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Review

Chiral pyridine-containing ligands in asymmetric catalysis

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Contents

Abstract

In this article, an overview of the investigations on chiral py-containing ligands in asymmetric catalysis that have carrried out in the past years is provided. The ligands are divided into different types based on the number of pyridine moieties present and the way they are connected in the ligands, namely monopyridine, dipyridine (two pyridines connected by a bridge), bipyridine, phen(1,10-phenanthroline), terpyridine and oligopyridine.

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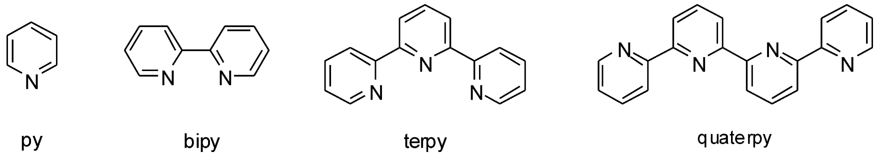
In each section, the synthetic strategies for each type of ligands are discussed and representative examples are given. Selected important results in asymmetric catalysis are mentioned.

Keywords: Pyridine; Bipyridine; Phenanthroline; Terpyridine; Oligopyridine; Chiral ligand; Asymmetric catalysis

1. Introduction

Pyridine (py) is a six-member heterocyclic aromatic ring with a lone pair of electrons at the nitrogen atom. A ligand which forms complexes with various metals, it is important and arguably the best known motif in coordination chemistry. When pys are connected together with linkers, a large variety of polydentate ligands are possible [1]. When pys are connected through a single bond at the 2- and/or 6-position, 2,2′-bipyridine (bipy), 2,2′:6′,2″-terpyridine (terpy), and other oligopyridines are formed. These ligands have played a very important role in the study of the coordination chemistry of transition metal ions [2–7].



Chiral py-containing ligands have been known for some time [8,9] but the development of their applications in asymmetric catalysis had been lacking until 1981, when the first report of chiral py-containing ligands for asymmetric catalysis appeared [10]. The situation further improved as more reports focused on the syntheses of chiral py-containing ligands and their applications in asymmetric catalysis have appeared since then [11–15]. Their high stability against moisture and oxygen in the atmosphere and their interesting coordination chemistry probably account for their attractiveness. The diversity of their structures is the major reason why we are interested in studying them.

In this article, we provide an overview of the investigations on chiral py-containing ligands in asymmetric catalysis that we and other groups have carried out in the past years. Because of our interest in the diversity of the structure, the chiral py-containing ligands are divided into different types based on the number of py moieties present and the way they are connected in the ligands, namely monopy, dipy (two pys connected by a bridge), bipy, phen (1,10-phenanthroline), terpy and polypy. To limit the scope of the article, in each section, we focus on the synthetic strategies for each type of ligand and give representative examples; selected important results in asymmetric catalysis are mentioned to highlight the achievement of these ligands.

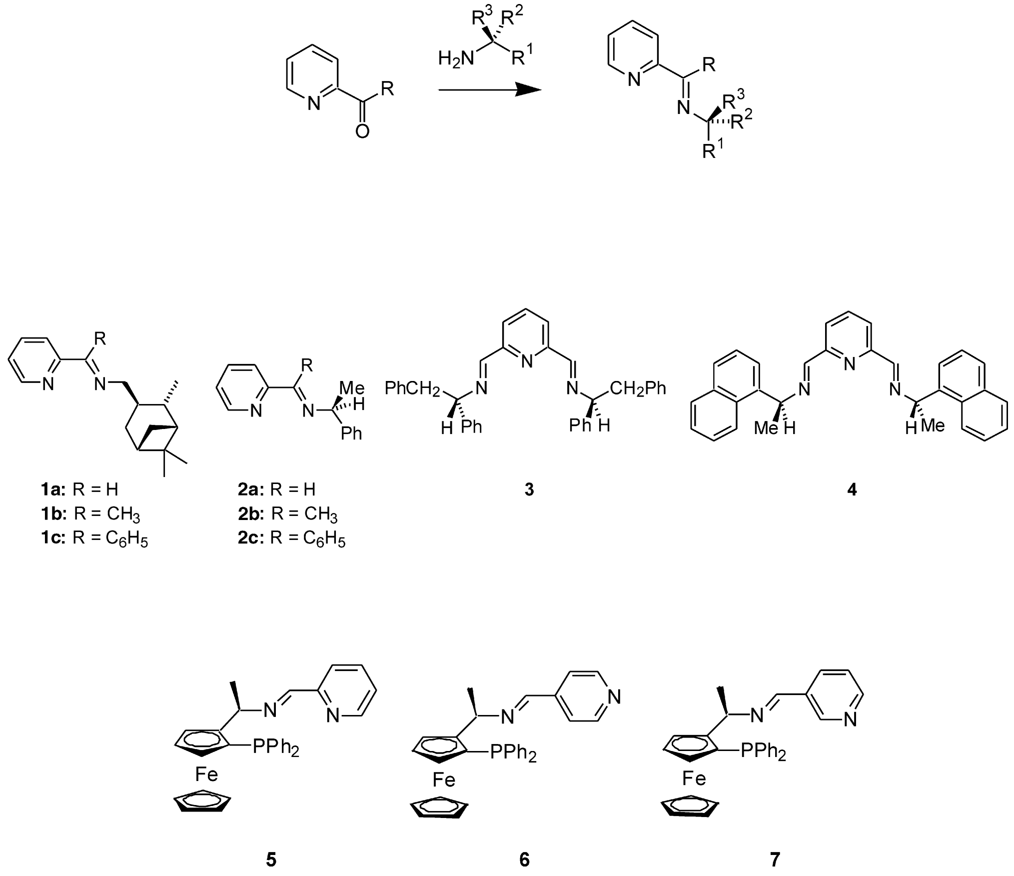
2. Chiral monopyridines

In general, there are four ways to introduce chirality into the py ligands: (I) reaction between achiral pyridyl precursors and chiral reagents, usually a naturally occurring substance or a commercially available compound; (II) asymmetric reduction and dihydroxylation of achiral py substrates; (III) formation of chiral fused py ring by Kröhnke condensation; (IV) resolution of racemate.

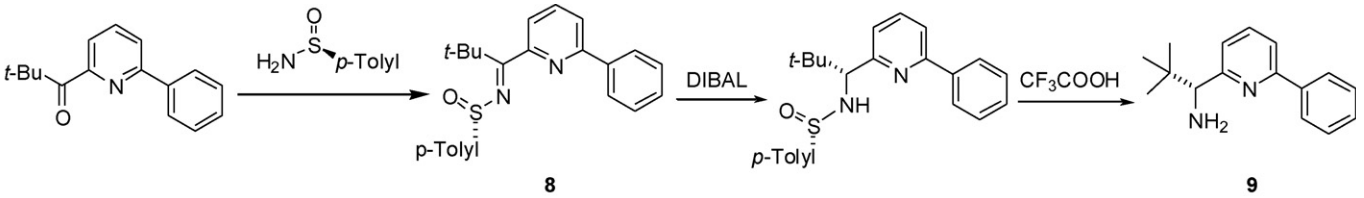
2.1. From achiral pyridines

Most of the reported chiral monopy ligands were prepared from an achiral pyridyl precursor and a chiral reagent, which contains another hetero atom for chelation. Synthetic procedures are usually quite simple, probably because the chiral reagents were most often prepared from naturally occurring substances or commercially available compounds. Typical examples are chiral py-imine ligands **1** and **2**, which were prepared easily by condensation of pyridyl carbonyl compounds with various chiral amines (Scheme 1). Ligands **2a** [8] and **2b** [9] were first synthesized in 1974 but no catalysis was reported. Ligand **1a**, reported by Zassinovich et al. in 1981, was the first chiral py ligand used in asymmetric catalysis; an Ir-catalyzed transfer hydrogenation reaction [10]. Brunner et al. later reported the use of **1** and **2** in the Rh-catalyzed asymmetric hydrosilylation of acetophenone with diphenylsilane [16–18]. Zassinovich et al. then reported the use of **2a** for the Ir-catalyzed asymmetric transfer hydrogenation of *t*-butyl phenyl ketone using propan-2-ol [19]. Ligand **3**, prepared using a similar strategy, was utilized in the Ir-catalyzed asymmetric transfer hydrogenation of 4-phenyl-3-buten-2-one [20]. Monopyridyl bisimine ligand **4**, reported by Bianchini and Lee, was used in the Ru-catalyzed cyclopropanation of styrene with ethyl diazoacetate [21]. Using the same strategy, a number of ferrocenylphosphine-pyridylimine ligands **5**–**7** were synthesized and used in the Pd-catalyzed allylic alkylation of 1,3-diphenylprop-2-enyl pivalate [22].

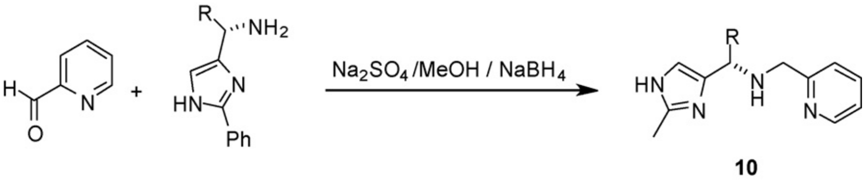
Using pyridylimine as an intermediate, py ligands having amine or aziridine groups were synthesized. The pyridyl amine ligand **9**, obtained by diastereoselective reduction of enantiopure *N*-*p*-toluenesulfinyl ketimine **8** (Scheme 2) [23], was shown to be an efficient catalyst in the Ru-catalyzed transfer hydrogenation reactions [24]. Chiral pyridyl amine **10**, obtained through sodium borohydride reduction of the pyridylimine intermediate (Scheme 3), was used in Cu(II)-catalyzed Henry reaction [25]. Pyridyl aziridine **12**, prepared through stereoselectively addition of *i*PrMgCl to chiral pyridyl imine **11** followed by cyclization to form an aziridine ring (Scheme 4), was used in the Pd-catalyzed allylic alkylation [26].



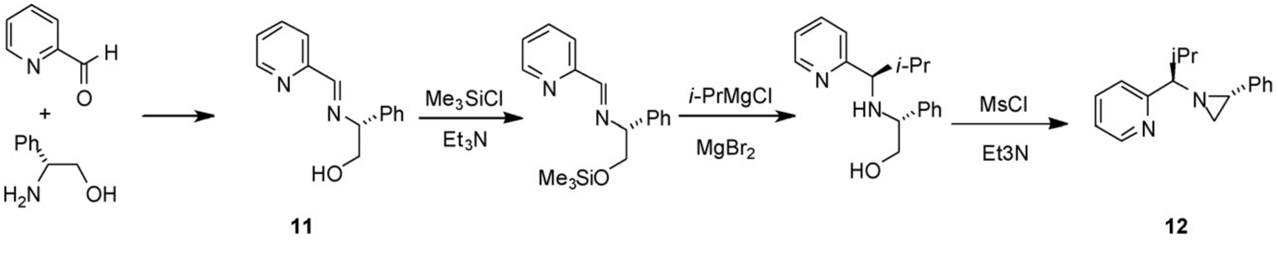
Scheme 1.



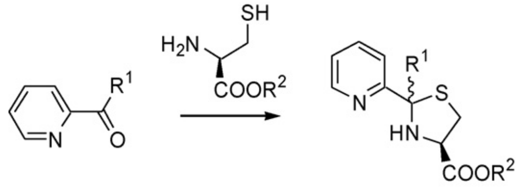
Scheme 2.



Scheme 3.



Scheme 4.



13a: R^1 = H, R^2 = Me
13b: R^1 = H, R^2 = Et
13c: R^1 = Me, R^2 = Me

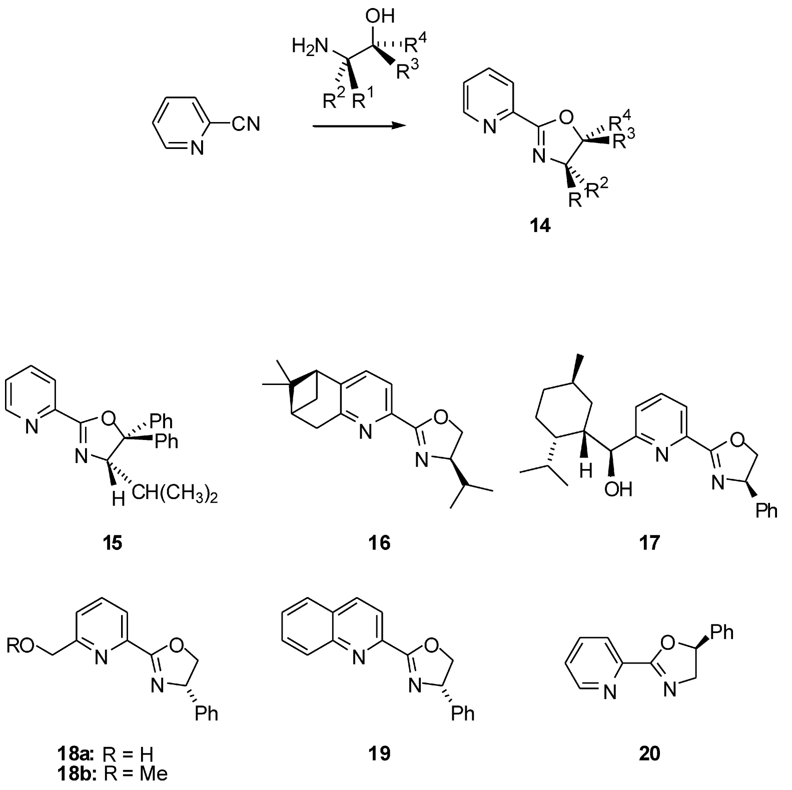
Scheme 5.

Brunner et al. prepared monopys **13a–c**, in 1983, by one-step condensation of pyridyl carbonyl compounds with cysteine derivatives (Scheme 5) [27–29]. Excellent enantioselectivity (up to 97% ee) was obtained when **13b** was used in the Rh-catalyzed asymmetric hydrosilylation of acetophenone with diphenylsilane [28]. They later introduced 2-oxazolinylpys **14**, which was prepared by converting 2-cyanopy to the imidate by addition of methanol followed by addition of the corresponding optically active amino alcohol (Scheme 6) [30,31]. Ligands **15**–**20** are examples, which contain an oxazoline ring at the 2-position of the py. Ligand **15** was used in the Rh-catalyzed enantioselective hydrosilylation of ketones with diphenylsilane [31]. Ligands **16** and **17**, containing another chiral substituent on the other side of the py ring, were used in the Rh-catalyzed enantioselective hydrosilylation [32] and the Pd-catalyzed asymmetric allylic alkylation of 1,3-diphenylprop-2-enyl pivalate with dimethyl malonate, respectively [33]. Ligands **18a,b** and **19** prepared with similar strategies, were active in the Pd-catalyzed asymmetric allylic alkylation [34]. Ligand **20**, with a remote chiral substituent, was obtained from (*S*)-mandelic acid. It was used in the Rh-catalyzed hydrosilylation of 4-methylacetophenone with trichlorosilane [35].

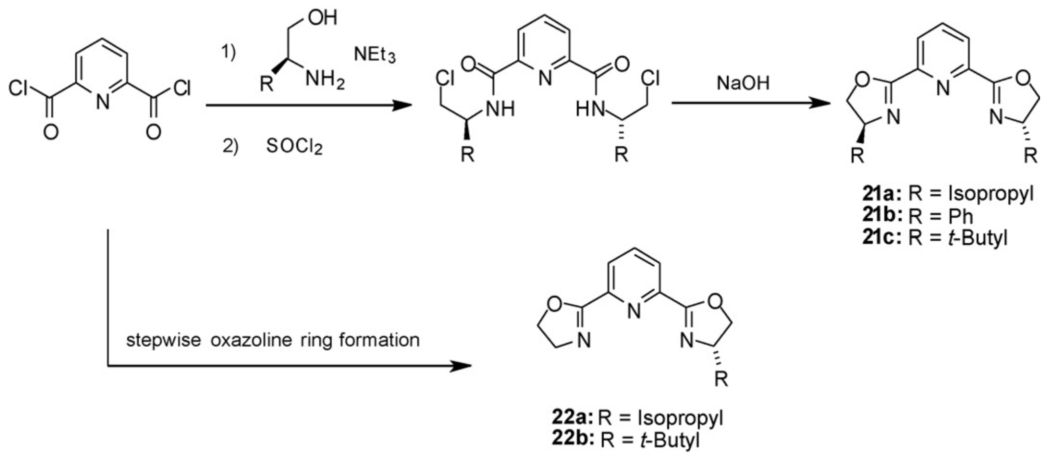
Ligands **21a–c**, reported by Nishiyama et al. using a slightly different cyclization procedure from Brunner's method (Scheme 7), were highly effective ligands in a number of asymmetric reactions, such as Rh(**21a**)-catalyzed hydrosilylation [36,37], Ru(**21a**)-catalyzed cyclopropanation [38], Ru(**21a**)-catalyzed epoxidation [39], Cu(**21b**)-catalyzed Aldol reaction [40,41], and Cu(**21c**)-catalyzed Diels–Alder reaction [42]. C_1-symmetric ligands **22a,b**, synthesized through stepwise cyclization with different amino acids, were used in the Ru-catalyzed cyclopropanation [43]. These ligands have been previously reviewed [44].

Beller and co-workers reported a new type of ligand, pyboxazine **23**, which was synthesized by reacting chiral amino alcohol with dimethyl pyridine-2,6-dicarboximidate (Scheme 8). This ligand was used in the Ru-catalyzed epoxidation 2-methyl-1-phenyl-1-propene of with H_2O_2 [45].

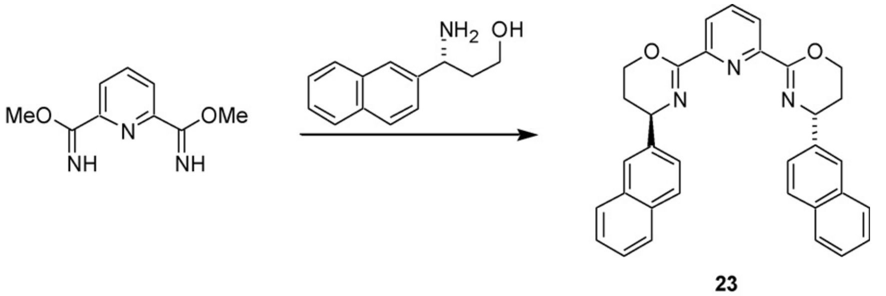
Similar ligands with heterocycles other than oxazolines were reported by Beller's group [46,47]. Pyridyl bisimidazole ligand **24**, was easily prepared in good yields by reacting bisimidate with chiral diamine followed by treatment of carbonyl chloride or chloroformate (Scheme 9). Ligand **25** and **26** were used in the Ru-catalyzed asymmetric epoxidation of alkenes with H_2O_2 [46,47]. Apart from bisimidazole, monoimidazoline **27** was used in the Ru-catalyzed Diels–Alder reaction of bromoacrolein with cyclopentadiene [48,49] and py



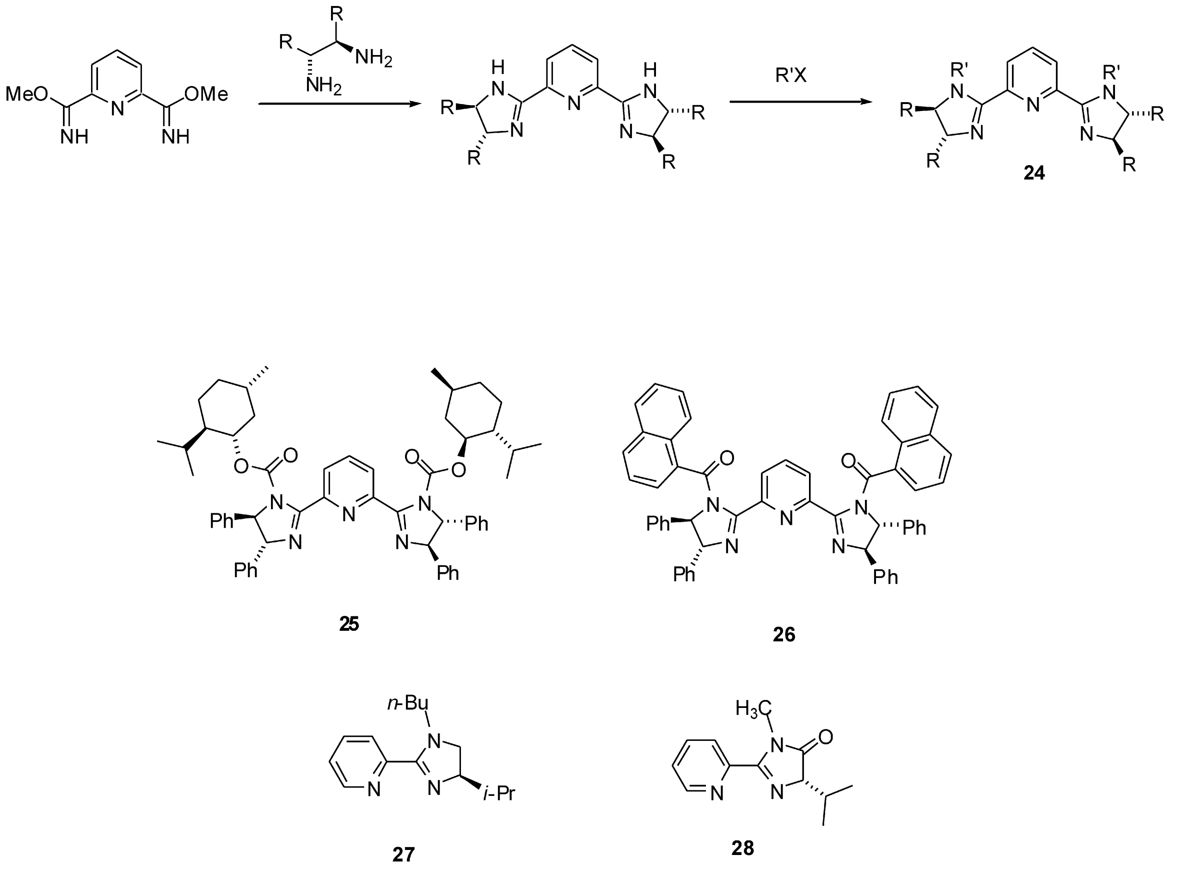
Scheme 6.



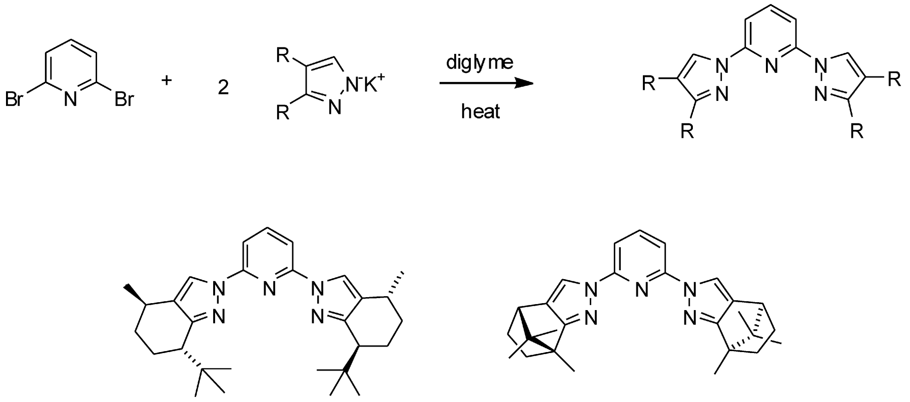
Scheme 7.



Scheme 8.



Scheme 9.



29 **30**

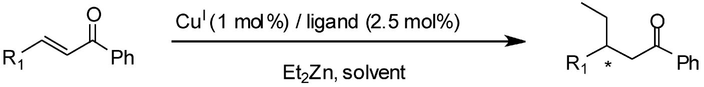
Scheme 10.

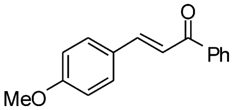
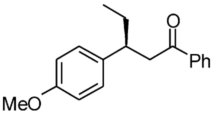
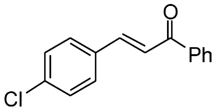
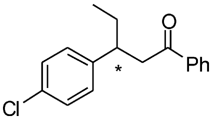
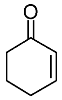
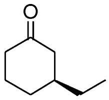
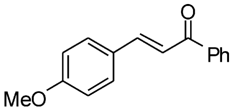
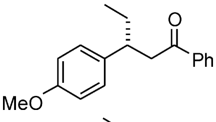
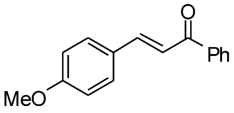
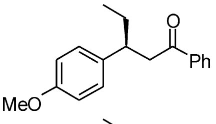
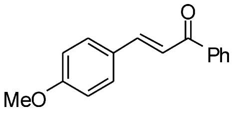
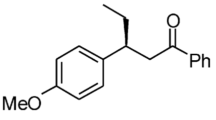
imidazol ligand **28** was used in the asymmetric Henry reaction [50].

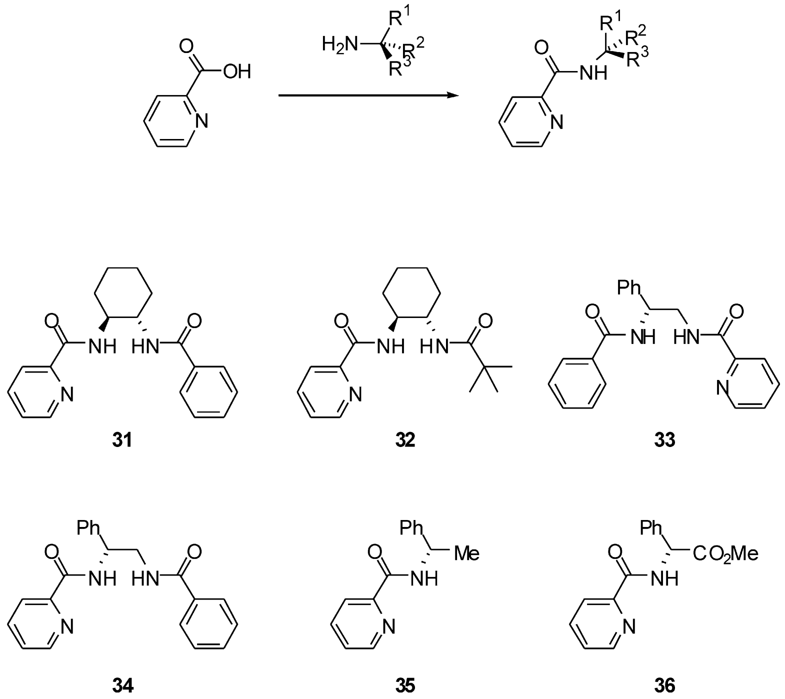
One of the major approaches for preparation of monopy ligands is to utilize a chiral nucleophile and an achiral py precursor. Bispyrazoylpy ligand **29**, prepared by Tolman and co-workers via addition of chiral potassium pyrazolates to 2,6-dibromopy (Scheme 10), was applied in both Cu- and Rh-catalyzed enantioselective cyclopropanation of styrene with ethyl diazoacetate [51]. Che and co-workers used ligand **30** for ruthenium asymmetric epoxidation [52].

Chiral pyridyl amide is another type of chiral monopy, which can be easily obtained by a condensation reaction of commercially available chiral amine with pyridyl carboxylic acid (Scheme 11). They are easily prepared in large quantities and some examples of C_1-symmetric pyridylamide ligand are **31–33** [53], **34** [54,55], **35** [56], and **36** [53,54]. They showed excellent potential in the Mo-catalyzed asymmetric allylic alkylation of unsymmetrical substrate as demonstrated independently by Trost et al. [53] and Kočovský [53,54].

Table 1
Cu-catalyzed enantioselective 1,4-conjugate addition of Et_2Zn to α,β-unsaturated ketones

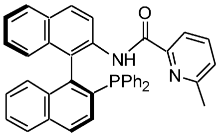


Entry	Ligand	Substrate	Product	%Yield	%Ee
1	**37**			97	98
2	**37**			72	95
3	**37**			98	92
4	**38**			76	95
5	**39**			76	98
6	**40**			80	97



Scheme 11.

A number of chiral pyridyl amides with a phosphorus donor group were developed by Zhang and co-workers (**37**) [57] and Hu (**38** [58], **39** [59] and **40** [60]). The chiral moieties were introduced to the py by reacting picolinic acid with chiral amino biphenyls or binaphthyls. All of these ligands were utilized in the Cu-catalyzed 1,4-conjugate addition to enones with excellent results (Table 1).



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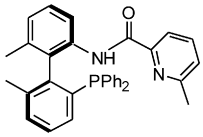

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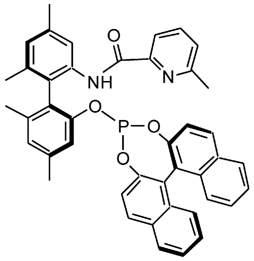

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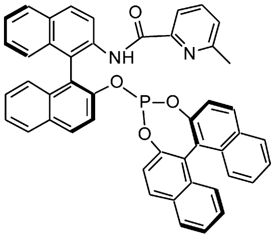
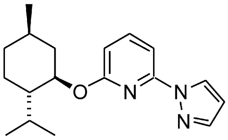


40

Ligands **41–44** represent some other examples where the chirality of the ligands was introduced from a chiral nucleophile. Burke investigated the methyltrioxorhenium(VII) and the Mo-catalyzed asymmetric epoxidation with chiral bidentate ligand **41** [61,62] and **42** [61]. Pyridyl bis(amino alcohol) **43** achieved high enantioselectivities in the Cu-catalyzed asymmetric cyclopropanation of styrene with ethyl diazoacetate [63]. Moberg introduced ligand **44** for enantioselective Pd-catalyzed allylic alkylation [64].



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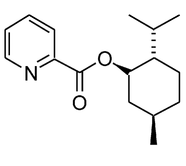

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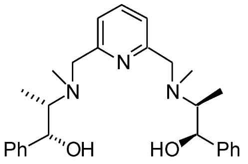

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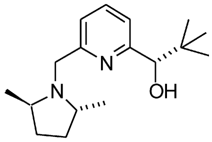

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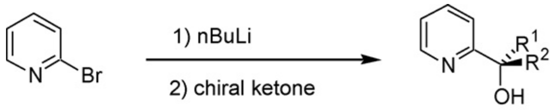
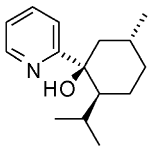


Scheme 12.

Another type of chiral induction method is nucleophilic addition of a lithiated pyridyl anion to chiral ketones (Scheme 12). Monopyridyl alcohols **45–47**, first prepared by Chelucci and Soccolini via condensation of 2-pyridyllithium with optically active naturally occurring ketones, were utilized in diethylzinc addition to aldehydes [65]. Apart from C_1-symmetric ligands, C_2-symmetric ligand **48**, which was reported by Osborn was



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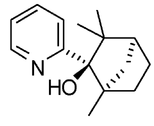

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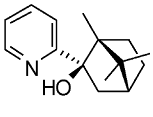

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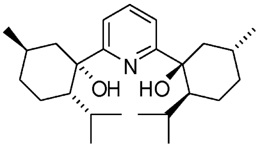

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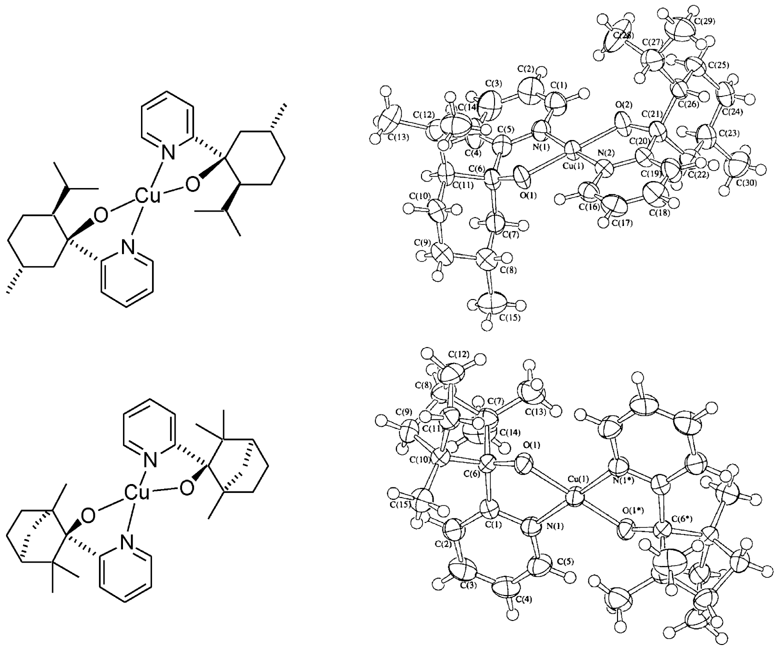
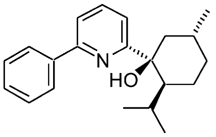


Fig. 1. Synthesis and X-ray crystallography of [Cu(**45-H**)$_2$] and [Cu(**46-H**)$_2$] complexes.

utilized in the Mo- and V-catalyzed asymmetric epoxidation of olefins with *t*BuOOH as oxidant [66]. The coordination chemistry of ligands **45** and **46** with Cu ions were studied and the two complexes, [Cu(**45-H**)$_2$] and [Cu(**46-H**)$_2$] were characterized by X-ray crystallography (Fig. 1). The ligands were effective in the Cu-catalyzed asymmetric cyclopropanation and allylic oxidation [67].

In addition, the phenyl-substituted pyridyl alcohols **49–51** prepared by Pd(0)-catalyzed cross couplings of different bromopy alcohols with phenylboronic acid, were efficient catalysts towards asymmetric Cu-cycloproanation [67] diethylzinc addition reaction [68].



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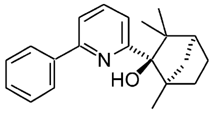

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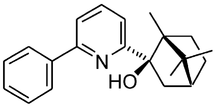
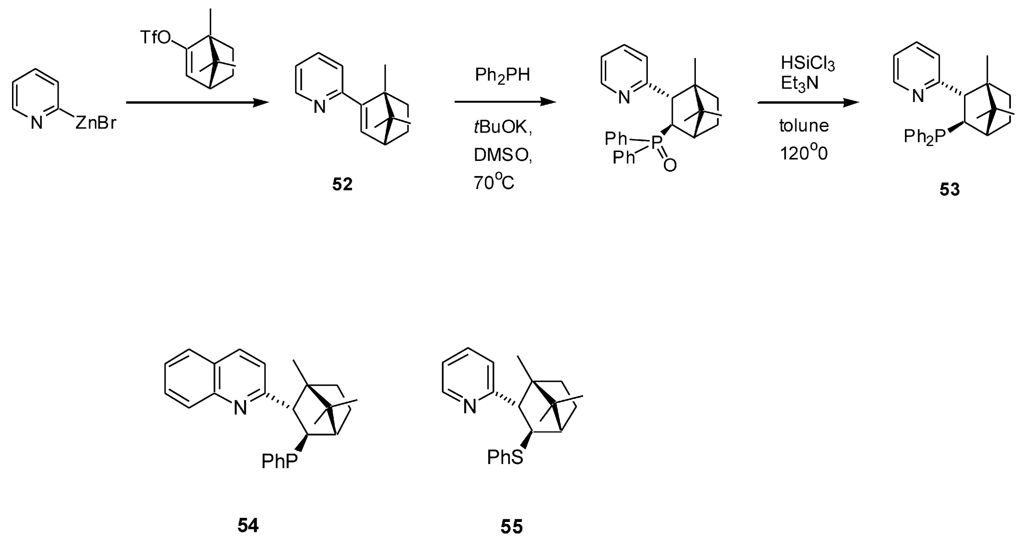


51

Other than pyridyl alcohol ligands, reacting unsaturated pyridines **52**, obtained via Negishi crossing-coupling of alkene triflate and pyridyl intermediate, with diphenylphosphane and then HSiCl$_3$/Et$_3$N (Scheme 13) gave P,N-ligand **53** [69]. Ligands **53** and **54**, obtained similarly, were applied in the Ir-catalyzed enantioselecive hydrogenation of alkenes (Table 2). Through the same intermediate **52**, obtained from **47**, Chelucci et al. reported thioether ligand **55** for Pd-catalyzed allylic alkylation [70].

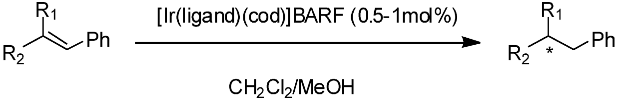
Apart from chiral ketone, other chiral substrates can also react with lithiated pyridyl anions. For example, Moberg and co-workers utilized neomenthyl-1-nitrile for preparation of optically pure pyridyl alcohols **56a** and **56b**, which were easily separated by column chromatography (Scheme 14) [71]. With mandelic acid methyl ester derivative, a single isomer of chiral pyridyl alcohol **57** was easily obtained from the stereospecific reduction of the pyridyl ketone (Scheme 15). With these chiral backbones, they developed a number of ligands with different functionality. The arylated-substituted ligands **58** and **59** were assessed in diethylzinc reactions and pyridyl phosphinites **60** and pyridyl phosphites **61** were used in the Pd-catalyzed allylic substitution reaction [72].

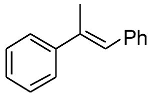
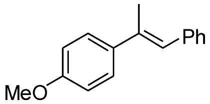
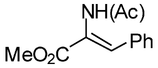
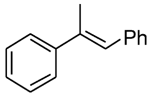
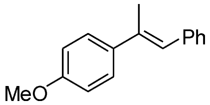
Chiral py ligands with different donor atoms could be easily prepared by addition reaction between lithiated methylpyridyl compounds and chiral ketones/thioketones (Scheme 16). Ligands **62** and **63**, first reported by Kellogg and co-workers [73], were used later independently by Pale and co-workers [74] and Pan and co-workers [75] in asymmetric diethylzinc addition to aldehydes. Other types of pyridyl-alcohol ligands **64** and **65**, reported by Zheng using D-fructose as chiral inducers, were applied in the diethylzinc reaction [76]. Sulfide-containing ligands **66** and **67**, also reported by Kellogg and co-workers [77], were successfully applied to the Pd-catalyzed asymmetric allylic substitution of different acetates, especially 1,3-diethylprop-2-enyl acetate with dimethyl malonate [78]. Similarly, the pyridyl sulfoxide **68** and sulfide **69**, synthesized by reacting lithi-

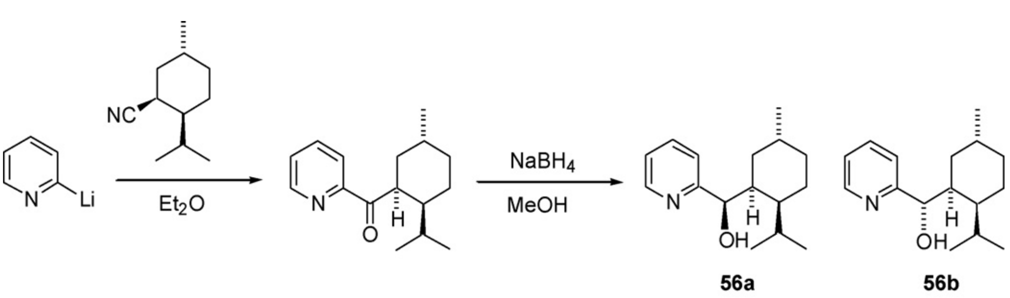


Scheme 13.

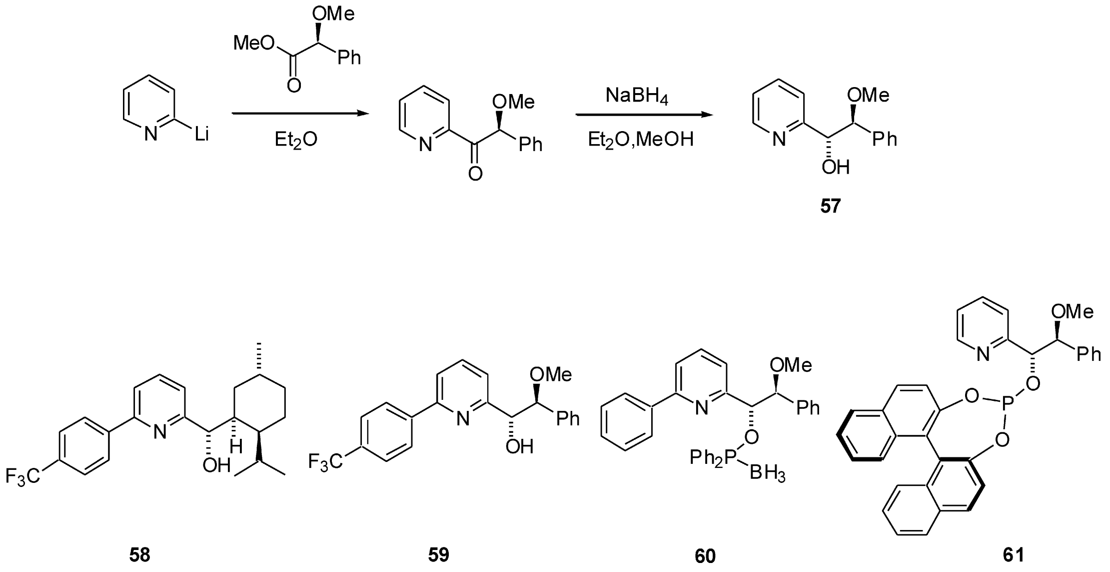
Table 2
Asymmetric hydrogenation of alkenes



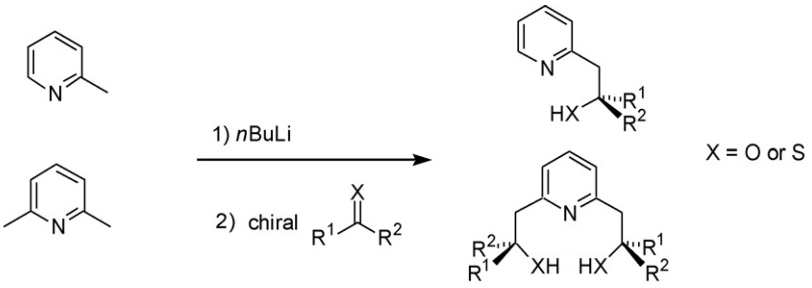
Entry	Ligand	Alkene	*P* (bar)	Time (h)	Conversion (%)	%Ee
1	**53**		50	12	100	95
2	**53**		50	2	87	91
3	**53**		1	12	100	97
4	**54**		1	5	100	95
5	**54**		50	2	100	95



Scheme 14.



Scheme 15.



Scheme 16.

ated alkyl pyridine with (−)-(*R*)-menthyl (*S*)-*p*-tolysulfinate (Scheme 17), were used in the Pd-catalyzed allylic substitution and diethylzinc addition [79].

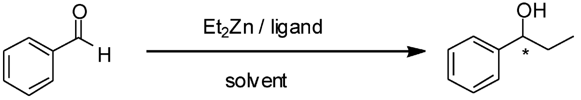
Another way of constructing monopy is through nucleophilic addition using the O- or N-functionalities of the py derivatives. Ligand **70** [80], **71** [80,81] and **72** [81], prepared by reacting 2-hydroxypyridine, 2-hydroxymethylpyridine or 2-aminopyridine with chiral (*S*)-2,2′-binaphthol phosphorochlorite, respectively (Scheme 18), were utilized in allylic alkylation. Ligand **73**, synthesized by an exchange reaction between tris(dimethylamino)phosphane and (*S*)-2-anilinomethylpyrrolidine and hydroxyquinoline [82], was used in the Cu-catalyzed conjugate addition of diethylzinc to enones [83] and Pd-catalyzed allylic alkylation of phenyl 2-oxocyclopentanecarboxylate with allyl acetate [84]. The chirality of the pyridine moiety can also be introduced with planar chiral ferrocene-based intermediates. Ligand **74**, prepared by reaction of ferrocene-acetate with 2-(methylamino)pyridine in the presence of DMAP, was applied in the Pd-catalyzed allylic alkylation [85].

2.2. *From asymmetric reduction and dihydroxylation*

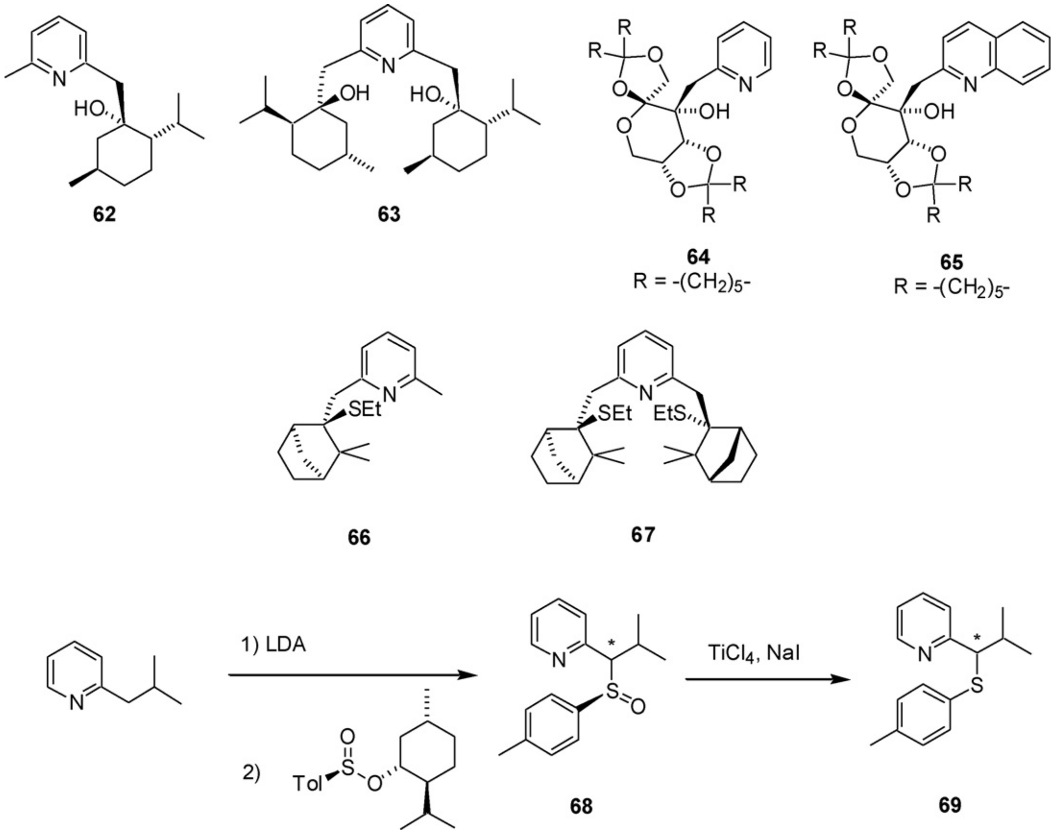
Bolm et al. reported the use of asymmetric reduction for making chiral py ligands (Scheme 19). Prochiral ketones were obtained by lithiation of bromopy with *n*-butylithium followed by treatment with pivalonitrile or *N*,*N*-dimethylacetamide. Asymmetric reduction of the ketones with (+) or (−)-isopinocamphenylborane gave their corresponding chiral pyridylalcohols **75a,b** and **76a,b** [86]. With this method, a number of substituted pyridylalcohols **77–79** were developed and applied in asymmetric diethylzinc addition to aldehydes (Table 3) [87] and Ni-catalyzed conjugate addition reactions of diethylzinc to chalcone [88,89].

Substituted phosphine and phosphite groups were further added to the chiral pyridine diol ligand, obtained by asymmetric reduction of the corresponding ketone, to give the P,N-ligands **80** [90] and **81** [91] (Scheme 20). Ligand **80** was used in Ru-

Table 3
Diethylzinc addition to benzaldehyde



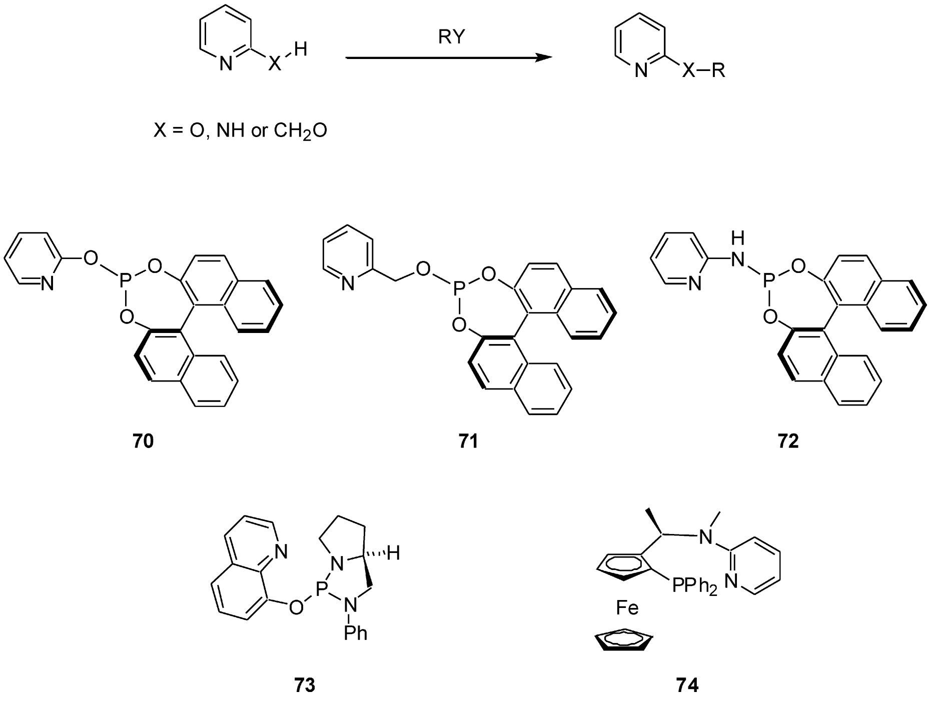
Entry	Ligand	Cat. (mol%)	Time (h)	%Yield	%Ee	Configuration
1	**75a**	11	6	61	78	*R*
2	**77**	8	3	73	93	*R*
3	**78**	5	3	77	95	*S*
4	**79**	5	6	58	82	*S*



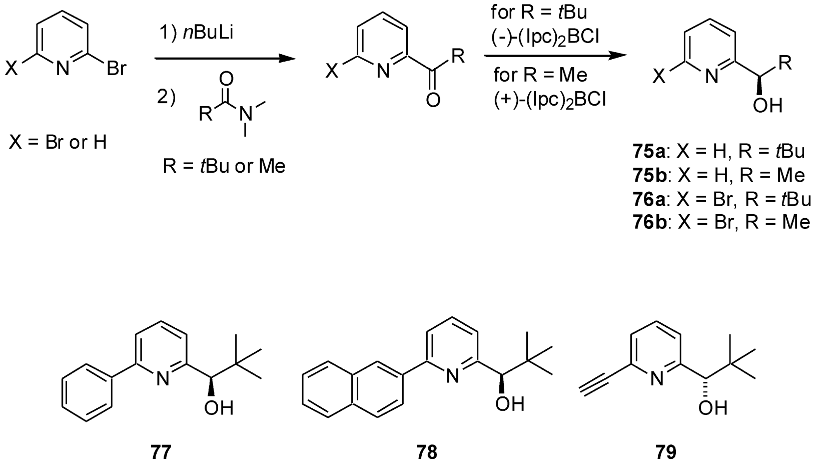
Scheme 17.

catalyzed transfer hydrogenation [90] and ligand **81** was used in Ir- and Rh-catalyzed asymmetric hydrogenation of imines [92].

Another type of ligand with chirality based on asymmetric reduction was developed by Pfaltz. The pyridyl phosphane **83** was synthesized by a series of steps and chirality was introduced with (−)-Ipc$_2$BCl by reduction of ketone **82** to the corresponding alcohol (Scheme 21). Similar ligands with a quinoline moiety can also be prepared by a different strategy. The chirality of

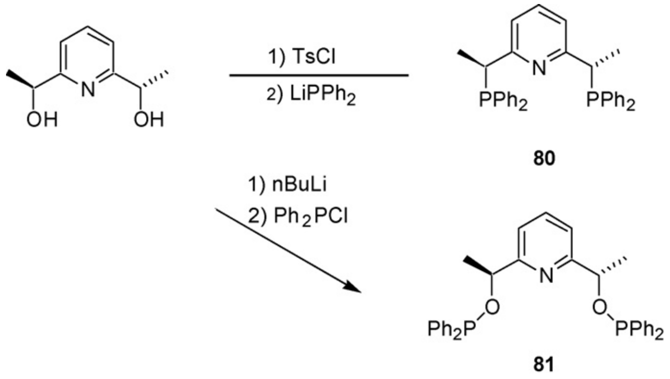


Scheme 18.



77 **78** **79**

Scheme 19.



80 **81**

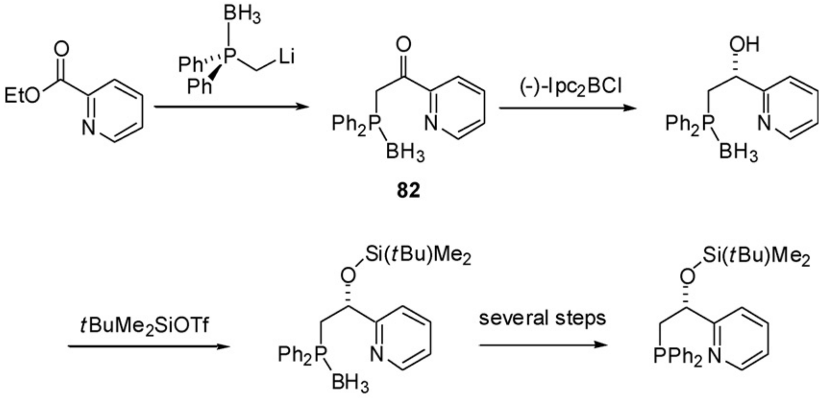
Scheme 20.

phosphanyl ligand **84** could be introduced via Sharpless dihydroxylation of 2-vinylquinoline (Scheme 22). These ligands were used in the Pd-catalyzed Heck reaction and their Iridium complexes were prepared and utilized in catalytic hydrogenation of olefins [93].

2.3. *From Kröhnke condensation*

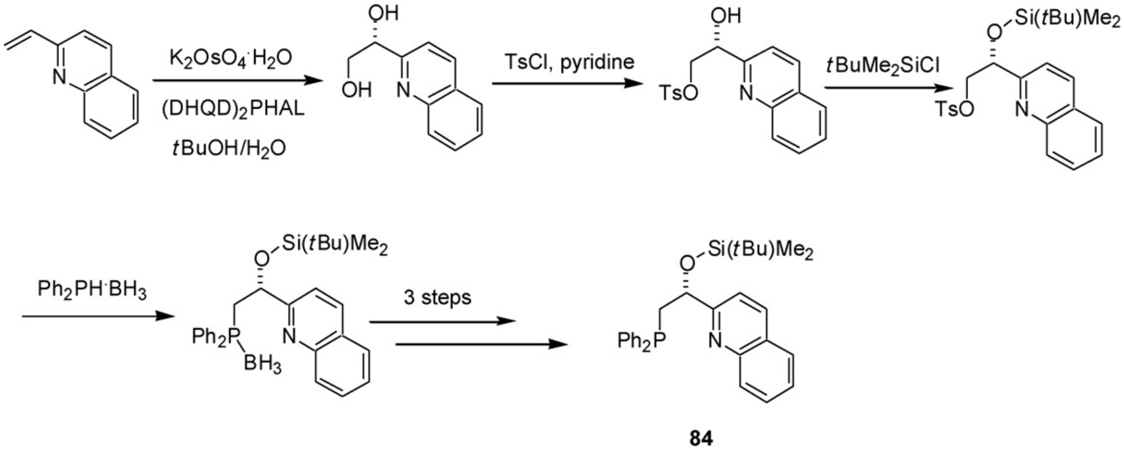
Kröhnke condensation is an important method for the construction of a py ring, Collomb and von Zelewsky were the first group to apply this strategy in the preparation of monopy **85**, which has a rigid substituent fused to the py ring (Scheme 23) [94]. Ligands having oxygen, sulfur or phosphorus donor atoms can be easily prepared by lithiation of **85** followed by trapping with various ketones. Ligands **86** [94] and **87a,b** [95] were used in the asymmetric diethylzinc addition reactions. Ligand **88**, developed by Chelucci and Cabras was used in the Pd-catalyzed allylic substitution reactions [96]. Phosphine ligand **89**, reported independently by Kočovský and co-workers [97] and Chelucci et al. [98], was applied in asymmetric Pd-catalyzed Heck reaction [97]. Other examples of this type of ligands are **90** and **91**; both of them were used in the Pd-catalyzed allylic alkylation and Cu-catalyzed cyclopropanation [99–101].

Using von Zelewsky's synthetic strategy, several series of chiral pyridyl-thiazole ligands, **92a,b**, **93a–e** and **94a–d**, were prepared. Ligand **92a** formed a stable complex with $Cu(ClO_4)_2{\cdot}6H_2O$ and the structure was characterized by X-ray



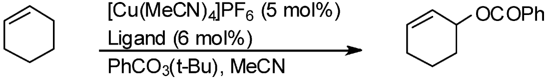
82 **83**

Scheme 21.



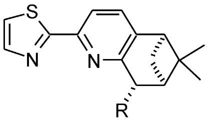
Scheme 22.

Table 4
Cu catalyzed asymmetric allylic oxidation of cyclohexene promoted by pyridyl-thiazole ligands

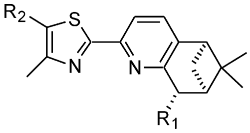


Entry	Ligand	Time (h)	%Yield	%Ee
1	**93a**	48	75	62 (*R*)
2	**93b**	60	95	61 (*R*)
3	**93c**	48	62	62 (*R*)
4	**93d**	48	63	55 (*R*)
5	**93e**	48	95	58 (*R*)

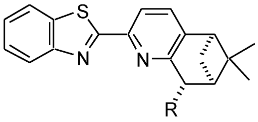
crystallography (Fig. 2). Ligands **93a–e** and **94a–d** were used in Cu(I)-catalyzed allylic oxidations of cyclohexene (Table 4) [102].



92a R = H
92b R = Me



93a: R_1 = iPr, R_2 = H
93b: R_1,R_2 = nPr
93c: R_1,R_2 = nBu
93d: R_1,R_2 = Benzyl
93e: R_1,R_2 = CH_2SiMe_3



94a: R = H
94b: R = nPr
94c: R = Benzyl
94d: R = CH_2SiMe_3

More recently, adopting a similar synthetic procedure, a number of pyridyl-thiazoles **95a,b**, pyridyl-imidazole **96–97** and pyridyl-pyrazine **98–99** were reported. The coordination with metal ion was demonstrated by reacting $CuCl_2$ with **97** (Fig. 3). The copper complex [Cu_2(**97**)$_2$(μ-Cl)$_2$$Cl_2$], was successfully characterized by X-ray crystallography as a dimer which is bridged by two chloride ligands. These ligands were applied in the asymmetric Pd-catalyzed allylic substitution [103].

Other cyclizations, such as cocyclotrimerization of nitrile and acetylene (Scheme 24), have also been used. In the presence of [CpCo(COD)] as catalyst, pyridine **100** can be obtained in good yield. Based on this compound, phosphino derivatives **101** and **102** were developed and utilized in metal catalyzed asymmetric addition of diethylzinc, hydroformylation, hydrocarbo ethoxylation and allylic alkylation [104].

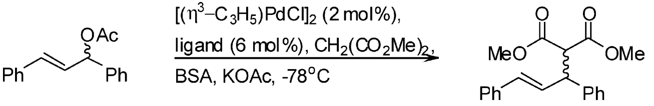
Apart from pyridines, chiral quinolines could be obtained by Friedlander-modified condensation between the camphor derivative and 2-nitrobenzaldehyde (Scheme 25). The corresponding phosphanyl ligands **103** and **104** were used in Pd-allylic substitution [105].

2.4. *From resolution of racemates*

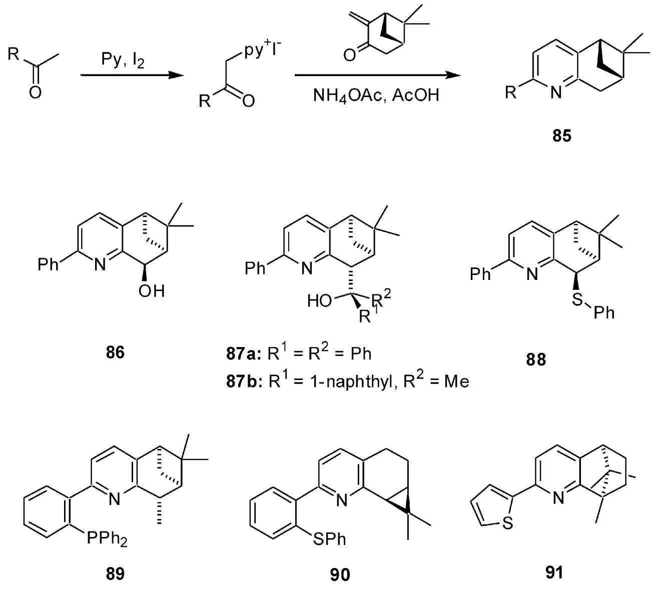
Resolution of a racemate is another method for obtaining enantiomerically pure chiral py ligands. This could be achieved by chromatographic separation or corecrystallization of racemates with chiral auxiliaries.

Chiral pyridyl oxazoline ligands can be prepared starting from the racemic alcohol **105** [106] (Scheme 26). Upon introduction of chiral oxazolinyl group into the ligands in three steps

Table 5
Enantioselective alkylation of (rac)-1,3-diphenylprop-2-enyl acetate with dimethyl malonate

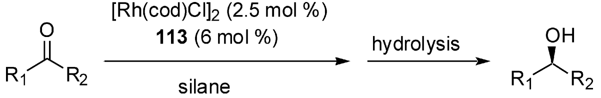


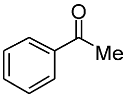
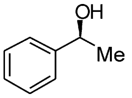
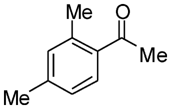
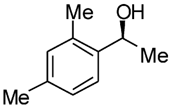
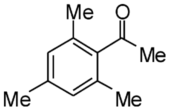
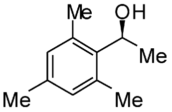
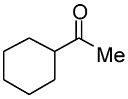
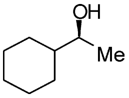
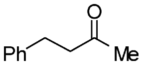
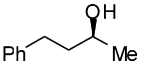
Entry	Ligand	%Yield	%Ee
1	**108a**	96	91 (*S*)
2	**108b**	91	99 (*S*)
3	**109c**	81	95 (*R*)
4	**109a**	96	90 (*R*)
5	**109b**	97	>99 (*R*)



Scheme 23.

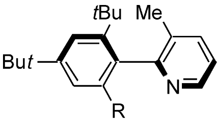
Table 6
Rh catalyzed asymmetric hydrosilylation of ketones



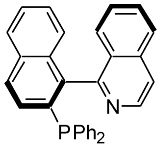
Entry	Ketone	Product	%Yield	%Ee
1			94	98
2			97	95
3			99	98
4			91	94
5			98	82

via *N*-oxide **106** and nitriles **107**, the diastereomeric pyridyl oxazoline ligands were purified as optically pure ligands by column chromatography. Following similar strategy, interesting ligands **108a,b** and **109a–c** were synthesized and utilized in diethylzinc reactions and Pd-catalyzed allylic substitutions (Table 5) [33,34].

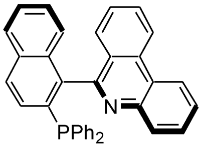
Planar chiral pyridyl phenol ligand **110a**, reported by Chan and co-workers, was prepared by resolution through chromatographic separation of the sulfonyl derivatives followed by alkaline hydrolysis. It is an efficient catalyst in diethylzinc addition reactions [107]. Later, the same group reported that optically active pyphos ligand **110b** could be obtained by resolution through fractional recrystallization of the diastereomeric Pd complexes. This ligand was used in the Rh-catalyzed hydroboration of vinylarenes [108]. Brown et al. found the P,N-ligands **111** and **112**, resolved via the chiral Pd complexes, to be active in the Rh-catalyzed hydroboration of arylalkenes with catecholborane [109,110]. The enantiomers of the planar chiral py **113**, resolved by chiral HPLC, were reported by Fu and co-workers. It has been applied in the Rh-catalyzed asymmetric hydrosilylation of ketones (Table 6) [111].



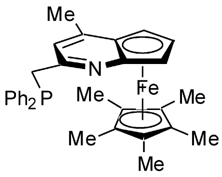
110a: R = OH
110b: R = PPh3



111



112



113

3. Chiral dipyridines

3.1. Dipyridine with achiral bridge

Though many different types of monopy ligands have been studied, relatively few dipy ligands have been reported. The

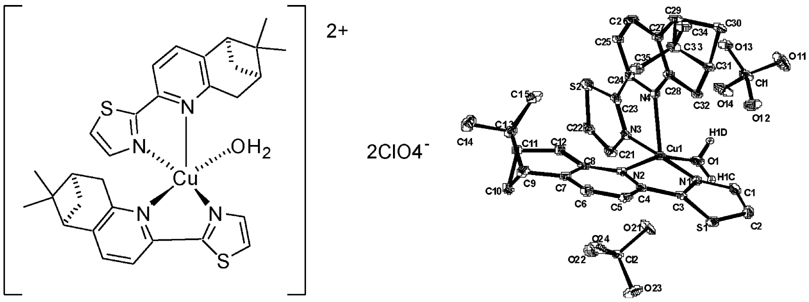
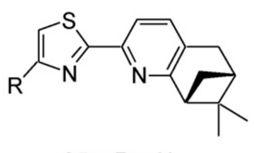
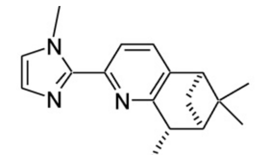


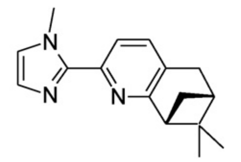
Fig. 2. X-ray crystal structure of [Cu(**92a**)$_2$(H$_2$O)](ClO$_4$)$_2$.



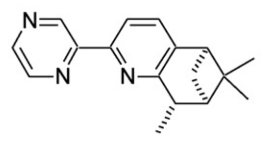
95a: R = H
95b: R = Me



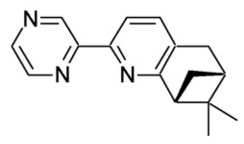
96



97



98



99

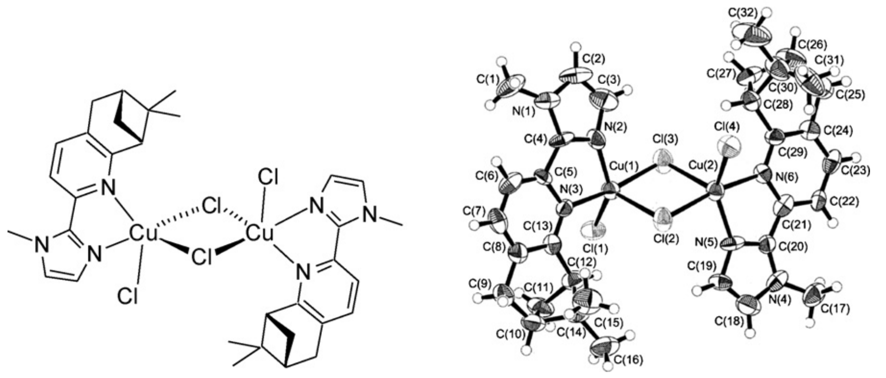
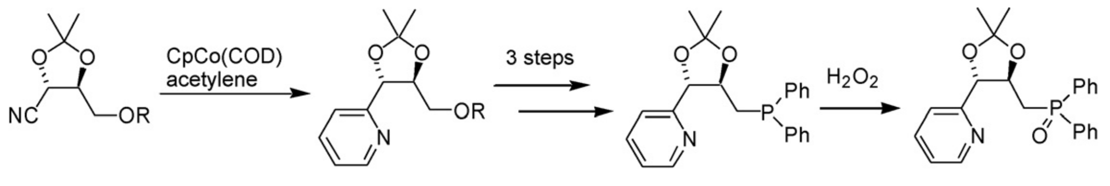
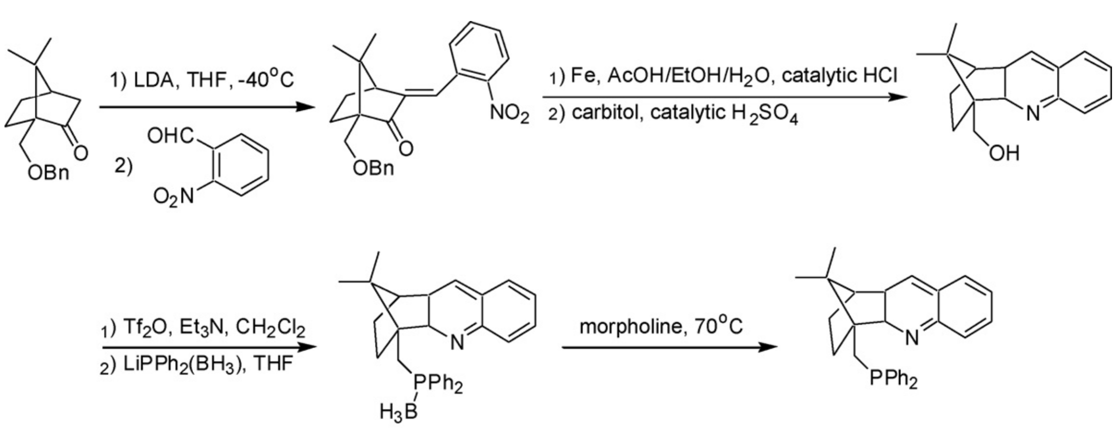


Fig. 3. X-ray crystal structure of $[Cu_2(\mathbf{97})_2(\mu\text{-}Cl)_2Cl_2]$.



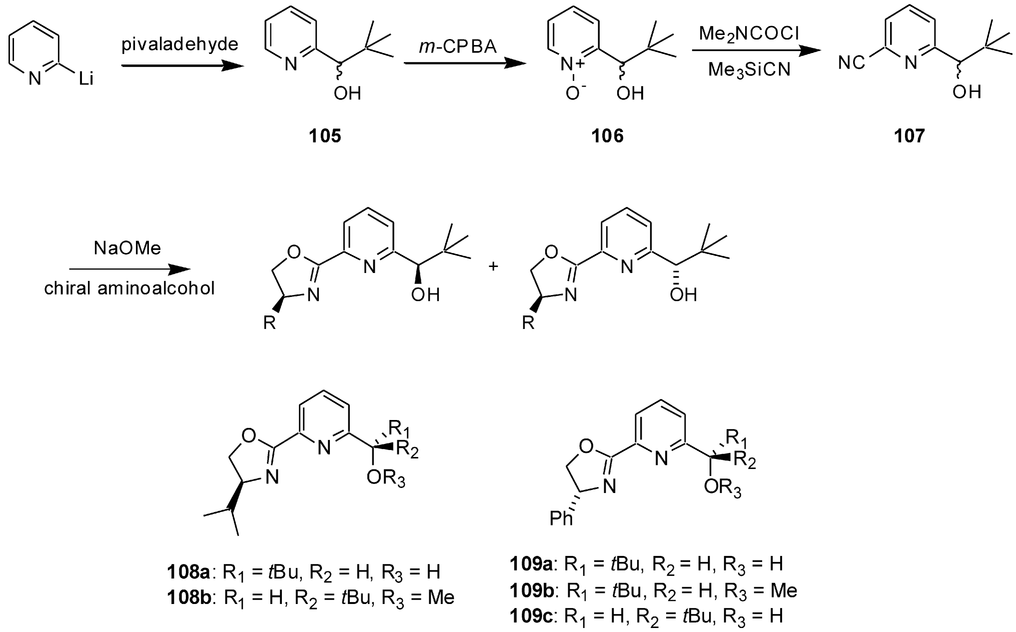
100 **101** **102**

Scheme 24.



103 **104**

Scheme 25.



Scheme 26.

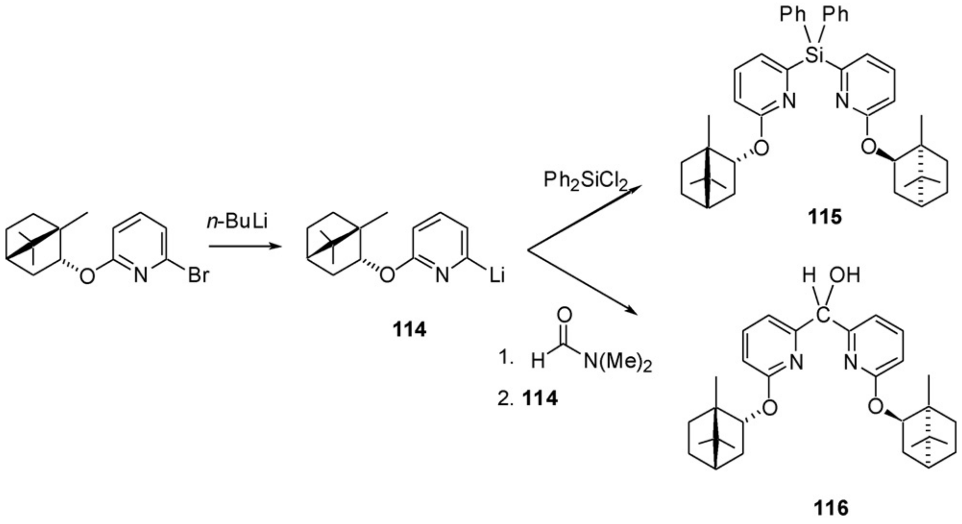
preparation of dipy ligands with different backbones permits facile access to a large variety of ligands. Moreover, ligands with equal or different rings can be conveniently prepared, commonly through nucleophilic attack of lithiated species of chiral py onto the bridging atoms.

Using this method, Wright et al. introduced ligands **115** and **116**, which are dipy ligands with Si and C bridging atoms, respectively (Scheme 27). Reactions with Ph_2SiCl_2 gave chiral bis(pyridyl)silane **115** in excellent yield (85–95%). The ligands were used in the Cu-cyclopropanation and Rh-hydrosilylation [112].

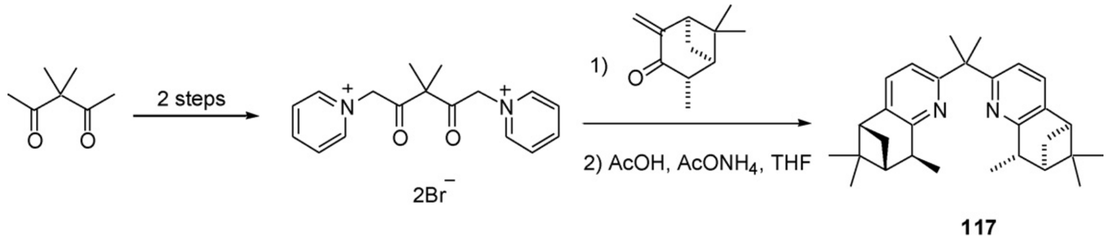
Using Kröhnke methodology, starting from 3,3-dimethylpenta-2,4-dione in which the dimethylcarbon acted as a bridging unit, Chelucci et al. prepared chiral dipyridylpropane **117** (Scheme 28) [113]. The ligand has been utilized in the Cu-catalyzed asymmetric cyclopropanation [113] and Pd-catalyzed allylic alkylation [114].

Apart from the tetrahedral shape of the silicon and carbon bridging atoms, nitrogen atom is another choice for connection between the two py rings. Ligands **118** and **119**, recently reported by Bolm et al., were easily prepared through the Pd-catalyzed coupling of chiral py to the 2-aminopy (Scheme 29). Ligands **118**–**121** were active in the Cu-catalyzed allylic oxidation of cyclohexene [115,116].

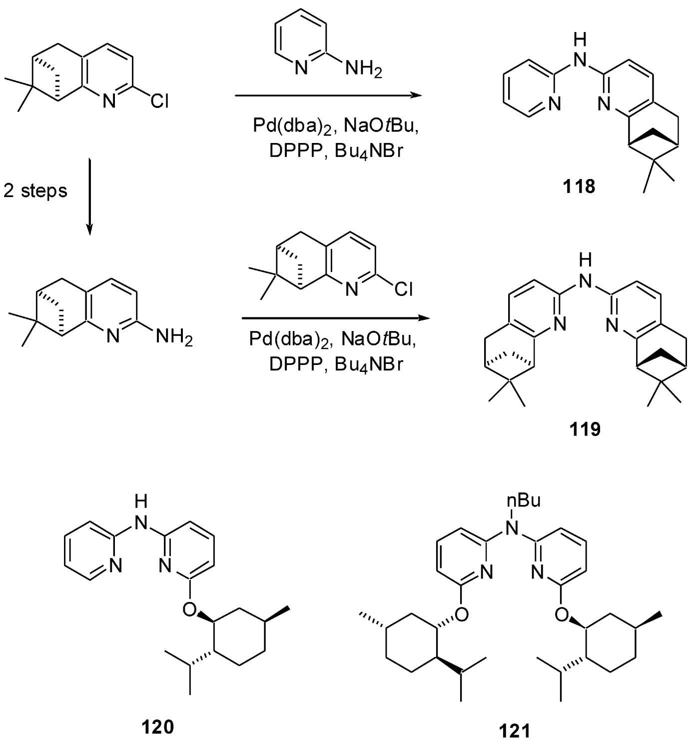
Chiral pyridyl ketone **122**, prepared via reactions between chiral 2-bromopy and chloroformate (Scheme 30), formed a 1:1 Cu(II)–ligand complex which was successfully characterized by X-ray crystallography (Fig. 4). The ligand was applied in the Cu-catalyzed cyclopropanation of different alkenes [117].



Scheme 27.



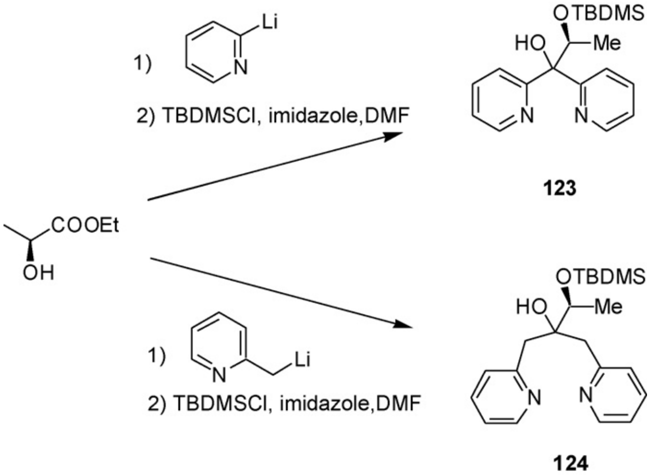
Scheme 28.



Scheme 29.

3.2. Dipyridine with chiral bridge

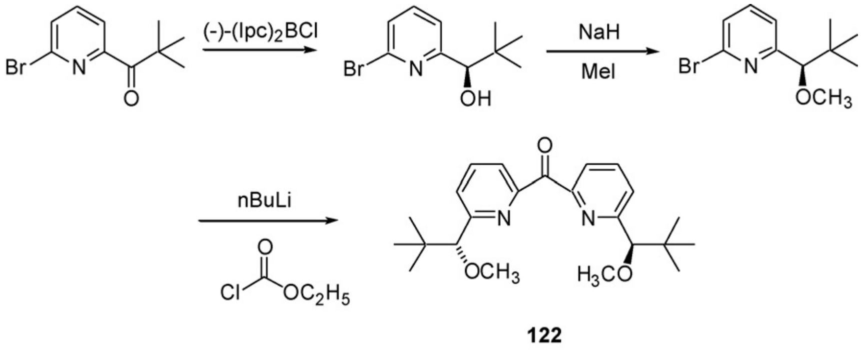
Kotsuki et al. synthesized ligands **123** and **124** by reacting 2-lithopy and 2-picolyllithium with ethyl L-lactate (Scheme 31). They were active catalysts towards enantioselective addition of diethylzinc to benzaldehyde [118].



Scheme 31.

Starting from diethyl tartrates, Chelucci and co-workers prepared ligands **125** and **126** (Scheme 32) [119–121]. Using Co(I) as catalyst, the corresponding bis-cyano substituted dioxolane underwent cyclotrimerization with acetylene. Under high temperature and pressure, formation of the py ring was accomplished with 80–95% yields. Ligand **126** was used in the Pd-catalyzed asymmetric allylic substitution [121]. When applied in asymmetric cyclopropanation [122,123], ligand **126** was more active than **125**.

The chiral Schiff base ligands **127–128** were synthesized by condensation of chiral diamine with 2-pyridine carboxaldehyde (Scheme 33). Ligand **127**, first reported by Vögtle and Goldschmitt [124], was employed by Li et al. in the Ti-catalyzed asymmetric pinacol coupling of aromatic aldehydes [125]. Ligand **128** was applied in the Cu-catalyzed asymmetric Henry



Scheme 30.

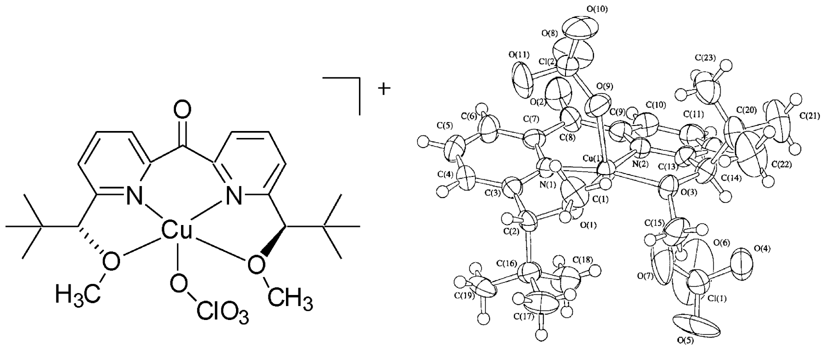
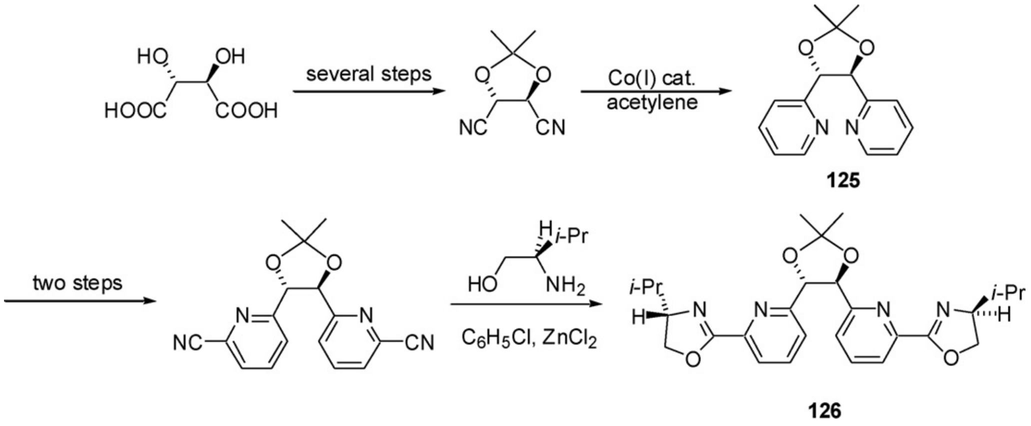


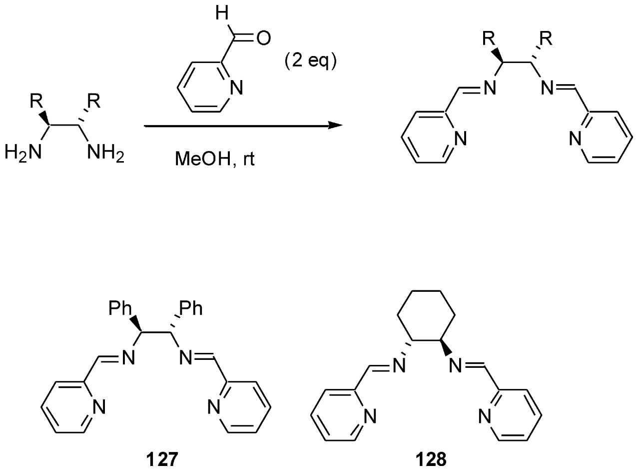
Fig. 4. X-ray crystallography of cation [Cu(**122**)(ClO_4)] complex.



Scheme 32.

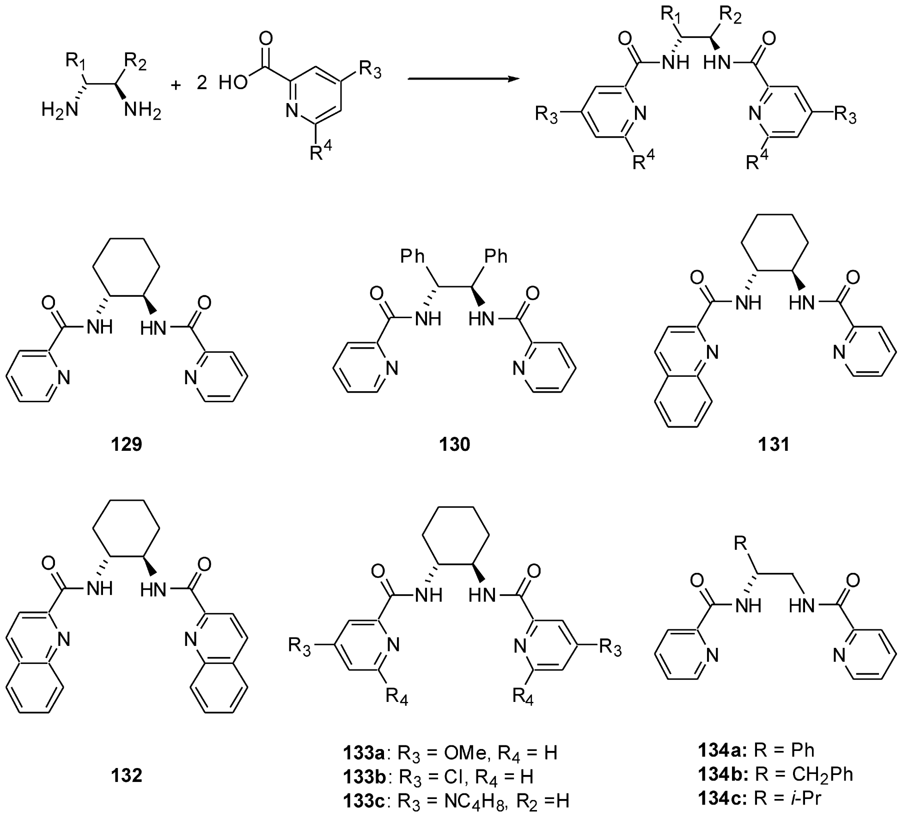
reaction [126] and Mn-catalyzed asymmetric sulfur oxidation [127].

Chiral bis(pyridylamide), synthesized using commercially available diamines, represents an important class of dipy ligands which have a chiral bridge (Scheme 34). Ligands **129** and **130**, first reported in 1978 [128] and 1991 [129], respectively, as racemate by Vagg and co-workers, are two examples. Moberg et al. first reported the use of these two ligands, together with other derivates, in asymmetric catalysis; a metal-catalyzed ring opening of cyclohexene oxide with trimethylsilyl azide as nucleophile [130]. Later, they reported the use of these ligands in the metal-catalyzed TMSCN addition to aldehydes [131]. The most important application of this class of ligands came when Trost and Hachiya reported the use of **129** [132] and its derivatives, like **131** and **132**, in the Mo-catalyzed asymmetric allylic alkylation of unsymmetrical substrates [53]. The same group also reported the use of **129** in the Mo-catalyzed asymmetric allylic alkylation of with polyenyl esters substrates [53,133]. Larhed and co-workers developed a new procedure for the Mo-catalyzed asymmetric allylic alkylation using microwave irradiation in air with $Mo(CO)_6$ as the catalyst [134] and they also studied the different electronic effects on ligands **133a–c** [135]. C_1-symmetric bis(pyridylamide) **134a–c**, first reported by Kočovský and co-workers [54,55], were used in the allylic substitution of unsymmetrical substrates. The remarkable achievements of these ligands on enantioselective Mo-catalyzed allylic substitution of unsymmetrical substrates have been reviewed recently (Table 7) [136].



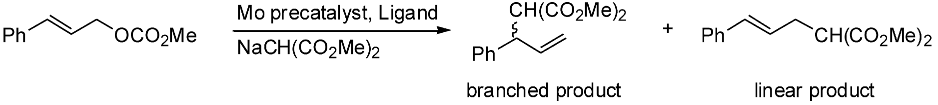
Scheme 33.

Bisacetal pyridinamide ligands **135** and **136**, in which the amide bonds were formed by the condensation of chiral bis(acyl chloride) and 2-pyridylamine (Scheme 35), were utilized in the Re-catalyzed asymmetric epoxidation of styryl substrates [61]. A longer bridge with phosphine is also possible. Dipy phosphine ligand **137**, reported by Minato et al. (Scheme 36), was applied in the Pd-catalyzed asymmetric allylic substitution [137].



Scheme 34.

Table 7
Mo-catalyzed allylic substitution



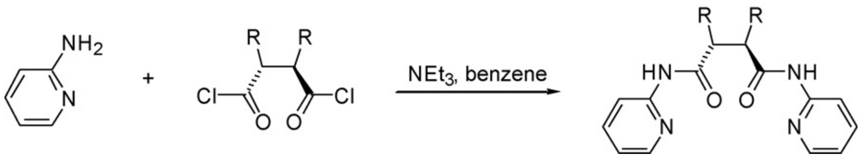
Entry	Ligand	%Yield	Branch:linear	%Ee
1	**129**	88	32:1	99 (*R*)
2	**130**	95	19:1	99 (*S*)
3	**133a**	88	41:1	>99 (*R*)
4	**133b**	89	74:1	96 (*R*)
5	**133c**	91	88:1	96 (*R*)
6	**134b**	59	38:1	97 (*R*)

4. Chiral 2,2′-bipyridines

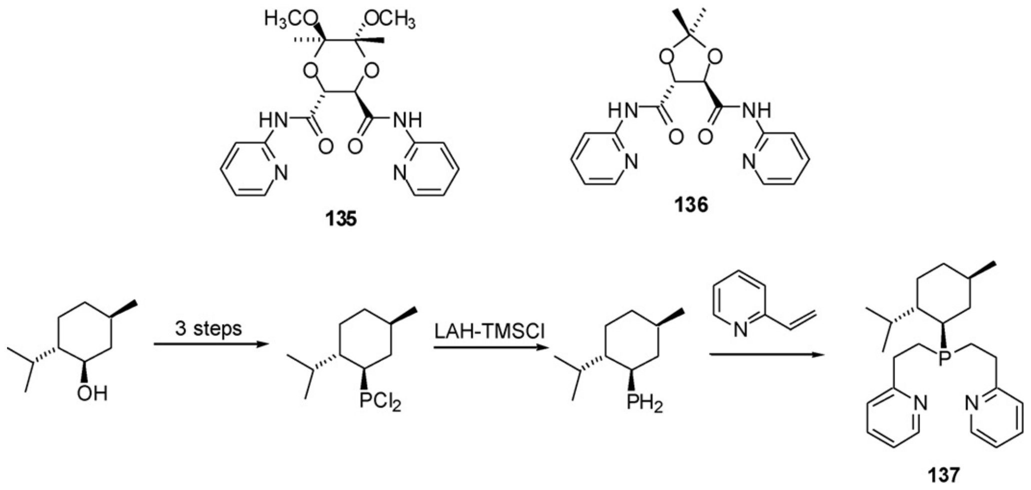
Chiral bipy ligands can be considered as dipyridyl compounds without a linker. They received the greatest attention over the past because of their great potential in asymmetric catalysis.

4.1. From cyclotrimerization

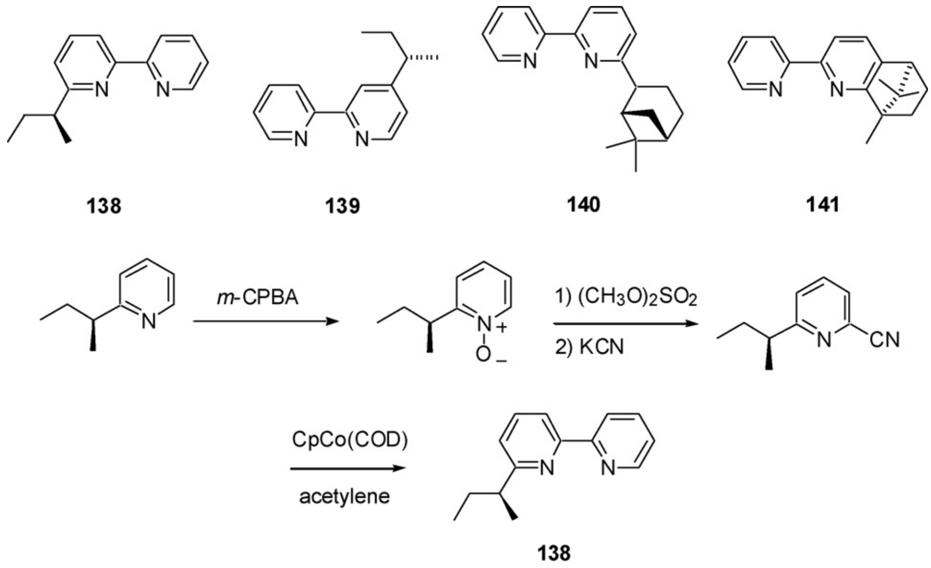
The early development of chiral bipy focused on C_1-symmetric monoalkyl substituted 2,2′-bipy. Ligands **138–141**, first reported by Botteghi et al. in the 1980s, were synthesized through co-cyclotrimerization of a cyano substituted py with



Scheme 35.



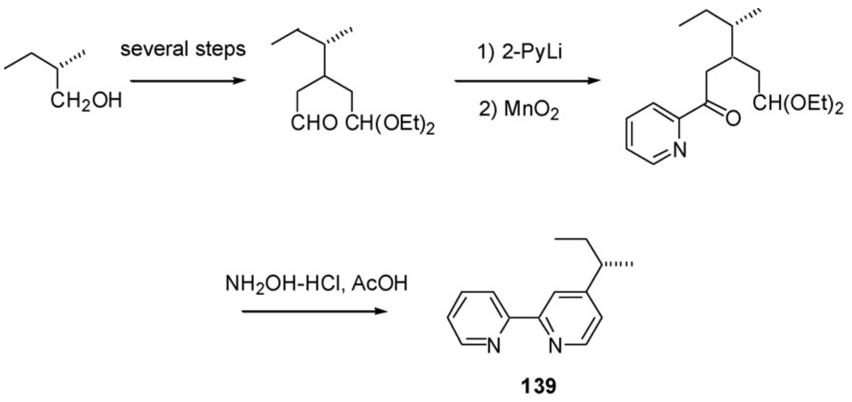
Scheme 36.



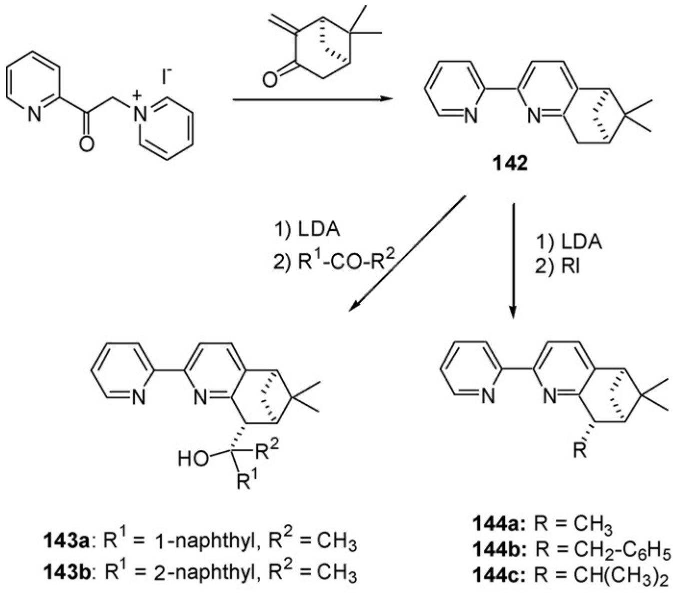
Scheme 37.

acetylene using Co(I) catalyst or through an aza anellation of a pyridyl ketone in the presence of hydroxylamine hydrochloride (Schemes 37 and 38) [138–142]. These ligands were active catalysts towards several asymmetric reactions such as transfer hydrogenation [140], cyclopropanation [141], hydrosilylation [142] and allylic substitution [143].

Chiral C_1-symmetric bipy ligand **142**, reported by Hayoz and von Zelewsky via a Kröhnke-type cyclization (Scheme 39)



Scheme 38.



Scheme 39.

[144], could be deprotonated using LDA and trapped with the appropriate ketones to afford ligands **143a,b**, which were studied in the diethylzinc addition to benzaldehyde [95].

C$_1$-Symmetric bipy ligands **144a–c**, reported by Chelucci et al. via adding different alkyl groups at the 8-position of the bipy ligand **142**, were used in the asymmetric Pd-catalyzed allylic substitution [145].

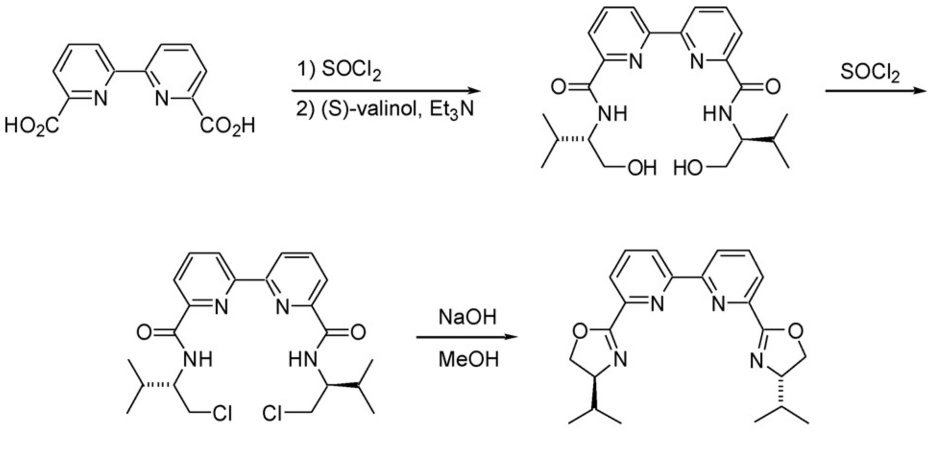
4.2. From achiral bipyridine

C$_2$-Symmetric bipy-bisoxazoline ligand **145**, reported by Nishiyama et al. (Scheme 40) [146], was an effective ligand in the asymmetric Rh-catalyzed asymmetric hydrosilylation of acetophenone with diphenylsilane. Bipy macrocycle **146** was prepared with achiral bipy building block, 6,6′-bis(chlorocarbonyl)-2,2′-bipyridine, through coupling in high dilution with the diamide (Scheme 41). The Fe complex of ligand **146** was applied in the asymmetric epoxidation of alkene [147].

4.3. From homocoupling

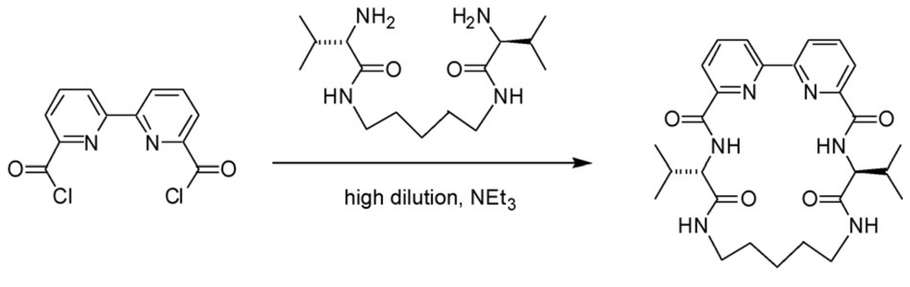
In 1990, Bolm et al. reported the first examples of *C*$_2$-symmetric chiral bipy ligands **147–148** which were obtained by nickel(0)/triphenylphosphine mediated homocoupling of chiral 2-bromopy (Scheme 42) [87,148,149]. These ligands were utilized in enantioselective Ni-catalyzed conjugate addition reactions [150] and diethylzinc addition to aldehydes [89,148].

Ligand **147** was shown later by Schneider et al. to be active in the Sc-catalyzed enantioselective addition of alcohols and amines to meso-epoxides (Table 8). The ligand was active



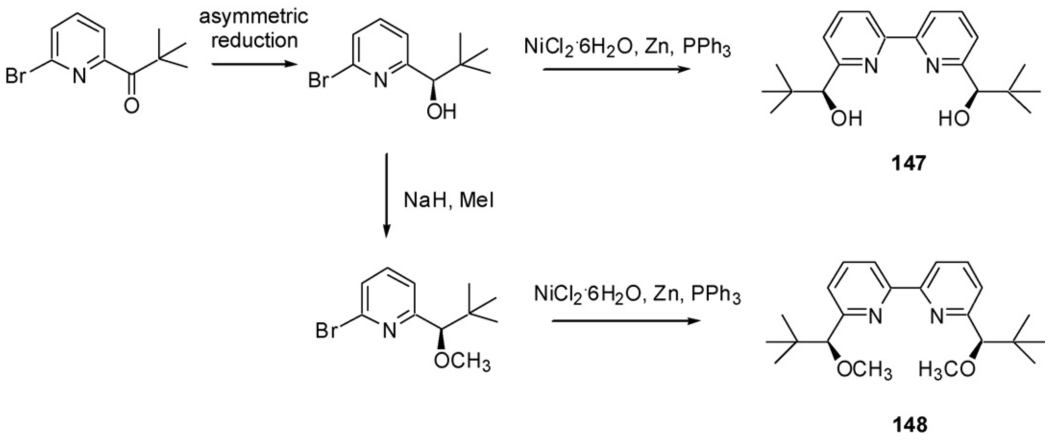
145

Scheme 40.



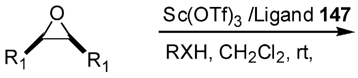
146

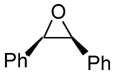
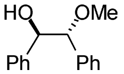
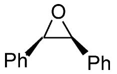
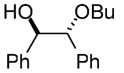
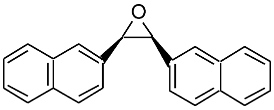
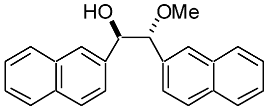
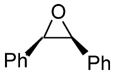
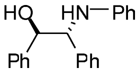
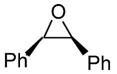
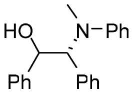
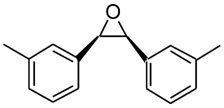
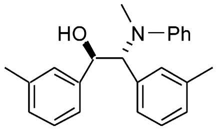
Scheme 41.



Scheme 42.

Table 8
Results of the scandium-catalyzed alcoholysis and aminolysis of meso-epoxides



Entry	Substrate	Nucleophile	Product	%Yield	%Ee
1		MeOH		81	92
2		*n*BuOH		80	94
3		MeOH		83	98
4		$PhNH_2$		95	93
5		$PhNHCH_3$		85	97
6		$PhNH_2$		93	91

with aliphatic alcohols and gave complete anti diastereoselectivity. It also gave the ring-opened products in excellent enantioselectivities upon aminolysis [151]. The bismuth and scandium complexes of ligand **147** were shown by Kobayashi and co-workers to be active catalysts in the asymmetric hydroxymethylation of silicon enolates (Table 9) [152,153].

Ligand **148** formed an orange complex with copper(II) and the X-ray crystal structure is shown in Fig. 5. The complex was

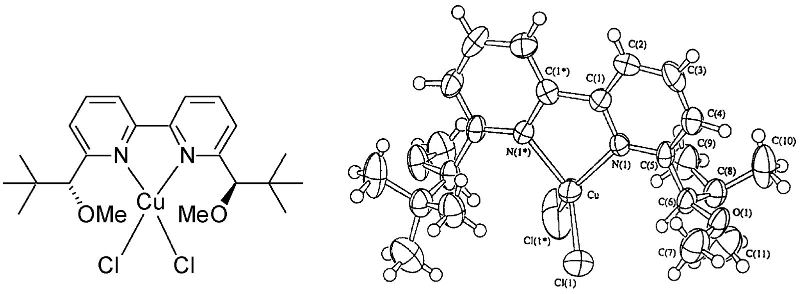
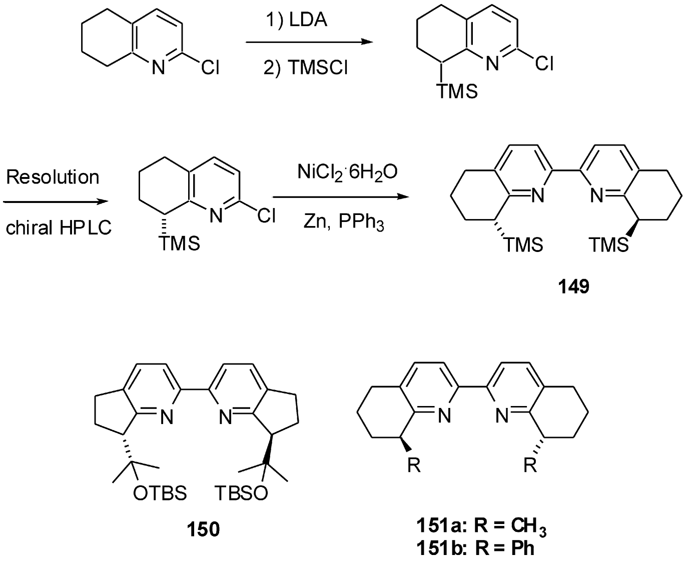


Fig. 5. X-ray crystallography of Cu(**148**)Cl_2 complex.



Scheme 43.

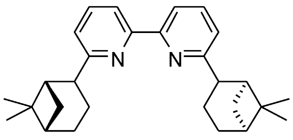
used in the asymmetric cyclopropanation of alkenes with ethyl diazoacetate [154].

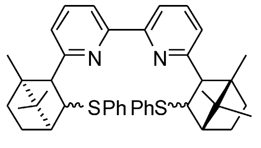
The X-ray crystallography of the Zn(II)–**148** complex (Fig. 6) was characterized and the use of its triflate complex in the asymmetric allylation of aldehydes with allyltributyltin gave very good yields of the homoallylic alcohols for a wide range of substrates, including aromatic, aliphatic and α,β-unsaturated aldehydes [155].

Katsuki and co-workers reported the synthesis of bipy ligands **149–151** using a homocoupling strategy (Scheme 43) [156–158]. Ligand **149** showed very good effectiveness in the Cu-catalyzed cyclopropanation (Table 10). They also investigated the utilities of ligand **150** and **151** on ring expansion of oxetane [159,160].

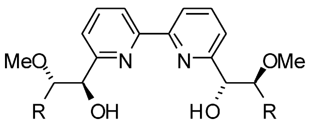
The chiral ligand **152**, reported by Chelucci et al. was used in Cu-catalyzed cyclopropanation [122] and Pd-catalyzed allylic substitution reactions [143]. Chiral C_2-symmetric sulfur-containing bipy ligand **153**, also prepared by Chelucci et al., was used in the Pd-catalyzed asymmetric allylic substitution [65]. Bipy ligands **154a,b** [72] and **155** [76] were utilized in the diethylzinc reaction of aldehyde and **156** [161] were applied in cyclopropanation.

Through similar homocoupling synthetic methods, Fu and Chan developed ligands **157** [162] and **158** [163] for asymmetric cyclopropanation, respectively. Excellent results in diastereoselectivity and enantioselectivity for the cyclopropanation of styrene with 2,6-di-*tert*-butyl-4-methylphenyl diazoacetate were obtained for **157**. Another type of ring-fused bipy, ligand **159**, was reported independently by von Zelewsky and co-workers [164] and Kočovský and co-workers [165]. Using a similar strategy, Kočovský developed a variety of bipys **160** and **161a–c**. Ligands **159** and **160** were active catalysts for the Cu-catalyzed allylic oxidation of cycloalkenes [166,167]. For the ligand **161b**, good results were obtained in the Cu-catalyzed asymmetric cyclopropanation [168].

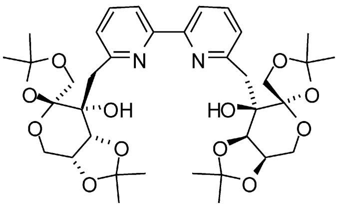

152



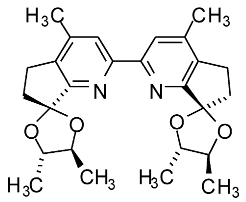
153



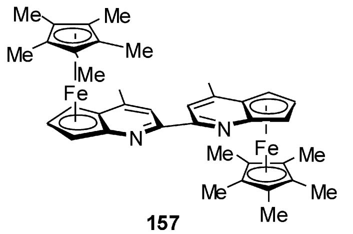
154a: R = Ph
154b: R = CH3



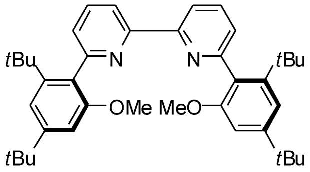
155



156

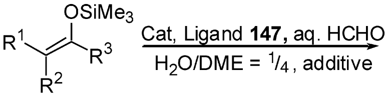


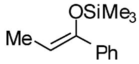
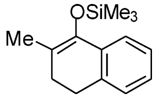
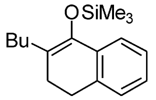
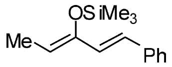
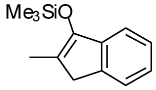
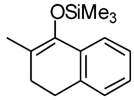
157

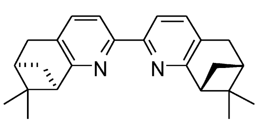


158

Table 9
Catalytic asymmetric hydroxymethylation of silicon enolates



Entry	Substrate	Catalyst	Additives	%Yield	%Ee
1		Bi(OTf)₃	Bipy	93	91
2		Bi(OTf)₃	Bipy	81	95
3		Bi(OTf)₃	Bipy	68	93
4		Sc(OTf)₃	–	73	92
5		Sc(OTf)₃	–	90	90
6		Sc(OTf)₃	–	80	94



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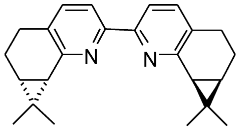
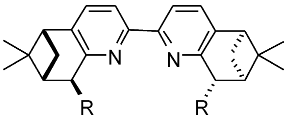
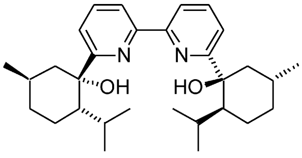


160

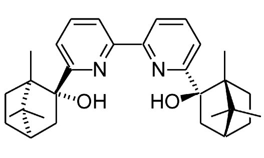


161a: R = H
161b: R = Et
161c: R = *n*-but

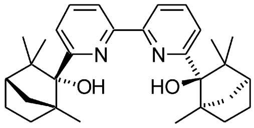
C_2-Symmetric chiral bipy ligands **162–165**, prepared using the nickel(0)-mediated homocoupling of the chiral bromopyridyl alcohols, showed good catalytic activity in diethylzinc addition to aldehydes [68].



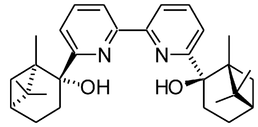
162



163



164



165

4.4. *From cross coupling*

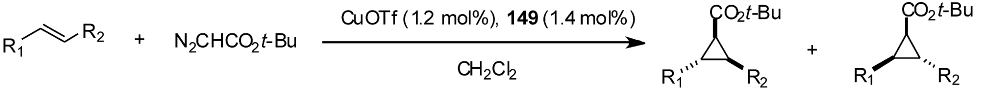
C_1-Symmetric bipy-alcohol ligands **166–169** were prepared via Pd(0) catalyzed cross-coupling of different chiral bromopyridyl alcohols with 2-pyridylzinc chloride (Scheme 44) [67,169]. These ligands were active catalysts in the Cu-catalyzed allylic oxidation of cyclohexene [169].

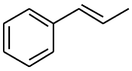
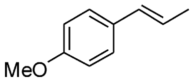
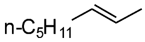
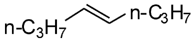
Table 11 highlights some of the catalytic results for ligand **159**, **160**, **162–169** in the Cu-catalyzed allylic oxidation of cyclic alkene. Ligands **159** and **160** reported by Kočovský showed fast reaction rate with good enantioselectivities [166,167]. C_1- and C_2-bipy N,O-ligands **162–169**, developed by this group gave comparable results [169].

4.5. *From resolution of racemates*

Enantiomerically pure bipy ligands can be obtained by the resolution method. Chiral bipy *N*,*N*-bisoxides are bipy-

Table 10
Asymmetric cyclopropanation of olefins



Entry	Substrate	%Yield	*Cis*:*trans*	%Ee (*cis*)	%Ee (*trans*)
1		54	60:40	>99	24
2		45	55:45	>99	35
3		67	50:50	75	69
4		55	–	53	

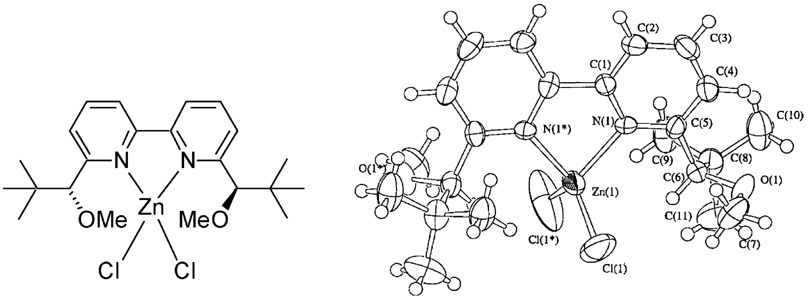
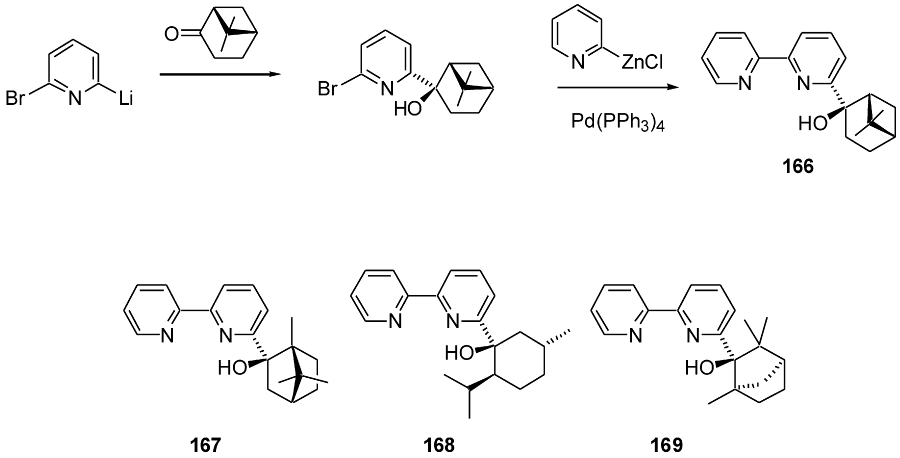


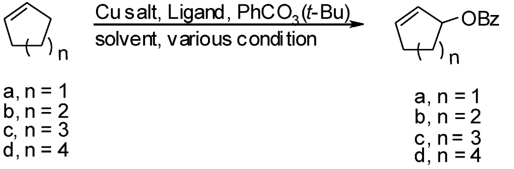
Fig. 6. X-ray crystallography of Zn(**148**)Cl$_2$ complex.



Scheme 44.

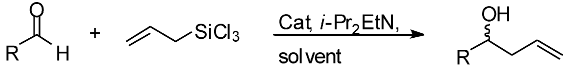
containing compounds that takes advantage of this method. Axial chiral *N*,*N*-bisoxides ligands **170** and **171**, derived from 1,1′-biisoquinoline and 2,2′-biquinoline, were separated into their enantiomers by complexation with chiral binaphthol [170–172]. They have been utilized in the enantioselective ring opening of meso-epoxides with tetrachlorosilane [173]. Different bipy *N*,*N*-bisoxide ligands **172a** and **172b**, developed by Hayashi and co-workers, were obtained through coupling of the diol with 2,2′-bis(chlorocarbonyl)-1,1′-binaphthalene [174,175]. Ligand **173** reported by Malkov and Kočovský, on the other hand, was obtained through cocrystallization with (*S*)-(−)-2,2′-dihydroxy-1,1′-biphenyl [176]. Ligands **170–173** were used as Lewis base in the enantioselective allylation of benzaldehyde with allyltrichlorosilane (Table 12) [170–176]. Chiral py *N*-oxide has been reviewed previously [177]. In addition, enantiomers of bipy ligands **174** and **175**, obtained by semipreparative HPLC on a chiral column, were applied in the Cu-catalyzed cyclopropanation [178,179].

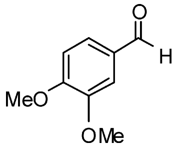
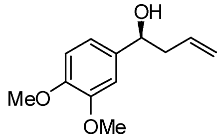
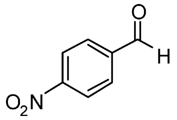
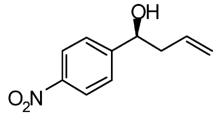
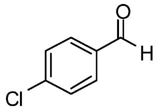
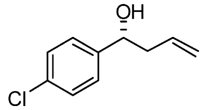
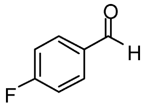
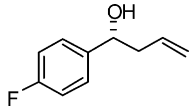
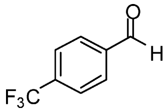
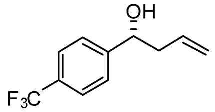
Table 11
Cu catalyzed asymmetric allylic oxidation of cyclohexene promoted by bpy ligands

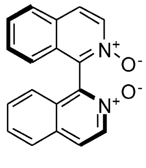


Entry	Ligand	Substrate	Time (h)	Temperature (°C)	%Yield	%Ee
1	**159**	c	12	0	66	75 (*R*)
2	**160**	c	96	−20	35	82 (*R*)
3	**162**	c	96	Room temperature	62	61 (*R*)
4	**162**	d	96	Room temperature	72	70 (*R*)
5	**166**	a	48	Room temperature	83	56 (*S*)
6	**166**	b	48	Room temperature	43	65 (*S*)

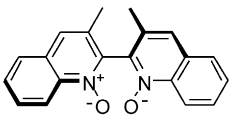
Table 12
Enantioselective allylation of benzaldehyde with allyltrichlorosilane



Entry	Ligand	Cat. (mol%)	Substrate	Product	%Yield	%Ee
1	**172a**	0.1			95	98 (*S*)
2	**173**	5			73	89 (*S*)
3	**173**	5			65	93 (*R*)
4	**173**	5			79	91 (*R*)
5	**173**	5			85	96 (*R*)



170



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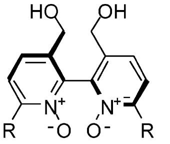
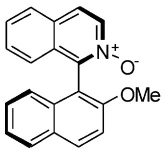
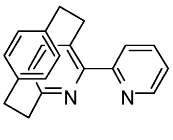


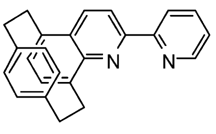
172a: Ph
172b: 3,5-Me$_2$-4-MeOC$_6$H$_2$



173



174



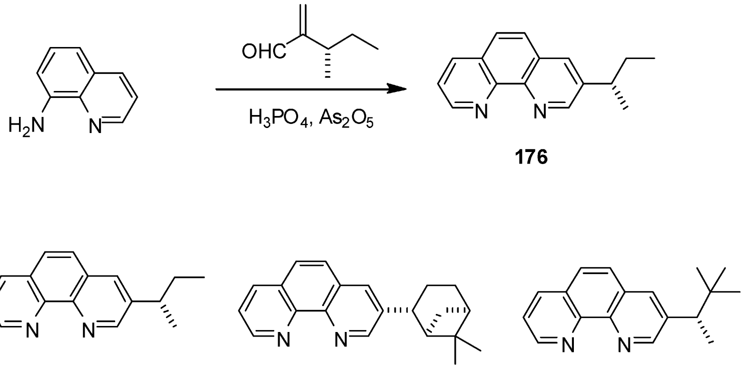
175

5. Chiral 1,10-phenanthrolines

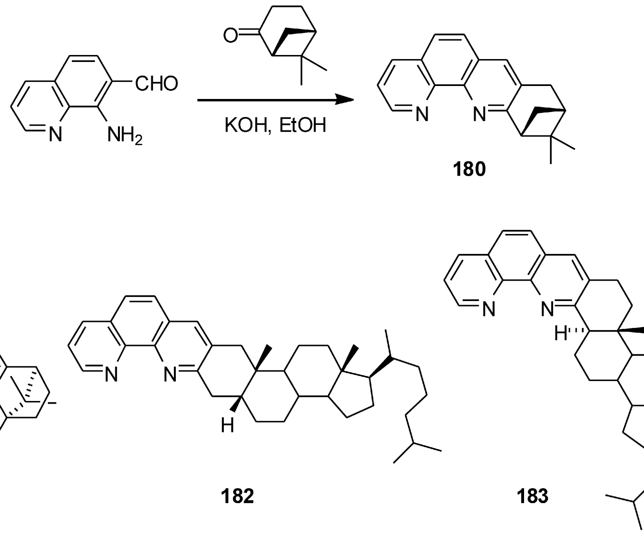
Chiral phens, structurally similar to the bipys ligands, have two py rings fused together through extensive π-conjugation. The introduction of chirality to phen can be achieved through cyclization reactions and using achiralphen as template.

5.1. From cyclization reactions

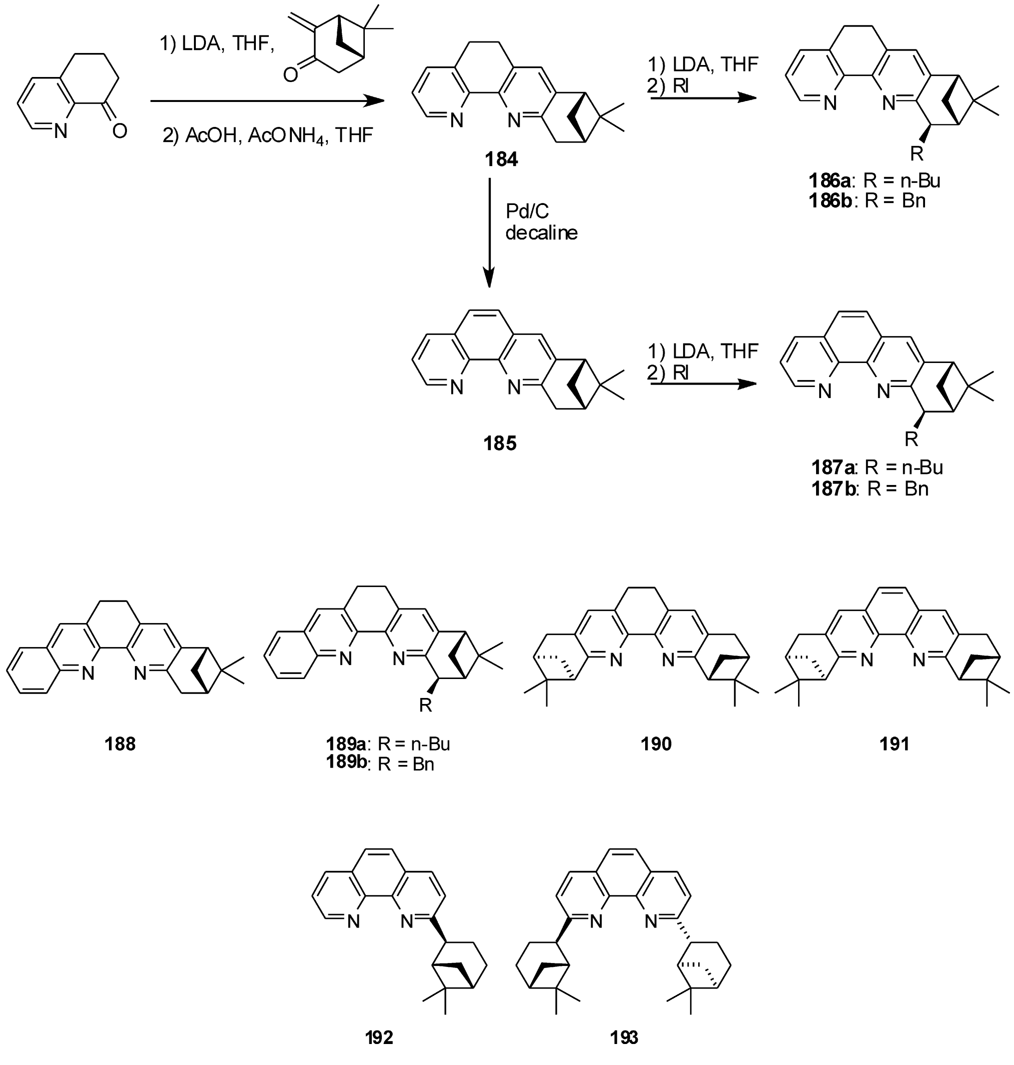
The preparation of 3-substituted chiral C_1- and 3,8-disubstituted C_2-symmetric phen ligands were obtained through stepwise ring cyclization reactions. Chiral ligand **176**, reported by Gladiali et al., was obtained via Doebner–Miller reaction (Scheme 45) [180]. C_2-symmetric **177**, another example, was obtained via stepwise cyclization starting from 2-nitroaniline [181]. Ligands **178** [182,183] and **179** [181] were



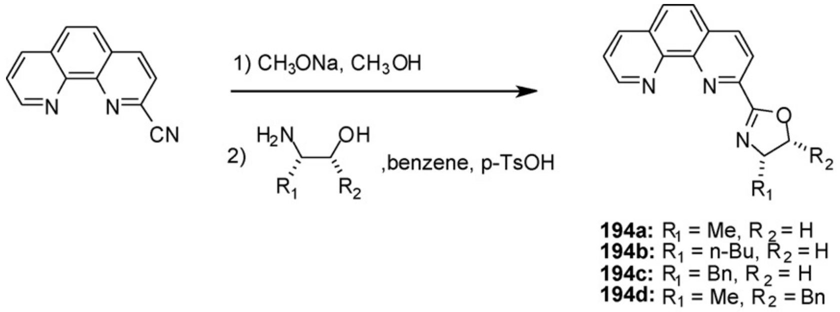
Scheme 45.



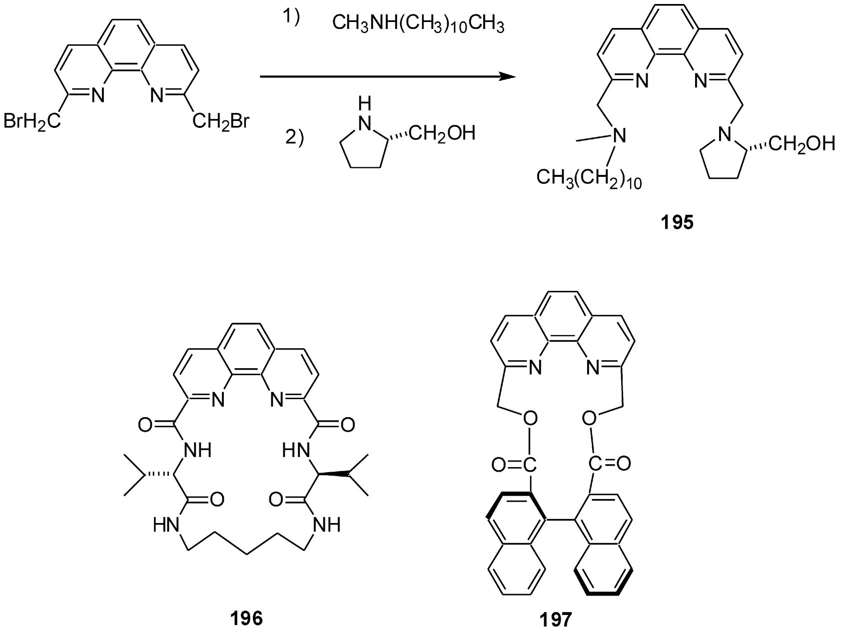
Scheme 46.



Scheme 47.



Scheme 48.



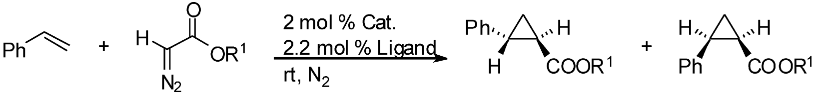
Scheme 49.

obtained using different α,β-unsaturated aldehydes. Ligands **176–179** were applied in enantioselective transfer hydrogenation [180,181,183] and hydrosilylation [184]. Ligand **178** was also utilized in Cu catalyzed asymmetric cyclopropanation [122] and Pd catalyzed allylic substitution [143].

Ligand **180** was first synthesized by Thummel using the Friedländer condensation reaction of 8-aminoquinoline-7-carbaldehyde with nopinone (Scheme 46) [185]. Chelucci later reported the utility of **180** in Cu catalyzed allylic oxidation [186]. The author also prepared ligand **181** [187] in high yield by a similar procedure and applied in the same allylic oxidation [186]. Using Friedländer condensation, Chelucci further introduced ligand **182** [188] and **183** [188,189] with a steroid backbone and used them in the Pd catalyzed allylic substitution of 1,3-diphenylprop-2-enyl acetate with dimethyl malonate [189]. Both of **182** and **183** were also applied in Cu catalyzed allylic oxidation of cyclohexene [186].

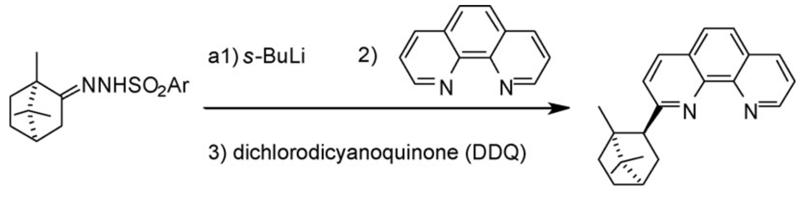
Table 13

Asymmetric cyclopropanation with chiral terpy and terpy mono- and di-*N*-oxides



Entry	Ligand	Catalyst	*R*	%Yield	*Trans*:*cis*	%Ee (*trans*)	%Ee (*cis*)
1	**204a**	$Cu(OTf)_2$	Et	87	64:36	75	84
2	**204a**	$RhCl_3$ [a]	Et	46	70:30	65	71
3	**204c**	$Cu(OTf)_2$	Et	96	69:31	90	94
4	**204c**	$Cu(OTf)_2$	*t*Bu	91	77:23	85	90
5	**204d**	$Cu(OTf)_2$	Et	97	61:39	72	75

[a] The active catalyst was treated with AgOTf (4 equiv.) before the reaction.



198

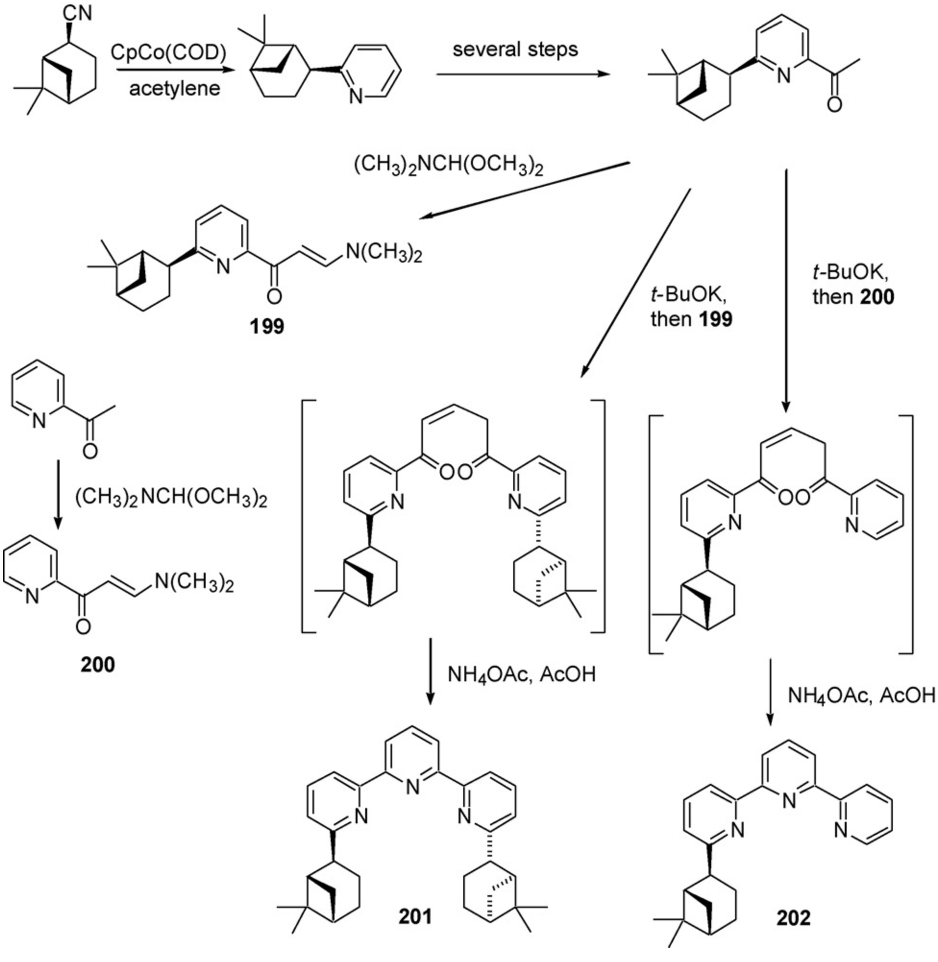
Scheme 50.

Other phens can be obtained via cyclization reactions starting from 8-quinolone and α,β-unsaturated ketone derived from terpene (Scheme 47) [190]. Dihydro-phen ligand **184**, obtained via cyclization, underwent dehydrogenation to give **185**. Ligand **186a,b** and **187a,b** were prepared by quenching of lithiated **184** and **185**, respectively, with suitable alkyl iodide. Using similar procedure, **188–189** [191], **190–191** [192] and **192–193** [193] were obtained. Ligands **184–193** were applied in a number of asymmetric catalysis such as Pd-allylic substitution reactions [143,190,191], Cu-cyclopropanation [194], Cu-allylic oxidation [186,192], and Rh-hydrosilylation [194].

5.2. *From achiral phenanthroline*

A chiral oxazoline ring was introduced to achiral phen by condensation of 2-cyanophen with different chiral amino alcohols via methoxyimidate intermediates (Scheme 48). Ligands **194a–d** prepared were found effective in Rh catalyzed enantioselective hydrosilylation reactions of acetophenone [185].

The C_1-symmetric ligand **195**, reported by Engbersen and co-workers, was obtained by reacting 2,9-bis(bromethyl)-1,10-phen sequential reactions with dialkyl amine and



Scheme 51.

(*S*)-2-pyrrolidinemethanol (Scheme 49). The Co(II) complex of **195** catalyzez the hydrolysis of D-*p*-nitrophenyl esters of picolinic acid faster than that of the L-enantiomer [195]. Following a similar strategy, other chiral macrocycle phen ligands **196** [196] and **197** [197] were developed and utilized in Cu(I) catalyzed cyclopropanation of styrene with EDA [196,197].

Ligand **198** was developed by reacting phen with a lithiated hydrazone followed by DDQ-induced rearomatization (Scheme 50). The ligand exhibited very good result in the Pd-catalyzed alkylation of 1-phenylbut-2-enyl acetate with dimethyl malonate with ee up to 96% [198].

6. Chiral terpyridines and other polypyridines

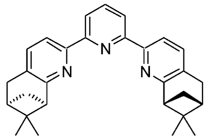
6.1. Terpyridines from cyclotrimerization and cyclization

The use of optically active terpy ligands for asymmetric catalysis was first reported by Chelucci [199]. They reported the synthesis of ligands **201** and **202** (Scheme 51) and their use in the Cu-catalyzed asymmetric cyclopropanation [122] and Pd-catalyzed allylic substitution [143].

6.2. Terpyridines from Kröhnke condensation

In the course of studying the coordination chemistry of terpys with transition metals, von Zelewsky and co-workers reported the synthesis through Kröhnke condensation of a new C_2-symmetric chiral terpy **203** and its ruthenium and rhodium complexes (Scheme 52) [200]. However, no catalysis was reported.

Encouraged by the result, chiral terpy ligands with different substituents on **203** and different fused rings on the two side of the pys were explored. Terpy ligands **204a–d** were prepared by deprotonation and alkylation (Scheme 53) [201]. Terpy ligands **205–207** [202] were prepared by Kröhnke condensation of different chiral α,β-unsaturated ketones with 2,6-bis(pyridinoacetyl)py diiodide. The Rhodium complex of **206**, $\mathrm{Rh(III)Cl_3}$(**206**), was successfully characterized by X-ray crystallography (Fig. 7). The copper and rhodium complexes of **203–207** were active catalysts towards asymmetric cyclopropanation (Table 13) [201,202]. Chelucci et al. also reported independently on the utilities of these terpy ligands in Ru-, Rh- and Cu-catalyzed asymmetric cyclopropanation and hydrosilylation [203,204].



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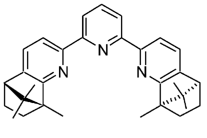

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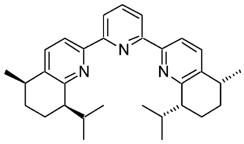
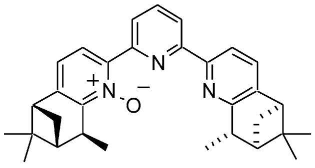


207

6.3. Terpyridine-N-oxides

The scope of study on asymmetric catalysis with terpy-based ligands was expanded by using *m*-CPBA to oxidize terpys **204a**, **205** and **206** to their corresponding mono-, di- and tri-*N*-oxides **208–215** [205,206]. Both mono- and di-*N*-oxide ligands showed effectiveness in asymmetric cyclopropanation [205], whereas tri-*N*-oxides were active as Lewis base-catalysts in the asymmetric allylation of aldehydes using allyltrichlorosilane [206].



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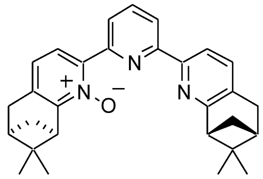

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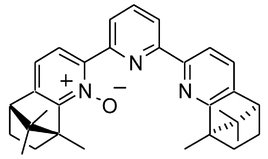

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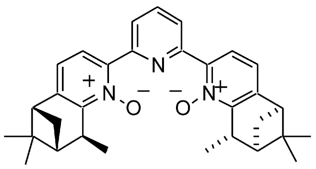

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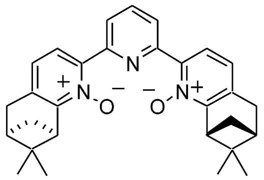

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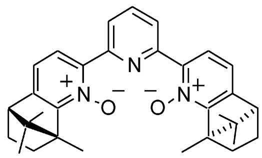

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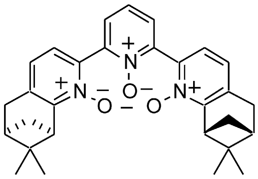

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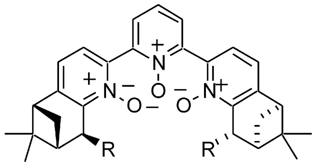
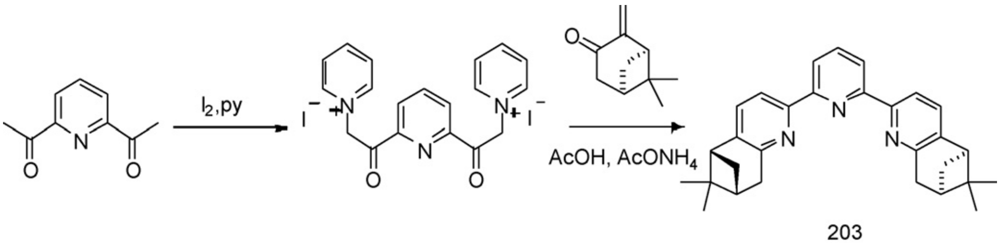
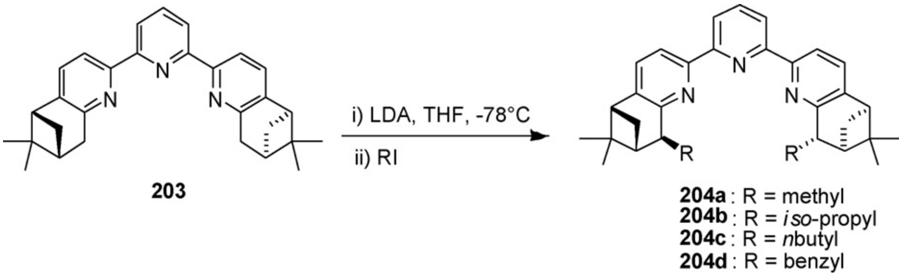


215a: R = Bn
215b: R = Me
215c: R = *n*-Butyl
215d: R = *i*-Propyl



Scheme 52.



Scheme 53.

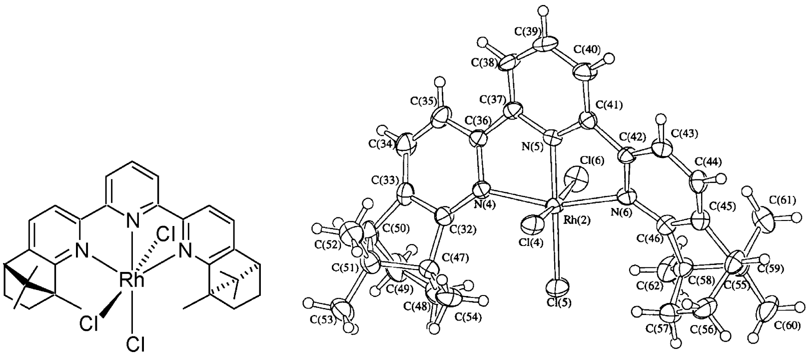


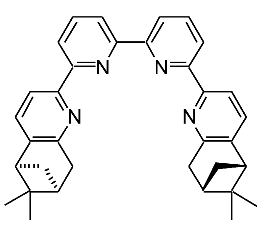
Fig. 7. X-ray crystallography of Rh(III)Cl_3(**206**) complex.

6.4. *Trispyridines*

Only one example of chiral tripodal py ligand was applied in asymmetric reaction. Rodríguez, von Zelewsky and Llobet recently reported the preparation of C_3-symmetric tripodal trispy ligand **218** (Scheme 54). Starting from a chiral 2-bromopy **216**, a dipy ketone **217** was obtained from reaction with chloroformate. Treatment of another equivalent of **216** afforded the trispy methanol ligand, which further underwent methyl alkylation to generate **218**. The Ru complex of **218** was attempted in enantioselective epoxidation of styrene [207].

6.5. *Tetra- and quarter-pyridines*

Compared with other chiral py-containing ligands, studies on tetrapys and quaterpys have been limited. The first syntheses of optically active quaterpy **219** was reported by Constable and co-workers and they isolated a disilver(I) double helical structure [208]. Tetrapys **220**, which has two bipy units bridged by a linker was developed by von Zelewsky and co-workers [209]. It showed interesting coordination chemistry [209] and was later employed by Moutet et al. in the electrocatalytic hydrogenation of acetophenone with Rh complexes [210].



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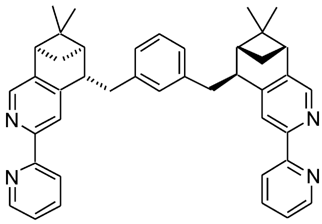
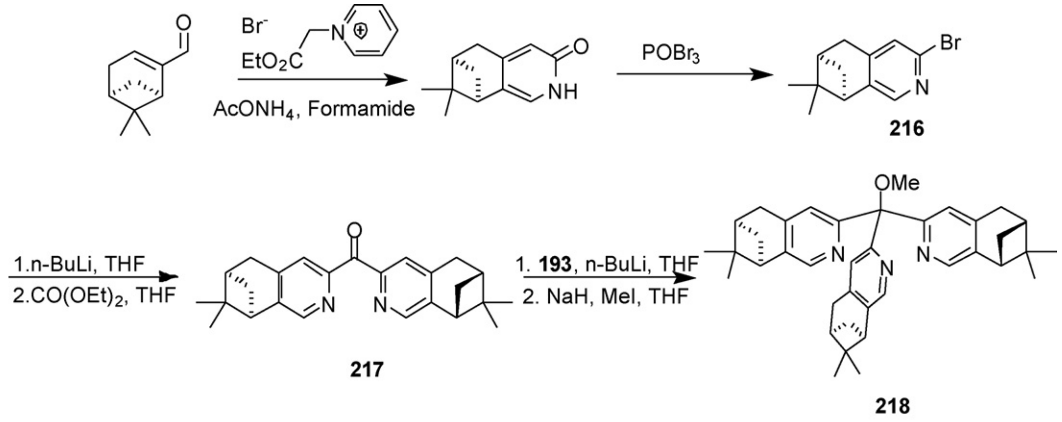
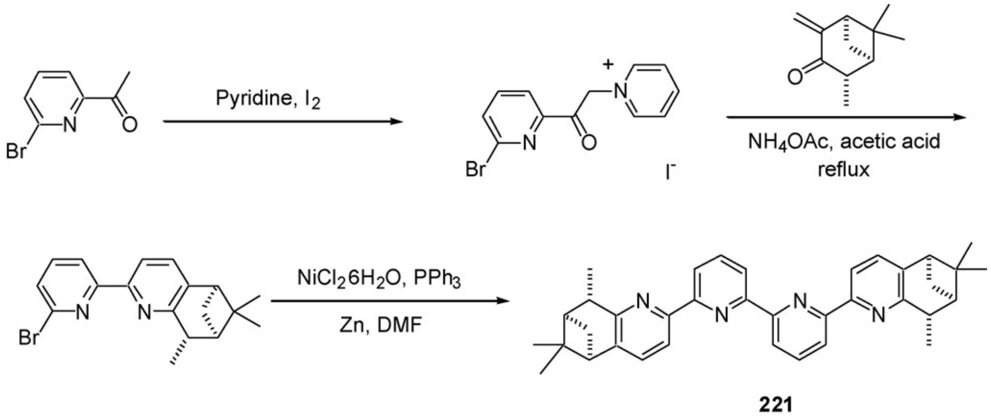


220

Recently, the synthesis of a new chiral quaterpy **221** through Kröhnke condensation and homocoupling was reported (Scheme 55). Interestingly, the self assembly of the single helicate was achieved by reacting [Pd(η^3-C_3H_5)$_2$Cl]$_2$ dimer and a CH_2Cl_2 solution of **221** in 1:1 ratio. The complex [Pd_2(η^3-C_3H_5)$_2$(**221**)]$^{2+}$ was isolated as a SbF_6 salt and the X-ray crystal structure revealed that the stereoselective formation was M-helix (Fig. 8). The helicate [Pd(η^3-C_3H_5)$_2$(**221**)]$^{2+}$ was used as catalyst for the allylic substitution of 1,3-diphenylprop-2-enyl



Scheme 54.



Scheme 55.

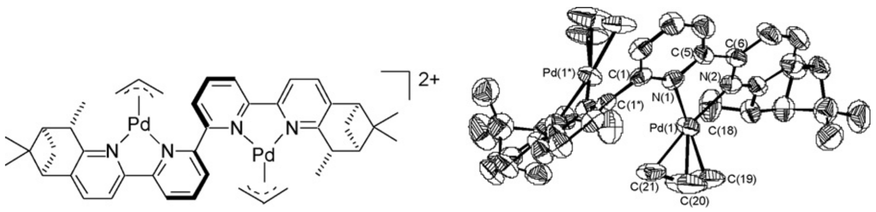


Fig. 8. X-ray crystallography of complex $[Pd(\eta^3\text{-}C_3H_5)_2(\mathbf{221})]^{2+}$.

acetate with dimethyl malonate; >99% in yield and up to 85% in enantioselectivities were obtained [211].

7. Conclusion

This article provides an overview of the rich variety of structures of chiral py-containing ligands and the various methods employed to construct them. From the number of catalytic reactions reported and some of the excellent results that were obtained through these ligands, it is evident that they play a very important role in asymmetric catalysis. The development of new chiral py-containing ligands is certainly of interest in research on new catalytic processes as well as in the improvement of existing catalytic reactions.

Acknowledgements

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