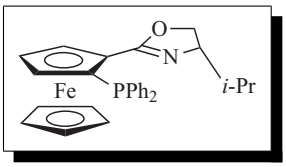
Ferrocene, 1-[(4*S*)-4,5-Dihydro-4-(1-methylethyl)-2-oxazolyl]-2-(diphenylphosphino)-, (2*R*)



[163169-10-6]

InChI = 1S/C23H23NOP.C5H5.Fe/c1-17(2)21-16-25-23(24-21)20-14-9-15-22(20)26(18-10-5-3-6-11-18)19-12-7-4-8-13-19;1-2-4-5-3-1;/h3-15,17,21H,16H2,1-2H3;1-5H;

InChIKey = FELCHLOAEMNOQM-UHFFFAOYSA-N

(a reagent used as a chiral ligand in a variety of transition metal-catalyzed reactions)

Alternate Name: (*S,S*)-[2-(4′-isopropyloxazolin-2′-yl)ferrocenyl]diphenylphosphine, (*S,S*)-ip-FOXAP.

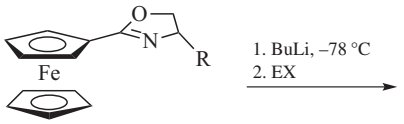
Physical Data: mp 164–165 °C.

Form Supplied in: an orange solid, commercially available.

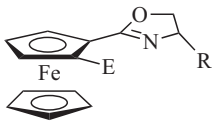
Purification: recrystallization from ethanol.

Handling, Storage, and Precautions: stored under nitrogen in the dark.

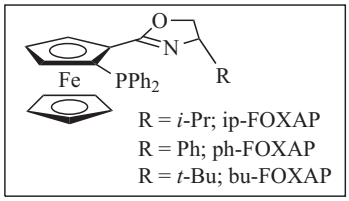
Preparation of Optically Active Ferrocenyloxazolinylphosphines (FOXAPs). Diastereoselective *ortho*-lithiation of optically active oxazolinylferrocenes, followed by quenching chlorodiphenylphosphine as an electrophile, gave the corresponding ferrocenyloxazolinylphosphines (abbreviated as FOXAPs) in high yields as a mixture of two diastereoisomers. The diastereoselectivity of the *ortho*-lithiation of optically active oxazolinylferrocenes depends on the nature of organolithium reagents (eq 1).[1] Steric repulsion between an organolithium reagent and a substituent on the oxazoline ring in the transition state is considered to be an important factor for the control of the diastereoselectivity.



R = *i*-Pr, Ph, *t*-Bu



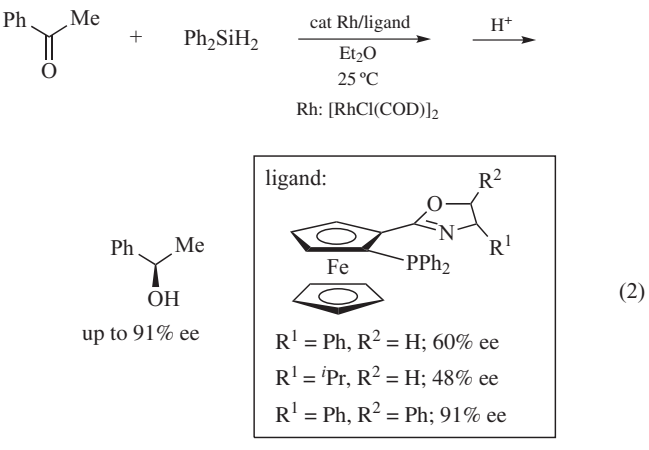
R = *i*-Pr	Diastereoselectivity
sec-BuLi	86–95% de
n-BuLi	71–94% de
n-BuLi/TMEDA	>99% de



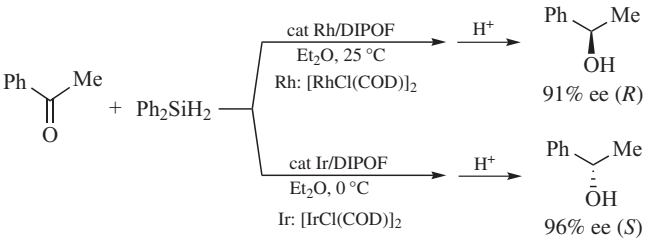
(1)

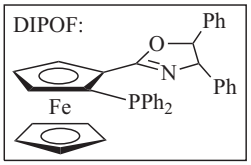
Optically pure ferrocenyloxazolinylphosphines were isolated by the recrystallization of a mixture of two diastereoisomers from ethanol. As a series of optically active ferrocenyloxazolinylphosphines, (*S,S*)-[2-(4′-isopropyloxazolin-2′-yl)ferrocenyl]-diphenylphosphine (abbreviated as ip-FOXAP) was obtained from the reaction of (*S*)-(4-isopropyloxazolin-2-yl)ferrocene with *n*-BuLi in the presence of *N,N,N′,N′*-tetramethylethylenediamine (TMEDA) in *n*-hexane at −78 °C for 2 h,[2] followed by addition of chlorodiphenylphosphine. ip-FOXAP is now commercially available from Wako Pure Chemical Co., Ltd and Tokyo Chemical Industry Co., Ltd (TCI).[3]

Rhodium- and Iridium-catalyzed Enantioselective Hydrosilylation of Ketones and Imines. Transition metal-catalyzed hydrosilylation of ketones is a useful method for the reduction of ketones. Thus, enantioselective hydrosilylation followed by hydrolysis provides an effective route to optically active secondary alcohols. In fact, some FOXAPs are effective ligands in the rhodium- and iridium-catalyzed enantioselective hydrosilylation of alkyl aryl ketones.[4] Although use of ip-FOXAP in the rhodium-catalyzed hydrosilylation affords only a moderate enantioselectivity, a related compound such as 4,5-diphenyloxazolinylferrocenylphsophine (abbreviated as DIPOF) was revealed to be the most effective ligand for rhodium- and iridium-catalyzed hydrosilylation (eq 2). Hydrosilylation of alkyl aryl ketones catalyzed by an iridium complex instead of a rhodium complex also proceeds smoothly to give the corresponding alcohols with a high enantioselectivity (eq 3).[5] Surprisingly, the absolute



(2)

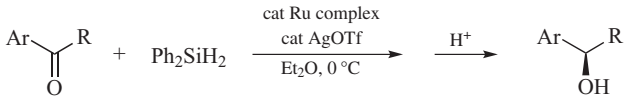


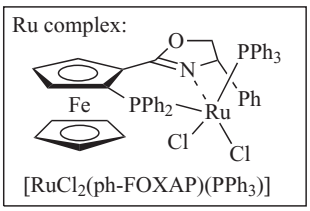


(3)

configuration of the produced alcohols was opposite to that in the case of rhodium-catalyzed reactions.[4,5] X-ray analysis of molecular structures of rhodium and iridium complexes bearing DIPOF as a ligand revealed that both complexes have almost similar chiral environments around the coordination site.[6] The exact reason why the absolute configuration of the produced alcohols from rhodium and iridium catalysis is different is not yet clear. As to the enantioselective hydrosilylation of imines, some FOXAPs worked as effective ligands only for iridium-catalyzed hydrosilylation of cyclic and acyclic imines.[7]

Ruthenium-catalyzed Enantioselective Hydrosilylation of Ketones and Imines. Ruthenium complexes bearing one of the FOXAPs as a chiral ligand are easily prepared from reactions of $[RuCl_2(PPh_3)_3]$ with FOXAPs.[8] A ruthenium complex bearing ip-FOXAP $[RuCl_2$(ip-FOXAP)$(PPh_3)]$ is now commercially available.[9] In the presence of a catalytic amount of ruthenium complexes, hydrosilylation of alkyl aryl ketones followed by acidic workup gave the corresponding alcohols with a high enantioselectivity (eq 4).[8] In contrast to rhodium- and iridium-catalyzed hydrosilylation, where only a moderate enantioselectivity was observed, use of FOXAPs was revealed to be effective ligands for ruthenium hydrosilylation. These results indicate that the triphenylphosphine ligand on the ruthenium atom of the ruthenium complexes plays an important role in construction of an effective chiral environment. Addition of metal triflates such as silver triflate and copper triflate was essential for the achievement of a high enantioselectivity. Ruthenium complexes bearing FOXAPs were also effective for the enantioselective hydrosilylation of a cyclic imine to give the corresponding chiral amine with a high enantioselectivity.[8]

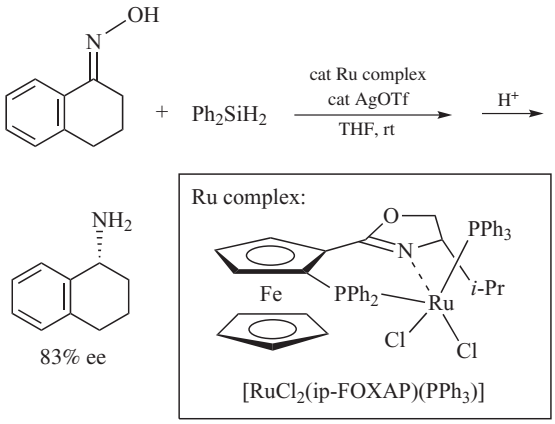




(4)

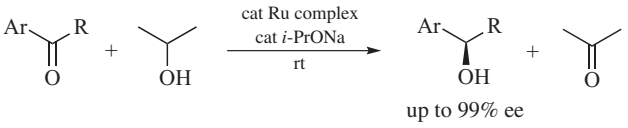
Ruthenium-catalyzed Enantioselective Hydrosilylation of Oximes. Hydrosilylation of oximes followed by hydrolysis provides the corresponding primary amines, while hydrolysis of hydrosilylated products of imines gave the corresponding secondary amines. In the presence of a catalytic amount of the ruthenium complexes bearing FOXAPs, hydrosilylation of 1-tetralone oxime followed by hydrolysis gave the corresponding amine with a high enantioselectivity (eq 5).[10] Addition of a catalytic amount of silver triflate slightly increased the yield of the amine. This ruthenium-catalyzed system is useful as a direct and catalytic preparative method for primary amines without protection of the nitrogen atom.

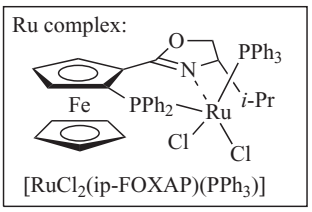
Ruthenium-catalyzed Enantioselective Redox Reactions of Ketones and Alcohols. The ruthenium complex bearing

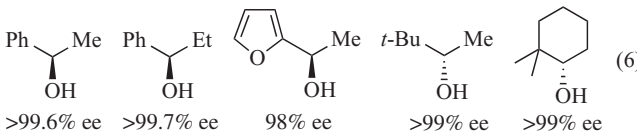


(5)

ip-FOXAP $[RuCl_2$(ip-FOXAP)$(PPh_3)]$ worked as an effective catalyst for the transfer hydrogenation of ketones.[11] 2-Propanol was the most effective hydrogen source in this reaction system. No reaction occurred at all when the combination of formic acid and triethylamine was used as a hydrogen source. Interestingly, the transfer hydrogenation of dialkyl ketones proceeded smoothly to give the corresponding alcohols with a high enantioselectivity (eq 6). On the other hand, kinetic resolution of racemic alcohols with acetone as a hydrogen acceptor took place to recover unreacted alcohols with a high enantioselectivity (eq 7).[11] It is







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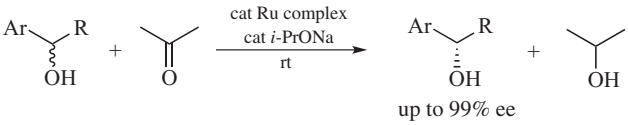
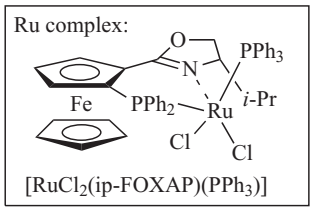
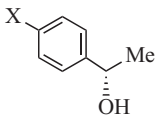


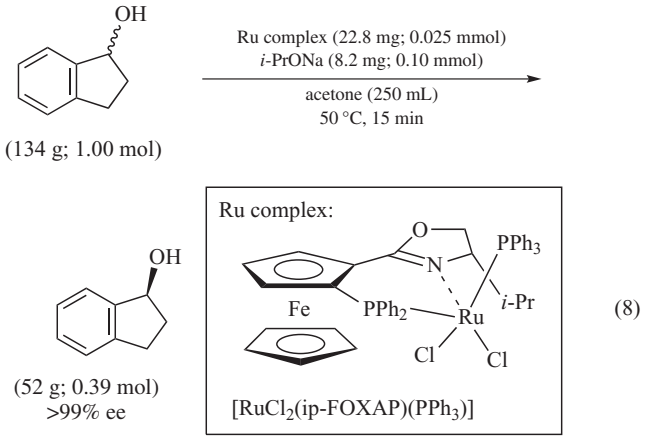


(7)



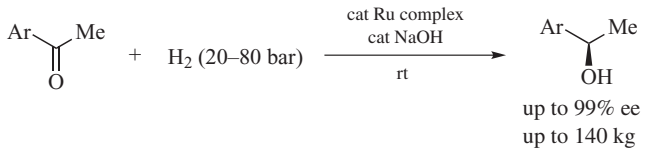
X = H; >99.6% ee (s >100)
X = Me; >99.7% ee (s >100)
X = Cl; >99.8% ee (s >50)
X = Br; >99.8% ee (s >100)

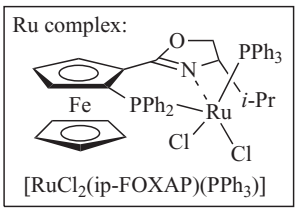
noteworthy that kinetic resolution of 1-indanol proceeded quite rapidly with the turnover frequency (TOF) exceeding 80 000 h^{-1} (eq 8). This reaction system provides a practically useful method for the preparation of optically active 1-indanol on a 100 g scale.



(8)

Ruthenium-catalyzed Enantioselective Hydrogenation of Ketones. The ruthenium complex bearing ip-FOXAP [$RuCl_2$(ip-FOXAP)(PPh_3)] also worked as an effective catalyst for the hydrogenation of alkyl aryl ketones (eq 9).[12,13] The catalytic hydrogenation in higher concentration and on a large scale was available with a high enantioselectivity and efficiency. This reaction system provides a large amount of optically active alcohols, where 140 kg of ketones could be fully converted into alcohols.





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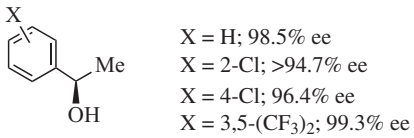
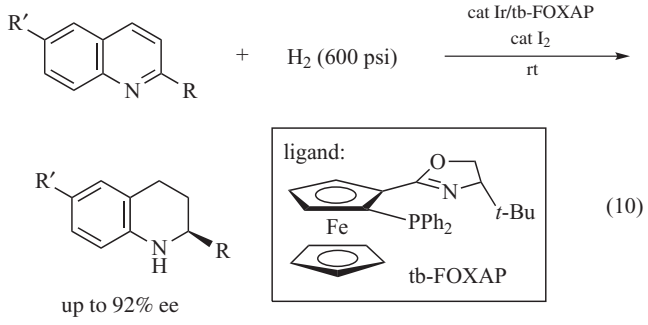


Iridium-catalyzed Enantioselective Hydrogenation of Imines and Alkenes. Some FOXAPs are effective ligands in the iridium-catalyzed enantioselective hydrogenation of 2-substituted quinolines and aryl-substituted alkenes (eqs 10 and 11).[14,15] Interestingly, the hydrogenation of both unfunctionalized and functionalized alkenes proceeds smoothly in the presence of iridium complexes bearing FOXAPs [Ir(COD)(FOXAP)]BAr_4 (Ar = C_6H_3-3,5-$(CF_3)_2$) to give the corresponding hydrogenated products with a high enantioselectivity.[15]



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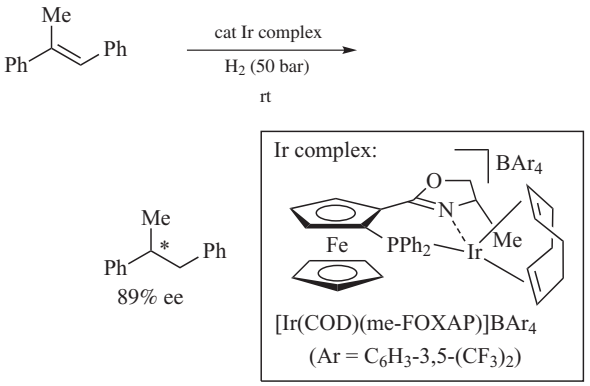
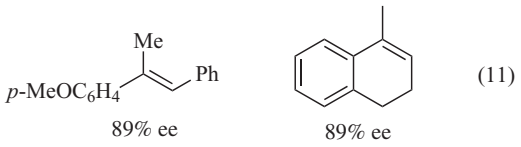
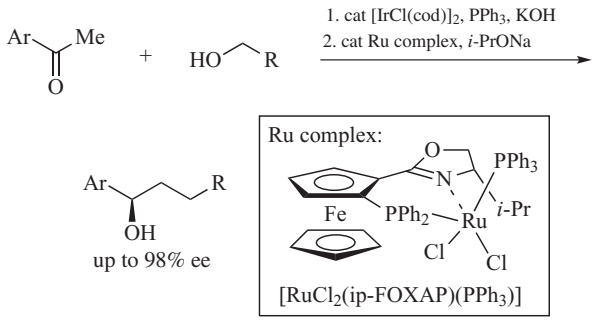
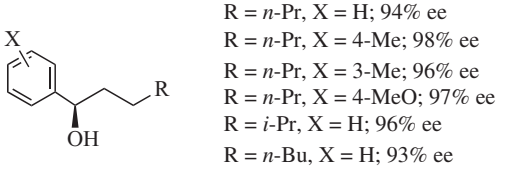




(11)

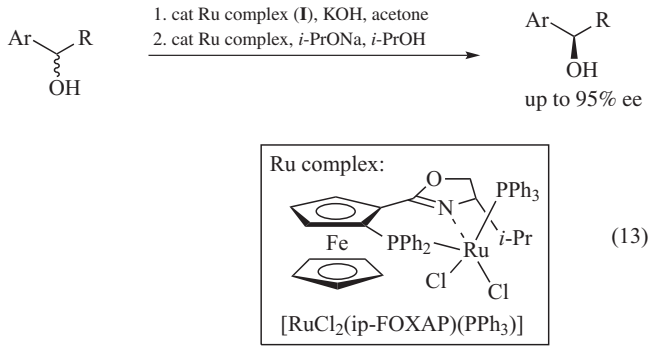
Transition Metal-catalyzed Enantioselective Sequential Reactions. The combination of iridium-catalyzed α-alkylation of ketones with alcohols[16] and ruthenium-catalyzed transfer hydrogenation of ketones[8] effected the enantioselective α-alkylative reduction of prochiral ketones with alcohols to directly give the chiral alcohols with elongation of the carbon skeleton in high yields with a high enantioselectivity (eq 12).[17] A similar reaction proceeded when only the ruthenium complex was used, but a low





(12)

enantioselectivity was observed in the produced alcohols. This result indicates that the combination of iridium and ruthenium complexes is appropriate in this reaction system. The methodology can also be applied to deracemization of racemic alcohols via a sequential oxidation and reduction system. The combination of two different ruthenium complexes promoted deracemization process smoothly, where chiral alcohols were obtained in high yields with a high enantioselectivity (eq 13).[18]



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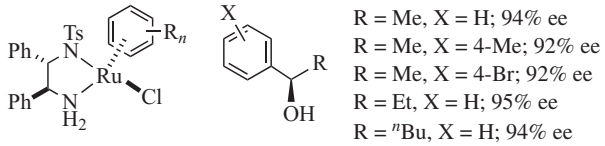
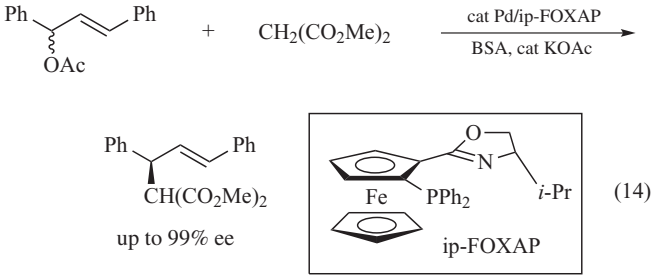


Palladium- and Nickel-catalyzed Enantioselective Allylic Substitution Reactions and Related Reactions. Transition metal-catalyzed allylic substitution reactions of allylic alcohol derivatives with nucleophiles are one of the most reliable methods in organic synthesis. The process is catalyzed by various transition metal complexes and a wide variety of nucleophiles are available for this reaction to afford the corresponding allylated products with high chemo- and regioselectivities. Enantioselective version of this process has also been extensively studied, providing useful methods for the synthesis of various optically active compounds including natural products.[19] Some FOXAPs worked as effective ligands for the palladium-catalyzed allylic alkylation with *soft* carbon-centered nucleophiles to give the corresponding allylic alkylated products with a high enantioselectivity (eq 14).[20] In contrast to the allylic substitution reactions with *soft* carbon-centered nucleophiles, successful examples using *hard* carbon-centered nucleophiles are limited. In this reaction system, *hard* carbon-centered nucleophiles first attack the metal center, followed by reductive elimination to afford the allylic substituted products. Thus, the reaction mechanism is quite different from that of *soft* carbon-centered nucleophiles. Some FOXAPs also worked as effective ligands for the nickel-catalyzed allylic alkylation with *hard* carbon-centered nucleophiles such as Grignard reagents to give the corresponding allylic alkylated products with a high enantioselectivity (eq 15).[21] The allylic substitution reactions with arylboric acids also proceeded, but only a moderate enantioselectivity was observed.[22] As related reactions, palladium-catalyzed enantioselective Mizoroki–Heck reactions and ring-opening reactions proceeded with a high enantioselectivity when FOXAPs were used as chiral ligands (eqs 16 and 17).[23,24]



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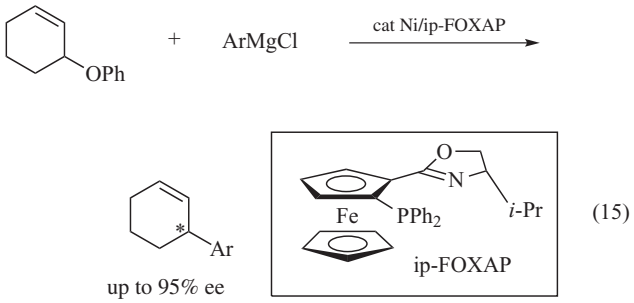

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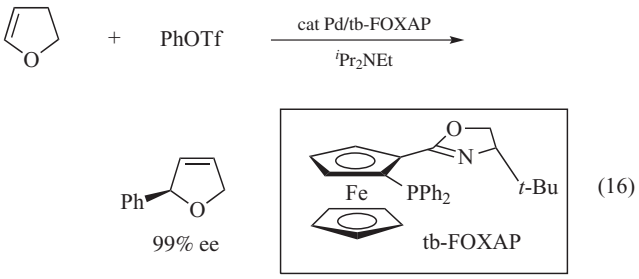

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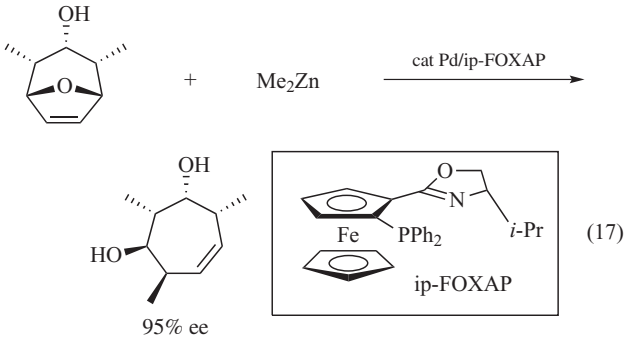
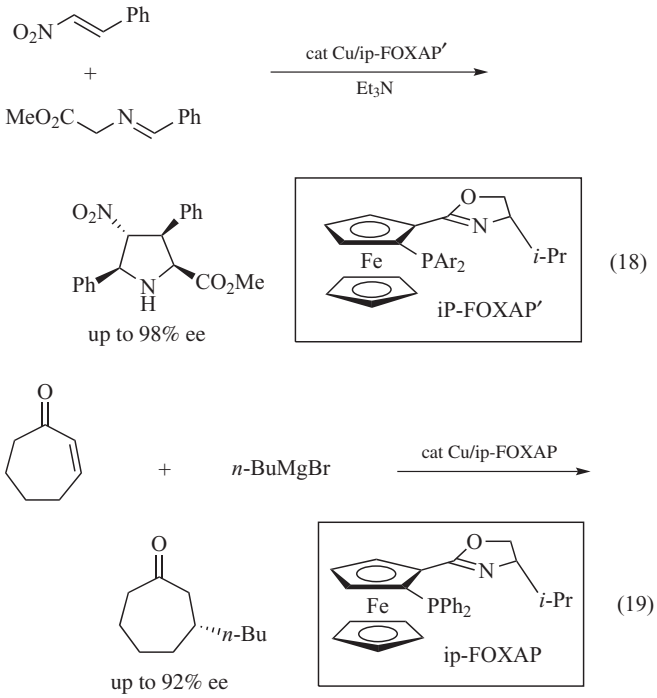
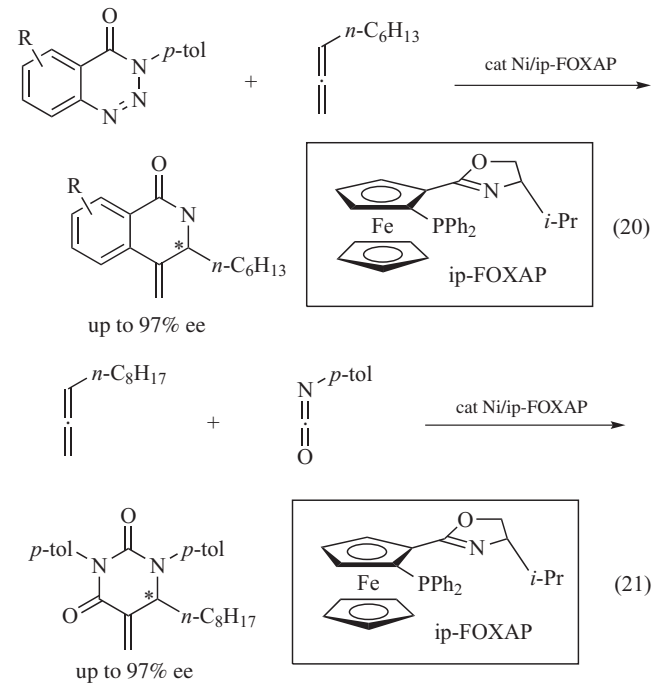


(17)

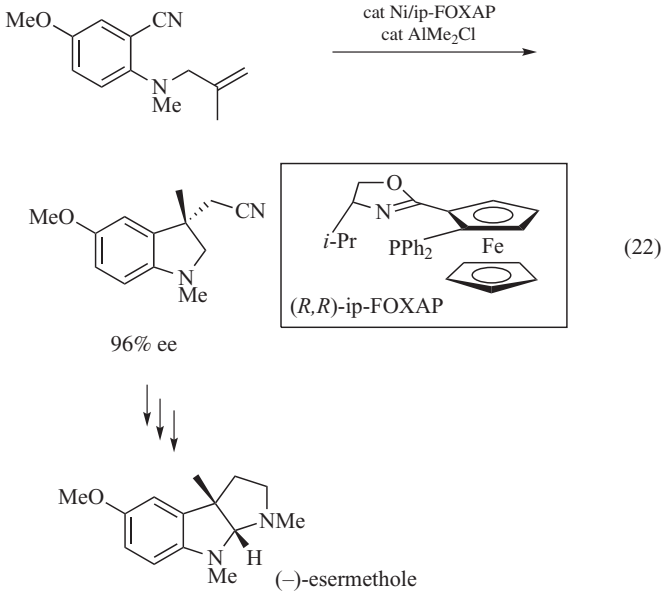
Copper-catalyzed Cycloaddition and Related Reactions. 1,3-Dipolar cycloaddition of azomethine ylides with electron-deficient alkenes is one of the useful methods for the construction of highly substituted pyrrolidines. Some FOXAPs also worked as effective ligands for the copper-catalyzed cycloaddition with a high enantioselectivity (eq 18).[25] The diastereoselectivity depends on the nature of aryl groups on the phosphorus atom in FOXAPs. On the other hand, the combination of copper(I) complex and FOXAPs can be applied to nucleophilic addition to electrophiles to give the addition products with a high enantioselectivity (eq 19).[26,27] In these reaction systems, useful levels of enantioselective induction were achieved. The planar chirality derived from the ferrocene skeleton was found to be essential for the achievement of high enantioselectivity.



Nickel-catalyzed Cycloaddition and Related Reactions. Nickel-catalyzed cycloaddition of 1,2,3-benzotriazin-4(3*H*)-ones with allene derivatives by using ip-FOXAP as a chiral ligand proceeded smoothly to give 3,4-dihydroisoquinolin-1(2*H*)-ones with a high enantioselectivity (eq 20).[28,29] Although bidentate phosphines such as Me-DuPhos exhibited reasonable enantioselectivity, only a moderate regioselectivity was observed in the cycloaddition products. High regio- and enantioselectivities were achieved when ip-FOXAP was used as a chiral ligand. ip-FOXAP also worked as the most effective ligand for the nicked-catalyzed [2 + 2 + 2] cycloaddition of isocyanates and allenes to give the corresponding dihydropyrimidine-2,4-diones with a high enantioselectivity (eq 21).[30]



Nickel-catalyzed Arylcyanation of Alkenes. The intramolecular arylcyanation of alkenes in the presence of a nickel complex and a Lewis acid such as $AlMe_2Cl$ is developed to be a versatile protocol to prepare a variety of synthetically useful nitriles bearing a benzylic quaternary carbon. The enantioselective version of the intramolecular arylcyanation of alkenes was achieved by using (*R,R*)-ip-FOXAP as a chiral ligand (eq 22).[31] This method can be applied to the synthesis of (−)-esermethole via three steps from the arylcyanated product.



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