

Stereoselective Asymmetric Hydrogenation Catalysts

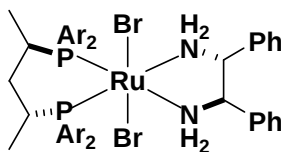
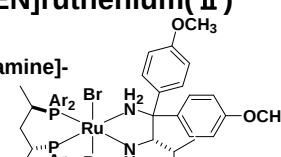
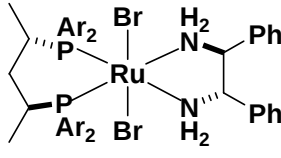
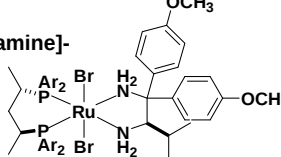
-XylSKEWPHOS Catalysts-



We Kanto Chemical launched asymmetric hydrogenation catalysts, which were discovered by NOY-ORI Molecular Catalysis Project of Exploratory Research for Advanced Technology (ERATO) by Japan Science and Technology Corporation (JST) and were developed by Exploitation and Application Study supported by JST. These catalysts have both diphosphine and diamine ligands, and the catalyst performance is tunable by the choice of these chiral ligands. Chiral Ru catalysts with BINAP ligand have high reactivity and enantioselectivity for the reaction of ketones¹, but higher enantioselectivity is desirable for certain ketonic substrates.

We examined the structure of the catalysts, and the chiral Ru catalysts with 1,3-diphosphine, Xyl-SKEWPHOS [(2,4-bis(di-3,5-xylylphosphino)pentane)] were found to have higher enantioselectivity than the prototype BINAP catalysts. Now we launch these asymmetric hydrogenation catalysts.

List of Products

Product	Product number	Package	
Dibromo[(<i>R,R</i>)-XylSKEWPHOS][(<i>R,R</i>)-DPEN]ruthenium(II) Dibromo[(<i>R,R</i>)-2,4-bis(di-3',5'-xylylphosphino)pentane]- [(<i>R,R</i>)-1,2-diphenylethanediamine]ruthenium(II) [RuBr₂[(<i>R,R</i>)-xylskewphos][(<i>R,R</i>)-dpen]] $C_{51}H_{62}Br_2N_2P_2Ru$ F.W : 1025.88	 Ar = 3,5-(CH ₃) ₂ C ₆ H ₃	10535-68 10535-95	100mg 500mg
Dibromo[(<i>R,R</i>)-XylSKEWPHOS][(<i>S</i>)-DAIPEN]ruthenium(II) Dibromo[(<i>R,R</i>)-2,4-bis(di-3',5'-xylylphosphino)pentane]- [(<i>S</i>)-1,1-bis(<i>p</i> -methoxyphenyl)-2-isopropyl-1,2-ethanediamine]- ruthenium(II) [RuBr₂[(<i>R,R</i>)-xylskewphos][(<i>S</i>)-daipen]] $C_{56}H_{72}Br_2N_2O_2P_2Ru$ F.W : 1128.01	 Ar = 3,5-(CH ₃) ₂ C ₆ H ₃	10537-68 10537-95	100mg 500mg
Dibromo[(<i>S,S</i>)-XylSKEWPHOS][(<i>S,S</i>)-DPEN]ruthenium(II) Dibromo[(<i>S,S</i>)-2,4-bis(di-3',5'-xylylphosphino)pentane]- [(<i>S,S</i>)-1,2-diphenylethanediamine]ruthenium(II) [RuBr₂[(<i>S,S</i>)-xylskewphos][(<i>S,S</i>)-dpen]] $C_{51}H_{62}Br_2N_2P_2Ru$ F.W : 1025.88	 Ar = 3,5-(CH ₃) ₂ C ₆ H ₃	10536-68 10536-95	100mg 500mg
Dibromo[(<i>S,S</i>)-XylSKEWPHOS][(<i>R</i>)-DAIPEN]ruthenium(II) Dibromo[(<i>S,S</i>)-2,4-bis(di-3',5'-xylylphosphino)pentane]- [(<i>R</i>)-1,1-bis(<i>p</i> -methoxyphenyl)-2-isopropyl-1,2-ethanediamine]- ruthenium(II) [RuBr₂[(<i>S,S</i>)-xylskewphos][(<i>R</i>)-daipen]] $C_{56}H_{72}Br_2N_2O_2P_2Ru$ F.W : 1128.01	 Ar = 3,5-(CH ₃) ₂ C ₆ H ₃	10538-68 10538-95	100mg 500mg

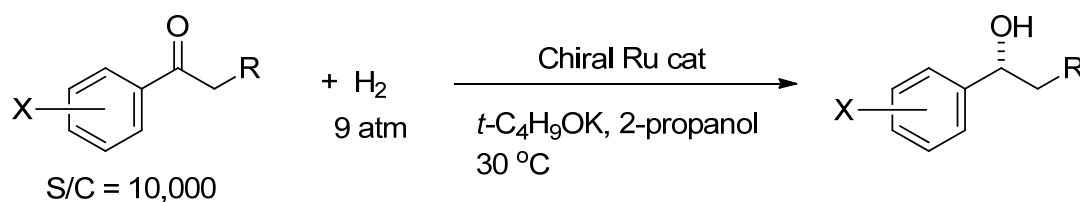


Asymmetric Hydrogenation

1 Hydrogenation of Aromatic Ketones

XylSKEWPHOS catalysts hydrogenate aromatic ketones effectively with high enantioselectivity to yield optically active alcohols². For example, acetophenone is hydrogenated with RuCl₂(tolbinap)(daipen) to afford 1-phenylethanol in 91% ee and RuBr₂(xylskewphos)(daipen) gives 98% ee for the same substrate. Aromatic ketones with several substituents on aromatic rings are also hydrogenated efficiently to give optically active alcohols in high ee. To obtain high enantioselectivity, proper selection of the catalysts is necessary. The DPEN catalyst usually gives higher enantioselectivity than the DAIPEN catalyst for the reaction of *o*-substituted aromatic ketones. For example, *o*-chloroacetophenone is hydrogenated with the DPEN and the DAIPEN catalyst in 97% ee and 94% ee, respectively. *o*-Bromoacetophenone is hydrogenated in >99% ee with both of the DPEN and the DAIPEN catalyst. For the reaction of *m*- and *p*-substituted aromatic ketones, the DAIPEN catalyst is assumed to have better enantioselectivity than the DPEN catalyst. For example, *m*-chloro and *m*-bromoacetophenone are hydrogenated with the DAIPEN catalyst in 97% ee and 99% ee, respectively. *p*-Fluoro, *p*-chloro and *p*-bromoacetophenone are also hydrogenated with the DAIPEN catalyst in 97% ee, 97% ee and 99% ee, respectively.

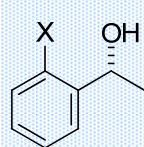
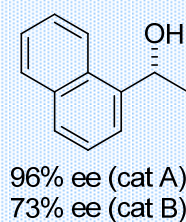
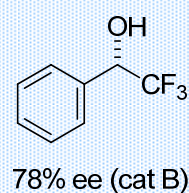
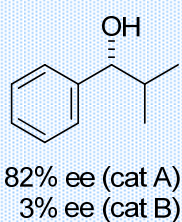
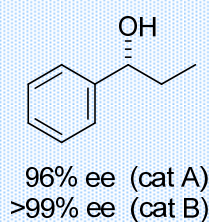
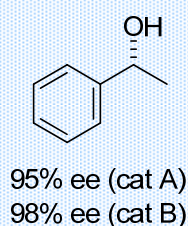
Example



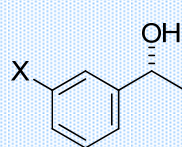
Chiral Ru cat: RuBr₂[(*S,S*)-xylskewphos][(*S,S*)-dpn] (cat A)

RuBr₂[(*S,S*)-xylskewphos][(*R*)-daipen] (cat B)

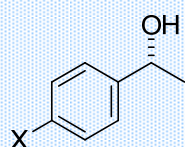
Conditions: ketone/Ru/*t*-C₄H₉OK = 10,000:1:40, [ketone]₀ = 2.0-2.5 mol/L



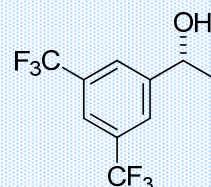
	A, % ee	B, % ee
X = CH ₃	95	77
F	92	78
Cl	97	94
Br	>99	>99
CH ₃ O	90	40



	A, % ee	B, % ee
X = CH ₃	96	96
F	95	93
Cl	94	97
Br	95	99



	A, % ee	B, % ee
X = CH ₃	88	92
F	96	97
Cl	94	97
Br	97	99

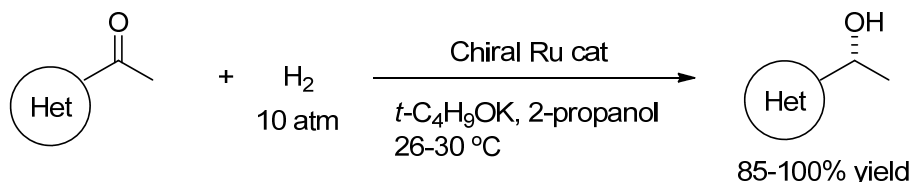


38% ee (cat A)
91% ee (cat B)

2 Hydrogenation of Heteroaromatic Ketones

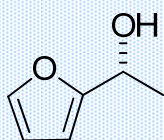
$\text{RuCl}_2(\text{binap})(\text{diamine})$ catalysts don't have high enantioselectivity for the asymmetric hydrogenation of heteroaromatic ketones. $\text{RuBr}_2(\text{xylskewphos})(\text{daipen})$ has higher enantioselectivity for these ketones. For example, 2-acetylfuran and 2-acetylthiophene are hydrogenated efficiently to give optically active 1-(2-furanyl)ethanol in 97% ee and 1-(2-thienyl)ethanol in 96% ee.

Example

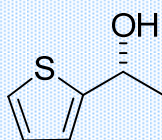


Chiral Ru cat: $\text{RuBr}_2[(S,S)\text{-xylskewphos}][(R)\text{-daipen}]$

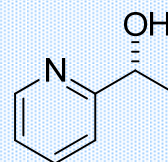
Conditions: ketone/ $\text{Ru}/t\text{-C}_4\text{H}_9\text{OK}$ = 2,000-10,000:1:40, $[\text{ketone}]_0$ = 2.5 mol/L



S/C = 5,000
 100% yield
 97% ee



S/C = 10,000
 85% yield
 96% ee

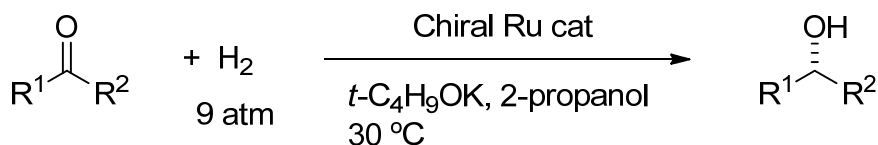


S/C = 2,000
 100% yield
 83% ee

3 Hydrogenation of Other Ketones

Carbonyl compounds with conjugated or unconjugated carbon-carbon multiple bonds can be converted effectively to the corresponding unsaturated alcohols. Cyclohexyl ketone, which is a difficult substrate to obtain with high enantioselectivity, can be hydrogenated in 94% ee with this catalyst. α -*N,N*-Dimethylaminoacetophenone is also hydrogenated enantioselectively with 97% ee.

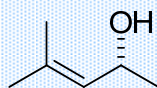
Example



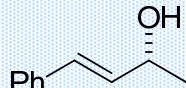
S/C = 2,000-10,000

Chiral Ru cat: $\text{RuBr}_2[(S,S)\text{-xylskewphos}][(R)\text{-daipen}]$

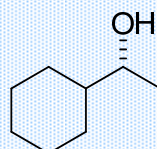
Conditions: ketone/ $\text{Ru}/t\text{-C}_4\text{H}_9\text{OK}$ = 2,000-10,000:1:10-40, $[\text{ketone}]_0$ = 2.0-2.5 mol/L



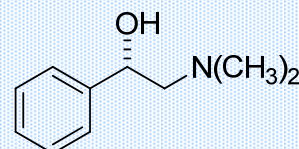
91% ee



94% ee



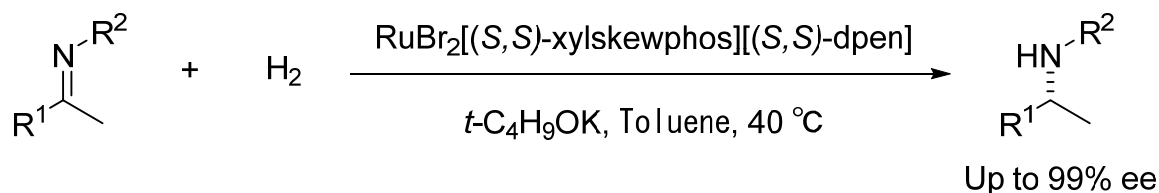
94% ee



97% ee

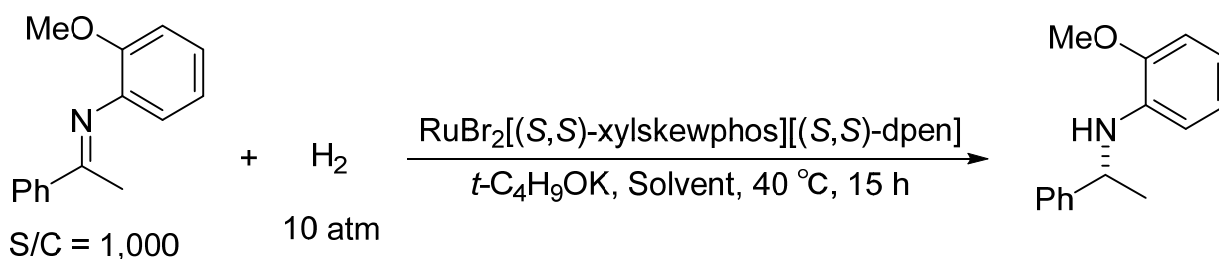
4 Hydrogenation of Imines

Catalytic asymmetric hydrogenation of imines is one of the most efficient and convenient method for the preparation of optically active amines. We found that $\text{RuBr}_2(\text{xylskewphos})(\text{dpen})$ is an excellent catalyst for the asymmetric hydrogenation of imines³ in the joint research project with Prof. Ohkuma in Hokkaido University. Various imines can be hydrogenated efficiently with this catalyst to afford the amine products in high ee's.



Example (reaction solvent)

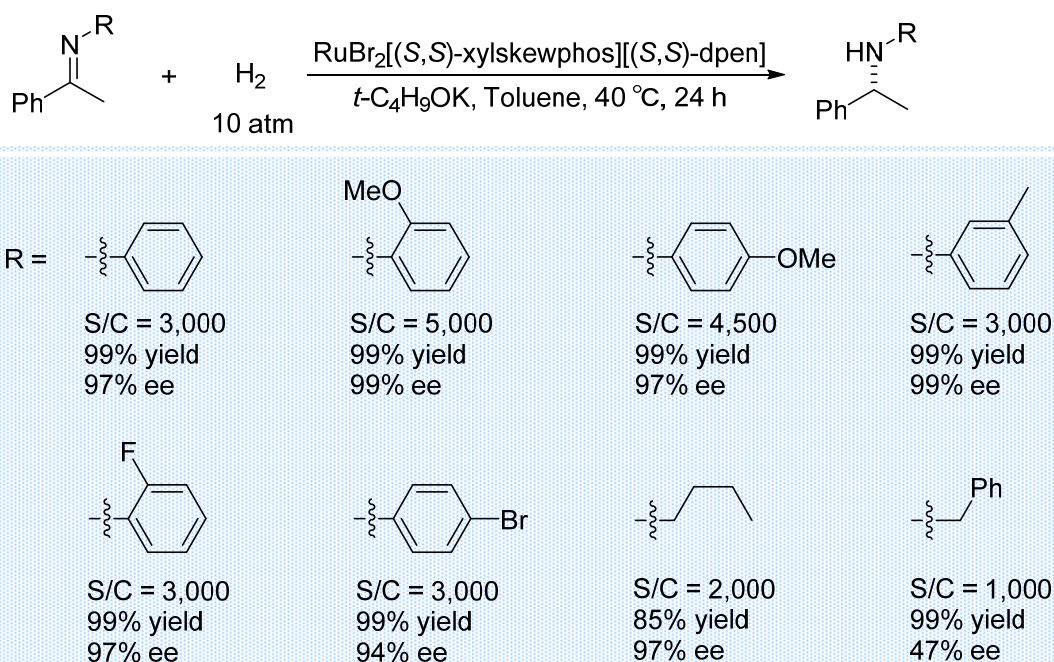
Reactions can be conducted in a variety of aprotic solvents such as toluene (hydrocarbon solvent) and THF (ether solvent). For example, hydrogenation of *N*-2-methoxyphenyl (OMP) acetophenone imine is completed within 15 h in various aprotic solvents in the presence of potassium *tert*-butoxide at a substrate-to-catalyst molar ratio (S/C) of 1,000 under 10 atm of hydrogen pressure, leading to the optically active phenethylamine in 99% ee.



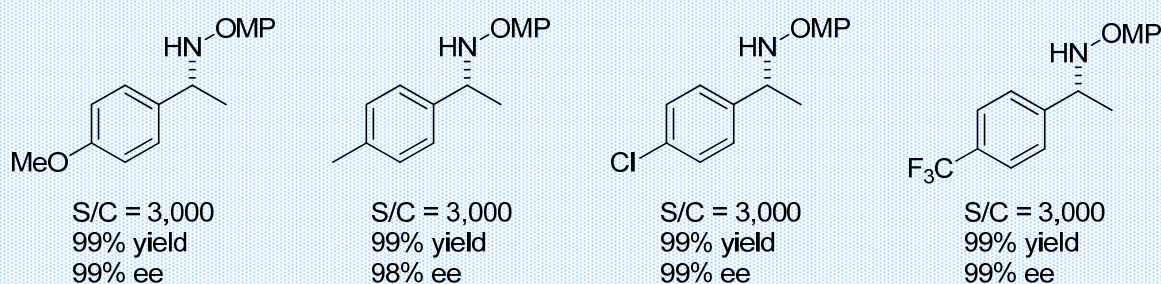
Solvent	yield (%)	ee (%)
toluene	>99	99
benzene	>99	99
THF	>99	99
MTBE	>99	99
2-propanol	<1	-
<i>t</i> -BuOH	<1	-

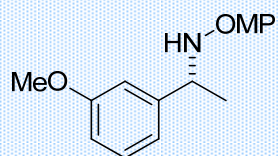
Example (scope of substrates)

The reaction of *N*-substituted imines, where the *N*-substituent is the phenyl group, electron-donating methoxyphenyl group, or electron-withdrawing halogenated phenyl group, also proceed efficiently. For example, *N*-OMP acetophenone imine is hydrogenated at a S/C of 5,000 under 10 atm of H₂ to afford the optically active amine in 99% ee quantitatively. The methoxyphenyl group is readily deprotected to give the optically active primary amines⁴. Although the reactivity of *N*-alkyl imines is relatively low, the enantioselectivity is maintained high. For example, *N*-*n*-Bu imine is reduced at a S/C of 2,000 to afford the amine product in 97% ee with 85% yield. The ee value of product from *N*-Benzyl imines is low.

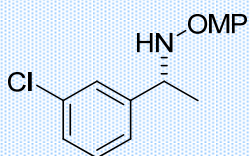


This catalyst system is applicable to a variety of *N*-aryl imines. *N*-OMP imines derived from acetophenones with an electron-donating or an electron-withdrawing group on the phenyl ring are hydrogenated at a S/C of 1,500–3,000 under 10 atm of H₂ to afford the optically active amines in up to 99% ee quantitatively. The reaction of imine with naphthalene ring, propiophenone imine and butyrophenone imine proceed efficiently. Heteroaromatic imines, such as pyridyl imines, ferrocenyl imine and α,β -unsaturated imine, are also hydrogenated in high ee's. The hydrogenation of diimine derived from 1,3-diacetylbenzene affords the diamine product with a high diastereo (*dl:meso* = 96:4) and high enantioselectivity (>99% ee) in 99% yield.

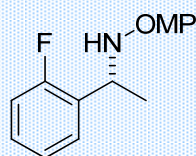




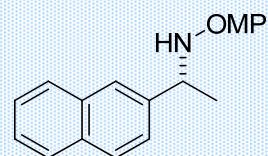
S/C = 3,000
99% yield
98% ee



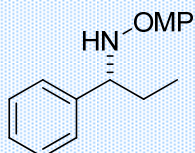
S/C = 3,000
99% yield
99% ee



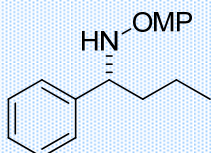
S/C = 1,500
99% yield
91% ee



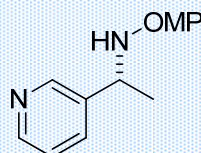
S/C = 3,000
99% yield
98% ee



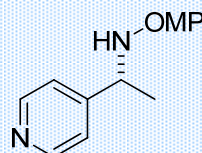
S/C = 3,000
99% yield
96% ee



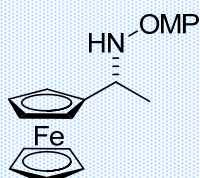
S/C = 3,000
98% yield
95% ee



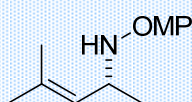
S/C = 500
99% yield
98% ee



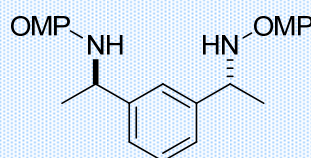
S/C = 500
98% yield
93% ee



S/C = 500
99% yield
96% ee

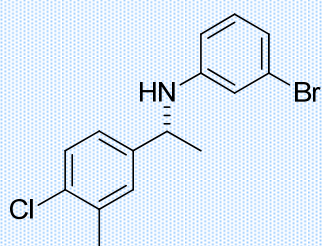


S/C = 3,000
99% yield
85% ee

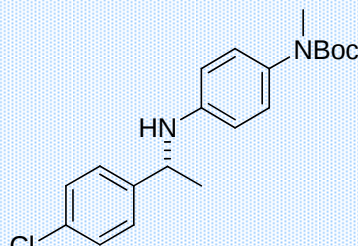


S/C = 1,000
99% yield
dl:meso = 96:4
>99% ee for *dl*

The *N*-substituted imine with 3-bromophenyl group or 4-Boc-aminophenyl group is converted to the corresponding chiral amine in 96% ee or 93% ee, respectively. These chiral amines are enable to the synthetic intermediates of biologically active compounds^{5,6}.



S/C = 2,500
99% yield
96% ee



S/C = 3,000
99% yield
93% ee

5 Procedure for Hydrogenation of Ketones

A representative procedure for the asymmetric hydrogenation of ketones using $\text{RuBr}_2(\text{xylskewphos})(\text{dpen})$ at a S/C of 2,000 is described below.

Reaction vessel (internal volume : 100 mL)^{*1}

← The ready-to-use Ruthenium(II) complex : ca. 10 mg (0.01 mmol)^{*2}
 (A solid substrate of ketone should be added with the Ruthenium(II) complex at this point.)
 Ketone substrate : 20 mmol)

Purging away the inside gas of the vessel and displacing with inert gas (1 time)

← Ketone substrate : 20 mmol^{*3}
 ← 0.01 mol/L Solution of potassium alkoxide in 2-propanol: 10 mL (0.1 mmol)^{*4*5*6}

Purging away the inside gas of the vessel and displacing with inert gas^{*7}

Replacing the inside gas of the vessel with hydrogen under stirring
 (H_2 is introduced into the vessel, before being reduced to 1 atm. This procedure is repeated 3~5 times)^{*8*9}

Pressurizing with H_2 (10 atm)

Hydrogenation (stirring, 30~60 °C)^{*10*11}

Isolating the alcohol product^{*12}



*1: Pressure resistant glass vessel with guard jacket can be used as well as stainless steel autoclave. In case of the poor reactivity, pretreatment of vessel may be effective.

*2: Ruthenium complexes can be handled in the air. It should be kept under inert gas in a long term preservation.

*3: Because this hydrogenation is sensitive even small amount of acid, acidic impurities should be carefully removed. By the following step, washing the substrate dissolved in appropriate solvent with an aqueous KOH solution then drying over Na_2SO_4 anhydride, the substrate can be purified with an appropriate purity.

*4: Please use commercial 0.01 mol/L potassium *tert*-butoxide/2-propanol solution by our company, or prepare it from commercially available potassium *tert*-butoxide and dry 2-propanol.

*5: The strong base more than 2 equivalent of Ruthenium complex is required for the hydrogenation. Some enones might be unstable against a base may polymerize in this conditions. In that case, the concentration of the strong base should be optimized, or using a weak base such as K_2CO_3 instead of the strong base is effective.

- *6: When a ketone substrate is hard to dissolve in 2-propanol, toluene or THF can be used as a cosolvent.
- *7: Repeated operation as follows (remove the internal gas by vacuum line and replace with inert gas) is enable.
- *8: For safety, replacing the internal gas of the vessel with H₂ should be operated in a draft-chamber (refer to the procedure in the literature: M. Kitamura, M. Tokunaga, T. Ohkuma and R. Noyori, *Org. Synth.* **1993**, 71, 1-13).
- *9: Please keep hydrogen pressure of vessel at more than 1 atm to prevent contamination by air.
- *10: High speed reaction is enable with a high speed stirring.
- *11: After the completion of hydrogenation, the hydrogen pressure will be decreased to the range of 4-5 atm.
- *12: The residual catalyst can be removed from the product by filtration through a silica-gel pad.

Hydrogenation with more than S/C = 3,000 can be operated by the same procedure. Quantitative conversion is possible with S/C>100,000 at optimized conditions. For a detail, please consult us.

Hydrogenation under atmospheric pressure of hydrogen is possible with these Noyori type asymmetric hydrogenation catalysts. In case of the atmospheric pressure reaction, reaction at a S/C of 500-1,000 is the first recommended trial, as the reaction rate is largely decreased than the reaction under the pressure of hydrogen.

6 Procedure for Hydrogenation of Imines

A representative procedure for the asymmetric hydrogenation of *N*-OMP acetophenone imine at a S/C of 3,000 is described below using RuBr₂(xylskewphos)(dppe) as a catalyst.

Reaction vessel (internal volume: 100 mL)^{*1}

← Imine substrate: 6.8 g (30 mmol)^{*2}
 (a liquid substrate of imine should not be added at this point)
 ← The ready-to-use Ruthenium complex: ca. 10 mg (0.01 mol)^{*3}
 ← Potassium *tert*-butoxide: 110 mg (1.0 mmol)

Purging away the internal gas of the vessel and displacing with inert gas

← Toluene: 7.5 mL^{*4}
 (A liquid substrate of imine should be added at this point. imine substrate: 30 mmol)

Purging away the internal gas of the vessel and displacing with inert gas^{*5}

Replacing the internal gas of the vessel with hydrogen under stirring

(H₂ is introduced into the vessel, before being reduced to 1 atm. This procedure is repeated 3~5 times)^{*6*7}

Pressurizing with H₂ (10~50 atm)^{*8}

Hydrogenation (stirring, 30~60 °C)^{*9}

Isolating the amine product

*1: Stainless steel autoclave (~50 atm) and pressure resistant glass vessel(~10 atm) can be used dependent on the pressure of hydrogen.

*2: The hydrogenation of imines doesn't proceed with alcohols. Coexistence of ketones in imine substrates causes producing alcohols from the hydrogenation of ketones. Please carefully remove ketones from imine substrates.

*3: Ruthenium complexes can be handled in the air. It should be kept under inert gas in a long term preservation.

*4: A variety of aprotic solvents such as benzene, THF and MTBE can be used as a solvent.

*5: Repeated operation as follows (remove the internal gas by vacuum line and replace with inert gas) is enable.

*6: For safety, replacing the internal gas of the vessel with H₂ should be operated in a draft-chamber (refer to the procedure in the literature: M. Kitamura, M. Tokunaga, T. Ohkuma and R. Noyori, *Org. Synth.* **1993**, 71, 1-13).

*7: Please keep hydrogen pressure of vessel at more than 1 atm to prevent contamination by air.

*8: Higher pressure of hydrogen leads to increase reaction rate as keeping enantioselectivity high.

*9: Higher temperature leads to increase reaction rate, but enantioselectivity is slightly decreased.

[References]

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[Conformation of Complexes]

RuBr₂(xylskewphos)(diamine) complexes present as two diastereomeric mixtures by ³¹P-NMR analysis.

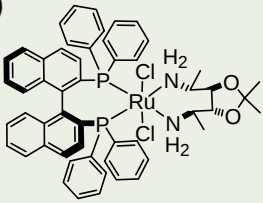
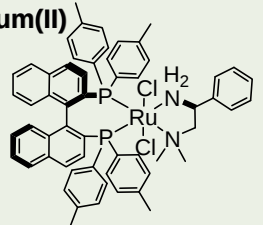
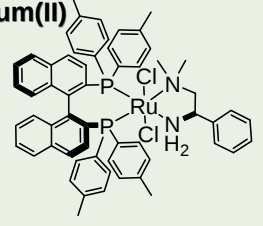
●RuBr₂[(*S,S*)-xylskewphos][(*S,S*)-dpen] and RuBr₂[(*R,R*)-xylskewphos][(*R,R*)-dpen]

³¹P-NMR(CDCl₃, -25 °C) δ ppm, major: 65.0 (d, *J* = 42.6 Hz), 41.6 (d, *J* = 43.6 Hz),
minor: 59.8 (d, *J* = 43.6 Hz), 49.0 (d, *J* = 48.0 Hz). major/minor = 10:9

●RuBr₂[(*S,S*)-xylskewphos][(*R*)-daipen] and RuBr₂[(*R,R*)-xylskewphos][(*S*)-daipen]

³¹P-NMR(CDCl₃, -25 °C) δ ppm, major: 60.0 (d, *J* = 48.0 Hz), 46.8 (d, *J* = 43.6 Hz),
minor: 60.6 (d, *J* = 48.0 Hz), 48.2 (d, *J* = 48.0 Hz). major/minor = 10:3

Related Products, Asymmetric Hydrogenation Catalysts

Product	Product number	Package
Dichloro[(R)-2,2'-bis(diphenylphosphino)-1,1'-binaphthyl]-[(R,R)-1,2-diphenylethanediamine]ruthenium(II) RuCl ₂ [(R)-binap][(R,R)-dpen]	11404-65	1g
	11404-95	200mg
Dichloro[(R)-2,2'-bis(diphenylphosphino)-1,1'-binaphthyl]-[(S,S)-1,2-diphenylethanediamine]ruthenium(II) RuCl ₂ [(R)-binap][(S,S)-dpen]	11405-65	1g
	11405-95	200mg
Dichloro[(S)-2,2'-bis(diphenylphosphino)-1,1'-binaphthyl]-[(R,R)-1,2-diphenylethanediamine]ruthenium(II) RuCl ₂ [(S)-binap][(R,R)-dpen]	11402-65	1g
	11402-95	200mg
Dichloro[(S)-2,2'-bis(diphenylphosphino)-1,1'-binaphthyl]-[(S,S)-1,2-diphenylethanediamine]ruthenium(II) RuCl ₂ [(S)-binap][(S,S)-dpen]	11403-65	1g
	11403-95	200mg
Dichloro[(R)-2,2'-bis(diphenylphosphino)-1,1'-binaphthyl]-[(R)-1,1-bis(p-methoxyphenyl)-2-isopropyl-1,2-ethanediamine]ruthenium(II) RuCl ₂ [(R)-binap][(R)-daipen]	11408-65	1g
	11408-95	200mg
Dichloro[(S)-2,2'-bis(diphenylphosphino)-1,1'-binaphthyl]-[(S)-1,1-bis(p-methoxyphenyl)-2-isopropyl-1,2-ethanediamine]ruthenium(II) RuCl ₂ [(S)-binap][(S)-daipen]	11409-65	1g
	11409-95	200mg
Dichloro[(S)-binap][(R)-iphan]Ruthenium(II) RuCl ₂ [(S)-binap][(R)-iphan] 	10532-68	100mg
	10532-95	500mg
※NIPPON SODA CO., INC.		
Dichloro[(R)-tolbinap][(S)-dmapen]Ruthenium(II) RuCl ₂ [(R)-tolbinap][(S)-dmapen] 	10533-68	100mg
	10533-95	500mg
※NIPPON SODA CO., INC.		
Dichloro[(S)-tolbinap][(R)-dmapen]Ruthenium(II) RuCl ₂ [(S)-tolbinap][(R)-dmapen] 	10534-68	100mg
	10534-95	500mg
※NIPPON SODA CO., INC.		

Related Products, Chiral Ligands

Product Name	Product number	Package
(R)-(+)-2,2'-Bis(diphenylphosphino)-1,1'-binaphthyl (R)-(+)-BINAP	04969-35	25g
	04969-55	5g
	04969-65	1g
(S)-(-)-2,2'-Bis(diphenylphosphino)-1,1'-binaphthyl (S)-(-)-BINAP	04970-35	25g
	04970-55	5g
	04970-65	1g
rac-2,2'-Bis(diphenylphosphino)-1,1'-binaphthyl rac-BINAP	05069-35	25g
	05069-55	5g
	05069-65	1g
(R)-(+)-2,2'-Bis(di-<i>p</i>-tolylphosphino)-1,1'-binaphthyl (R)-(+)-Tol-BINAP	41105-55	5g
	41105-65	1g
(S)-(-)-2,2'-Bis(di-<i>p</i>-tolylphosphino)-1,1'-binaphthyl (S)-(-)-Tol-BINAP	41106-55	5g
	41106-65	1g
(R)-1,1-Bis(<i>p</i>-methoxyphenyl)-2-isopropyl-1,2-ethanediamine (R)-DAIPEN	11407-65	1g
	11407-95	200mg
(S)-1,1-Bis(<i>p</i>-methoxyphenyl)-2-isopropyl-1,2-ethanediamine (S)-DAIPEN	11406-65	1g
	11406-95	200mg
(1<i>R</i>,2<i>R</i>)-(+)-1,2-Diphenyl-1,2-ethanediamine (<i>R,R</i>)-DPEN	11444-35	25g
	11444-55	5g
	11444-65	1g
(1<i>S</i>,2<i>S</i>)-(-)-1,2-Diphenyl-1,2-ethanediamine (<i>S,S</i>)-DPEN	11445-35	25g
	11445-55	5g
	11445-65	1g


KANTO CHEMICAL CO., INC.

Performance Chemicals Dept.

 East Muromachi Mitsui BLDG, 2-1, Nihonbashi Muromachi 2-chome,
Chuo-ku, Tokyo, 103-0022 JAPAN

Telephone +813-6214-1092

Telefax +813-3241-1053

<http://www.kanto.co.jp> E-mail: kanto-61@gms.kanto.co.jp