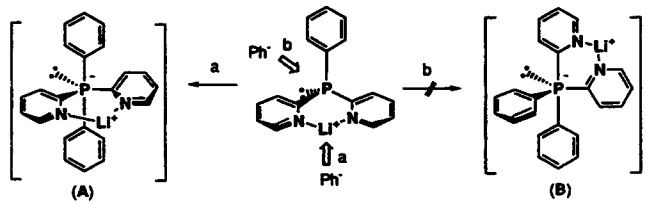


FIGURE 1

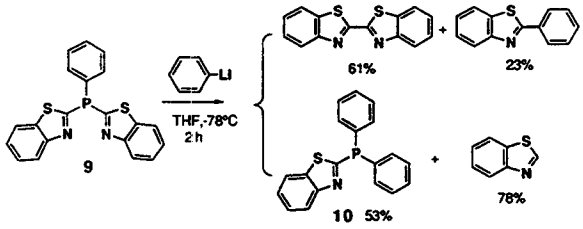
The latter (attack b) can therefore be excluded from further consideration, since both 2,2′-bipyridyl and diphenyl(2-pyridyl)phosphine **3** were not formed in the reaction of **2a** with phenyllithium. Since lithium ion probably chelates with the nitrogen atoms of both pyridyl rings, the axial attack (b) by Ph^-, to form a TBP intermediate (**B**), would be more unfavorable than the axial attack (a) because it requires the placement of one chelated pyridyl ring at an axial and the other at an equatorial position. Although a pyridyl group is more electron-withdrawing than a phenyl group (i.e., a pyridyl group has greater apicophilicity), conversion from the intermediate (**A**) to (**B**) by a pseudorotation process is very unlikely because of the high energy requirement. Thus, the chelated pyridyl rings would both be located at equatorial positions in the TBP (**A**). Hence, the one pyridyl group would have no chance to couple with the other 2-pyridyl group within the intermediate (**A**), and the exchanged phosphine (**3**) would not be formed in the reaction of **2a** with phenyllithium.

On the other hand, for the reaction of the triarylphosphine (**3**) with one pyridyl group attached to the phosphorus atom, the nucleophilic attack on the phosphorus atom results in the formation of two possible TBP intermediates (**C**) and (**D**), depending on the direction of approach of the nucleophile, as shown Figure 2. In this case, however, both directions of nucleophilic approach, i.e., (c) and (d), are possible, because the formed TBP intermediates do not form any chelate ring, as mentioned for **2**. Both attacks would lead to ligand coupling or ligand exchanged products, because interconversion of (**C**) $\rightleftharpoons$ (**D**) can be attained by only one Berry pseudorotation with a lone electron pair (or a P–Li bond) as pivot.

Triarylphosphines Bearing the 2-Benzothiazyl Group(s)

The reaction of tris(2-benzothiazyl)phosphine with phenyllithium has been found to involve both cou-

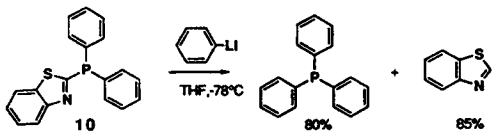
pling and exchange reactions to give 2,2'-bibenzothiazyl, 2-phenylbenzothiazole, and benzothiazole [2]. In this study, we have examined the reactions of bis(2-benzothiazyl)phenylphosphine (**9**) or 2-benzothiazyldiphenylphosphine (**10**) with phenyllithium in THF. When the reaction of **9** with 0.8 equivalent of phenyllithium in THF at −78°C was carried out, both ligand coupling and ligand exchange reactions were found to take place concomitantly, giving 2,2'-bibenzothiazyl (61%), 2-phenylbenzothiazole (23%), **10** (53%), and benzothiazole (78%), as shown in Equation 3.



(3)

The mode of the reaction of **9** is different from that of the phosphines bearing two pyridyl groups mentioned earlier, in which 2,2'-bipyridyl was not formed but only a 2-substituted pyridine as the coupling product. In this reaction, both 2,2'-bibenzothiazyl and 2-phenylbenzothiazole were formed as the coupling products. Furthermore, this reaction was found to yield **10** by ligand exchange of the 2-benzothiazyl group of **9** with a phenyl group. This can be explained in terms of the lack of chelating ability of the two nitrogen atoms of the benzothiazyl rings with lithium ion. There would be no necessity to locate two 2-benzothiazyl groups at the equatorial positions of the constrained TBP intermediate formed by the nucleophilic attack of phenyl anion on the phosphorus atom of **9**. The 2-benzothiazyl group, being a better electron-withdrawing group and having a better leaving ability than a phenyl group, can be placed in the favored axial position of the TBP intermediate. Hence, the intermediate can lead to the formation of the two observed coupling products and the exchanged phosphine **10**.

For the reaction of **10** with phenyllithium, only the exchange products, triphenylphosphine (80%) and benzothiazole (85%), were obtained (Equation 4).



(4)

Since the 2-benzothiazyl group is strongly electron-withdrawing and hence apicophilic, it is quite natural that the reaction gives benzothiazole as the ligand exchange product. The reason why the reaction did not give any ligand coupling product is probably due to the short life of the intermediate hypervalent species, unlike the former case in which there are at least two 2-benzothiazyl groups in the intermediate hypervalent species.

EXPERIMENTAL

General

All melting points are uncorrected. Mass spectra were recorded on a JEOL JMS-AX505W spectrometer at 70 eV. High-performance liquid chromatography (HPLC) was performed on a Shimazdu LC-5A instrument.

Triarylphosphines

Triarylphosphines bearing the 2-pyridyl group(s), i.e., **1**, **2**, and **3**, were prepared by treating phosphorus trichloride, the corresponding aryldichlorophosphines, or diphenylchlorophosphine, respectively, with 2-pyridyllithium according to the previous procedures [6].

1: mp 113–114°C (Ref. [16], mp 114°C); MS *m/z* (*rel* intensity) 265 (M^+, 15), 188 (12), 187 (100), 186 (21).

2a: mp 94–95°C (Ref. [17], mp 96°C); MS *m/z* (*rel* intensity) 265 (12), 264 (M^+, 65), 236 (22), 187 (44), 186 (60), 185 (100), 107 (22), 78 (29).

2b: mp 86–87°C (Ref. [6], mp 86.5–87.5°C); MS *m/z* (*rel* intensity) 279 (15), 278 (M^+, 74), 277 (20), 201 (19), 200 (51), 199 (100), 198 (18), 187 (27), 185 (31), 109 (16).

2c: mp 103–104°C; MS *m/z* (*rel* intensity) 300 ($(M + 2)^+$, 32), 299 (26), 298 (M+, 89), 297 (26), 222 (23), 221 (44), 220 (63), 219 (100), 187 (66), 186 (24), 185 (47), 184 (16), 109 (14). Anal. calcd for $C_{16}H_{12}ClN_2P$: C, 64.34; H, 4.05; N. 9.38. Found: C, 64.44; H, 4.03; N, 9.29.

3a: mp 85–86°C (Ref. [17], mp 84–85°C); MS *m/z* (*rel* intensity) 264 (17), 263 (M^+, 97), 262 (100), 186 (16), 185 (30), 183 (23)

Triarylphosphines bearing 2-benzothiazyl group(s) **9** and **10** were prepared by the method of Moore and Whitesides [1] from phosphorus trichloride, dichlorophenylphosphine, or chlorodiphenylphosphine, respectively, with 2-trimethylsilylbenzothiazole.

9: mp 128–129°C (Ref. [1], mp 127–128°C); MS (70 eV) *m/z* (*rel* intensity) 376 (M^+, 48), 299 (100), 241 (56).

10: mp 86.5–87.5°C (Ref. [1], mp 87–88°C); MS (70 eV) *m/z* (*rel* intensity) 319 (M^+, 100), 318 (52), 242 (14), 183 (37).

Preparation of Diphenyl(6-phenylpyrid-2-yl)phosphine Oxide (**5**)

To a solution of 2-bromo-6-phenylpyridine (4.75 g, 20 mmol) in dry diethyl ether (130 mL) was added dropwise a solution of butyllithium (20 mmol) in hexane (1.6 N, 12 mL) with stirring at −40°C under

argon atmosphere. After addition of a solution of dichlorophenylphosphine (4.41 g, 20 mmol) in diethyl ether (40 mL), the mixture was stirred for 15 hours at −40°C and dilute sulfuric acid (1.5 N, 150 mL) was added to the reaction mixture with cooling. The ethereal layer was washed with water, dried over Na_2SO_4, and then the solvent was evaporated. The residue was dissolved in methanol (30 mL), and oxygen was bubbled through the solution for 1 hour. After evaporation of the solvent, the crude product was chromatographed (silica-gel/ethyl acetate) to give diphenyl(6-phenylpyrid-2-yl)phosphine oxide (3.20 g, 45%) which was recrystallized from methanol; mp 159–160°C. MS *m/z* (*rel* intensity); 356 ($(M + 1)^+$, 21), 355 (M^+, 89), 354 (100), 279 (12), 278 (55), 230 (13), 201 (11), 77 (11). Anal. calcd for $C_{23}H_{18}NOP$: C, 77.74; H, 5.11; N, 3.94. Found: C, 77.73; H, 4.95; N, 3.94.

General Procedure for the Reaction of Triarylphosphines Bearing the 2-Pyridyl Group(s) with Lithium Reagents

A typical procedure for the reaction of a triarylphosphine bearing one or more 2-pyridyl groups, with organolithium reagents, is as follows. To a solution of **2a** (264 mg, 1 mmol) in dry THF (40 mL) was added dropwise a solution of phenyllithium (0.8 mmol) in diethyl ether (3 mL) with stirring at −78°C under an argon atmosphere. After having been stirred for 2 hours at −78°C, water (0.5 mL) was added to the reaction mixture, together with acenaphthene (100 mg) as an internal standard. The reaction mixture was filtered, and a portion of the filtrate was subjected to GC and HPLC analyses.

Isolation of the Products from Reaction of Diphenyl(2-pyridyl)phosphine (**3**) *with Phenyllithium*

To a stirred solution of diphenyl(2-pyridyl)phosphine (**3**) (5.26 g, 20 mmol) in dry THF (80 mL) was added a solution of phenyllithium (20 mmol) in diethyl ether (4.5 mL) at room temperature under an argon atmosphere, and stirring was maintained for 12 hours. Water (1 mL) was added to the reaction mixture, and the solvent was evaporated under reduced pressure. The residue was dissolved in acetone (30 mL), and 35% hydrogen peroxide (0.5 mL) was added to the solution. After 1 hour, acetone was evaporated under reduced pressure and the residue was chromatographed on alumina with ethyl acetate as the eluent. The first fraction contained 2-phenylpyridine and 2,6-diphenylpyridine (0.03 g) which were purified by column chromatography (silica-gel/dichloromethane). The second fraction gave diphenyl(6-phenylpyrid-2-yl)phosphine oxide (0.72 g, 10%) which was recrystallized from methanol; mp 159–160°C.

Reaction of Phosphines Bearing the 2-Benzothiazyl Group(s) with Lithium Reagents

A typical procedure is as follows. To a solution of bis(2-benzothiazyl)phenylphosphine (**9**) (376 mg, 1 mmol) in dry THF (40 mL) was added dropwise a solution of phenyllithium (0.8 mmol) in diethyl ether (3 mL) with stirring at −78°C under an argon atmosphere. After having been stirred for 2 hours at −78°C, water (0.5 mL) was added to the reaction mixture, together with acenaphthene (100 mg) as an internal standard. A portion of the mixture was subjected to HPLC analysis. The solvent was evaporated, and diethyl ether (30 mL) was added to the residue. The precipitated 2,2′-bibenzothiazyl was filtered off and washed with water and diethyl ether.

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