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Cyclopropanation of styrene with diazoacetates catalyzed by copper and rhodium complexes of new chiral 2,2′:6′,2″-terpyridines derived from natural occurring compounds

Giorgio Chelucci*, Antonio Saba, Franco Soccolini, Davide Vignola

Dipartimento di Chimica, Università di Sassari, Via Vienna 2, I-07100 Sassari, Italy

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Abstract

Three new chiral 2,2′:6′,2″-terpyridines (terpy) were prepared from (−)-β-pinene, (+)-camphor and (+)-2-carene and the corresponding copper and rhodium complexes were assessed as chiral catalysts for the enantioselective cyclopropanation of styrene with diazoacetates. Enantioselectivities up to 64% were obtained.

Keywords: Chiral terpyridines; Copper complex; Rhodium complex; Cyclopropanation reaction; Enantioselectivity

1. Introduction

There has been a continuous interest in the 2,2′:6′,2″-terpyridine (terpy) compounds due to their rich coordination chemistry. Indeed, complexes of terpys afford compounds which are useful in supramolecular chemistry [1,2], in molecular biology [3,4] or photochemistry [5]. Despite the large field of application of this type of ligand, only recently some chiral terpys and their application in asymmetric catalysis have been reported. We prepared and assessed the terpys **1** and **2** (Scheme 1) as chiral controllers in some enantioselective processes such as the Pd-catalyzed allylic alkylation of 1,3-diphenylprop-2-enyl acetate with dimethyl malonate [6], the cyclopropanation of styrene with diazoacetates [7] and the hydrosilylation of acetophenone with diphenylsilane [8]. Recently, Kwong and we have independently introduced the use of chiral terpys **3** as ligands for asymmetric catalysis [9,10] (the synthesis of the unsubstituted ligand **3** (Scheme 1: R = H) has been reported by Von Zelewsky [11]). Thus, while we have reported the application of **3** in the rhodium-catalyzed hydrosilylation of acetophenone with diphenylsilane and in the ruthenium and rhodium-catalyzed cyclopropanation of styrene with ethyl diazoacetate [10], obtaining enantioselectivities up to 59%, Kwong and Lee obtained, in the copper(II)-catalyzed cyclopropanation of olefins, much higher enantiomeric excesses (up to 94%) [9].

In order to extend the applications of chiral terpy ligands in asymmetric reactions we report here the synthesis of the three new chiral terpys **6**–**8** and the study of their catalytic activities in the copper and rhodium-catalyzed cyclopropanation of styrene with diazoacetates [12,13].

The design of these new ligands is such that it is possible to obtain C_2-symmetric terpys bearing a stereogenic center on the carbons bonded to the 6 and 6″-positions of the two side pyridine rings.

* Corresponding author. Tel.: +39-79-229-539; fax: +39-79-229-559.
E-mail address: chelucci@ssmain.uniss.it (G. Chelucci).

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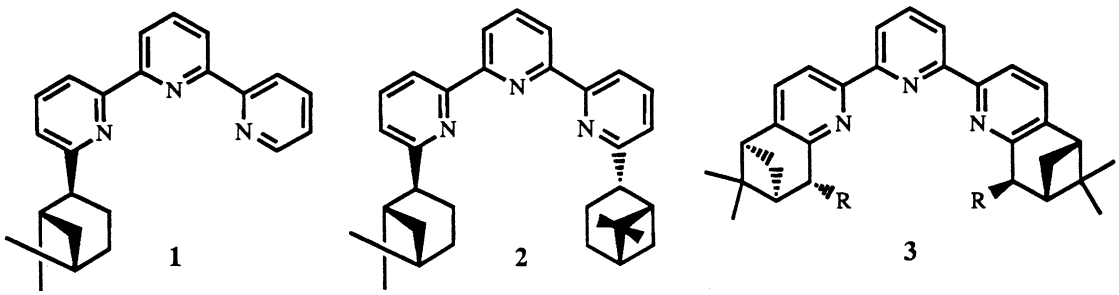


Scheme 1.

In fact, it is indicated in the literature that effective chiral controllers are those ligands in which the substituents at the asymmetric centers are forced to be directed toward the metal ion on its complex-formation [14,15]. To this purpose, (−)-β-pinene, (+)-camphor and (+)-2-carene, easily available building blocks originating from the chiral pool, were selected as appropriate starting materials.

2. Results and discussion

2.1. Synthesis of the ligands

The terpys **6–8** were readily accessible by reaction of the α,β-methylene ketones **9–11** with 2,6-bis(pyridinioacetyl)pyridine iodide (**5**) [16] following the Kröhnke methodology [17] (16–26% yields) (Scheme 2). Ketones **9-11** were in turn obtained from (−)-β-pinene, (+)-camphor and (+)-2-carene following a well-described procedure [18].

With the new ligands in hand, the Rh(terpy)Cl_3 complexes **6a–8a** were prepared in satisfactory yield by heating a methanolic solution of **6–8** with $RhCl_3 \cdot 3H_2O$ (60–68%) under reflux.

2.2. Copper-catalyzed asymmetric cyclopropanation

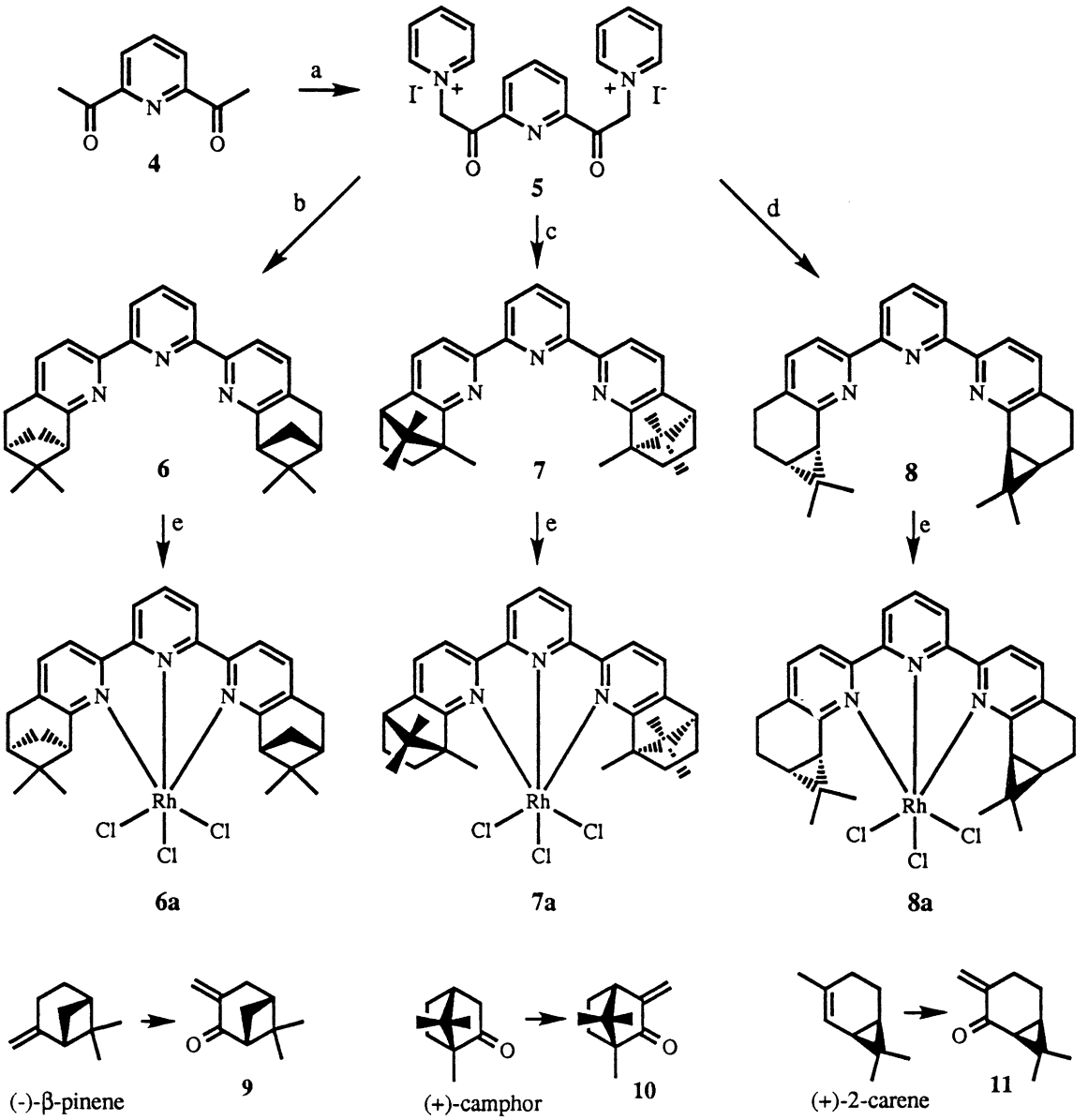
Firstly we compared the results obtained with ligands **3** for the asymmetric cyclopropanation of styrene using copper(II)-terpy catalysts prepared in situ from copper(II) triflate and the ligands **6–8** (Scheme 3). The reaction was carried out at room temperature by slow addition (2 h) of ethyl diazoacetate to a solution of styrene in methylene chloride containing the copper(II)-ligand adduct which was previously activated by stirring with a few equivalents of ethyl diazoacetate. The results obtained in these runs are summarized in Table 1. While the copper(II)-(terpy **6** and **7**) complexes exhibited an acceptable efficiency and afforded diastereomeric cyclopropanes **12** and **13** with moderate yields, that derived from ligand **8** gave poor yield.

The enantioselectivities were low for ligands **6** and **7** (22–33%) and very low (7%) for ligand **8**. The result obtained with **8** was particularly disappointed because, to our expectation, the dimethylcyclopropane ring on the 7,8- and 7′,8′-positions of the two tetrahydroquinoline rings should be able to give to the copper(II)-complex a very high stereodifferentiating ability.

With these results in hand, we decided to evaluate the efficiency of these ligands in the copper(I)-catalyzed asymmetric cyclopropanation of styrene. The results obtained are reported in Table 2. The substitution of copper(II)-complexes with those of copper(I) did not change substantially the yields and diastereoselectivities, but the enantioselectivities were lower.

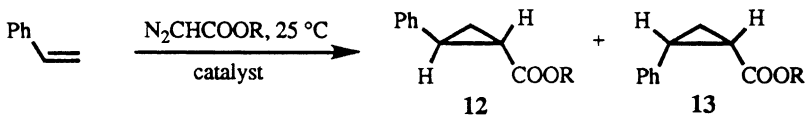
2.3. Rhodium(III)-catalyzed asymmetric cyclopropanation

The particularly disappointing results obtained with Cu(I)- and C(II)-catalysts prompted us to examine the ability of the Rh(III) complexes of these ligands in the cyclopropanation reaction of styrene. This research was inspired by our findings [8] and by the works of Nishiyama et al. who showed that trivalent rhodium complexes, derived from terdentate C_2-symmetrical



a: I_2, pyridine, 100 °C; b: **9**, AcOH, $AcONH_4$, 120 °C, 12h, 16%; c: **10**, AcOH, $AcONH_4$, 140 °C, 20h, 26%; d: **11**, AcOH, $AcONH_4$, 80 °C, 20h, 26%; e: $RhCl_3$ H_2O, EtOH, reflux, 4h, 60-68%.

Scheme 2.



Scheme 3.

Table 1
Enantioselective cyclopropanation of styrene with ethyldiazoacetate using $Cu(OTf)_2$[a]

Ligand	Yield[b] (**12** + **13**) (%)	*Trans*:*cis*[c] (**12**:**13**)	e.e.[c] (%)		Configuration[d]	
			12	**13**	**12**	**13**
6	78	72:28	22	20	1*R*,2*R*	1*S*,2*R*
7	73	75:25	32	34	1*S*,2*S*	1*R*,2*S*
8	33	67:33	7	4	1*R*,2*R*	1*S*,2*R*

[a] The ligand (35 mmol) in CH_2Cl_2 (1.5 ml) was added to a suspension of $Cu(OTf)_2$ (11.4 mg, 31 mmol) in CH_2Cl_2 (1.5 ml). After 2 h styrene (0.715 ml, 6.25 mmol) and diazoacetate (0.315 mmol) were added. After 30 min diazoacetate (2.5 mmol) in CH_2Cl_2 (2.5 ml) was added dropwise over a period of 2 h and then stirred for 16 h.

[b] Isolated yield, based on the diazoacetate, for the mixture of *trans*- and *cis*-cyclopropanes.

[c] Determined by GC analysis on a chiral column.

[d] Assignment according to [20].

bis(oxazolinyl)pyridines (pybox) and $RhCl_3$ with the aid of $AgBF_4$, are effective chiral catalysts for the hydrosilylation of ketones [19].

Thus, the reactions were carried out at room temperature for 24 h by slow addition of ethyl or *t*-butyl diazoacetate (2 h) to a solution of styrene in THF containing the active catalyst which was prepared by the addition of two equivalents of silver triflate to the $Rh(terpy)Cl_3$ complex in THF [19].

The cyclopropanes recovered from the reactions were obtained in moderate yield as a mixture of *trans* and *cis* isomers in a ratio which varies appreciably with the nature of the substituents present on the tetrahydroquinoline rings (Table 3). Enantioselectivities were also moderate with the best result being obtained with the complex **8a** (64%). Unexpectedly, the use of the *t*-butyl diazoacetate in the case of ligand **8a** reduced both the *trans–cis* diastereoselectivity and the enantioselectivity. Moreover, the presence of the *t*-butyl group on the diazoester caused a chiral switch of the configuration of the *cis*-cyclopropane.

In conclusion we have reported the synthesis of three new 2,2′:6′,2″-terpyridine starting from compound originating from the chiral pool and

Table 2
Enantioselective cyclopropanation of styrene with ethyldiazoacetate using Cu(OTf)[a]

Ligand	Yield[b] (**12** + **13**) (%)	*Trans*:*cis*[c] (**12**:**13**)	e.e.[c] (%)		Configuration[d]	
			12	**13**	**12**	**13**
6	73	64:36	20	18	1*R*,2*R*	1*S*,2*R*
7	71	67:33	18	16	1*S*,2*S*	1*R*,2*S*
8	22	70:30	6	4	1*S*,2*S*	1*R*,2*S*

[a] The ligand (34 μmol) in CH_2Cl_2 (2.5 ml) was added to a suspension of $[Cu(OTf)(C_6H_6)_{0.5}]$ (8 mg, 32 μmol) in CH_2Cl_2 (2.5 ml). After 30 min, the mixture was filtered-through packed adsorbent cotton under argon and, to the filtrate, was added styrene (1.59 ml, 13.87 mmol). Ethyl diazoacetate (2.5 μmol) in CH_2Cl_2 (2.5 ml) was added dropwise over a period of 1 h and then la mixture was stirred for 24 h.

[b] Isolated yield, based on the diazoacetate, for the mixture of *trans*- and *cis*-cyclopropanes.

[c] Determined by GC analysis on a chiral column.

[d] Assignment according to [20].

Table 3
Enantioselective cyclopropanation of styrene with diazoacetates using Rh(terpy)Cl_3 complexes[a]

Complex	Diazoacetate	Yield[b] (**12** + **13**) (%)	*Trans*:*cis*[c] (**12**:**13**)	e.e.[c] (%)		Configuration[d]	
				12	**13**	**12**	**13**
6a	Et	73	71:29	10	12	1*S*,2*S*	1*R*,2*S*
7a	Et	71	46:54	8	32	1*R*,2*R*	1*S*,2*R*
8a	Et	78	37:63	52	64	1*R*,2*R*	1*S*,2*R*
8a	^{t}Bu	78	52:48	40	12	1*R*,2*R*	1*R*,2*S*

[a] To solution of the Rh(terpy)Cl_3 complex (0.05 mol) in THF (2 ml) was added AgOTf (0.1 mmol) under argon atmosphere. After 30 min stirring, styrene (12.5 mmol) was added and then a solution of the diazoacetate (2.5 mmol) in THF (2.5 ml) was added dropwise over a period of 2 h and then stirred for 24 h.
[b] Isolated yield, based on the diazoacetate, for the mixture of *trans*- and *cis*-cyclopropanes.
[c] Determined by GC analysis on a chiral column.
[d] Assignment according to [20].

demonstrated that they are poorly suitable chiral controllers in the Cu-catalyzed cyclopropanation of styrene, while they seem to deserve attention for their possible applications in the same reaction when the corresponding Rh(III)-complexes are used. The use of these ligands in other catalytic processes are currently in progress in our laboratory.

3. Experimental

3.1. General methods

Boiling points are uncorrected. Melting points were determined on a Buchi 510 capillary apparatus and are uncorrected. The ^{1}H NMR (300 MHz) spectra were obtained with a Varian VXR-300 spectrometer. Optical rotations were measured with a Perkin-Elmer 241 polarimeter in a 1 dm tube. Elemental analyses were performed on a Perkin-Elmer 240 B analyzer. Gas chromatographic analyses were performed with a HP 5900 chromatograph using He (60 kPa) as the carrier gas.

$RhCl_3 \cdot 3H_2O$, ethyl and *t*-butyl diazoacetate were purchased from Aldrich. **5** was obtained from 2,6-diacetylpyridine (**4**) (Aldrich) following the Ortoleva–King procedure [16].

(1*R*,5*R*)-6,6-dimethyl-3-methylenebicyclo[3.1.1]heptan-2-one (**9**), (1*S*,6*R*)-7,7-dimethyl-3-methylenebicyclo[2.2.1]heptan-2-one (**10**), (1*R*,4*S*)-3-methylene-1,7,7-trimethylbicyclo[4.1.0]heptan-2-one (**11**) were prepared from (1*S*)-(−)-β-pinene $[\alpha]_D^{25}$ −22.0 (neat) (99%, Aldrich), (1*R*)-(+)-camphor $[\alpha]_D^{25}$ +44.1 (ca. 10, C_2H_5OH), (98%, Aldrich) and (1*R*)-(+)-2-carene $[\alpha]_D^{25}$ +90.0 (ca. 6, C_2H_5OH), (97%, Aldrich), respectively, following published methods [18].

3.2. (6S,8S)-2,6-bis(7,7-dimethyl-5,6,7, 8-tetrahydro-6,8-methanoquinolin-2-yl)pyridine (6)

A solution of **5** (12 g, 21 mmol), α,β-methylene ketone **9** (6.3 g, 42 mmol), ammonium acetate (32.3 g, 0.42 mol) in glacial acetic acid (120 ml) was heated at 120–125 °C for 12 h under nitrogen. Then, most of the acetic acid was removed under reduced pressure and the residue taken up with H_2O (600 ml) and extracted with CH_2Cl_2 (2 × 150 ml). The organic phase was washed with a 5% NaOH solution and then extracted with a 10% HCl solution. The acid solution was alkalinized with a 10% NaOH solution and extracted with CH_2Cl_2 (2 × 150 ml). The organic phase was dried on anhydrous Na_2SO_4, the solvent was evaporated and the residue was recrystallized from dichloromethane-diethyl ether to gave pure **6** as a white solid: 1.42 g (16%); m.p. > 250 °C; $[\alpha]_D^{25}$ −9.8 (ca. 0.8, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ 8.39 (d, 2H, J = 8.6 Hz), 8.38 (d, 2H, J = 8.6 Hz), 7.88 (t, 1H, J = 8.6 Hz), 7.57 (d, 2H, J = 8.6 Hz), 3.11 (t, 2H, J = 6.3 Hz), 3.01–3.00 (m, 4H), 2.77 (t, 1H, J = 6.3 Hz), 2.75 (t, 1H, J = 6.3 Hz), 2.38–2.34 (m, 2H), 1.45 (s, 6H), 1.36 (d, 2H, J = 9.9 Hz), 0.71 (s, 6H). Anal. Calcd. for $C_{29}H_{31}N_3$: C, 82.61; H, 7.42; N, 9.97. Found: C, 82.67; H, 7.55; N, 9.92.

3.3. (5S,8R)-2,6-bis(8,9,9-trimethyl-5,6,7,8-tetrahydro-5,8-methanoquinolin-2-yl)pyridine (7)

Compound **7** was obtained as a white solid following the procedure described for the preparation of **6** using the α,β-metylene ketone **10** and carrying out the reaction at 140 °C for 20 h: 2.45 g (26%); m.p. 204–5 °C; $[\alpha]_D^{25}$ −15.8 (ca. 0.6, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ 8.47 (d, 2H, $J = 7.8$ Hz), 8.34 (d, 2H, $J = 7.5$ Hz), 7.88 (t, 1H, $J = 7.8$ Hz), 7.50 (d, 2H, $J = 7.5$ Hz), 2.89 (d, 2H, $J = 3.9$ Hz), 2.20–2.09 (m, 2H), 1.90 (dt, 2H, $J = 12.3$ Hz, 3.6 Hz), 1.40 (s, 6H), 1.34–123 (m, 2H), 1.23–1.13 (m, 2H), 1.02 (s, 6H), 0.60 (s, 6H). Anal. Calcd. for $C_{31}H_{35}N_3$: C, 82.80; H, 7.85; N, 9.35. Found: C, 82.89; H, 7.64; N, 9.44.

3.4. (7R,8S)-2,6-bis(9,9-dimethyl-5,6,7,8-tetrahydro-7,8-methanoquinolin-2-yl)pyridine (8)

Compound **8** was obtained as a white solid following the procedure described for the preparation of **6** using the α,β-metylene ketone **11** and carrying out the reaction at 80 °C for 20 h: 2.29 g (26%); m.p. 210–212 °C; $[\alpha]_D^{25}$ −148.7 (ca. 1.0, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ 8.45 (d, 2H, $J = 8.3$ Hz), 8.28 (d, 2H, $J = 8.3$ Hz), 7.91 (t, 1H, $J = 8.3$ Hz), 7.47 (d, 2H, $J = 7.8$ Hz), 2.92–2.79 (m, 2H), 2.64–2.54 (m, 2H), 2.12–2.03 (m, 4H), 1.94–1.80 (m, 2H), 1.42–1.36 (m, 2H), 1.29 (s, 6H), 0.85 (s, 6H). Anal. Calcd. for $C_{29}H_{31}N_3$: C, 82.61; H, 7.42; N, 9.97. Found: C, 82.77; H, 7.66; N, 9.74.

3.5. Rh(terpy 6)Cl_3 (6a)

A mixture of the terpyridine **6** (1.0 mmol) and $RhCl_3 \cdot 3H_2O$ (0.263 g, 1.0 mmol) in methanol (8 ml) was heated under reflux for 4.5 h. After cooling the precipitate was filtered-off, and recrystallized from dichloromethane-ethyl ether. Finally the crystals were washed with ethyl ether and dried under vacuo to give pure **6a** as an orange solid: 0.410 g (65%); IR (KBr) $\nu_{C=N}$ 1600 cm^{-1} (m); ^{1}H NMR ($CDCl_3$) δ 8.09 (t, 1H, $J = 8.6$ Hz), 7.95 (d, 2H, $J = 8.6$ Hz), 7.66 (d, 2H, $J = 8.6$ Hz), 5.55 (t, 2H, $J = 6.0$ Hz), 3.09–3.07 (m, 4H), 2.81–2.76 (m, 2H), 2.32–2.28 (m, 2H), 1.60 (s, 6H), 0.95 (s, 6H). Anal. Calcd. for $C_{29}H_{31}Cl_3N_3Rh$: C, 55.21; H, 4.95; N, 6.66. Found: C, 55.09; H, 4.87; N, 6.77.

3.6. Rh(terpy 7)Cl_3 (7a)

Compound **7a** was obtained as an orange solid starting from the terpyridine **7** and following the procedure described for the preparation of **6a:** 0.447 g (68%); IR (KBr) $\nu_{C=N}$ 1610 cm^{-1} (m); ^{1}H NMR ($CDCl_3$) δ 8.12 (t, 1H, $J = 8.0$ Hz), 7.82 (d, 2H, $J = 8.0$ Hz), 7.51 (d, 2H, $J = 8.0$ Hz), 7.60 (d, 2H, $J = 8.0$ Hz), 2.85 (d, 2H, $J = 4.0$ Hz), 2.79–2.73 (m, 2H), 2.26–2.11 (m, 2H), 2.10–2.03 (m, 2H), 1.44–1.36 (m, 2H), 1.25 (s, 6H), 0.96 (s, 6H), 0.40 (s, 6H). Anal. Calcd. for $C_{31}H_{35}Cl_3N_3Rh$: C, 56.61; H, 5.37; N, 6.39. Found: C, 56.55; H, 5.30; N, 6.42.

3.7. Rh(terpy 8)Cl_3 (8a)

Compound **8a** was obtained as an orange solid starting from the terpyridine **8** following the procedure described for the preparation of **6a:** 0.378 g (60%); IR (KBr) $\nu_{C=N}$ 1610 cm^{-1} (m); ^{1}H NMR ($CDCl_3$) δ 8.08 (t, 1H, $J = 8.7$ Hz), 7.96 (d, 2H, $J = 8.6$ Hz), 7.77 (d, 2H, $J = 8.6$ Hz), 7.56 (d, 2H, $J = 8.6$ Hz), 4.79 (d, 2H, $J = 8.1$ Hz), 3.00–2.85 (m, 2H), 2.62–2.54 (m, 2H), 2.27–2.16 (m, 2H), 1.78–1.66 (m, 2H), 1.44 (s, 6H), 1.28–1.25 (m, 2H), 0.69 (s, 6H). Anal. Calcd. for $C_{29}H_{31}Cl_3N_3Rh$: C, 55.21; H, 4.95; N, 6.66. Found: C, 55.12; H, 4.77; N, 6.87.

3.8. Asymmetric cyclopropanation of styrene using Cu(I)-complexes: typical procedure

A solution of the ligand (34 μmol) in CH_2Cl_2 (2.5 ml) was added to a suspension of [Cu(OTf)$(C_6H_6)_{0.5}$] (8 mg, 32 μmol) in CH_2Cl_2 (2.5 ml). After 30 min, the mixture was filtered through packed adsorbent cotton under argon and, to the filtrate, styrene (1.59 ml, 13.87 mmol) was added. Then a solution of the diazoacetate ester (2.5 μmol) in CH_2Cl_2 (2.5 ml) was added dropwise over a period of 1 h. The mixture was stirred for 24 h at room temperature and then concentrated in vacuo. The residue was purified by column chromatography on silica gel (hexane/ethyl acetate = 15/1) to afford a mixture of ethyl *trans*- and *cis*-2-phenyl-cyclopropane-1-carboxylates as a colourless oil. The *trans*:*cis* ratio and the e.e. were determined by GC analysis on a diethyl-*t*-butylsilyl-β-cyclodextrin capillary column 25 m × 0.25 mm operated at 60 °C for 5 min, then programmed at

3 °C/min to 160 °C. Retention times: 33.2 min (1*S*,2*S*) and 33.5 min (1*R*,2*R*) for *trans*-**12**; retention times: 31.4 min (1*R*,2*S*) and 31.8 min (1*S*,2*R*) for *cis*-**13**.

3.9. Asymmetric cyclopropanation of styrene using Cu(II)-complexes: typical procedure

The ligand (35 mmol) in CH_2Cl_2 (1.5 ml) was added to a suspension of $Cu(OTf)_2$ (11.4 mg, 31 mmol) in CH_2Cl_2 (1.5 ml). After 2 h, styrene (0.715 ml, 6.25 mmol) and diazoacetate (0.315 mmol) were added. After 30 min, diazoacetate (2.5 mmol) in CH_2Cl_2 (2.5 ml) was added dropwise over a period of 2 h and then the mixture was stirred for 16 h. The solvent was evaporated under vacuo and the residue was then worked up as described above.

3.10. Asymmetric cyclopropanation of styrene using Rh(terpy)Cl_3 complexes: typical procedure

To solution of Rh(terpy)Cl_3 complex (0.05 mol), in THF (2 ml) was added AgOTf (25.7 mg, 0.1 mmol) under argon atmosphere. After 30 min stirring, styrene (1.43 ml, 12.5 mmol) was added and then a solution of ethyl diazoacetate (0.263 ml, 2.5 mmol) in THF (2.5 ml) was added dropwise over a period of 2 h. The mixture was stirred for 24 h and then the solvent and excess olefin were removed under vacuum. The residue was purified by column chromatography on silica gel (hexane/ethyl acetate = 15/1) to afford a mixture of ethyl *trans*- and *cis*-2-phenylcyclopropane-1-carboxylates as a colurless oil. The *trans*:*cis* ratio and the e.e. were determined by GC analysis on a diethyl-*t*-butylsilyl-β-cyclodextrin capillary column 25 m × 0.25 mm operated at 60 °C for 5 min, then programmed at 3 °C/min to 160 °C. Retention times: 33.2 min (*1S,2S*) and 33.5 min (*1R,2R*) for *trans*-**13**; retention times: 31.4 min (*1R,2S*) and 31.8 min (*1S,2R*) for *cis*-**14**.

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