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# New indenyl phosphinooxazoline complexes of iron and their catalytic activity in the Mukaiyama aldol reaction

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ABSTRACT

New phosphinooxazoline (PHOX) $\eta^5$-indenyl complexes of iron were synthesized and applied as catalysts in the Mukaiyama aldol reaction. Reaction of three different PHOX ligands with $[Fe(\eta^5\text{-Ind})I(CO)_2]$ afforded the iodide salts of three complexes of the general formula $[Fe(\eta^5\text{-Ind})(CO)(PHOX)]^+$ in 73–81% isolated yields. The molecular structure of one of the new complexes was determined, revealing a pseudo octahedral coordination geometry about the iron center. The iron complexes are catalytically active in the Mukaiyama aldol reaction between aldehydes and 1-(*tert*-butyldimethylsilyloxy)-1-methoxyethene to give the corresponding aldol adducts (3 mol % catalyst, 15 min, room temperature, 48–83% isolated yields). A previously synthesized iron complex of the general formula $[Fe(\eta^5\text{-Cp})(CO)(PHOX)]^+$ was found to be catalytically active in the title reaction as well, but needed three hours at room temperature to convert the starting materials to the products.

Iron catalysis is a field of intensive research in organometallic chemistry.[1] Iron based catalyst systems have a number of advantages over other transition metals typically employed in catalysis. Iron is cheap, abundant, nontoxic and environmentally friendly. Its non-toxicity is especially important for applications in the pharmaceutical industry.[2]

Consequently, iron catalysts have been applied in a number of organic transformations, such as oxidations,[3a,b] reductions,[3c] carbon–carbon bond forming reactions,[3d–f] carbon–heteroatom bond forming reactions,[3g,h] polymerizations[3i] in addition to other reactions.[3k,3l] Typically in situ catalyst systems are applied,[3a,3d] but preformed, well defined catalyst systems allow for easier investigations of the catalytically active species and for mechanistic studies.[3i] In order for an iron complex to be catalytically active, open coordination sites are required. They can be provided in situ by combining a substoichiometric amount of the ligand and a metal salt or by preformed complexes, in which coordination sites are only loosely occupied by solvents or donating atoms of chelating ligands.

Phosphinooxazolines (PHOX, **1**, Fig. 1) which represent a bidentate ligand class,[4] have widely been applied in transition metal catalyzed organic transformations.[5] We have recently synthesized and structurally characterized the first chelating iron PHOX complexes **2** (Fig. 1).[6] These complexes are coordinatively saturated, but more significantly, they showed less catalytic activity in the oxidation of benzylic methylene groups by *t*-BuOOH than iron complexes of the general formula $[Fe(PHOX)_2]^+OTf^-$ (**3**, $OTf^- = CF_3SO_3^-$).[7] We tentatively ascribed this difference in reactivity to the fact that the latter group of complexes already has open coordination sites available, whereas an open site must first be created for the complexes **2**.[7]

We were interested in alternative ways to create open coordination sites on iron. The indenyl ligand $C_9H_7^-$ (Ind) is known to easily undergo an $\eta^5$ to $\eta^3$ haptotropic shift in metal complexes,[8] and thus opening coordination sites at the metal (Scheme 1).

Consequently, indenyl complexes tend to be more active in ligand exchange reactions, than their cyclopentadienyl analogs (such as **2** in Fig. 1), sometimes referred to in the literature as the 'indenyl effect'.[8,9] The indenyl effect has been described to participate in catalytic reactions.[10] We thus set out to synthesize iron

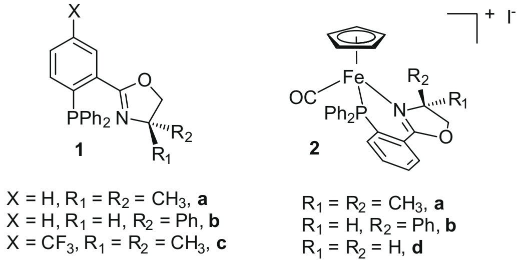

**Figure 1.** Phosphinooxazoline ligands (**1**) and iron complexes (**2**) thereof.

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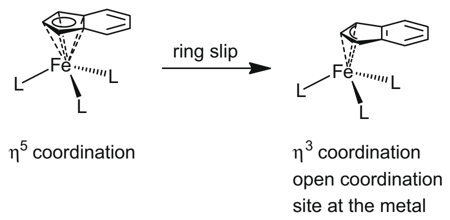


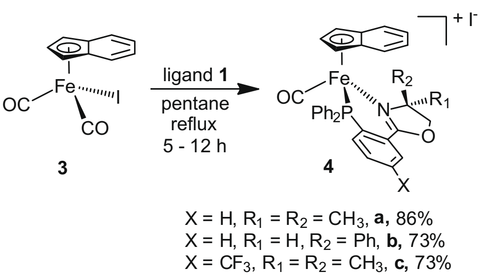
**Scheme 1.** The indenyl effect.

PHOX indenyl complexes and anticipated that these might act as gentle Lewis acids in solution. The Mukaiyama aldol reaction is a Lewis acid catalyzed coupling reaction of silyl enol ethers and carbonyl compounds.[11] Accordingly, the catalytic activity of the new iron complexes in the Mukaiyama aldol reaction was tested.

First, synthetic access to indenyl PHOX iron complexes was targeted. Accordingly, when the known complex [Fe($\eta^5$-Ind)I(CO)$_2$] (**3**)[12] was refluxed with one equivalent of the PHOX ligand **1a**[13] in pentane for 3 h, the iodide salt of the complex [Fe($\eta^5$-Ind)(CO)(**1a**)]$^+$ (**4a**) was isolated in 81% yield as a light red solid after washing with diethyl ether (Scheme 2). Applying similar conditions with ligands **1b** and **1c** gave the iodide salts of the complexes [Fe($\eta^5$-Ind)(CO)(**1b**)]$^+$ (**4b**) and [Fe($\eta^5$-Ind)(CO)(**1c**)]$^+$ (**4c**) as orange-red solids, both in 73% isolated yield (Scheme 2).

The new iron complexes **4a**, **4b** and **4c** were characterized by NMR ($^1$H, $^{13}$C, $^{31}$P), MS (including HRMS), and IR. The coordination of the PHOX ligand was best seen by the large downfield shift of the phosphorus signal in the $^{31}$P NMR spectrum. The free ligands have chemical shifts of around −4.5 ppm, whereas the iron complexes exhibited signals between 61.3 and 67.0 ppm. The cyclopentadienyl portion of the coordinated indenyl ligand shows a characteristic pattern of signals in the $^1$H and $^{13}$C NMR spectra.[14] The three protons gave distinct signals between 5.70 and 4.88 ppm in the $^1$H NMR spectrum and the five carbon atoms gave distinct signals between 110.0 and 64.5 ppm in the $^{13}$C NMR spectrum. The IR spectra also clearly show coordination of the ligands. The free ligands **1a**, **1b** and **1c** show characteristic $\upsilon_{C=N}$ stretching frequencies between 1630 and 1655 cm$^{-1}$, respectively,[6] which shift significantly to lower wavenumbers (1607 to 1617 cm$^{-1}$), when coordinated to the iron center. The IR spectra of each of the complexes also showed a single $\upsilon_{C\equiv O}$ stretch between 1941 and 1959 cm$^{-1}$, as expected for an iron complex with a single carbonyl ligand. The carbonyl ligand was also observed in the $^{13}$C NMR spectra of the complexes and gives a distinct doublet between 217.6 and 221.2 ppm ($^2J_{CP}$ = 29.4 to 28.0 Hz) as previously observed in structurally related iron complexes.[6] The FAB MS spectra showed molecular ion peaks for the new complexes as well as ions for CO loss.

The resulting complexes **4** are stereogenic at the metal. Consequently, for the achiral ligands **1a** and **1c**, racemic mixtures of the complexes **4a** and **4c** were isolated. Ligand **1b** is chiral, and thus, two diastereomers can form during synthesis. The diastereomeric ratio of the isolated material of **4b** was determined to be 94:6, as assessed by NMR ($^1$H, $^{31}$P).



**Scheme 2.** Iron indenyl PHOX syntheses.

To unequivocally establish the structures of the new iron complexes, an X-ray structure for **4a** was determined (details are given in Supplementary data).[15] The molecular structure is shown in Figure 2, along with key structural data. Bond length and angles are similar to those of the structurally related complex **2a**.[6] The molecular structure of **4a** confirms the piano stool type coordination geometry around the iron, in which one of the carbonyl ligands and the iodo ligand in the precursor complex [Fe($\eta^5$-Ind)I(CO)$_2$] **3** is substituted by the PHOX ligand **1a**. The bond angles around iron range from 83.76(11)° for the N(1)–Fe–P(1) angle to 99.61(18)° for the C(10)–Fe–N(1) angle. Thus, the coordination geometry of the complexes is best described as slightly distorted octahedral.

We next employed the new complexes as catalysts for initial screening in the Mukaiyama aldol reaction of aldehydes (**5**) and 1-(*tert*-butyldimethylsilyloxy)-1-methoxyethene (**6**), which exhibits enhanced reactivity, to obtain the product **7** (Table 1). The title reaction proceeded only in acetonitrile as the solvent, and only trace amounts of products were detected, when diethyl ether, THF, CH$_2$Cl$_2$ and toluene solvents were employed. For complexes **4**, after a 15 min reaction time at room temperature, only traces of the aldehyde starting material were detected by GC/MS, and the products were observed in about 50–90% yields. Complex **2a** (Fig. 1) was employed as a catalyst as well; most significantly, it showed decreased catalytic activity in the title reaction (Table 1, entries 1 and 2). The precursor complex **3** showed slightly less catalytic activity than the iron PHOX complexes in the test reaction in Table 1 (entry 3). Aliphatic aldehydes formed a number of unidentified side products (Table 1, entry 6); only butyraldehyde exhibited a clean reaction to the corresponding aldol adduct (Table 1, entry 5).

Acetophenone reacted much slower under the conditions in Table 1 and gave only an inseparable mixture of products (Table 1, entries 7 and 8).

Next, the catalyst **4a** was applied in a number of reactions of aromatic aldehydes **5** with 1-(*tert*-butyldimethylsilyloxy)-1-methoxyethene **6** and the corresponding aldol products **7** were isolated

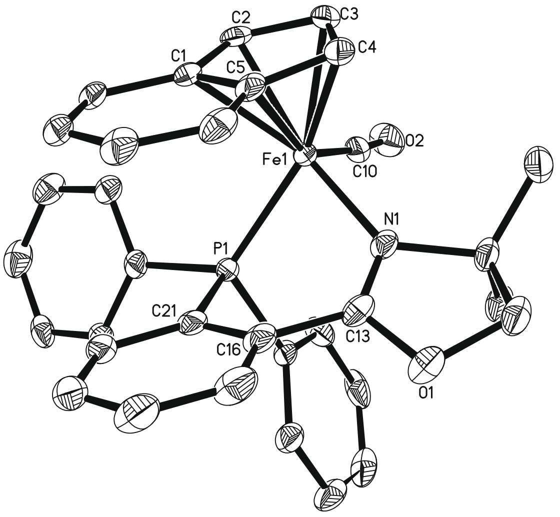
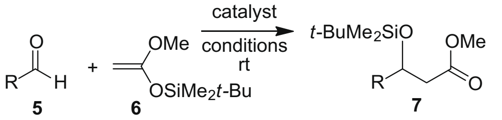


**Figure 2.** Molecular structure of **4a** (depicted with 65% probability ellipsoids; H atoms and the acetone solvate are omitted for clarity). Key bond lengths (Å) and bond angles (°): Fe–C10, 1.746(5); Fe–N1, 1.999(4); Fe–P1, 2.2091(12); N1–C13, 1.285(6); Fe–C2, 2.100(4); Fe–C3, 2.080(4); Fe–C4, 2.100(4); Fe–C1, 2.241(4); Fe–C5, 2.228(4); C10–O2, 1.148(6); C10–Fe–N1, 99.61(18); N1–Fe–P1, 83.76(11); C10–Fe–P1, 95.18(14); O2–C10–Fe, 174.1(4).

in 48–83% yields (Table 2). The catalytic performance of complexes **4a** and **4c** were comparable, but complex **4a** was used for further studies, as its ligand **1a** is synthetically more easily accessed than the other ligands.

A variety of aromatic substitution patterns in the aldehyde is compatible with the catalyst. The catalysts loads were 3% and the turnover frequencies ranged from 64 to 106 h$^{-1}$. The new iron indenyl complexes are thus highly active in the title reaction. Nishiyama recently revealed that 8 mol % of the rhenium complex $Re(CO)_5Br$ catalyzes the conversion of the silyl enol ether **6** and aromatic aldehydes to the corresponding aldol products (1 h, 80 °C, 50–80% isolated yields).[16] Mukaiyama reported that 10 mol % of an $InCl_3 \cdot AgClO_4$ mixture is catalytically active in the reaction between **6** and benzaldehyde (2 h, −78 °C, 96% isolated yield).[17] Reetz described the cationic complex $[Fe(Cp)(dppe)(CO)]^+$ as a catalyst in the reaction between **6** and benzaldehyde after photolytic activation (2.5 mol % catalyst, 20 h, −20 °C, 77%, dppe = diphenylphosphinoethane).[18] The catalytic activity of the new indenyl complexes **4** described herein is, thus, comparable to other transition metal complexes that catalyze the reactions shown in Table 2.

**Table 1**
Initial Screening of Iron catalyzed Mukaiyama aldol reactions



| Entry | Aldehyde | Catalyst | Conditions[a] | Yield[b] (%) |
|---|---|---|---|---|
| 1 | R = Ph | **2a** | $CH_3CN$, 25 min | 3 |
| 2 | R = Ph | **2a** | $CH_3CN$, 3 h | 61 |
| 3 | R = Ph | **3** | $CH_3CN$, 15 min | 89 |
| 4 | R = Ph | **4a**[c] | $CH_3CN$, 15 min | 90 |
| 5 | R = $CH_3(CH_2)_4$ | **4a** | $CH_3CN$, 5 h | 48 |
| 6 | 2-Ethylbutanal | **4a** | $CH_3CN$, 5 h | 0[d] |
| 7 | Acetophenone | **4a** | $CH_3CN$, 20 min | 5 |
| 8 | Acetophenone | **4a** | $CH_3CN$, 5 h | 50 |

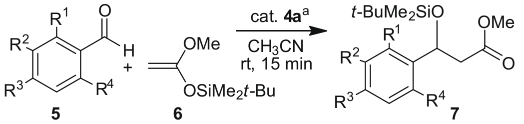
[a] Conditions: The aldehyde (0.377 mmol) was dissolved in the solvent (1.0 mL). The catalyst (0.011 mmol) was added, followed by 1-(*tert*-butyldimethylsilyloxy)-1-methoxyethene (0.377 mmol). After the time indicated, the reaction mixture was filtered through a short pad of silica gel and analyzed by GC/MS. All reactions were carried out at room temperature.

[b] Determined by GC/MS.

[c] Complexes **4b** and **4c** showed comparable activity.

[d] No aldol product was obtained, but unidentified side products were observed, presumably due to elimination reactions.

**Table 2**
Iron catalyzed Mukaiyama aldol reaction



| Entry | Aldehyde | Yield[b] (%) | TOF/h$^{-1}$ [c] |
|---|---|---|---|
| 1 | $R^1 = R^2 = R^3 = R^4$ = H, **a** | 74[d] | 98 |
| 2 | $R^1 = R^2 = R^4$ = H, $R^3 = NO_2$, **b** | 59 | 78 |
| 3 | $R^1$ = OMe, $R^2 = R^3 = R^4$ = H, **c** | 62 | 80 |
| 4 | $R^2$ = OMe, $R^1 = R^3 = R^4$ = H, **d** | 78 | 101 |
| 5 | $R^3$ = OMe, $R^1 = R^2 = R^4$ = H, **e** | 70 | 91 |
| 6 | $R^1$ = Cl, $R^2 = R^3 = R^4$ = h, **f** | 83 | 104 |
| 7 | $R^3 = CH_3$, $R^1 = R^2 = R^4$ = H, **g** | 76 | 101 |
| 8 | $R^1 = R^3 = R^4 = CH_3$, $R^2$ = H, **h** | 64 | 86 |
| 9 | $R^1 = CH_3$, $R^2 = R^3 = R^4$ = H, **i** | 70 | 93 |
| 10 | $R^2 = CH_3$, $R^1 = R^3 = R^4$ = H, **k** | 79 | 106 |
| 11 | $R^1 = R^2 = R^4$ = H, $R^3$ = Et, **l** | 48 | 64 |
| 12 | Butyraldehyde, **m** | 54 | 71 |

[a] Conditions: The aldehyde **5** (0.377 mmol) was dissolved in $CH_3CN$ (1.0 mL). The catalyst **4a** (0.011 mmol) was added, followed by 1-(*tert*-butyldimethylsilyloxy)-1-methoxyethene (**6**, 0.377 mmol). After 15 min at rt, the solvent was removed and the products were isolated by column chromatography.

[b] Isolated yields after column chromatography.

[c] Turnover frequency determined from isolated yields: number of moles (product) over (number of moles (catalyst) times reaction time).

[d] Employment of the chiral catalyst **4b** resulted in essentially no enantiomeric excess for this reaction, as determined by chiral GC.

The complex **4b** was isolated in high diastereomeric purity ($^1$H, $^{31}$P NMR). The Mukaiyama aldol reaction in Table 2 produces chiral aldol products **7**. We anticipated that the chiral complex **4b** could be catalytically active in enantioselective formation of the products. However, essentially no enantiomeric excess was observed for the reaction of benzaldehyde with 1-(*tert*-butyldimethylsilyloxy)-1-methoxyethene **6** (Table 2, entry 1).

In conclusion, the present study shows for the first time the catalytic activity of new iron indenyl PHOX complexes of the general formula $[Fe(\eta^5\text{-Ind})(CO)(PHOX)]^+$ in the Mukaiyama aldol reaction of 1-(*tert*-butyldimethylsilyloxy)-1-methoxyethene and a variety of aromatic aldehydes to give the corresponding aldol products in 48–83% isolated yields. The new complexes show high activity, as the reaction proceeds at room temperature within 15 min. The substitution of a Cp ligand by an indenyl ligand enhanced the catalytic activity in the $[Fe(L)(CO)(PHOX)]^+$ system. Ligand and metal complex modifications to improve catalytic activity for other silane substrates and to obtain enantiomeric excesses are currently underway.

## Acknowledgments

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## Supplementary data

Crystallographic data (excluding structure factors) for the structure in this paper has been deposited with the Cambridge Crystallographic Data Centre as supplementary publication CCDC 766377. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, (fax: +44-(0)1223-336033 or e-mail: deposit@ccdc.cam.ac.uk). Experimental details, characterization data and $^1$H and $^{13}$C NMR spectra for ligand **1c**, all new complexes and for the catalysis products in Table 2; experimental details and crystallographic data for the X-ray determination of **4a** are available. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.tetlet.2010.03.090.

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15. *Crystal data of complex* **4a**: $(C_{33}H_{29}FeINO_2P)_2\cdot(C_3H_6O)$, $M = 1428.66$, $T = 100(2)$ K, monoclinic, $P2_1/n$, $a = 10.1851(10)$ Å, $b = 16.1312(16)$ Å, $c = 18.6639(17)$ Å, $\beta = 96.338(4)^\circ$, $V = 3047.7(5)$ Å$^3$, $Z = 2$, density (calculated) 1.557 Mg/m$^3$, absorption coefficient 1.594 mm$^{-1}$, crystal size 0.21 × 0.21 × 0.18 mm$^3$, theta range for data collection 1.67–28.85°, 69837 reflections collected, 7802 independent reflections [$R(\text{int}) = 0.0371$], absorption correction semi-empirical from equivalents, max. and min. transmission 0.7645 and 0.7266, data/parameters 7802/399, goodness-of-fit = 1.139, final $R$ indices $R_1$ [$I > 2\sigma(I)$] = 0.0450 and $wR_2$ (all data) = 0.1238, largest difference in peak and hole 1.714 and −0.698 e Å$^{-3}$. Experimental details and crystallographic data for the X-ray determination can be found in Supplementary data.
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