

Efficient Solvent-Free Hydrosilylation of Aldehydes and Ketones Catalyzed by $Fe_2(CO)_9/C_6H_4-o-(NCH_2PPh_2)_2BH$

Fei Fang¹ · Jiarui Chang¹ · Jie Zhang¹ · Xuenian Chen^{1,2}

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Abstract

An efficient solvent-free catalyst system for hydrosilylation of aldehydes and ketones was developed based on iron pre-catalyst $Fe_2(CO)_9/C_6H_4$ -o- $(NCH_2PPh_2)_2BH$. The reactions were tolerant of many functional groups and the corresponding alcohols were isolated in good to excellent yields following basic hydrolysis of the reaction products. The reaction is likely catalyzed by an in situ generated pincer ligated iron hydride complex.

Graphic Abstract



Keywords Hydrosilylation \cdot Aldehydes \cdot Ketones \cdot Solvent-free \cdot Iron \cdot PBP ligand

1 Introduction

Hydrosilylation of carbonyl compounds is of great importance in the field of synthetic organic chemistry. This specific type of reaction makes it possible to hydrosilylate the carbonyl groups in an organic molecule under mild conditions without affecting other functional groups in the same molecule [1, 2]. Recently, in order to develop environmentally

 Jie Zhang jie.zhang@htu.edu.cn
Xuenian Chen xnchen@htu.edu.cn

- ¹ Henan Key Laboratory of Boron Chemistry and Advanced Energy Materials, Key Laboratory of Green Chemical Media and Reactions, Ministry of Education, Collaborative Innovation Center of Henan Province for Green Manufacturing of Fine Chemicals, School of Chemistry and Chemical Engineering, Henan Normal University, Xinxiang 453007, Henan, China
- ² College of Chemistry and Molecular Engineering, Zhengzhou University, Zhengzhou 450001, Henan, China

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friendly processes for this type of reaction, significant efforts have been made to explore new catalysts based on iron complexes [3–30] due to the fact that iron is earth-abundant, inexpensive, biocompatible and nontoxic [31, 32]. Another consideration to develop greener chemical transformation process is to perform the reaction under a solvent-free condition. However, hydrosilylation of carbonyl compounds under solvent-free condition has rarely been reported [20, 21, 29, 33–35].

We have been interested in the catalytic application of transition metal complexes supported by different types of pincer ligands [36–41]. In an attempt to prepare a diphosphinoboryl-based pincer iron complex, we treated $Fe_2(CO)_9$ with C_6H_4 -o-(NCH₂PPh₂)₂BH in toluene at room temperature. Although the expected iron pincer complex could not be isolated, we found that the resultant reaction mixture is very effective in catalyzing the hydrosilylation of aldehydes and ketones. The reactions proceeded smoothly at 65 °C under a solvent-free condition and were tolerant of many functional groups. The corresponding alcohols were isolated in good to excellent yields following basic hydrolysis of the hydrosilylation products. The present work is of relevance to green and sustainable chemistry.

2 Experimental

2.1 General Information

All of the reactions and manipulations were performed by using the standard Schlenk techniques under a nitrogen atmosphere. All of the solvents were distilled from standard drying agents and degassed before use. NMR spectra were recorded on a Bruker Advance 600 or 400 MHz spectrometer. The residual solvent resonances were used to reference the chemical shift values internally for ¹H and ¹³C NMR spectra. H₃PO₄ (85%) was used as a standard to calibrate the chemical shifts externally for ³¹P NMR spectra. GC–MS analyses were carried out by using a SHIMADZU-(GCMS-QP2020) instrument. The PBP pincer ligand C₆H₄-*o*-(NCH₂PPh₂)₂BH was synthesized by following a published method [42].

2.2 General Procedure for Hydrosilylation Reactions

C₆H₄-o-(NCH₂PPh₂)₂BH (257 mg, 0.5 mmol), Fe₂(CO)₉ (364 mg, 1 mmol) and toluene (30 mL) were mixed in a 50 mL Schlenk flask. The mixture was stirred at room temperature for 12 h. A portion of this resultant reaction mixture was transferred quantitatively to a 10 mL flame-dried Schlenk flask and the volatiles were removed under vacuum. Aldehyde or ketone substrate and PhSiH₃ (1.2 or 1.5 equiv.) were then added. The mixture was stirred at 65 °C and the reaction was monitored by using GC-MS. The reaction was stopped after aldehyde or ketone substrate disappeared completely. The resulting residue was treated with a 10% aqueous NaOH solution. The solution containing the alcohol product was extracted with diethyl ether for three times, dried over anhydrous Na2SO4, and concentrated under vacuum. The desired alcohol was further purified by flash column chromatography on silica gel using petroleum ether/ EtOAc as the eluent. Conversions were calculated based on the GC analysis results. The isolated alcohol products were characterized by ¹H NMR, ¹³C{¹H} NMR and HRMS. More details about the characterization of the reduction products are provided in the Supporting Information.

3 Results and Discussion

3.1 Catalytic Hydrosilylation Reactions

Hydrosilylation of benzaldehyde with phenylsilane catalyzed by $Fe_2(CO)_9/C_6H_4$ -o-(NCH₂PPh₂)₂BH was explored

first under different conditions and the results are provided in Table 1. As indicated by Table 1, in most cases, benzyl alcohol could be isolated in good yields following basic hydrolysis of the resultant products. Under the same conditions, neither C_6H_4 -o-(NCH₂PPh₂)₂BH nor Fe₂(CO)₉ alone catalyzed the reaction (entries 5 and 6); no hydrosilylation occurred in the absence of the iron catalyst (entry 4) or using $Fe_2(CO)_0$ as the catalyst and C₆H₄-o-(NCH₂PPh₂)₂BH as an additive (entry 7). It seemed that the reaction can be performed in any solvents; however, CH₃CN is the most effective solvent especially at higher temperatures (entries 1-3). Most importantly, the reaction could be performed very smoothly and effectively under a solvent-free condition (entry 13), which has rarely been reported for efficient catalytic hydrosilylation of carbonyl compounds [20, 21, 29, 33-35]. It was also demonstrated that the reaction can be finished in a reasonable period of time with a lower catalyst loading (entry 14); however, other silanes, including the more environmentally benign polymethylhydrosiloxane, are less effective silyl reagents for this reaction.

Hydrosilylation of other aldehydes was explored at 65 °C using $Fe_2(CO)_0/C_6H_4$ -o-(NCH₂PPh₂)₂BH as the pre-catalyst and 1.2-1.5 equiv. of PhSiH₃ as the silyl reagent with a catalyst loading of 0.5 mol%. The results are provided in Table 2. It can be seen from Table 2 that the present catalyst system is very effective in catalyzing the hydrosilylation of different aldehydes, including substituted benzaldehydes and aromatic aldehydes. Except for nitro substituted benzaldehydes, the reactions proceeded smoothly and effectively without using any solvent at 65 °C although a slightly more excess of PhSiH₃ (1.5 equiv.) was required in some cases (entries 6, 11 and 12) in order to make the reaction mixture easier to be handled. The corresponding alcohols were isolated in good to excellent yields following basic hydrolysis of the hydrosilylation products. The reactions were tolerant of many functional groups including MeO (entries 2-4), halogens (entries 6-8), CN (entry 9) and C=C (entry 12). For nitro substituted benzaldehydes, significant side reactions occurred under the solvent-free condition as indicated by GC-MS. However, hydrosilylation of nitro substituted benzaldehydes was carried out successfully in CH₃CN and the expected products were isolated in good yields (entries 13 and 14). The catalyst system is very effective even under a solvent-free condition. Based on the isolated yield, a turnover frequency (TOF) of 384 h^{-1} was achieved at 65 °C (entry 7), which is higher than those of other iron based systems under solvent-free condition [21, 29] and lower than some leading TOF values for similar iron catalyzed hydrosilylation reactions [22, 43].

Under the same conditions hydrosilylation of ketones was also investigated. The results are provided in Table 3. With a slightly higher catalyst loading (2 mol % based on Fe), different ketones were effectively reduced in a reasonable period of time and the corresponding alcohols were isolated Table 1 Hydrosilylation of benzaldehyde with Fe₂(CO)₉/C₆H₄-o-(NCH₂PPh₂)₂BH ([Fe])

Ph/
$$\overset{U}{\overset{L}{\leftarrow}}_{H}$$
 + PhSiH₃ $(1) 2 \text{ mol% [Fe], solvent (5 mL)}$
2.0 mmol 2.4 mmol (2) 10% NaOH(aq)

Entry	Catalyst	Solvent	Temp (°C)	Time (h)	Conv (%) ^a	Yield (%) ^b
1	[Fe]	CH ₃ CN	25	30	>99	88
2	[Fe]	CH ₃ CN	65	5.0	>99	89
3	[Fe]	CH ₃ CN	80	1.5	>99	85
4	-	CH ₃ CN	65	30	0	0
5 ^c	$Fe_2(CO)_9$	CH ₃ CN	65	30	0	0
6 ^d	PBP	CH ₃ CN	65	30	0	0
7 ^e	Fe ₂ (CO) ₉ /PBP	CH ₃ CN	65	30	0	0
8	[Fe]	Toluene	65	18	>99	93
9	[Fe]	Benzene	65	18	>99	75
10	[Fe]	THF	65	5.0	>99	84
11	[Fe]	DMF	65	22	>99	72
12	[Fe]	Et_2O	65	18	>99	92
13 ^f	[Fe]	-	65	5.0	>99	90
14 ^g	[Fe]	CH ₃ CN	65	9.0	>99	88

Catalyst was made by treating $Fe_2(CO)_9$ with 0.5 equiv. of C_6H_4 -o-(NCH₂PPh₂)₂BH in toluene at room temperature for 12 h followed by removal of the solvent; based on Fe, 2 mol % of catalyst was used

^aConversions were based on the GC-MS analysis results

^bisolated yields

^c0.16 mmol of Fe₂(CO)₉ was used

^d0.048 mmol of PBP was used

^e2 mol % of Fe₂(CO)₉ was used with 5 equiv. of PBP as an additive

^f5.0 mmol of benzaldehyde and 6.0 mmol of PhSiH₃ were used

^gBased on Fe, 0.5 mol % of catalyst was used

in good yields following basic hydrolysis of the reaction products. No significant side reactions occurred as indicated by GC–MS analysis. Functional groups such as OMe and halogens were not affected.

As in other catalytic systems for hydrosilylation of aldehydes and ketones [40, 41, 44], the relationship between the nature of the substrates and the relative reaction rates would be difficult to rationalize. For the present solvent-free catalytic system, in order to fully understand this relationship, the solubility of the substrates and reaction products in the liquid reaction mixture must also be considered apart from detailed kinetic data.

3.2 Mechanistic Considerations

As mentioned in the above section, neither C_6H_4 -o-(NCH₂PPh₂)₂BH nor Fe₂(CO)₉ alone catalyzed the reaction under the same conditions and no hydrosilylation occurred when Fe₂(CO)₉ was used as the catalyst

and C_6H_4 -o-(NCH₂PPh₂)₂BH was used as an additive. Further investigation indicated that the residue developed from the reaction of Fe₂(CO)₉ with 0.5 equiv. of C_6H_4 -o-(NCH₂PPh₂)₂BH in toluene at room temperature was the most effective and residues developed by treating these two complexes in other ratios under the same conditions were either less effective or completely inactive especially when an excess amount of C_6H_4 -o-(NCH₂PPh₂)₂BH was used. The catalytic activity was not influenced if elemental mercury was added in the reaction system implying that the reaction was not catalyzed by elemental iron materials such as iron nanoparticles [45]. The above information clearly demonstrated that the reaction was catalyzed by some homogeneous species derived from the reaction of Fe₂(CO)₉ with C_6H_4 -o-(NCH₂PPh₂)₂BH.

In order to gain information about the reaction between $Fe_2(CO)_9$ and C_6H_4 -o- $(NCH_2PPh_2)_2BH$, we treated $Fe_2(CO)_9$ with 0.5 equiv. of C_6H_4 -o- $(NCH_2PPh_2)_2BH$ in toluene- d_8 at room temperature in a NMR tube and monitored the

Table 2Solvent-freehydrosilylation of aldehydescatalyzed by $Fe_2(CO)_9/C_6H_4$ -o-(NCH_2PPh_2)_2BH

$\begin{array}{c} O \\ H \\ R \\ \hline C \\ H \\ 5.0 \text{ mmol} \end{array} + \begin{array}{c} PhSiH_3 \\ 6.0 \text{ mmol} \end{array} \xrightarrow{(1) 0.5 \text{ mol }\% \text{ [Fe]}, 65^{\circ}C \\ (2) 10\% \text{ NaOH(aq)} \end{array} RCH_2OH \\ \hline \end{array}$						
Entry	Substrate	Product	Time (h)	Yield (%) ^a		
1	CHO	CH2OH	7.0	89		
2	MeO	MeO CH ₂ OH	8.0	94		
3	CHO	CH ₂ OH OMe	3.0	87		
4	CHO	CH ₂ OH OMe	7.0	90		
5	СНО	CH2OH	10	89		
6 ^b	F CHO	F CH2OH	35	97		
7	CI	CI CH2OH	0.5	96		
8	Br	Br CH2OH	0.5	92		
9	NC	NC CH2OH	3.0	93		
10	CHO	CH2OH	12	78		
11 ^b	СНО	CH ₂ OH	29	85		
12 ^b	CHO	CH ₂ OH	35	83		
13°	O ₂ N CHO	O ₂ N CH ₂ OH	2.0	92		
14°	CHO NO ₂	CH ₂ OH	3.0	68		

The catalyst was made similarly as described in Table 1; based on Fe, 0.5 mol % of catalyst was used ^a isolated yields

^b7.5 mmol of PhSiH₃ was used

^cthe reactions were performed in 5 mL of CH₃CN

reaction by using NMR spectroscopy at room temperature. The starting PBP pincer ligand disappeared completely after 12 h as indicated by ³¹P{¹H} NMR spectrum; a few new ³¹P NMR resonances were displayed in the range of 60–90 ppm, including a singlet at 86.3 ppm (Fig. 1, right, spectrum a). The ¹H NMR spectrum showed several resonances in the range of -15 to -7 ppm, including a sharp triplet at -10.34 ppm with a coupling constant of 36 Hz (Fig. 1, left, spectrum a). The sealed NMR tube was kept at room temperature and the reaction was further monitored.



$R \xrightarrow{C} C_{H_3} + PhSH_3 \xrightarrow{(1) 2 \text{ Into it is press, oscilar or }} RCHCH_3OH$ 5.0 mmol 6.0 mmol 6.0 mmol								
Entry	Substrate	Product	Time (h)	Yield (%) ^a				
1	C) L	OH	5	91				
2	CI C	CI	4	90				
3	F _{3C}	P ₃ C OH	3	85				
4	Meo	ОН Мео	2	87				
5		OH	5	83				
6	Ů	ОН	30	80				

0

The catalyst was made similarly as described in Table 1; based on Fe, 2 mol % of catalyst was used ^a isolated yields

After 48 h, all of the ¹H NMR resonances in the range of -15 to -7 ppm disappeared completely. It seemed that the sharp triplet at -10.34 ppm in the ¹H NMR spectra and the singlet at 86.3 ppm in the ³¹P{¹H} NMR spectra disappeared simultaneously (Fig. 1).

We then checked the catalytic activities of different residue samples derived from the reaction of $Fe_2(CO)_9$ with 0.5 equiv. of C_6H_4 -o-(NCH₂PPh₂)₂BH in toluene- d_8 at room temperature. It was found that the residues with NMR spectra of (a), (b) and (c) are all active in catalyzing the reaction while the residue with NMR spectrum of (d) is inactive. It was also found that the triplet at -10.34 ppm

can hardly be seen in the ¹H NMR spectra when the residue was made by treating $Fe_2(CO)_9$ with more than 0.5 equiv. of C_6H_4 -o-(NCH₂PPh₂)₂BH. This indicated that the reaction was catalyzed by a species with the sharp triplet at -10.34 ppm in the ¹H NMR spectra. Preparative scale experiments were subsequently carried out in order to isolate any products for the inaction between $Fe_2(CO)_9$ and C_6H_4 -o-(NCH₂PPh₂)₂BH. However, all of the experiments, including a few low temperature reactions and isolations, were unsuccessful due to the instability of these species.

It was demonstrated that $Fe_2(CO)_9$ can react with olefins, dithiane and pyridine under very mild conditions to

Fig. 1 ¹H (left) and ³¹P{¹H} (right) NMR spectra of the interaction of Fe₂(CO)₉ with C_6H_4 -o-(NCH₂PPh₂)₂BH in toluene- d_8 at room temperature. Spectra recorded 12 h (a), 24 h (b), 36 h (c) and 48 h (d) after mixing the two reactants



form $Fe(CO)_4L$ monomer species [46–50]. In the present case, it was very likely that $Fe_2(CO)_0$ reacted with C₆H₄-o-(NCH₂PPh₂)₂BH through phosphorus coordination followed by oxidative addition of the B-H bond to the iron center to form a PBP pincer iron hydride complex C_6H_4 -o-(NCH₂PPh₂)₂BFe(H)(CO)₂ (1) (Scheme 1). Similar reactivity was reported by Ke, Langer and co-workers for the reactions between Fe(CO)₅ and (PR₂XPR₂)₂B(H)₂Br $(X = NH, CH_2; R = Ph, Et)$, in which a series of cationic PBP pincer iron complexes, $[(PR_2XPR_2)_2B(H)Fe(H)]$ $(CO)_2$ ⁺, formed [51]. The ruthenium counterpart of complex (1), C_6H_4 -o-(NCH₂PPh₂)₂BRu(H)(CO)₂ (2), was previously reported by Hill and co-workers. The ¹H NMR spectrum for complex 2 showed a sharp triplet at -8.20 ppm (²J_{H-P} = 23 Hz) for the hydride. X-ray crystallography experiment of 2 showed two cis-CO ligands coordinated to the metal center [52]. The formation of complex 1 in the present system was confirmed by HRMS (see Supporting Information). The reactions were likely catalyzed by complex 1; detailed mechanistic investigation including further characterization of complex 1 is in progress in our laboratory.

4 Conclusions

In conclusion, we have successfully used $Fe_2(CO)_9/C_6H_4$ o-(NCH₂PPh₂)₂BH as a pre-catalyst for hydrosilylation of aldehydes and ketones under a solvent-free condition. The reaction is highly efficient and tolerant of many functional groups such as MeO, halogens, CN and C=C; the corresponding alcohols have been isolated in good to excellent yields following basic hydrolysis of the resultant hydrosilylation products. The reaction is likely catalyzed by an in situ generated PBP iron hydride complex C_6H_4 -o-(NCH₂PPh₂)₂BFe(H)(CO)₂. The present work represents a good example of developing greener catalytic processes for important chemical transformations based on environmentally benign earth-abundant first-row transition metals and is of relevance to green and sustainable chemistry.



Scheme 1 Reaction of $Fe_2(CO)_9$ with C_6H_4 -o-(NCH₂PPh₂)₂BH

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s10562-021-03578-9.

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Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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