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SHORT COMMUNICATION

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Highly active cobalt complex catalysts used for alkene hydrosilylation

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A series of nitrogen phosphine ligands were synthesized, and the hydrosilylation reaction of alkenes catalyzed using MCl_2 in the presence of these ligands was investigated. $FeCl_2$/**1**(*N*^{1}, *N*^{1}, *N*^{2}, *N*^{2}-Tetrakis[(diphenylphosphino)methyl]ethane-1,2-diamine) showed low catalytic activity. $MnCl_2$/**1,** $CrCl_3$/**1** and $NiCl_2$/**1** showed some catalytic activity. The $CoCl_2$/*N*,*P*-ligand catalyst system showed high activity as well as excellent selectivity (The selectivity of the *β*-adduct was ∼100%.) in the hydrosilylation reaction. $CoCl_2$/**1** showed the highest catalytic activity (∼ >99.9% conversion of 1-octene). Additionally, no α-adduct, dehydrogenative silylation product and octane were detected.

KEYWORDS
alkene, $CoCl_2$, hydrosilylation, phosphine ligands

1 | INTRODUCTION

The hydrosilylation of alkenes has emerged as a powerful method in organic synthesis.[1–3] The catalytic hydrosilylation of alkenes is a valuable transformation because it generates silicon-containing compounds, such as silane coupling agents. Platinum catalysts, such as Speier et al.'s[4] and Karstedt and Scotia's[5] catalysts, which exhibit high catalytic activity, have been widely used in various hydrosilylation reactions. However, platinum catalysts have some drawbacks, such as their high price, low compatibility towards substrates, and the occurrence of side reactions. In addition, many transition metal complexes, such as rhodium, iridium, iron, and cobalt, are known to be catalysts for hydrosilylation of alkenes.[6–9] However, they still possess many drawbacks, such as low catalytic activity and reaction selectivity. Therefore, there has been an increasing demand for cheap metal catalysts with high catalytic activity and reaction selectivity.[10–16] Verhoeven et al.[17] reported the catalytic activity of well-characterized Co (II) and Co(I) complexes bearing *p*-Toldpbp ligands observed in the hydrosilylation of 1-octene using phenylsilane. The cobalt complexes featuring N-heterocyclic carbene, *β*-diketiminato, and 2, 6-diiminopyridine ligands are found effective in catalyzing alkene hydrosilylation reactions. In addition, there are some studies on hydrosilylation reactions catalyzed by cobalt salts.[18–30]

Our group has paid a great deal of attention to cobalt complexes, which are recognized as earth-abundant metal catalysts, which can be used to replace precious metals, for use as catalysts in the hydrosilylation of alkenes. The utility of cobalt complexes as hydrosilylation catalysts has been known for a long time, although most of the examples suffer from low reaction selectivity. In this paper, we report that several cobalt *P*-ligand complex catalysts act as effective alkene hydrosilylation catalysts under mild reaction conditions.

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2 | EXPERIMENT

2.1 | General methods

Styrene was washed with 5% NaOH and dried with Na_2SO_4 and then was distilled under reduced pressure after filtration. All other chemicals were purchased from Aldrich and were used as received.

Gas chromatography (GC): Trace DSQ GC column = DB-5 30 m × 2.5 mm × 0.25 μm, split ratio = 50:1, flow rate = 1 ml min^{-1}, inlet temperature = 260°C, column temperature = 50°C (hold for 1 min) then increased to 260 15°C min^{-1} up C at a heating rate of 15°C min^{-1} up (hold 10 min). GC–MS: Trace DSQ GC–MS column.

^1H, ^{13}C, ^{29}Si and ^{31}P nuclear magnetic resonance (NMR) spectra were measured using a Bruker AV400-MHz spectrometer operating at 400.13, 100.62, 79.49, and 161.97 MHz, respectively.

IR spectra were recorded on a Nicolet 5700 instrument and Elemental analyses were performed on a VARIO EL-3 elemental analyzer.

2.2 | Synthesis of the *N*,*P*-ligands

An oven-dried, 100-ml three-necked flask equipped with a magnetic stir bar, rubber septum, and reflux condenser was charged with toluene (40 ml), followed by diamine (11.0 mmol) or amine (22.0 mmol), and 37% HCHO (5.3 ml 65.0 mmol) under an Ar atmosphere. After stirring for 5 min, the reaction mixture was heated at 60°C, and diphenylphosphine (7.5 ml 43.0 mmol) was added at once. The reaction mixture was heated at 61°C for 1.5–2 h and then cooled to room temperature. The combined organic extracts were dried using anhydrous Na_2SO_4. The filtrate was concentrated in vacuo and the resulting crude product purified by recrystallization from ethanol to give a yellow solid. The ^1H, ^{13}C, and ^{31}P NMR, infrared (IR), and elemental analyses were consistent with those reported in the literature.[15]

2.3 | Catalytic hydrosilylation of alkene with silane

The typical hydrosilylation reaction procedure was as follows: Given amounts of the catalyst and ligand were charged into a 10-ml round bottomed flask equipped with a magnetic stirrer, followed by the alkene and silane. The resulting mixture was heated to the specified temperature, and the hydrosilylation reaction was allowed to proceed with constant stirring for 5 h. At the end of the reaction, the conversion of the alkene and selectivity were determined using GC.

2.4 | X-ray structures of 1, the $NiCl_2$/1 and $CoCl_2$/6 complexes

Colorless single crystals of the compounds (dimensions: N^1,N^1,N^2,N^2-tetrakis[(diphenylphosphino)methyl]ethane-1,2-diamine (**1**): 0.35 × 0.25 × 0.25 mm, Ni (*N*,*N*-bis((diphenylphosphino)methyl)benzenamine)$_2$$Cl_2$ ($NiCl_2$/**6**): 0.31 × 0.35 × 0.23 mm, and CoO(*N*,*N*-bis((diphenylphosphino)methyl)benzenamine)$_2$$Cl_2$ ($CoCl_2$/**6**): 0.39 × 0.34 × 0.25 mm) were produced by slow evaporation of the solvents from a solution of the compounds in hexane/dichloromethane at 25°C. Diffraction data were collected at room temperature using the φ-scan and ω-scan technique on a Smart Apex Duo diffractometer with graphite-monochromated Mo–K_α radiation ($\lambda = 0.71073$ Å). The structures were solved using direct methods (SHELXTL-PLUS)[31] and subsequent Fourier difference syntheses, which were then refined by full-matrix least-squares on F^2 (SHELXL-97).[32] All non-hydrogen atoms were refined anisotropically, while the hydrogen atoms were placed in their geometrically idealized positions. Crystal data and details of the structures are given in Tables 1–3. (Tables 1–3) (Figures 1–3).

TABLE 1 Single crystal structure data of ligand **1**

Empirical formula	$C_{54}H_{52}N_2P_4CH_2Cl_2$
Formula weight	937.78
Temperature (K)	296 (2)
Space group	*P* −1
Unit cell dimensions	
a (Å)	10.062 (4)
b (Å)	10.274 (4)
c (Å)	13.358 (6)
Alpha (deg)	75.163 (10)
Beta (deg)	79.198 (10)
Gamma (deg)	81.614 (10)
Volume (Å^3)	1304.2 (10)
Z, calculated density	1
Abs. coeff. (mm^{-1})	0.284
F(000)	492
Reflections collected/unique	4270/5875[*R* (int) = 0.0505]
Goodness-of-fit on $F^2{}_{\theta max\ 25.2}$	1.006
Final *R* indices[$I > 2\sigma(I)$]	$R_1 = 0.1329, wR_2 = 0.4721$
R indices (all data)	$R_1 = 0.1535, wR_2 = 0.990$

FIGURE 1 Single crystal structure of ligand **1**

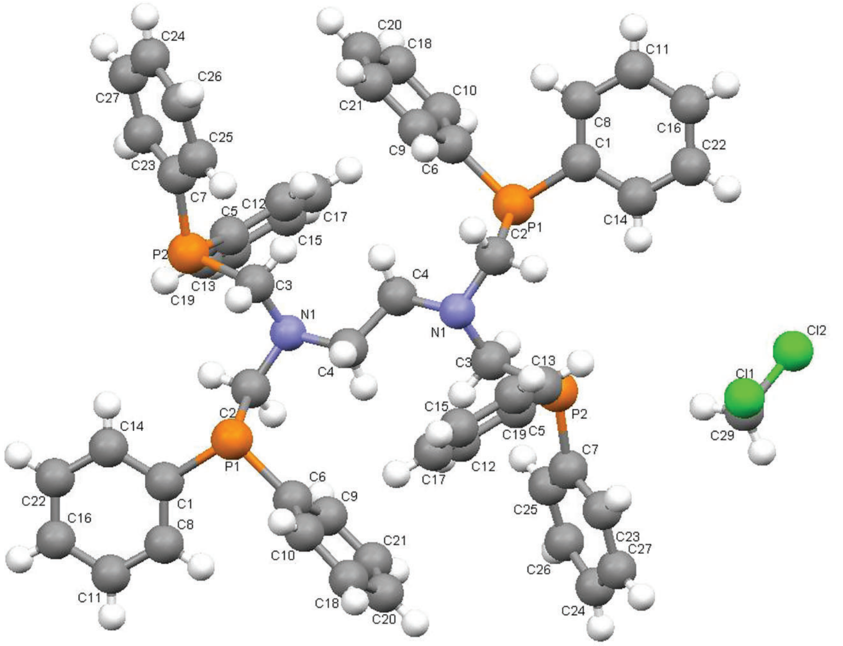


TABLE 2 Single crystal structure data of complex $NiCl_2$/**6**

Empirical formula	$2(C_{32}H_{29}Cl_2NNiP_2),CH_2Cl_2$
Formula weight	2646.22
Temperature (K)	293 (2)
Space group	P21/c
Unit cell dimensions	
a (Å)	9.7297 (14)
b (Å)	13.1974 (18)
c (Å)	26.581 (4)
Alpha (deg)	90
Beta (deg)	106.652 (5)
Gamma (deg)	90
Volume (Å^3)	3270.0 (8)
Z, calculated density	1
Abs. coeff. (mm^{-1})	0.959
F(000)	1364.0
Reflections collected/unique	5079/7445[*R* (int) = 0.0500]
Goodness-of-fit on $F^2{}_{\theta max\ 25.2}$	1.081
Final *R* indices[$I > 2\sigma(I)$]	$R_1 = 0.0826, wR_2 = 0.2651$
R indices (all data)	$R_1 = 0.1250, wR_2 = 1.080$

3 | RESULTS AND DISCUSSION

A series of highly efficient protocols used for the synthesis of *N*,*P*-ligands (**1**–**22**) are shown in Scheme 1. These products (**1**–**22**) were synthesized upon the reaction of an amino compound, formaldehyde, and diphenylphosphine (or dialkylphosphine). *N*,*P*-ligands **1**–**22** were characterized using NMR and IR spectroscopy and elemental analyses. The structure of ligand **1** was also determined using X-ray analysis (Figure 1). Furthermore, the NMR, IR, and elemental analyses data of the compounds were in agreement with their assigned structures.

3.1 | Effect of different metal salts on the hydrosilylation reaction

A series of different transition metal halides/$(CH_2N(CH_2PPh_2)_2)_2$ (**1**) were screened in the hydrosilylation reaction of 1-octene using diphenylsilane. The results were summarized in Table 4. $CuCl_2$/**1**, $ZnCl_2$/**1**, and $TiCl_4$/**1** showed no catalytic activity upon heating at 100°C for 8 h. $FeCl_2$/**1** showed low catalytic activity (~35.5% conversion of 1-octene). $MnCl_2$/**1,** $CrCl_3$/**1**, and $NiCl_2$/**1** showed some catalytic activity. $CoCl_2$/**1** showed the highest catalytic activity (~ >99.9% conversion of 1-octene) under the reaction conditions. Additionally, no α-adduct, dehydrogenative silylation product and octane were detected.

A series of different cobalt salts/**1** were then examined in the hydrosilylation reaction of 1-octene using diphenylsilane. The selectivity of the *β*-adduct catalyzed by CoF_2/**1** or $CoBr_2$/**1** was relatively low (89.1% and 78.9%, respectively). No α-adduct, dehydrogenative silylation product, and octane were detected in the reaction catalyzed by Co $(CH_3COO)_2$/**1**, Co $(BF_4)_2$/**1**, and cobalt 2-ethylhexanoate/**1**, respectively. Although the selectivity of the reaction catalyzed by cobalt salt/**1**

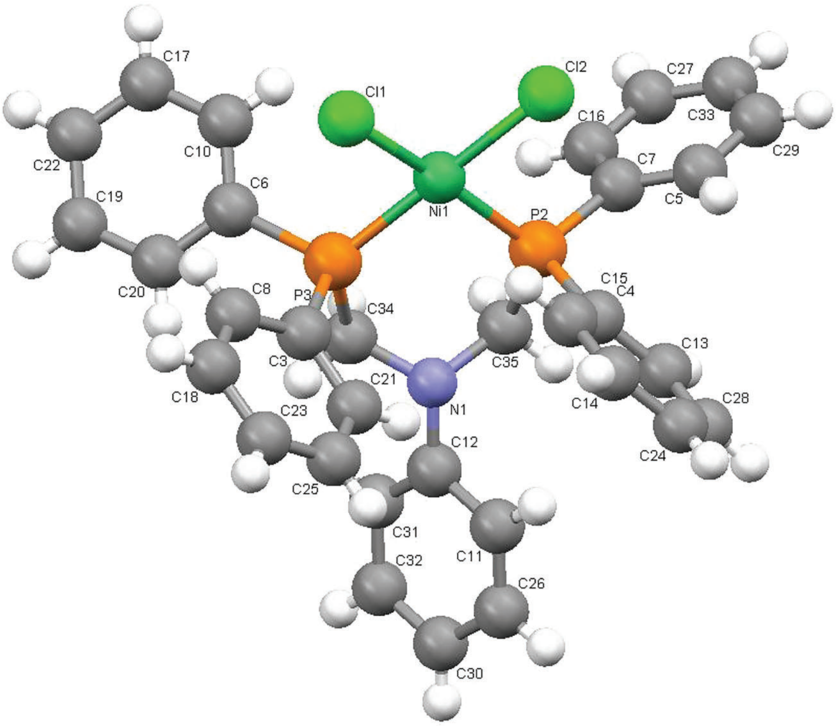


FIGURE 2 Effect of reaction temperature and reaction time on the hydrosilylation reaction of $CoCl_2$/**1** complex

TABLE 3 Single crystal structure data of complex $CoCl_2$/**1**

Empirical formula	$C_{32}H_{29}Cl_2CoNOP_2$
Formula weight	635.33
Temperature (K)	296
Space group	P21/c
Unit cell dimensions	
a (Å)	9.4662 (5)
b (Å)	16.8908 (9)
c (Å)	19.3207 (10)
Alpha (deg)	90
Beta (deg)	103.541 (1)
Gamma (deg)	90
Volume (Å^3)	3003.3 (3)
Z, calculated density	4
Abs. coeff. (mm^{-1})	0.882
F(000)	1308.0
Reflections collected/unique	3513/3877[*R* (int) = 0.5340]
Goodness-of-fit on $F^2{}_{\theta max\ 25.2}$	1.014
Final *R* indices[$I > 2\sigma(I)$]	$R_1 = 0.0456, wR_2 = 0.1237$
R indices (all data)	$R_1 = 0.0763, wR_2 = 1.014$

was better, the conversion of 1-octene was different due to the different counter anions used. The conversion of 1-octene catalyzed by $CoBr_2$/**1** was 50.8%, while in the reactions catalyzed by $CoCl_2$/**1**, Co $(CH_3COO)_2$/**1**, Co $(BF_4)_2$/**1**, and cobalt 2-ethylhexanoate/**1**, the conversion of 1-octene was extremely high (close to 100%). Therefore, after comprehensive consideration, cobalt chloride was chosen as the catalyst used in our subsequent experiments.

3.2 | Effect of the different *N,P*-ligands on the hydrosilylation reaction

The catalytic properties of $CoCl_2$/*N,P*-ligands **1–22** in the hydrosilylation reaction of 1-octene using diphenylsilane were investigated, and the results are listed in Table 5. By comparing the data, it was found that the cobalt catalyst system containing four *P*-ligands presented excellent catalytic performance in terms of both the conversion and *β*-adduct selectivity. However, the product conversion of the cobalt catalyst system containing a single *P*-ligand was generally low; the conversion was only 32.5% when using $CoCl_2$/**15** as the catalyst. At the same time, in the *P*-ligands whose groups directly connected to the nitrogen atoms are phenyl or alkyl groups, the conversion and selectivity of the reaction were better than those observed using *P*-ligands directly connected with a pyridine ring ligand (Entries 6–9 and 15–17, Table 5). Moreover, when the group attached to the nitrogen atom is an alkyl group, the length of the alkyl chain did not affect the activity of the catalyst (Entries 11–13, Table 5). In addition, alkyl phosphine was used to replace

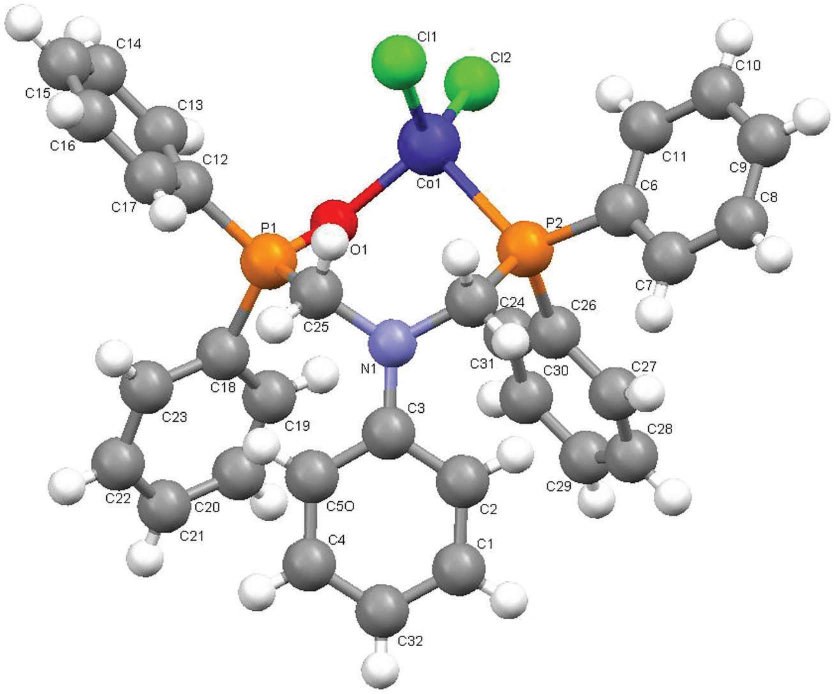
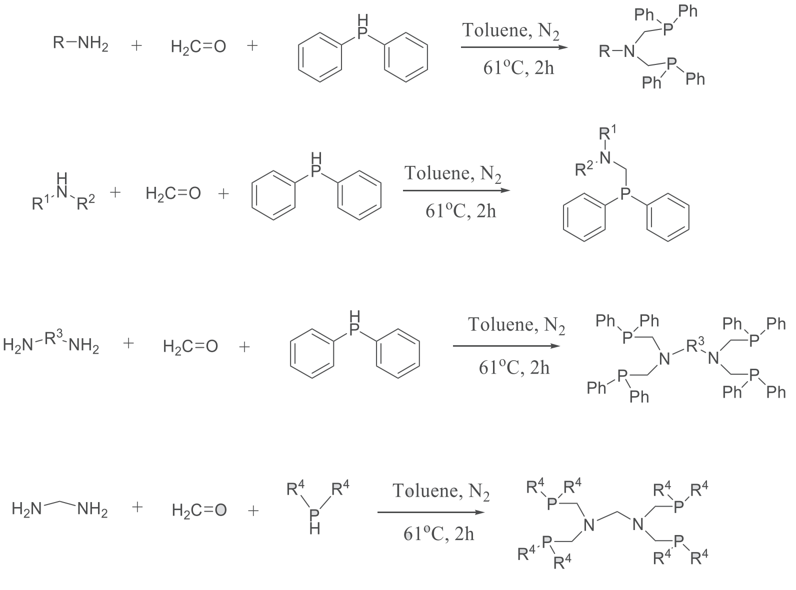


FIGURE 3 Effect of the amount of complex $CoCl_2$/**1** on the hydrosilylation reaction



SCHEME 1 The synthesis of N,P-ligands

R: R= $(CH_2)_2N(Me)_2$; Ph; *o*-Py; *o*-CH_2Py; Bn; *p*-MeO-Ph; ${}^tBu$; C_8H_{17}; $C_{12}H_{25}$;

R^1: Ph; C_6H_{11}; *o*-Py; o-CH_2Py; Bn;

R^2: Ph; C_6H_{11}; *o*-Py; o-CH_2Py; Bn; CH_3;

R^3: C_2H_4; Ph; $C_6H_{10;}$ C_4H_8;

R^4: Cy; o-CH_3OPh; o-ClPh;

diphenylphosphine in the ligand during the reaction of 1-octene using diphenylsilane catalyzed by $CoCl_2$. The conversion of 1-octene was 91.9%, and the selectivity of the *β*-adduct was ~100%. The activity of the catalyst was only a minor affected by the groups (phenyl or alkyl) linked on the phosphine atom (Schemes 2 and 3).

3.3 | The effect of different reaction conditions on the hydrosilylation reaction

The effects of the reaction temperature and time are illustrated in Figure 4. It shows that the conversion of 1-octene increases upon increasing the reaction

TABLE 4 The catalysts consisting of different metal salts/**1** used to catalyze the hydrosilylation reaction of 1-octene using diphenylsilane

			Select. (%)			
Entry	**Catalyst**	**Conv. (%)**	**β**	**α**	**Octane**	**Dehydrogenative silylation**
1	$CoCl_2$/**1**	~100	~100	/	/	/
2	$MnCl_2$/**1**	83	~100	/	/	/
3	$CrCl_3$/**1**	79	~100	/	/	/
4	$NiCl_2$/**1**	72	~100	/	/	/
5	$FeCl_2$/**1**	36	71	/	29	/
6	$ZnCl_2$/**1**	/	/	/	/	/
7	$CuCl_2$/**1**	/	/	/	/	/
8	$TiCl_4$/**1**	/	/	/	/	/
9	CoF_2/**1**	69	89	/	11	/
10	$CoBr_2$/**1**	51	79	/	21	/
11	Co $(CH_3COO)_2$/**1**	~100	~100	/	/	/
12	Co $(BF_4)_2$/**1**	~100	~100	/	/	/
13	$Co(C_{10}H_7COO)_2$/**1**	~100	~100	/	/	/

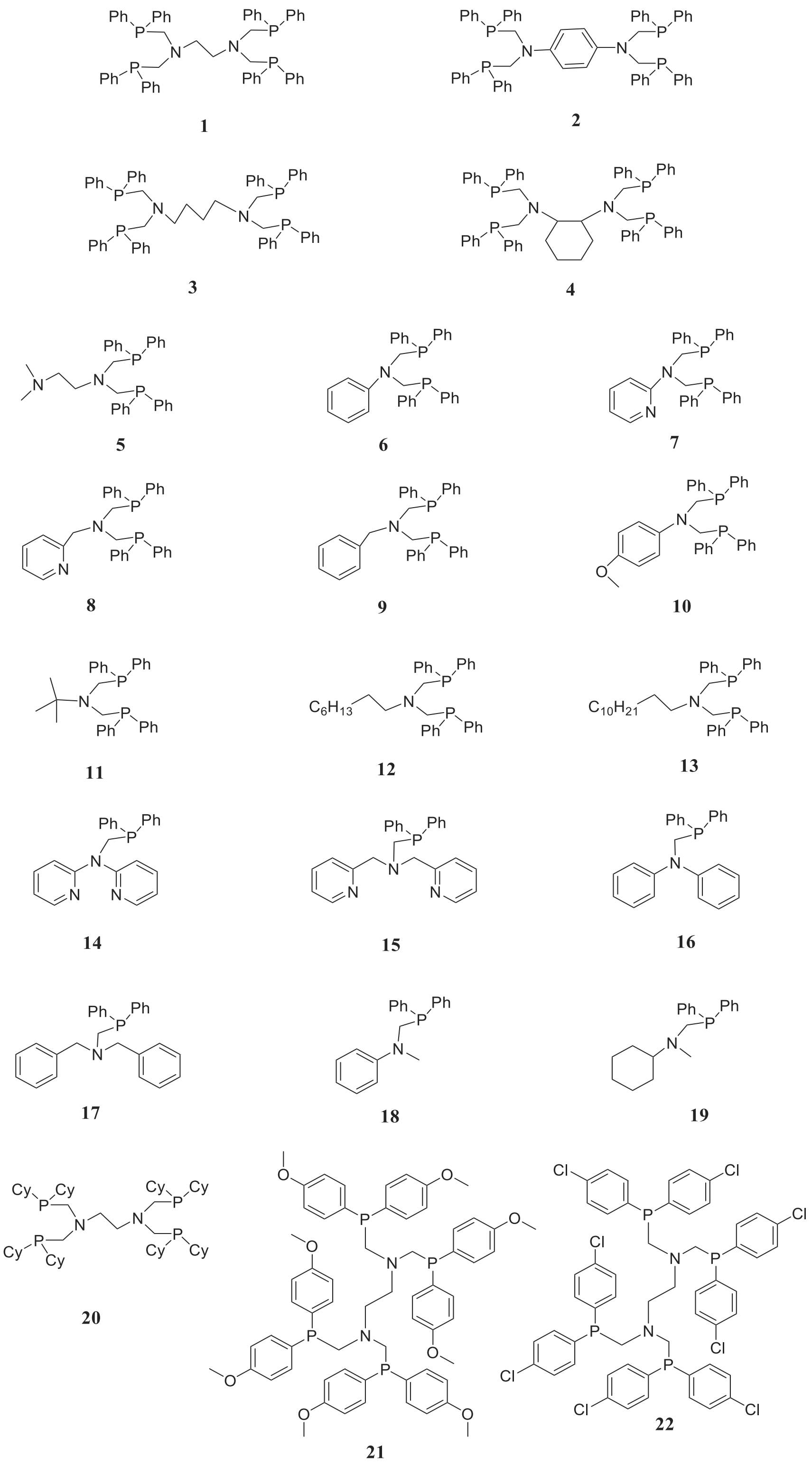
Note: Reaction conditions: 1-Octene (4 mmol), Ph_2SiH_2 (4.4 mmol), catalyst: metal salts/**1** (0.02 mol% with respect to 1-octene), and metal salts:**1** = 1:1, 8 h, 100°C.

TABLE 5 Cobalt chloride complexes prepared using different *N,P*-ligands used to catalyze the hydrosilylation reaction of 1-octene using diphenylsilane

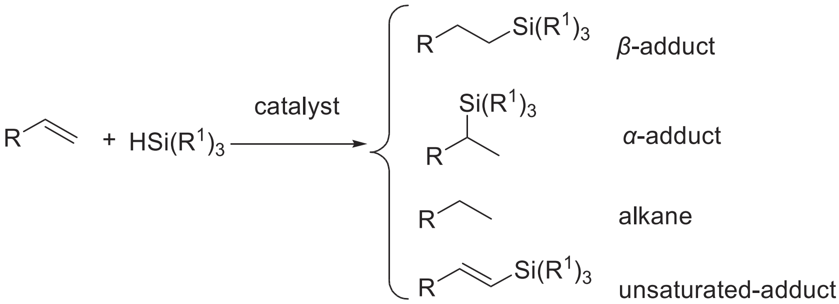
			Select. (%)			
Entry	**Ligands**	**Conv. (%)**	**β**	**α**	**Octane**	**Dehydrogenative silylation**
1	**1**	~100	~100	/	/	/
2	**2**	~100	~100	/	/	/
3	**3**	~100	~100	/	/	/
4	**4**	~100	~100	/	/	/
5	**5**	~100	~100	/	/	/
6	**6**	95	97	/	3	/
7	**7**	91	90	/	10	/
8	**8**	40	76	/	24	/
9	**9**	~100	~100	/	/	/
10	**10**	~100	96	/	4	/
11	**11**	~100	94	/	6	/
12	**12**	95	~100	/	/	/
13	**13**	~100	~100	/	/	/
14	**14**	57	85	/	15	/
15	**15**	33	87	/	13	/
16	**16**	64	73	/	27	/
17	**17**	88	88	/	12	/
18	**18**	~100	~100	/	/	/
19	**19**	85	92	/	8	/
20	**20**	92	~100	/	/	/
21	**21**	~100	~100	/	/	/
22	**22**	91	~100	/	/	/

Note: Reaction conditions: 1-Octene (4 mmol), Ph_2SiH_2 (4.4 mmol), and catalyst $CoCl_2$/Ligand (0.02 mol% with respect to 1-octene), **1–13**, **20–22**: $CoCl_2$/Ligand = 1:1, **14–19**: $CoCl_2$/Ligand = 1:2, 8 h, 100°C.

SCHEME 2 N,P-ligands



temperature and prolonging the reaction time. When the temperature is ≥90°C, a 100% conversion can be obtained within a suitable reaction time. When the temperature was 80°C, even if the reaction time is increased, the maximum conversion was <30%. At 90°C, 100°C, and 110°C, respectively, the reaction was particularly intense over the first 2 h with the conversion rapidly reaching >80%. After 2 h, the reaction rate slowed down, but the conversion continued to increase until the complete conversion of 1-octene. Taking all things into consideration, the optimal reaction temperature and time was 100°C and 8 h, respectively.



SCHEME 3 The hydrosilylation reaction

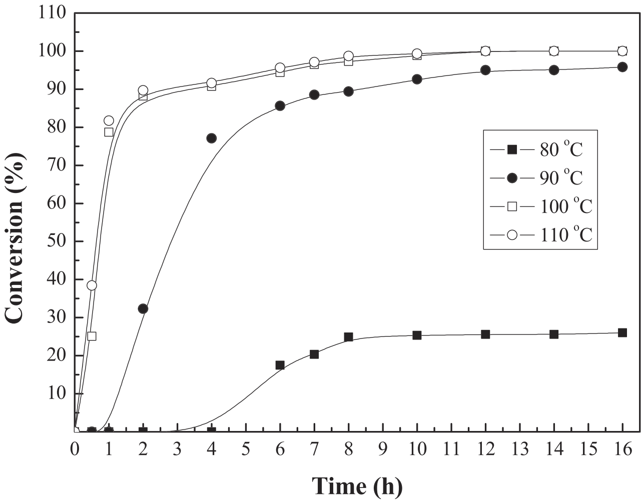


FIGURE 4 Crystal structure of the catalyst $NiCl_2$/**1**

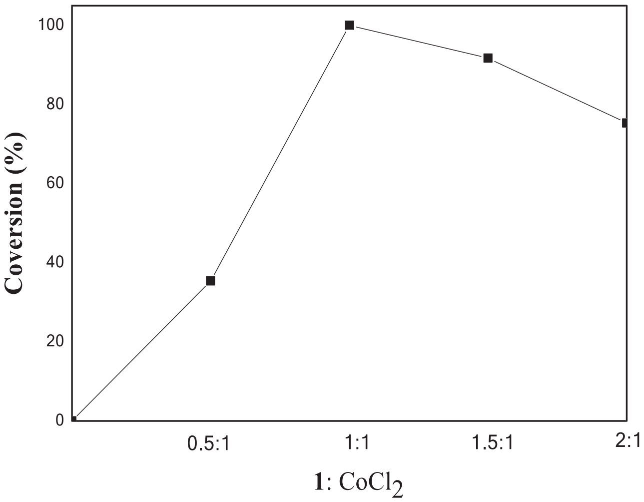


FIGURE 6 Crystal structure of the complex $CoCl_2$/**6**

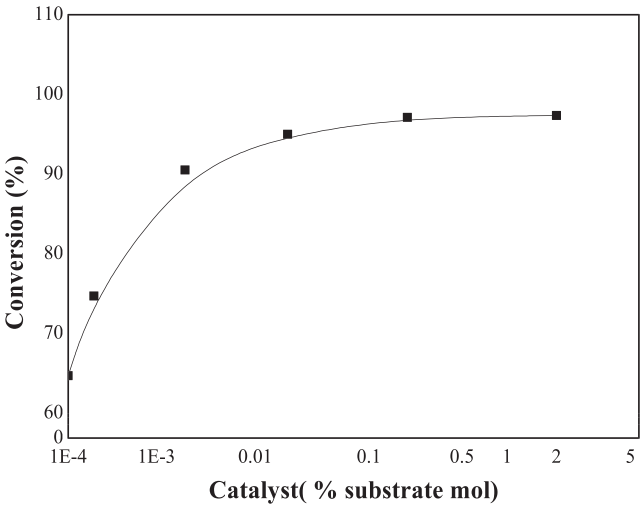


FIGURE 5 Effect of the amount of ligand **1** on the hydrosilylation reaction

The reactions of 1-octene and diphenylsilane carried out in the presence of different quantities of $CoCl_2$/**1** (2, 0.2, 0.02, 0.002, 0.0002, and 0.0001 mol%) at 100°C were investigated, and the results shown in Figure 5. The reaction conversion increased upon increasing the amount of $CoCl_2$/**1** used in the reaction. When the amount of $CoCl_2$/**1** was 0.02 mol% with respect to 1-octene, the reaction conversion was >95%. When the amount of $CoCl_2$/**1** was increased 10-fold, the reaction conversion increased, but the increase was not obvious. When the catalyst loading was 0.0001 mol% with respect to the olefin, nearly 65% of 1-octene was converted in the reaction. The effects of amounts of $CoCl_2$: ligands were illustrated in Figure 6. When the amount of $CoCl_2$/**1** was 1:1, the reaction conversion was >99.9%.

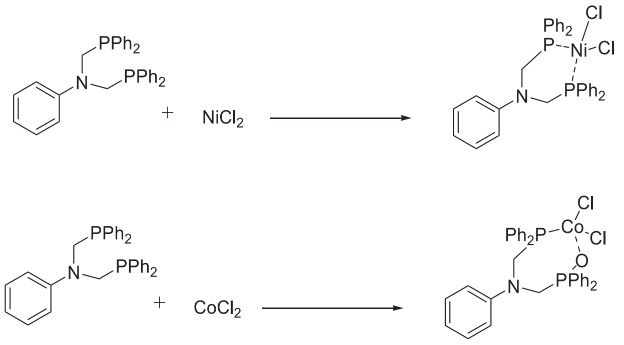
3.4 | Scope of the hydrosilylation reaction

The hydrosilylation reactions of a variety of alkenes using diphenylsilane were performed under the optimized reaction conditions in Table 6. When other alkenes such as 1-butene, 1-hexene, 1-octene, 1-dodecene, 1-tetradecene, and styrene were used as the substrate, excellent conversions were obtained using the $CoCl_2$/**1** catalyst system. In addition, good conversions were obtained when phenylsilane, dimethylphenylsilane, and diphenylmethylsilane were used to replace diphenylsilane.

TABLE 6 $CoCl_2$/**1** catalyzed hydrosilylation reaction of different olefins using silanes

				Select. (%)		
Entry	**Olefin**	**Silane**	**Conv. (%)**	**β**	**α**	**Alkane**
1	$CH_3(CH_2)_3CH{=}CH_2$	Ph_2SiH_2	~100	~100	/	/
2	$CH_3(CH_2)_5CH{=}CH_2$	Ph_2SiH_2	~100	~100	/	/
3	$CH_3(CH_2)_7CH{=}CH_2$	Ph_2SiH_2	96	83	/	17
4	$CH_3(CH_2)_{11}CH{=}CH_2$	Ph_2SiH_2	89	72	/	28
5	$CH_3(CH_2)_{13}CH{=}CH_2$	Ph_2SiH_2	83	64	/	36
6	$PhCH{=}CH_2$	Ph_2SiH_2	66	40	/	60
7	$CH_3(CH_2)_5CH{=}CH_2$	$PhSiH_3$	91	77	/	23
8	$CH_3(CH_2)_5CH{=}CH_2$	$PhMeSiH_2$	80	70	/	30
9	$CH_3(CH_2)_5CH{=}CH_2$	$PhMe_2SiH$	75	68	/	32

Note: Reaction conditions: Olefin (4 mmol), silane (4.4 mmol), and catalyst $CoCl_2$/**1** (0.02 mol% with respect to the olefin) $CoCl_2$:**1** = 1:1, 8 h, 100°C.

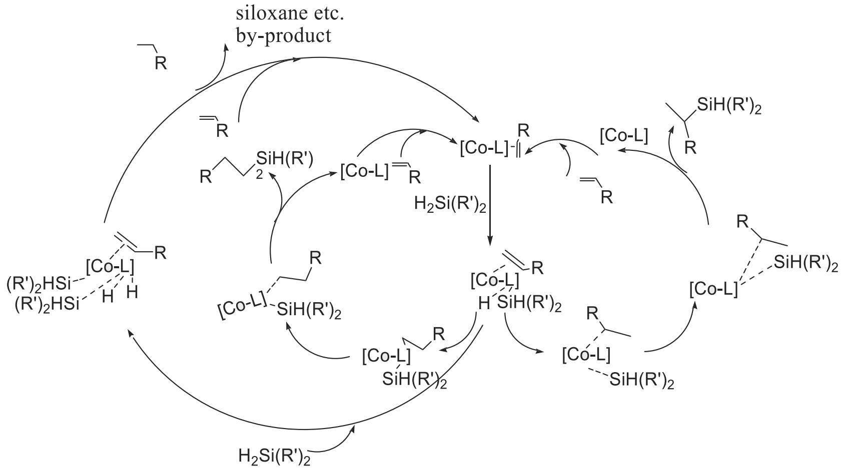


SCHEME 4 The synthesis of Ni(N,N-bis((diphenylphosphino)methyl)benzenamine)$_2$Cl$_2$ and CoO(N,N-bis((diphenylphosphino)methyl)benzenamine)$_2$Cl$_2$

3.5 | Mechanism

On the basis of our experimental results, we continued to study the ways in which the metal salts bind to the ligands. The single crystal structure of Ni(*N,N*-bis((diphenylphosphino)methyl)benzenamine)$_2$Cl$_2$ ($NiCl_2$/**6**) was determined using crystals produced by slow evaporation of the solvents from the catalyst solution in hexane/dichloromethane(Figure 5). One Ni atom coordinated to two *N,P*-ligands. Different from the coordination mode of $NiCl_2$ and ligand **6**, it was found that when $CoCl_2$ and the *N,P*-ligand coordinate with one another, one oxygen atom was inserted between the cobalt and phosphine

SCHEME 5 Mechanism



L: *N, P*-ligand

atoms in the catalysts obtained at the end of the reaction. The single crystal structure of CoO(*N,N*-bis((diphenylphosphino)methyl)benzenamine)$_2$Cl$_2$ (CoCl$_2$/**6**) was also determined (Figure 6). The oxygen atom was easily inserted between cobalt and ligands.[33] The oxygen atom did some good to the catalytic hydrosilylation reaction of alkenes.[8,34,35] We speculate that the insertion of the oxygen atom was beneficial to the catalytic activity of the cobalt complex catalyst and the catalytic hydrosilylation reaction of alkenes. A reaction may occur between the phosphine ligand and the Co metal center, which will help activate the alkene with the Co-phosphine ligand stabilizing the intermediate formed. On the basis of our results, we propose the following reaction mechanism (Scheme 4) (Scheme 5).

4 | CONCLUSIONS

In summary, a series of *N,P*-ligands (**1–22**) were synthesized. FeCl$_2$/**1**(*N^1*, *N^1*, *N^2*, *N^2*-Tetrakis[(diphenylphosphino)methyl]ethane-1,2-diamine) showed low catalytic activity. MnCl$_2$/**1**, CrCl$_3$/**1**, and NiCl$_2$/**1** showed some catalytic activity. The CoCl$_2$/*N,P*-ligand catalyst system showed high activity and excellent selectivity (The selectivity of the β-adduct was ~100%.) in the hydrosilylation reaction. CoCl$_2$/**1** showed the highest catalytic activity (~ >99.9% conversion of 1-octene). And the hydrosilylation reaction of alkenes using diphenylsilane catalyzed using CoCl$_2$ in the presence of these ligands was investigated. The catalyst complexes formed using CoCl$_2$ and *N,P*-ligands **1–22** exhibited high activity and could catalyze the hydrosilylation reaction of a range of olefin using a variety of silanes. The conversion of the reaction was high and the selectivity of the products was good.

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AUTHOR CONTRIBUTIONS

Liu Yu: Data curation. **Jiayun Li:** Conceptualization; data curation. **Ying Bai:** Formal analysis; resources. **Jiajian Peng:** Conceptualization.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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