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Salen Ligands Revisited: Synthesis and Application of a Planar Chiral "Ferro-Salen" Ligand

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Condensation of the O-protected planar chiral hydroxyferrocene carbaldehyde (S_p)-**1** with ethylenediamine, followed by deprotection gave rise to the "ferro-salen" ligand (S_p,S_p)-**3** in good yield. This scaffold was used for the preparation of a series of metal complexes [(S_p,S_p)-**4**/**5**/**6**], which were subsequently applied for the Lewis acid catalyzed asymmetric trimethylsilylcyanation of benzaldehyde.

Introduction

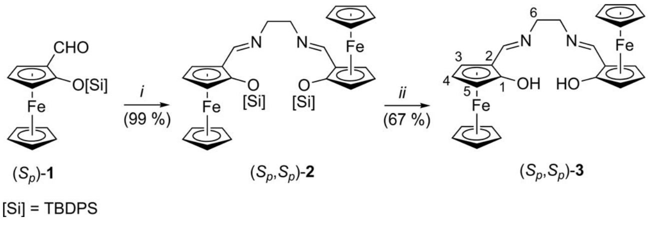
Since the seminal contributions by Katsuki and Jacobsen on the use of salen ligands in asymmetric epoxidation reactions, these ligands have been established as widely used systems in asymmetric catalysis.[1] The success of these "privileged" ligands[2] partly relies on the easy accessibility of the enantiopure derivatives by condensation of suitable salicylic aldehyde derivatives with enantiopure diamines, yielding the corresponding optically active C_2-symmetric ligands.[3]

Further development of these "parent" salen-type ligands has been attempted to enhance the reaction scope and selectivity of salen–metal-complex catalyzed reactions, for example, by introduction of additional sources of chirality into the ligand backbone. This concept has convincingly been pursued, for example, by Rozenberg, Belokon and Bräse, showing that planar chiral *p*-cyclophane derivatives[4] can be used as building blocks for the synthesis of salen-type ligands. This led to rather selective catalysts, which showed a marked *matched*/*mismatched* behaviour upon combination of the planar chiral *p*-cyclophane fragments with chiral 1,2-diamines.[5]

In a similar approach, 1,2-disubstituted ferrocene derivatives have been used as an element of planar chirality for the construction of tetradentate salen-like ligands. This was especially applied for phosphanyl-substituted ferrocenes, which gave rise to the corresponding P_2N_2-ligands.[6] While Bildstein later described the use of hydroxymethyl-substituted ferrocenes for the synthesis of the related planar chiral O_2N_2-salen ligands,[7] the more simple hydroxyferrocene analogues have to the best of our knowledge so far only been mentioned by Ito.[8] The synthesis and use of these "ferro-salen" ligands was hampered by the high sensitivity of the hydroxyferrocene moiety towards oxidative decomposition, which has prevented further application of these highly interesting salen analogues.

Results and Discussion

In a recent publication, we described the convenient synthesis of a protected ferrocene analogue of salicylic aldehyde in enantiomerically pure form [(S_p)-**1**, see Scheme 1]. In this account, we would now like to present the use of this building block for a revised synthesis of the "Ito-ligand" [(S_p,S_p)-**3**]. In addition, selected first examples of the coordination chemistry of this ligand system are described, together with the application of these complexes in the asymmetric trimethylsilylcyanation of benzaldehyde.



Scheme 1. Reagents and conditions: (i) Ethylenediamine (0.5 equiv.), EtOH, 78 °C; (ii) $Et_3N{\cdot}(HF)_3$, thf, room temp., (scheme includes NMR numbering scheme, TBDPS = *tert*-butyldiphenylsilyl).

The key building block [(S_p)-**1**] for the synthesis of ligand (S_p,S_p)-**3** is available from Kagan's ferrocene acetal[9] in four steps, as described previously.[10] Reaction of the (S_p)-isomer of this O-silyl-protected 2-hydroxyferrocene carbaldehyde with half an equivalent of ethylenediamine in ethanol gave rise to the bisprotected ligand precursor (S_p,S_p)-**2** in almost quantitative yield. This could then cleanly be transformed into the corresponding free diol by treatment

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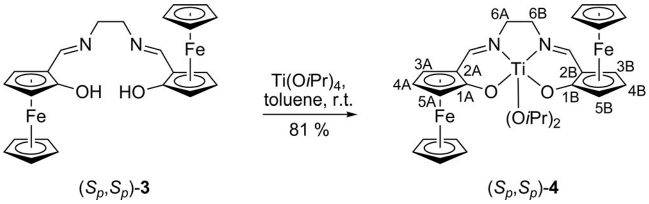
with triethylamine trihydrofluoride, yielding the "ferro-salen" ligand ((S_p,S_p))-**3** as an orange solid in 67% yield (see Scheme 1).

Free ligand ((S_p,S_p))-**3** was characterized by NMR and IR spectroscopy and elemental analysis.[11] As expected, the ligand exhibits a C_2-symmetric structure in solution (C_6D_6), resulting in a set of three signals for the substituted ferrocene Cp-units [δ = 4.40 (H-3), 3.63 (H-4) and 3.74 ppm (H-5), each 2 H], and one signal arising from the unsubstituted Cp-units (δ = 4.07 ppm, 10 H). The hydroxy protons can be observed as a broad resonance at δ = 9.41 ppm, indicating the formation of a hydrogen bridge to the imino nitrogen atoms. The signals of the CHN groups appear at δ = 7.87 ppm (^{13}C NMR: δ = 167.7 ppm), whereas the diastereotopic geminal methylene protons (H-6/H-6′) of the bridging ethylene unit show resonances at δ = 3.28 and 3.15 ppm (each 2 H), respectively (^{13}C NMR: δ = 60.7 ppm).

The "ferro-salen" ligand ((S_p,S_p))-**3** was subsequently treated with titanium tetraisopropoxide in an equimolar ratio, instantly giving the corresponding (ferro-salen)Ti(O*i*Pr)$_2$ complex ((S_p,S_p))-**4** by elimination of two equivalents of 2-propanol (Scheme 2). The highly sensitive complex could be isolated as a deep-red solid in good yield (81%).



Scheme 2. Synthesis of Ti complex ((S_p,S_p))-**4** (including NMR numbering scheme).

Assuming an octahedral coordination geometry of the central Ti atom, three different coordination isomers can potentially result for complex ((S_p,S_p))-**4**, namely, a *trans*-isomer, a *cis*-α- and a *cis*-β-isomer.[12] The latter two exhibit an octahedrally chiral metal centre, which in combination with the enantiomerically pure ((S_p,S_p))-configured ligand system can yield the corresponding ((S_p,S_p,Λ))- and ((S_p,S_p,Δ))-diastereoisomers [giving a total of five possible isomers for ((S_p,S_p))-**4**; Figure 1]. NMR spectroscopic analysis of complex ((S_p,S_p))-**4** reveals the presence of two different isomers in solution (C_6D_6), which are approximately formed in a 1.2:1 ratio.

The minor isomer in solution exhibits a C_2-symmetric structure, thus indicating a *trans*- or a *cis*-α-coordination geometry. The ligand system shows the characteristic set of signals for both the substituted Cp-rings (δ = 4.34, 3.93, 3.99 ppm, each 2 H) and the unsubstituted Cp-rings (δ = 4.19 ppm, 10 H), whereas the signal for the CHN group is slightly shifted to higher field (δ = 7.69 ppm) compared to the free ligand system. Accordingly, the Ti-isopropoxy ligands give rise to one septet at δ = 4.39 ppm (2 H) for the CH protons, whereas the methyl groups exhibit diastereotopic splitting, resulting in two doublets at δ = 1.06 and 1.03 ppm (each ^3J = 6.0 Hz, 6 H).

The major isomer is of lower symmetry (C_1), thus displaying separate sets of signals for the two ferrocenyl units [δ = 4.22, 4.32 (H-3$^{A/B}$), 3.86, 3.80 (H-4$^{A/B}$), 4.02, 3.89 (H-5$^{A/B}$), 4.25, 3.78 ppm (Cp$^{1/2}$)].[13] For the isopropoxy ligands, this leads to the observation of four doublets arising from the isopropyl methyl groups (δ = 1.51, 1.40, 1.31, 1.03 ppm, each 3 H), and two septets resulting from the corresponding methine protons (δ = 5.17, 4.93 ppm, each 1 H). The C_1 symmetry observed for the major isomer indicates the selective formation of one of the two possible isomers (Λ or Δ) of a *cis*-β-structure.

Repeated attempts to obtain crystalline material of ((S_p,S_p))-**4** in order to gain further structural information by X-ray single crystal analysis led to the isolation of a partial hydrolysis product. This was characterized as a dimeric structure containing a central four-membered cyclic Ti_2O_2 unit,[14] with the intact "ferro-salen"-ligands completing the octahedral coordination sphere around the Ti atoms (see Figure 2). Both Ti centres show a (*cis*-β)-(Δ)-configuration (compare Figure 1), resulting in a [(*cis*-β)-((S_p,S_p,Δ))-(ferro-salen)Ti]$_2$-(μ-O)$_2$ homodimer. This indicates the formation of the observed dimer by hydrolytic cleavage of the Ti(O*i*Pr) groups of the C_1-symmetric major isomer in solution, which can thus tentatively be assigned a (*cis*-β)-((S_p,S_p,Δ)) configuration.

The Ti_2O_2 core of the dimer is nearly planar [dihedral angle Ti1–O1–Ti2–O2 0.4(1)°], with the Ti_2O_2 unit distorted from a square arrangement [Ti1–O1 1.944(3), Ti1–O2 1.768(3), Ti2–O1 1.770(3), Ti2–O2 1.950(3) Å]. The two six-membered [N,O]-chelate rings of the "ferro-salen" subunits show different arrangements, with one chelate ring being nearly planar [dihedral angles Ti1–O1A–C1A–C2A –8.5(6)°, O1A–C1A–C2A–C11A 5.0(7)°, C1A–C2A–C11A–N1A –10.6(7)°, bond lengths Ti1–O1A 1.890(3), Ti1–N1A 2.314(3) Å], whereas the other chelate ring is distorted from planarity, with the Ti atom lying "above" the ferrocene unit [dihedral angles Ti1–O1B–C1B–C2B 31.2(6)°, O1B–C1B–C2B–C11B 14.0(7)°, C1B–C2B–C11B–N1B –23.9(7)°, bond lengths Ti1–O1B 1.972(3), Ti1–N1B 2.167(4) Å]. The bridging ethylene unit adopts a *gauche*

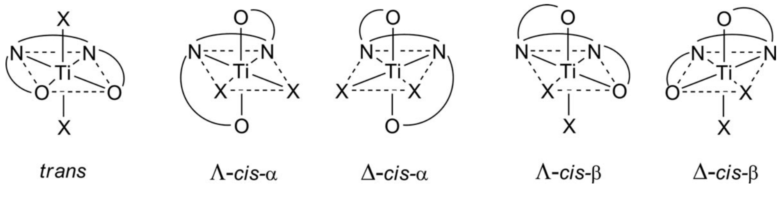


Figure 1. Potential octahedral isomers (sketches) for complex ((S_p,S_p))-**4** (X = O*i*Pr).

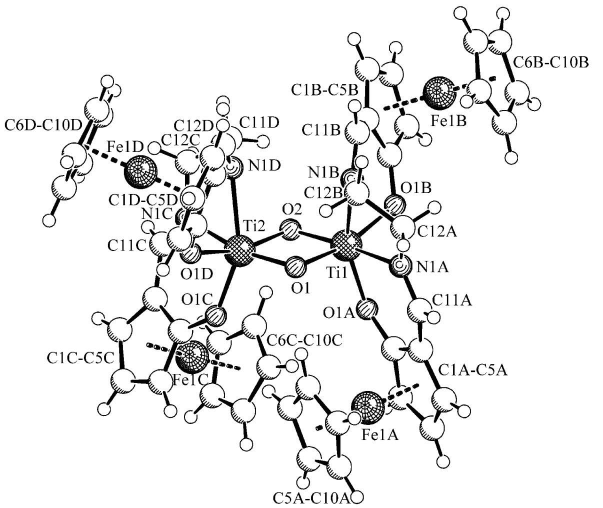
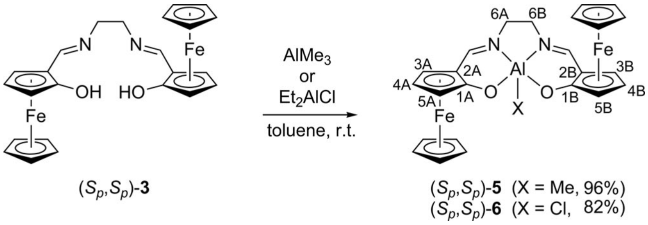


Figure 2. A view of the molecular structure of the partial hydrolysis product of (S_p,S_p)-**4**.

conformation, with a typical dihedral angle of –44.9(5)° (N1A–C12A–C12B–N1B).[15]

To further explore its coordination chemistry, the planar chiral “ferro-salen” ligand (S_p,S_p)-**3** was treated with equimolar amounts of trimethylaluminum or diethylaluminum chloride, yielding the corresponding methylaluminum [(S_p,S_p)-**5**] and chloroaluminum [(S_p,S_p)-**6**] complexes, respectively (see Scheme 3). Both complexes were obtained as purple solids in good yields and characterized by NMR and IR spectroscopy and elemental analysis.



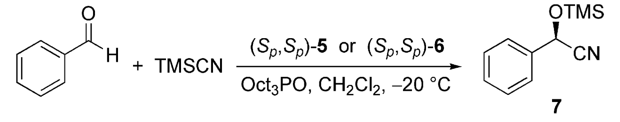
Scheme 3. Synthesis of complexes (S_p,S_p)-**5** and (S_p,S_p)-**6** (including NMR numbering scheme).

Complex (S_p,S_p)-**5** exhibits a C_1-symmetric structure in solution, as expected for a five-coordinate aluminum complex. In the ^{1}H NMR spectrum (C_6D_6, 298 K), the Al methyl group shows a typical resonance at high field (δ = –0.46 ppm, 3 H), the resonances for the CHN groups can be found as singlets at δ = 7.62 and 7.60 ppm (each 1 H), respectively (^{13}C NMR: δ = 172.4 and 172.2 ppm). The protons of the substituted Cp-rings give rise to six separate signals (δ = 4.63, 4.59, 3.93, 3.90, 3.83, 3.80 ppm, each 1 H), the unsubstituted Cp-rings show two singlets at δ = 4.18 and 3.96 ppm (each 5 H). In addition, all four protons of the ethylene bridge give clearly separated resonances (δ = 3.21, 2.75, 2.53, 2.47 ppm), showing large ^2J coupling constants between the diastereotopic geminal methylene protons [^2J(H-6^A,H-6$^{A'}$) = 12.0 Hz, ^2J(H-6^B,H-6$^{B'}$) = 12.7 Hz].

In contrast to these findings, the analogous chloroaluminum complex (S_p,S_p)-**6** seems to possess a fluctuating structure at room temperature. This leads to the observation of a single set of signals in the ^{1}H NMR spectrum at room temperature (CD_2Cl_2), according to a pseudo C_2-symmetric structure. Upon cooling of the solution to –85 °C, the dynamic fluctuation can be slowed down to an extent that allows the observation of separate signals for both ferrocene subunits [e.g., δ = 8.60, 8.54 (CHN$^{A/B}$), 4.20, 4.08 ppm (Cp$^{1/2}$)], which is in agreement with an expected C_1-symmetric structure of the pentacoordinate Al complex. Presumably, the ionic character of the Al–Cl bond allows dissociation of the Cl$^-$ anion, leading to fast equilibration between two identical C_1-symmetric structures by a chloride dissociation/reassociation process.[16] The activation energy for the equilibration process was determined to be 11.4 ± 0.3 kcal mol^{-1} by VT-NMR studies (for details see the Supporting Information).

The chiral (ferro-salen) metal complexes were subsequently tested for their potential use in catalytic asymmetric cyanohydrin synthesis.[17] We therefore investigated the addition of trimethylsilylcyanide to benzaldehyde catalyzed by complexes (S_p,S_p)-**4**/**5**/**6**, a reaction which has been shown to be catalyzed by a variety of (salen)titanium[18] and (salen)aluminum[19] complexes with high levels of asymmetric induction.

The use of Ti complex (S_p,S_p)-**4** for the asymmetric silylcyanation of benzaldehyde only resulted in poor enantiomeric excesses (<20% *ee*) and was additionally hampered by the extremely high sensitivity of the catalyst towards hydrolysis. We thus focused on the use of the aluminum complexes (S_p,S_p)-**5** and (S_p,S_p)-**6** (see Scheme 4), which after optimization of the reaction conditions gave rise to desired protected cyanohydrin **7** in excellent yields (97–99%) and in enantiomeric excesses of up to 62% *ee*.



Scheme 4. Asymmetric cyanohydrin synthesis using (ferro-salen)Al complexes (S_p,S_p)-**5**/(S_p,S_p)-**6**.

The best stereoselectivities for cyanide addition were found when performing the reactions in dichloromethane at –20 °C, and using rather low catalyst loadings of 1–5% (see Table 1). The use of phosphane oxide additives was found to profoundly enhance the stereoselectivity of the addition reaction, as had been noted earlier by other groups.[19] The best results were obtained with the use of trioctylphosphane oxide[20] (as compared to triphenylphosphane oxide), giving enantiomeric excesses of 53% *ee* for (S_p,S_p)-**5** and 62% *ee* for (S_p,S_p)-**6**. In both cases, the (*R*)-enantiomer of the product was obtained as the major isomer.

Table 1. Results of the asymmetric addition of TMSCN[a] to benzaldehyde by using complexes (S_p,S_p)-**5** and (S_p,S_p)-**6**.

Catalyst	Cat. loading [mol-%]	Oct_3PO [mol-%]	Time [h]	Yield [%]	ee[b] (conf.)[c] [%]
(S_p,S_p)-**5**	5	100	72	97	53 (*R*)
(S_p,S_p)-**6**	1	5	24	99	62 (*R*)

[a] 2.5 equiv. of TMSCN were used. [b] Determined by GC on a β-cyclodextrin phase. [c] Determined by comparison of the optical rotation with literature values.[19a]

Conclusions

In conclusion, we have described an improved revised synthesis of the "Ito-ligand" (S_p,S_p)-**3**, which allows the synthesis of this planar chiral salen analogue in enantiomerically pure form and in high yield. First examples of the coordination chemistry of this ligand have been described, showing the versatile coordination chemistry of this ligand set. The (ferro-salen)Al complexes (S_p,S_p)-**5** and (S_p,S_p)-**6** have successfully been used for the asymmetric trimethylsilylcyanation of benzaldehyde, giving the target cyanohydrin **7** in modest enantiomeric excesses. This shows that the use of this special ligand bearing "only" planar chirality can allow significant chiral induction in asymmetric catalysis. We think that this will allow the successful use of these "ferro-salen" ligands in the future, especially upon combination of the planar chiral ferrocene units with further elements of chirality, which is under current investigation in our group.

Supporting Information (see footnote on the first page of this article): Experimental details for the preparation of all new compounds (including detailed analytical data); detailed procedures for the cyanation reactions and crystallographic information.

Acknowledgments

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[11] Analytical data for (S_p,S_p)-**3**: M.p. 161 °C (DSC). $[\alpha]_D^{20}$ = –2120 (c = 0.200, CH_2Cl_2). ^1H NMR (499.8 MHz, C_6D_6, 298 K, TMS): δ = 9.41 (br. s, 2 H, O*H*), 7.87 (s, 2 H, C*H*N), 4.40 [dd, ^3J(H,H) = 2.5 Hz, ^4J(H,H) = 1.3 Hz, 2 H, 3-H], 4.07 (s, 10 H, Cp), 3.74 [dd, ^3J(H,H) = 2.7 Hz, ^4J(H,H) = 1.3 Hz, 2 H, 5-H], 3.63 [dd, ^3J(H,H) = 2.7 Hz, ^3J(H,H) = 2.5 Hz, 2 H, 4-H], 3.28 (m, 2 H, 6-H), 3.15 (m, 2 H, 6′-H) ppm. ^{13}C NMR (125.7 MHz, C_6D_6, 298 K, TMS): δ = 167.7 (*C*HN), 127.3 (C-1), 70.1 (Cp), 64.0 (C-2), 62.8 (C-4), 62.2 (C-5), 60.7 (C-6), 58.3 (C-3) ppm. IR (KBr): $\tilde{\nu}$ = 1631 cm^{-1} (C=N). $C_{24}H_{24}Fe_2N_2O_2$ (484.15): calcd. C 59.54, H 5.00, N 5.79; found C 59.01, H 5.11, N 5.57.

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