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Bis(pyridyl)-silane and -methanol ligands

IV *. Catalytic application of nickel(II) complexes in the Kumada cross-coupling reaction

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Abstract

Treatment of the bis(2-pyridyl)silane ligands $MeRSi(2\text{-}C_5H_4N)_2$ with $(MeCN)_2NiBr_2$ afforded $\{MeRSi(2\text{-}C_5H_4N)_2\}NiBr_2$ (**3a**, R = Me; **3b**, R = Ph) in excellent yield. The latter complexes were bright purple crystalline solids with paramagnetic line broadening in the NMR spectra; although, no ESR signal could be detected in solution or in the solid state. Complex **3a** was found to be an effective catalyst for the cross-coupling of Grignard reagents with aryl and vinyl halides. The catalytic system also coupled alkyl Grignard reagents with chlorobenzene provided a dipolar aprotic solvent such as HMPA was employed. Optically pure ligands, {6-alkoxy-2-pyridyl}(Ar)CH(OMe) (**5**), were employed in the asymmetric cross-coupling reaction of (1-phenylethyl)magnesium chloride and bromoethene. Conversion of the Grignard reagent ranged from 73 to 88% with ~ 50% chemical yields of 3-phenyl-1-butene with optical yields ranging from ~ 0 to 11%.

Introduction

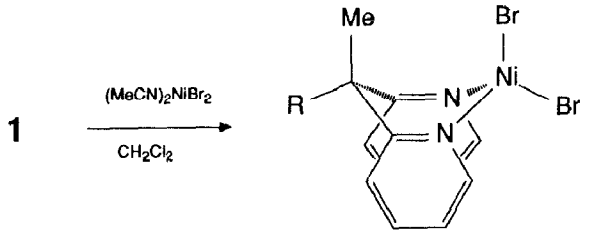
Recently we reported the synthesis of a new class of chelating ligands of achiral bis(2-pyridyl)silanes (**1**) [1a] and chiral bis(6-alkoxy-2-pyridyl)methanols (**2**) [1b] and demonstrated these ligands formed stable 1/1 complexes with $PdCl_2$. Although some analogs of the achiral bis(2-pyridyl)CR(OH) ligands have been reported [2], the catalytic aspects of the ligand-transition metal systems has received little attention [3]. In this paper we present the synthesis of two nickel(II) complexes **3** from our bis(2-pyridyl)silane (**1**) ligands and report on their catalytic activity in the Kumada cross-coupling reaction [4]. In addition, the optically active ligands {6-al-

* For part III see ref. 1b.

koxy-2-pyridyl}(Ar)CH(OMe) (**5**) were prepared and their utility in the asymmetric Kumada cross-coupling reaction is reported.

Results and discussion

Treatment of $(MeCN)_2NiBr_2$ with an excess of ligand **1** in dichloromethane gave nearly quantitative conversion (based on the nickel(II) bromide) to complex **3** (eq. 1). The complexes were isolated as purple powders after precipitation by pentane from the reaction mixture. The complexes were soluble in organic solvents however no NMR data could be obtained due to extreme broadening of the signals. Attempts to detect an ESR signal in solution or in the solid state at ambient temperature were unsuccessful. The lack of an observable ESR signal is not uncommon for the $^3T_1$ ground state configuration [5].



(1)

3a, R = Me

3b, R = Ph

Nickel(II) and palladium(II) complexes containing chelating phosphorus ligands have been used extensively as catalysts for the cross-coupling of Grignard reagents with vinyl or aryl halides [4]. Examples of catalytic systems utilizing chelating nitrogen ligands have been limited to the cross-coupling of aromatic partners for the synthesis of polymers [6]. As noted previously the {2,2′-bipyridine}$NiCl_2$ precatalyst appears to form stable diorganonickel intermediate which shuts down the catalytic cycle [7].

The nickel catalyzed cross-coupling between Grignard reagents and alkyl, vinyl, and aryl halides has been studied utilizing complex **3** as the catalyst (eq. 2), (Table 1). In tetrahydrofuran (THF) chlorobenzene reacts efficiently with the aryl-Grignard reagent but not well with the alkyl-Grignard reagent. This reactivity pattern parallels that reported for $Ni(acac)_2$ in the Kumada cross-coupling reaction [8]. This apparent limitation can be overcome by a change in solvent to a dipolar aprotic solvent hexamethylphosphoramide (HMPA). Dipolar aprotic solvents have been shown to decrease the energy of activation for reductive-elimination processes [9]; therefore, the previously stable diorganonickel complex now undergoes reductive-elimination to regenerate the active catalytic species. Complex **3** still remains less efficient than the typical and versatile Kumada catalyst (dppp)$NiCl_2$.

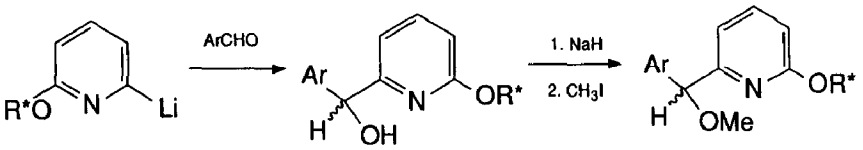
$$RMgX + RX' \xrightarrow[-10\,^{\circ}C]{\text{complex } 3} R\text{–}R' + MgXX' \qquad (2)$$

In an earlier paper we reported the synthesis of the N–N ligands (6-alkoxy-2-pyridyl)(Ar)CH(OH) (**2a**, Ar = 6-borneoxy-2-pyridyl; **2b**, Ar = 2-pyridyl) by treat-

Table 1

Results for Kumada cross-coupling reaction catalyzed by **3**

Catalyst	RMgX	R'X	Solvent	Yield (%)
3a	PhMgBr	PhCl	THF	100
	PhMgBr	BuBr	THF	17
	PhMgBr	BuBr	HMPA	40
	PhMgBr	CH_2=CHBr	THF	72
	BuMgCl	PhCl	ether	~ 0
	BuMgBr	PhCl	THF	5
	BuMgBr	PhCl	HMPA	82
3b	PhMgBr	PhCl	THF	100
	PhMgBr	BuBr	THF	~ 0
	PhMgBr	CH_2=CHBr	THF	46



4; Ar = 2-thiophenyl, OR* = (-)-borneoxy

5

Scheme 1

ment of the appropriate aldehyde with 2-lithio-6-alkoxypyridine [1b]. In a similar manner, treatment of 2-thiophenecarboxaldehyde with 6-fenchoxy-2-lithiopyridine produced the new chiral N–S ligand **4** (Scheme 1). The diastereomeric pair **4(i)** and **4(ii)** (ratio of 4/3, respectively) could be efficiently separated by column chromatography. Carbinols **2** and **4** were converted to their respective methyl ether derivatives **5a–5c** by treatment with sodium hydride and then methyl iodide.

Ligand **5** was employed in the asymmetric Kumada cross-coupling reaction [10]. The chiral Ni catalysts were prepared in situ by treatment of $(MeCN)_2NiBr_2$ with 1

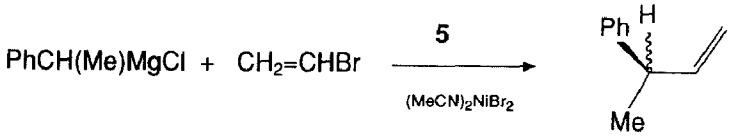
Table 2

Results for the asymmetric Kumada Grignard cross-coupling of 1-phenylethylmagnesium chloride using ligands **5a**,**5b(i)** and **5b(ii)** with $(MeCN)_2NiBr_2$ at $-10\,^{\circ}C$.

Ligand	Ligand/Rh ratio	Conversion (%)	Product [a] (Yield (%))		Optical yield [b] (% ee)
			A	**B**	
5a	1/1	76	46	30	11.4 (*R*)
	2/1	73	52	32	3.8 (*R*)
(5b(i)	1/1	81	52	29	0.3 (*R*)
	2/1	88	56	32	6.2 (*R*)
5b(ii)	2/1	86	50	36	5.2 (*S*)

[a] *A*: 3-phenyl-1-butene *B*: styrene. [b] Configuration of the 3-phenyl-1-butene obtained is given in parentheses after the % ee. Optically pure (*R*)-3-phenyl-1-butene has $[\alpha]^{20}$ $-52.6°$ ($c = 3$, benzene) [10a].

or 2 mol-equiv. of ligand **5** (eq. 3, Table 2). The stoichiometric **5a**/$NiBr_2$ complex exhibited better asymmetric induction than the 2/1 ligand/Ni system. On the other hand, the use of excess ligand was more desirable in the case of using **5b**. These results reveal that the choice of ligand and the ratio of ligand and Ni are quite empirical. Ligand **5c(i)** did not form stable nickel(II) complexes and was found not to be an effective catalyst in the Kumada cross-coupling reaction. A significant amount of styrene was always obtained through competing β-hydride elimination. A solvent change to HMPA resulted in a decrease of both conversion and optical yield. Chemical yields of 3-phenyl-1-butene were ~ 50% and optical yields ranged from 0 to 11.4%. These results would indicate our chiral ligand design affords low optical yields in comparison to the previous results of Kumada and coworkers [4b].



(3)

Experimental

General. All manipulations of compounds and solvents were carried out by standard Schlenk techniques. Solvents were degassed and purified by distillation under nitrogen from standard drying agents [11]. Spectroscopic measurements utilized the following instrumentation: ^1H NMR, Varian XL 300; ^{13}C NMR, Varian XL 300 (at 75.4 MHz); infrared, Perkin Elmer 1750 FT-IR spectrometer. Yields (Table 1) were calculated by GC analysis using naphthalene as an internal standard. GC response factors were determined by averaging three separate runs of purified product samples and naphthalene. The $NiBr_2$, chlorobenzene, phenylmagnesium bromide, n-butylmagnesium bromide, and n-butylmagnesium chloride were purchased from Aldrich Chemical Co. and used as received. The bis(2-pyridyl)silane ligands were prepared by the literature method [1a]. Elemental analyses were performed by Atlantic Microlab Inc., Norcross, Georgia.

{Bis(2-pyridyl)dimethylsilane}nickel(II) bromide (3a). To a solution of **1a** (1.0 g, 4.7 mmol) in CH_2Cl_2 (20 ml) $(MeCN)_2NiBr_2$ (1.2 g, 4.0 mmol) was added. The solution was stirred for an additional hour. The nickel complex was precipitated with pentane (60 ml), filtered, and washed with pentane. The complex was dried under reduced pressure to yield **3a** as a violet powder in quantitative yield. Anal. Found: C, 33.19; H, 3.26; Br, 37.1. $C_{12}H_{14}Br_2N_2NiSi$ calcd.: C, 33.22; H, 3.25; Br, 36.8%.

{Bis(2-pyridyl)methylphenylsilane}nickel(II) bromide (3b). To a solution of **1b** (1.0 g, 3.6 mmol) in CH_2Cl_2 (20 ml) $(MeCN)_2NiBr_2$ (0.81 g, 2.7 mmol) was added. The solution was stirred for an additional hour. The nickel complex was precipitated with pentane (60 ml), filtered, washed with pentane under nitrogen. The complex was dried under reduced pressure to yield **3b** as a violet powder in quantitative yield. Anal. Found: C, 41.59; H, 3.68. $C_{17}H_{16}Br_2N_2NiSi$ calcd.: C, 41.18; H, 3.25%.

Preparation of {(6-[(1R,2R,4S)-1,3,3-trimethyl-2-norbornanoxy]-2-pyridyl)-2-thiophenyl}CH(OH) (4). To a chilled (−78°C) THF (30 ml) solution containing 6-[(*1R,2R,4S*)-1,3,3-trimethyl-2-norbornanoxy]-2-bromopyridine (3.00 g, 9.67 mmol)

was added n-BuLi (3.9 ml, 2.5 *M* solution in hexanes) dropwise by syringe. The mixture was stirred at −78°C for 50 min and then a chilled (−78°C) THF solution (10 ml) containing 2-thiophenecarboxaldehyde (1.08 g, 9.67 mmol) was cannulated into the mixture. The cooling bath was removed and vessel allowed to warm to room temperature over 1.5 h. The mixture was diluted with ether (150 ml), washed with H_2O (120 ml), brine (100 ml), and the organic layer dried over K_2CO_3. The solvents were removed under reduced pressure and the diastereoisomers were separated by chromatography on silica gel (4 × 70 cm) with 10% ethyl acetate in hexanes to afford a 70% yield of **4(i)/4(ii)** (4/3 ratio). **4(i)**: $([\alpha]^{20}$ −37.7° (*c* = 1, CH_2Cl_2)) ^{1}H NMR ($CDCl_3$): δ 7.54 (dd, *J* 8.2, 7.3 Hz, 1H), 7.24 (dd, *J* 5.0, 1.3 Hz, 1H), 7.00 (m, 1 H), 6.95 (dd, *J* 4.9, 3.4 Hz, 1H), 6.76 (d, *J* 7.3 Hz, 1H), 6.68 (d, *J* 8.2, 1H), 5.92 (d, *J* 5.7 Hz, 1H), 4.93 (d, *J* 5.9 Hz, 1H), 4.72 (d, *J* 1.8 Hz, 1H), 2.00 (m, 1H), 1.74 (m, 3H), 1.46 (m, 1H), 1.23 (s, 3H), 1.20 (m, 2H), 1.10 (s, 3H), 0.75 (s, 3H). ^{13}C NMR ($CDCl_3$): δ 164.0, 157.4, 147.9, 139.5, 126.5, 125.4, 124.7, 113.1, 110.3, 87.1, 70.7, 48.9, 48.8, 41.5, 39.9, 30.0, 26.7, 25.8, 20.4, 19.8. **4(ii)**: $([\alpha]^{20}$ 146.3° (*c* = 1, CH_2Cl_2)) ^{1}H NMR ($CDCl_3$): δ 7.53 (dd, *J* 8.3, 7.3 Hz, 1H), 7.26 (dd, *J* 3.7, 1.3 Hz, 1H), 6.99 (m, 1H), 6.95 (dd, *J* 4.9, 3.4 Hz, 1H), 6.77 (d, *J* 7.3 Hz, 1H), 6.69 (d, *J* 8.2, 1H), 5.93 (d, *J* 5.7 Hz, 1H), 4.77 (d, *J* 5.1 Hz, 1H), 4.75 (d, *J* 1.6 Hz, 1H), 2.00 (m, 1H), 1.74 (m, 3H), 1.46 (m, 1H), 1.22 (s, 3H), 1.20 (m, 2H), 1.10 (s, 3H), 0.76 (s, 3H). ^{13}C NMR ($CDCl_3$): δ 164.0, 157.5, 147.3, 139.5, 126.5, 125.5, 125.1, 113.0, 110.4, 87.0, 70.9, 48.9, 48.8, 41.5, 39.9, 30.1, 26.7, 25.9, 20.5, 19.8. Anal. Found: C, 69.87; H, 7.36. $C_{20}H_{25}NO_2S$ calcd.: C, 69.86; H, 7.27%.

{6-Alkoxy-2-pyridyl}{Ar}CH(OMe) (5). A DMF (20 ml) solution containing **2** or **4** (2.0 mmol) was treated with NaH (53 mg, 2.2 mmol) and stirred at 0°C for 15 min. Methyl iodide (0.28 g, 2.0 mmol) was added, the cooling bath removed, and the mixture was allowed to react for 1 h. The mixture was diluted with H_2O (30 ml) and Et_2O (100 ml). The organic layer was separated and washed with H_2O (100 ml), brine (50 ml), and dried over K_2CO_3. The solvents were removed under reduced pressure and the residue was chromatographed on neutral alumina (4 × 7 cm) with gradient elution (0–3% ethyl acetate in hexanes) to afford pure **5** in greater than 93% yield.

Bis[(6-(1S,2R,4S)-borneoxy-2-pyridyl)]CH(OMe) (5a). $([\alpha]^{20}$ −80.7° (*c* = 1, CH_2Cl_2)) ^{1}H NMR ($CDCl_3$); δ 7.42 (dt, *J* 7.9, 3.0 Hz, 2 H), 7.02 (dd, *J* 11.1, 7.3 Hz, 2H), 6.55 (d, *J* 8.2 Hz, 2H), 5.20 (s, 1H), 4.93 (m, 2H), 3.44 (s, 3H), 2.30 (m, 2H), 2.15 (m, 2H), 1.74 (m, 2H), 1.62 (m, 2H), 1.25 (m, 6H), 0.92 (s, 6H), 0.87 (s, 6H), 0.83, 0.80 (s, s, 6H). ^{13}C NMR ($CDCl_3$): δ 163.6, 158.1, 113.8, 113.6, 109.5, 87.4, 80.7, 80.5, 57.3, 48.8, 47.6, 47.5, 44.9, 37.0, 28.1, 27.1, 19.8, 19.0, 13.8. Anal. Found: C, 75.98; H, 8.82. $C_{32}H_{44}N_2O_3$ calcd.: C, 76.08; H, 8.72%.

{(6-[(1R,2R,4S)-1,3,3-Trimethyl-2-norbornanoxy]-2-pyridyl)-2-pyridyl}CH(OMe) (5b(i); 1st diastereomer). $([\alpha]^{20}$ 114.3° (*c* = 1, CH_2Cl_2)) ^{1}H NMR ($CDCl_3$): δ 8.56 (m, 1 H), 7.67 (dt, *J* 7.8, 1.6 Hz, 1H), 7.53 (m, 2H), 7.16 (m, 1H), 7.01 (d, *J* 7.3 Hz, 1H), 6.60 (dd, *J* 4.6, 0.8 Hz, 1H), 5.34 (s, 1H), 4.56 (s, 1H), 3.46 (s, 3 H), 1.95 (m, 1H), 1.63 (m, 3H), 1.45 (m, 1H), 1.11 (s, 3H), 1.09 (m, 2H), 0.99 (s, 3H), 0.68 (s, 3H). ^{13}C NMR ($CDCl_3$): δ 164.3, 160.3, 157.2, 149.9, 138.7, 136.3, 122.3, 121.8, 113.8, 110.1, 87.2, 86.3, 57.3, 48.7, 48.6, 41.4, 39.7, 29.4, 26.7, 25.8, 20.3, 19.6. Anal. Found: C, 74.68; H, 8.05. $C_{22}H_{28}N_2O_2$ calcd.: C, 74.90; H, 7.94%.

{(6-[(1R,2R,4S)-1,3,3-Trimethyl-2-norbornanoxy]-2-pyridyl)-2-pyridyl}CH(OMe) (5b(ii); 2nd diastereomer). $([\alpha]^{20}$ 94.5° (*c* = 1, CH_2Cl_2)) ^{1}H NMR ($CDCl_3$): δ 8.56

(m, 1H), 7.67 (dt, *J* 7.8, 1.6 Hz, 1H), 7.53 (m, 1H), 7.41 (d, *J* 8.1 Hz, 1H), 7.13 (m, 2H), 6.59 (dd, *J* 4.6, 0.8 Hz, 1H), 5.34 (s, 1H), 4.54 (s, 1H), 3.45 (s, 3H), 1.95 (m, 1H), 1.65 (m, 3H), 1.43 (m, 1H), 1.15 (m, 2H), 1.00 (s, 3H), 0.98 (s, 3H), 0.67, 0.52 (s, s, 3H). ^{13}C NMR ($CDCl_3$): δ 164.2, 160.4, 157.2, 148.9, 138.7, 136.3, 122.4, 122.2, 113.1, 110.0, 87.1, 86.3, 57.3, 48.7, 48.7, 41.4, 39.5, 29.0, 26.6, 25.7, 20.0, 19.6. Anal. Found: C, 74.78: H, 7.96. $C_{22}H_{28}N_2O_2$ calcd.: C, 74.90; H, 7.94%.

{(6-[(1R,2R,4S)-1,3,3-Trimethyl-2-norbornanoxy]-2-pyridyl)-2-thiophenyl}-CH-(OMe) (5c(i)). ($[\alpha]^{20}$ 143.0° ($c = 1$, CH_2Cl_2)) ^{1}H NMR ($CDCl_3$): δ 7.55 (t, *J* 7.4 Hz, 1H), 7.24 (dd, *J* 5.0, 1.5 Hz, 1H), 7.04 (d, *J* 7.3 Hz, 1H), 6.95 (m, 2H), 6.62 (d, *J* 7.3 Hz, 1H), 5.43 (s, 1H), 4.61 (s, 1H), 3.45 (s, 3H), 1.96 (m, 1H), 1.70 (m, 3H), 1.45 (m, 1H), 1.25 (s, 3H), 1.16 (m, 2H), 1.04 (s, 3H), 0.75 (s, 3H). ^{13}C NMR ($CDCl_3$): δ 164.2, 158.0, 144.9, 139.0, 126.4, 125.1, 112.2, 110.0, 86.7, 82.5, 57.3, 48.9, 48.7, 41.5, 39.8, 29.6, 26.7, 25.9, 20.5, 19.8. Anal. Found: C, 70.29; H, 7.64. $C_{21}H_{27}NO_2S$ calcd.: C, 70.50; H, 7.55%.

Cross-coupling reactions in THF. To a THF (10 ml) solution of chlorobenzene (1.0 g, 8.9 mmol) was added phenylmagnesium bromide (5.3 ml, 2.0 *M* in THF) in the presence of **3a** (40 mg, 0.09 mmol). The mixture was stirred for 18 h. The mixture was quenched with H_2O (150 ml) and then diluted with Et_2O (100 ml). The organic layer was separated and washed with H_2O (150 ml) and brine (50 ml). The organic layer was dried (K_2CO_3), filtered, and the solvents were removed under reduced pressure. Pure biphenyl (~ 100%) was obtained after flash chromatography on alumina.

Cross-coupling reactions in HMPA. To a solution of chlorobenzene (1.0 g, 8.9 mmol) in HMPA (10 ml) was added n-butylmagnesium bromide (5.8 ml, 2.0 *M* in THF) in the presence of **3a** (40 mg, 0.09 mmol). The mixture was stirred for 18 h. The mixture was quenched with H_2O (150 ml) and then diluted with Et_2O (100 ml). The organic layer was washed with H_2O (2 × 150 ml) and the aqueous layer washed with Et_2O (100 ml). The combined organic layers were washed with brine (100 ml). The organic layer was dried (K_2CO_3), filtered, and the solvents were removed under reduced pressure to give n-butylbenzene in 70% yield after flash chromatography on alumina.

Asymmetric Grignard cross-coupling reaction. 1-Phenylethylmagnesium chloride (1.0 *M*) was prepared in ~ 95% yield by slowly adding an ether solution of 1-phenylethyl chloride to excess magnesium (the magnesium was freshly ground in a mortar and pestle and then heated in the flask under a stream of nitrogen) suspended in ether. The nickel(II) catalyst was generated in CH_2Cl_2 by treatment of **5** (0.10 mmol, for ligand/$NiBr_2$ = 1/1) with $(MeCN)_2NiBr_2$ (33 mg, 0.11 mmol) for 30 min. The solution was filtered (to remove unreacted $NiBr_2$) through Celite into a 50 ml Schlenk tube and the solvent removed under reduced pressure. The vessel was then cooled to −40 °C and the vinyl bromide (2.14 g, 20 mmol) and 1-phenylethylmagnesium chloride (1 *M*, 10 mmol) added. The mixture was allowed to react with stirring at −10 °C for 4 h and then quenched with H_2O. The mixture was diluted with ether (50 ml) and washed with H_2O and brine. The organic layer was dried (K_2CO_3) and the solvents removed under reduced pressure. The crude product was purified by distillation at reduced pressure. The chemical and optical yields were calculated using the proton NMR data and optical rotations of the product mixtures.

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References

1 (a) M.E. Wright, Tetrahedron Lett., 28 (1987) 2322. M.E. Wright and C.K. Lowa-Ma, Organometallics, in press; (b) M.E. Wright, S.A. Svejda, M.J. Jin, and M.A. Peterson, Organometallics, 9 (1990) 136.
2 H.C. Beyerman and J.S. Bontekoe, Res. Trav. Chim., 74 (1955) 1395. J. Murphy and J.W. Bunting, Org. Prep. Proced., Int. 3 (1971) 255.
3 D. Elman and C.J. Moberg, J. Organomet. Chem., 294 (1985) 117.
4 (a) K. Tamao, K. Sumitani, M. Zembayashi, A. Fijioka, S-I. Kodama, I. Nakajima, A. Minato, and M. Kumada, Bull. Chem. Soc. Jpn., 49 (1976) 1958; (b) T. Hayashi and M. Kumada, Acc. Chem. Res., 15 (1982) 395.
5 F.A. Cotton and G. Wilkinson, Advanced Inorganic Chemistry, John Wiley & Sons, New York, 4th edit., 1980, p. 787–788.
6 T. Yamamoto, H. Yasuhiro, and R. Yamamoto, Bull. Chem. Soc. Jpn., 51 (1978) 2091; T. Yamamoto, K. Sanechika, and R. Yamamoto, J. Polym. Sci., Polym. Lett. Ed., 18 (1980) 9; S. Tanaka, M. Sato, 1 K. Kaeriyama, and H. Kanetsuna, Chemical Abstr., 103; 88278.
7 T.A. Uchida, A. Misono, A. Yamamodo, K. Morifuji, and S. Ikeda, J. Am. Chem. Soc., 88 (1966), 5198; G. Wilke and G. Herrmann, Angew. Chem., 78 (1966) 591.
8 R.J.P. Corriu and J.P. Masse, J. Chem. Soc. Chem. Commun., (1972) 144.
9 A. Moravskiy and J.K. Stille, J. Am. Chem. Soc., 103 (1981) 4182.
10 (a) T. Hayashi, M. Konishi, M. Fukushima, T. Mise, M. Kagotani, M. Tajika, and M. Kumada, J. Am. Chem. Soc., 104 (1982) 180. (b) T. Hayashi and M. Kumada, Acc. Chem. Res., 15 (1982) 395.
11 A.J. Gordon and R.A. Ford, The Chemists Companion, Wiley, New York, 1972.