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Asymmetric Transfer Hydrogenation of Aromatic Ketones Catalyzed by New Chiral C2-Symmetric Bis(sulfonyl) tetraaza Ligands Complexed with [Ru(η^6-*p*-cymene)Cl_2]$_2$ in Water

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Abstract In this report, a new series of C2-symmetric bis(sulfonyl) tetraaza ligands were synthesized from (1S,2S)-1,2-diarylethylenediamine analogues and tested in the asymmetric transfer hydrogenation (ATH) of aromatic ketones by complexing with [Ru(η^6-*p*-cymene)Cl_2]$_2$ employing sodium formate as hydrogen source in neat water. A moderate to excellent conversion (~99.8 %) and overall satisfying enantioselectivity (~92.8 %) were obtained with varied electronic and steric effects of the substituents on ligands and substrates.

Keywords Bis(sulfonyl) tetraaza · Asymmetric transfer hydrogenation · Ruthenium · Ketones · HCOONa

1 Introduction

Asymmetric transfer hydrogenation (ATH) is a typical and effective method to obtain chiral secondary aromatic alcohols, which are essential intermediates in synthesizing physiological active pharmaceuticals under its safe and mild reaction condition. Noyori et al. [1–4] have achieved high enantioselectivity up to 90 % with Ru–TsDPEN complexes in the reduction of ketones and imines for the first time since the transition-metal catalyzed ATH was reported in 1970s [5]. Since then, lots of efforts have been put into this field using monotosylated 1,2-ethylenediamine [6–12], amino alcohols [13–15], tetraaza [16–21], e.g. especially bis(sulfonyl) tetraaza ligands [18–21] to optimize the conversion and stereoselectivity of the catalytic system.

The previous study in our group has provided a useful insight for the design of efficient C2-symmetric chiral tetraaza ligands (Fig. 1) [22]. Inspired by these results, we aimed to explore the electronic and steric effects on the catalytic activity of ligands by developing a new set of bis(sulfonyl) tetraaza ligands derived from (1S,2S)-diphenylethylenediamine complexed with [Ru(η^6-*p*-cymene)Cl_2]$_2$ and used in the ATH of acetophenone and propiophenone as well as their analogues in water with sodium formate as the hydrogen source [23–25]. In this work, we report on the preparation of five C2-symmetric chiral bis(sulfonyl) tetraaza ligands, **3a–3e**, which are used in the Ru-catalyzed asymmetric transfer hydrogenation of aromatic ketones, giving the corresponding optically active secondary alcohols in moderate to excellent conversion together with favorable enantioselectivity.

2 Experimental

All ATH reactions were carried out under nitrogen atmosphere using oven-dried glassware in oil bath. Acetophenone and dichloromethane were distilled according to standard methods. (1R, 2R)-1,2-bis-(2-hydroxyphenyl)-1,2-diaminoethane and 1,3-benzenedisulfonyl chloride were purchased from Sigma-Aldrich (Steinem, Germany) and TCI (Shanghai, China), respectively. 4′-nitrobenzaldehyde, 4′-trifluoromethylbenzaldehyde and 2′-methyloxybenzaldehyde were obtained from J&K chemicals (Shanghai, China). [Ru(η^6-*p*-cymene) Cl_2]$_2$ was from Alfa Aesar (Ward Hill, Massachusetts, UK). The ^{1}H NMR and ^{13}C NMR spectra were recorded on a 500 MHz Bruker spectrometer. Chiral GC analysis was carried out using

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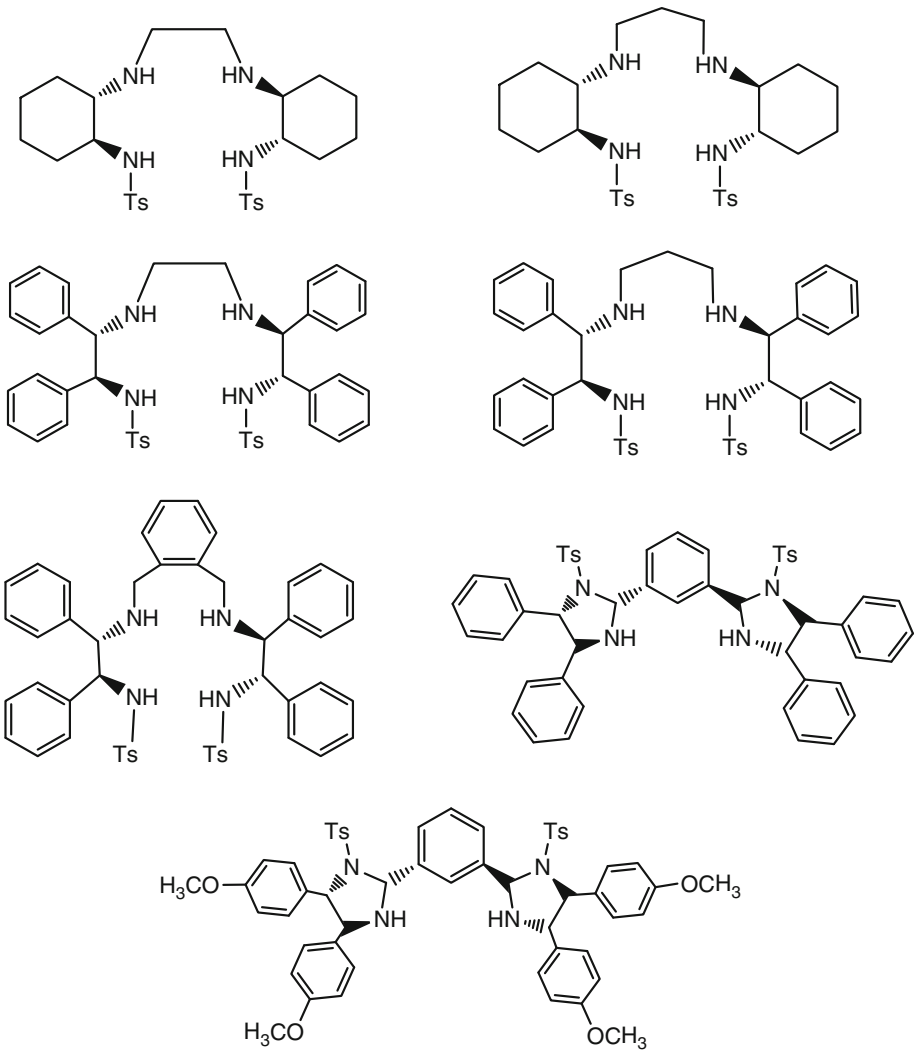


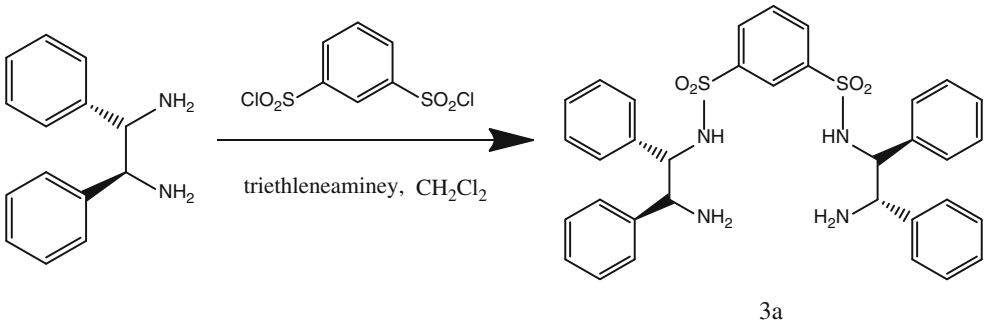
Fig. 1 The chiral tetraaza ligands in the previous study of our group (Ts = tosyl)

Agilent GC 7890A with CP-Chiralsil-Dex-CB Chiral column (Santa Clara, CA, USA). Mass spectra were measured with Agilent (Santa Clara, CA, USA) 5979 spectrometer. LC–MS was measured with Agilent (Santa Clara, CA, USA) Infinity 1290 LC and 6530 Accurate-Mass Q-TOF MS.

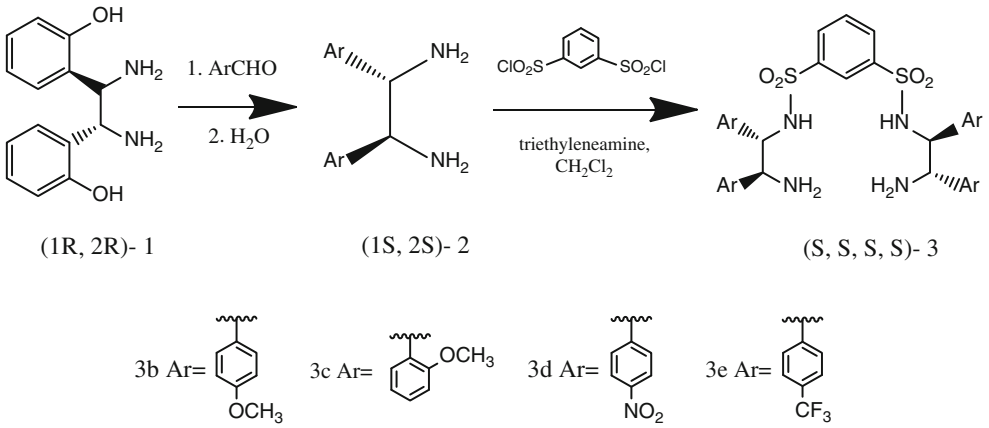
2.1 Preparation of the C2-Symmetric Bis(sulfonyl) tetraaza Ligands **3**–**3e**

*2.1.1 Preparation of (S,S,S,S)-N,N-bis(1,2-diphenylethylenediamino)-1,3-benzenedisulfonyl amine (**3a**) (Scheme 1)*

A solution of (1S,2S)-1,2-diphenylethylenediamine (3 mmol, 0.637 g) in dichloromethane (30 mL) was mixed with triethylamine (0.5 mL, 2.8 mmol). The mixture was cooled to 0 °C and 1,3-benzenedisulfonyl chloride (0.275 g, 1 mmol) dissolved in dichloromethane (10 mL) was added dropwise for 2 h. The mixture was stirred under room temperature for another 12 h. The solvent was removed under reduced pressure. The mixed solid was dissolved in dichloromethane (20 mL) and filtered, and then dichloromethane was removed under reduced pressure. Repeat the procedure of dissolving and filtering for 3 times until affording a yellow solution. Finally the solvent was removed under reduced pressure to obtain **3a** as light yellow powder (0.875 g, 96 % yield). $[\alpha]_D^{20}$: +88.4° (c 0.1, $CHCl_3$). ^{1}H NMR (500 MHz, DMSO-d_6): δ (ppm) 3.375 (6H, br singlet, N*H*), 3.993 (2H, d, J = 7.5 Hz, NH–C*H*), 4.318 (2H, d, J = 7.5 Hz, NH–C*H*), 6.869–6.920 (10H, m, C*H* of phenyl), 7.081–7.090 (10H, m, C*H* of phenyl), 7.192 (1H, m, C*H* of –SO_2–phenyl), 7.290 (2H, dd, J = 1.5 Hz, 8.0 Hz, C*H* of –SO_2–phenyl), 7.696(1H, singlet, C*H* of –SO_2–phenyl). ^{13}C NMR (125 MHz, DMSO-d_6): δ (ppm) 60.219(*C*HNH), 64.227(*C*HNH), 124.713, 127.310, 127.824, 127.984, 128.092, 128.138, 128.289, 128.577, 129.494, 138.412, 140.724, 140.087, 141.092. EI-MS: $[M + H]^+$: 633.



Scheme 1 Preparation of (S, S, S, S)-*N,N*-bis(1,2-diphenylethylenediamino)-1,3-benzenedisulfonyl amine (**3a**)



Scheme 2 Preparation of (S, S, S, S)-*N,N*-bis(1,2-diarylethylenediamino)-1,3-benzenedisulfonyl amine (**3b**–**3e**)

2.1.2 *Preparation of (S,S,S,S)-N,N-bis(1,2-bis (4′-methyloxybenzene) ethylenediamino)-1,3-benzenedisulfonyl amine (**3b**–**3e**) (Scheme 2)*

(1S,2S)-2 was prepared according to the literature procedure [26–31]. A solution of (1S,2S)-2 (3 mmol) in dichloromethane (30 mL) was mixed with excess triethylamine (1.5 mL). The mixture was cooled to 0 °C and a solution of 1, 3-benzenedisulfonyl chloride (0.275 g, 1 mmol) in dichloromethane (10 mL) was added dropwise over 2 h. After the addition, the mixture was warmed to room temperature and stirred for 12 h. The resulting reaction mixture was washed with hydrochloric acid (2 M, 4 × 5 mL). The dichloromethane layer was dried over anhydrous $MgSO_4$, filtered, and the solvent was removed under reduced pressure to obtain **3b**–**3e**.

3b (0.817 g, 62 % yield) as bright yellow powder. $[\alpha]_D^{20}$: −109° (c 0.1, $CHCl_3$). ^{1}H NMR (500 MHz, DMSO-d_6): δ (ppm) 3.580 (s, 6H, O*CH*$_3$), 3.648 (s, 6H, O*CH*$_3$), 3.991 (2H, d, J = 8.0 Hz, NH–C*H*), 4.283 (2H, d, J = 8.0 Hz, NH–C*H*), 6.441 (4H, d, J = 8.5 Hz, C*H* of phenyl), 6.659 (4H, d, J = 9.0 Hz, C*H* of phenyl), 6.761 (4H, d, J = 8.5 Hz, C*H* of phenyl), 7.014 (4H, d, J = 9.0 Hz, C*H* of phenyl), 7.057 (1H, t, J = 8.0 Hz, C*H* of –SO_2–phenyl), 7.300 (1H, d, J = 1.5 Hz, C*H* of –SO_2–phenyl), 7.316 (1H, d, J = 2.0 Hz, C*H* of –SO_2–phenyl), 7.655 (1H, s, C*H* of –SO_2–phenyl). ^{13}C NMR (125 MHz, DMSO-d_6): δ (ppm) 55.214(O*C*H$_3$), 55.321(O*C*H$_3$), 59.853(*C*HNH), 64.256(*C*HNH), 113.405, 113.576, 124.821, 128.908, 129.063, 129.400, 131.002, 133.251, 142.007, 158.251, 158.556. HR-ESI–MS: $[M + H]^+$: 747.2480.

3c (0.852 g, 65 % yield) as yellow powder. $[\alpha]_D^{20}$: +125° (c 0.1, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 3.421 (6H, s, O*CH*$_3$), 3.553 (6H, s, O*CH*$_3$), 4.811 (2H, s, NH–C*H*), 5.100 (2H, s, NH–C*H*), 6.275 (2H, d, J = 8.0 Hz, C*H* of phenyl), 6.415 (2H, d, J = 7.5 Hz, C*H* of phenyl), 6.714–6.797 (7H, m, C*H* of phenyl), 6.929 (2H, t, J = 7.5 Hz, 8.0 Hz, C*H* of phenyl and –SO_2–phenyl), 7.118 (2H, td, J = 1.0 Hz, 8.0 Hz, C*H* of phenyl), 7.197 (2H, dd, J = 1.5 Hz, 8.0 Hz, C*H* of phenyl), 7.296 (2H, s, C*H* of –SO_2–phenyl), 7.434 (1H, s, C*H* of –SO_2–phenyl). ^{13}C NMR (125 MHz, CDCl3): δ (ppm) 45.784(*C*HNH), 46.066(*C*HNH), 55.387(O*C*H$_3$), 55.936(O*C*H$_3$), 110.240, 111.316, 119.896, 120.326, 129.118, 129.359, 129.581, 130.457, 140.824, 155.838, 156.858. HR-ESI-MS: 747.2480.

3d (0.826 g, 59 % yield) as brownish red powder. $[\alpha]_D^{20}$: −107° (c 0.1, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$):

δ (ppm) 4.197 (4H, s, NH–C*H*), 6.815 (1H, t, J = 7.5 Hz, C*H* of –SO_2–phenyl), 6.940 (1H, d, J = 8.0 Hz, C*H* of –SO_2–phenyl), 6.978 (1H, d, J = 7.5 Hz, C*H* of –SO_2–phenyl), 7.209 (1H, t, J = 7.0 Hz, 8.0 Hz, C*H* of –SO_2–phenyl), 7.418 (8H, d, J = 8.5 Hz, C*H* of phenyl), 8.135 (8H, d, J = 8.5 Hz, C*H* of phenyl). ^{13}C NMR (125 MHz, DMSO-d_6): δ (ppm) 45.996(*C*HNH), 82.891, 89.176, 123.765, 123.853, 123.896, 124.168, 128.896, 128.925, 129.392, 129.664. HR-ESI-MS: $[M + H]^+$: 807.1454.

3e (1.056 g, 70 % yield) as pinkish purple solid. $[\alpha]_D^{20}$: −95.6° (c 0.1, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 4.204 (2H, d, J = 4.5 Hz, NH–C*H*), 4.490 (2H, d, J = 5.0 Hz, NH–C*H*), 6.818 (1H, t, J = 7.5 Hz, 8.0 Hz, C*H* of –SO_2–phenyl), 7.299 (4H, d, J = 8.0 Hz, C*H* of phenyl), 7.357 (4H, d, J = 8.0 Hz, C*H* of phenyl), 7.382 (4H, d, J = 8.5 Hz, C*H* of phenyl), 7.450 (4H, d, J = 8.5 Hz, C*H* of phenyl), 7.546 (3H, d, J = 8.0 Hz, C*H* of –SO_2–phenyl), 7.951 (1H, s, C*H* of –SO_2–phenyl). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 61.598(*C*HNH), 62.706(*C*HNH), 125.286(CF_3), 125.545(CF_3), 126.664 (CF_3), 126.920(CF_3), 127.275, 129.419, 129.527, 129.919, 130.172, 140.816, 142.535, 144.575, 146.998. HR-ESI-MS: $[M + H]^+$: 899.1587.

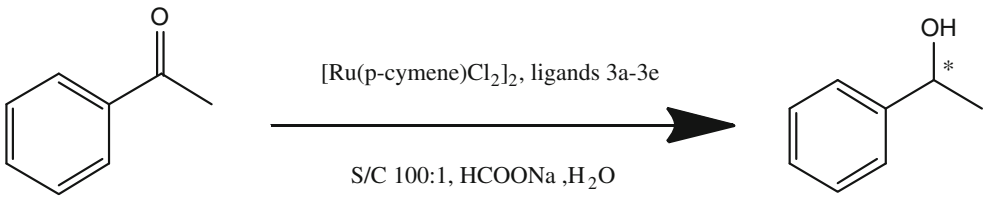
2.2 General Procedure of ATH in Water

The complex-precursor [Ru(η^6-*p*-cymene)Cl_2]$_2$ (0.005 mmol, 3.1 mg) and the ligands **3a**–**3e** (0.011 mmol) were mixed in degassed water (2 mL) and stirred at 40 °C for 1 h, subsequently, anhydrous sodium formate (10 mmol, 0.6801 g) and the substrate (1.0 mmol) were added. At 60 °C the mixture was heated for another 11 h. The suspension was extracted with dichloromethane (3 × 5 mL). The oganic layers were combined, dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure, and then passed through a short silicagel column. The enantiomeric excess (ee) was measured by Chiral GC analysis along with the conversion.

3 Results and Discussion

To investigate the electronic and steric effects of ligands, we prepared five bis(sulfonyl) tetraaza ligands with different substituents as indicated in Schemes 1 and 2, using the ATH of acetophenone to be a model reaction. The corresponding results are listed in Table 1. The ligands **3b**, carrying an electron donating group on the phenyl in para-position, displayed lower catalytic conversion (85.3 %) and enantioselectivity (82.6 % (S)-enantiomer) comparing to **3a** (entries 2 and 4). The ligands bearing an electron withdrawing group may have a complicated impact on the ligands' activities. As described in Table 1, the reduction under **3e** proceeded smoothly giving a conversion of 98.3 % better than that of **3a** (86.0 %) at 40 °C (entries 1 and 7). The case of entry 2 only displayed a slight loss of enantioselectivity about 2.6 % comparing to **3a** (90.0 %)

Table 1 A comparison of Ru(II)–Ligand **3a**–**3e** catalysts for the asymmetric transfer hydrogenation of acetophenone by sodium formate in water



Entry	Ligand	Conversion (%)[a]	ee (%)[a,b]	Configuration[a]
1[c]	**3a**	86.0	92.8	S
2	**3a**	99.8	90.0	S
3[c]	**3b**	78.8	91.7	S
4	**3b**	85.3	82.6	S
5	**3c**	72.4	78.4	S
6	**3d**	22.0	42.3	R
7[c]	**3e**	98.3	81.5	S
8	**3e**	99.8	87.4	S

Reaction conditions: Catalyst: 1.1×10^{-5} mol; HCOONa: 1.0×10^{-2} mol; substrate: 1.0×10^{-3} mol; water: 2 cm^3; temperature 60 °C; reaction time 11 h; nitrogen protection

[a] Determined by chiral GC (CP-Chiralsil-Dex-CB Chiral column)

[b] Enantiomeric excess

[c] Reaction temperature 40 °C

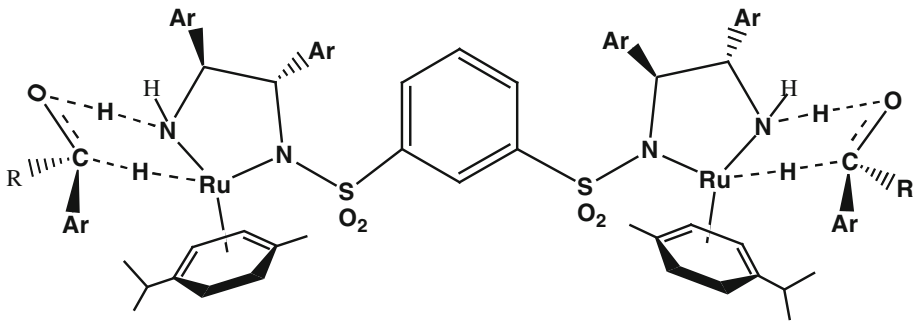
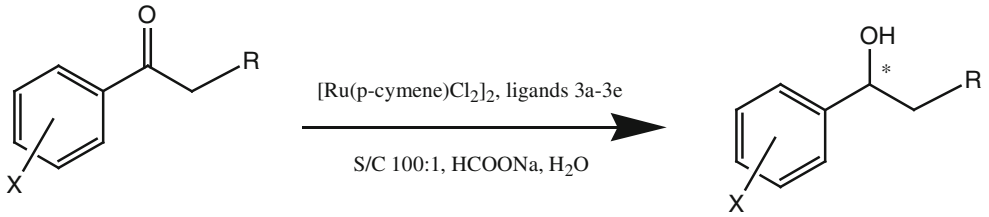


Fig. 2 The proposed six-membered metal-ligand-substrate transition state (TS)

Table 2 A comparison of Ru(II)–Ligand **3a**–**3e** catalysts for the asymmetric transfer hydrogenation of aromatic ketones by HCOONa in water



(X=Cl, NO_2, OCH_3; R= CH_3, Cl, CH_2Cl, $N(CH_3)_2$)

Entry	Ligand	Substrate	Conversion (%) (the reduction by-product)	ee (%)[a,b]	Configuration[a]
1	**3a**	4′-chloroacetophenone	99.8	80.4	S
2	**3b**	4′-chloroacetophenone	95.9	68.5	S
3	**3c**	4′-chloroacetophenone	78.5	79.6	S
4	**3d**	4′-chloroacetophenone	19.0	44.2	R
5	**3e**	4′-chloroacetophenone	99.7	72.3	S
6	**3a**	2′-chloroacetophenone	99.7	87.8	S
7	**3b**	2′-chloroacetophenone	94.7	75.6	S
8	**3c**	2′-chloroacetophenone	76.9	86.3	S
9	**3d**	2′-chloroacetophenone	36.5	48.5	R
10	**3e**	2′-chloroacetophenone	95.6	84.5	S
11	**3a**	4′-nitroacetophenone	78.2	79.4	S
12	**3b**	4′-nitroacetophenone	86.4	73.2	S
13	**3e**	4′-nitroacetophenone	67.5	79.2	S
14	**3a**	4′-methyloxyacetophenone	52.4	91.5	S
15	**3b**	4′-methyloxyacetophenone	48.6	86.4	S
16	**3a**	2-chloroacetophenone	56.2 (9.8)	91.0	S
17	**3b**	2-chloroacetophenone	51.9 (6.7)	87.7	S
18	**3c**	2-chloroacetophenone	52.0 (20.5)	82.6	S
19	**3d**	2-chloroacetophenone	65.8 (9.8)	60.8	R
20	**3e**	2-chloroacetophenone	57.2 (13.9)	87.7	S
21	**3a**	Propionphenone	55.6	85.2	S
22	**3b**	Propionphenone	51.8	79.4	S
23	**3e**	Propionphenone	96.2	80.2	S
24	**3a**	3-chloropropionphenone	33.8 (52.6)	90.4 (85.2)	S
25	**3a**	3-*N*,*N*-dimethylpionphenone	−(99.5)	–	–

Reaction conditions: Catalyst: 1.1×10^{-5} mol; HCOONa: 1.0×10^{-2} mol; substrate: 1.0×10^{-3} mol; water: 2 cm^3; temperature 60 °C; reaction time 11 h; nitrogen protection

[a] Determined by chiral GC (CP-Chiralsil-Dex-CB Chiral column)

[b] Enantiomeric excess

(Entry 8). It is suggested that the electron donating effect depress the stability of the six-membered metal-ligand-substrate transition state (TS, Fig. 2) [19, 20, 32, 33]. The double metal complex in TS is confirmed by the elemental analysis of the isolated Ru(II) complex with **3a** when mixing 1:1 ratio of Ru to ligand **3a** in water at 60 °C. However the electron withdrawing effect favors the formation of hydrogen bonds in TS via increasing the nucleophilicity of ligands to Ru(II). The electron effects on activity are in accord with Noyori's theoretical calculations [32]. Moreover, by building a more crowded space around the chiral center in the ligands, both steric hindrance and electron donating effect on the ortho-position (ligand **3c**, entry 5) exerted a more negative influence on conversion (72.4 %) and enantionselectivity (78.4 %, (S)-enantiomer). When (S,S,S,S)-tetraaza ligand and *p*-cymene were used as ancillaries, two (R)-configured Ru(II) centers were obtained, in which two parts of the C2 symmetric ligand were bound to metals independently. The transfer hydrogenation via this (R)-configured TS with S conformation of carbonyl carbon atom, resulting perhaps from the attractive CH/π interaction between *p*-cymene ligand of the Ru-complex and the aryl substituent in the substrate, afforded (S)-enantiomer.

Surprisingly, when nitro was imported in para-position (**3d**), the yield of the expected 1-phenethylalcohol fallen to 22.0 % and a reversion of enantioselectivity was observed. This was out of what we expected as only (S)-enantiomer being the predominant product in the previous reactions. The same reversion was caught in other substrates such as entries 4, 9 and 19 exhibited in Table 2. It is speculated that the delocalized π-bond of nitro causes this distinctness of enantioselectivity and conversion by dragging the hydrogen attached upon the prochiral carbon.

Furthermore, the ATH of different aromatic ketones (substrates) was explored under the same conditions as previous. The effect of electron withdrawing had a positive impact on the ATH over the electron donating groups on substrates as shown in entries 2, 7, 12 and 15 in Table 2. It may be attributed to the electron withdrawing effect which giving more stable hydrogen bonds consisting in TS. Comparing the ATH of 2-chloroacetophenone and propionphenone with acetophenone under the same ligands (such as entries 20 and 23 in Table 2, together with entry 8 in Table 1), we found that a methyl or chlorine atom on α-position of carbonyl group discouraged the performance of ATH. This revealed that the bulkier around the carbonyl, the lower stability of the TS. Notably, in entries 16–20, the ATH of 2-chloroacetophenone had furnished acetophenone as a substitution by-product (9.8–20.5 %). But we didn't trace the reduction by-product (1-phenylalcohol), indicating the priority of ATH to the side reaction of chlorine atom replacement. In entry 24, the ATH of 3-chloropropionphenone gave much lower yield but more replacement by-product (propionphenone, 13.5 %) and the reduction by-product (1-phenylpropanol, 52.6 %). It seems that the nucleophilicity of the R group on substrates enables the ketones to block the ligands to coordinate with the metal center. In order to prove what was hypothesized, we tried the 3-*N*,*N*-dimethylpionphenone as a substrate. We failed to get the expected alcohol but all propionphenone with its reduction outcome (1-phenylpropanol), which was in agreement with the results in Wills' and Qu's experiments [34, 35].

4 Conclusions

Herein we report a new series of C2-symmetric bis(sulfonyl) tetraaza ligands. By complexing with Ru(η^6-*p*-cymene)$Cl_2]_2$, the ligands exhibited good catalytic activities in the asymmetric transfer hydrogenation of aromatic ketones employing sodium formate as the hydrogen source in neat water at a modest temperature under insert gas protection. A moderate to excellent conversions and overall satisfying enantioselectivities of the chiral secondary alcohols were obtained under ligand **3a** and **3e**. The experiment results reveal that the electron donating effect and steric effect corrode the reactivity of the ligands but the electron withdrawing effect enhances the reactivity via consolidating the sixed-membered transition state. However, given the applicability of the catalytic system, the conditions for a wider range of aromatic ketones are still in a further performance optimization.

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