Available online at www.sciencedirect.com
SCIENCE DIRECT
Journal of Organometallic Chemistry 691 (2006) 2207–2212
Journal of Organometallic Chemistry
www.elsevier.com/locate/jorganchem

Resolution of BIPHEP on Rh with a chiral diene auxiliary

J.W. Faller *, Jeremy C. Wilt

Department of Chemistry, Yale University, P.O. Box 208107, New Haven, CT 06520-8107, USA

Received 13 July 2005; received in revised form 28 September 2005; accepted 28 September 2005
Available online 15 November 2005

Abstract

Resolution of the atropisomeric chiral BIPHEP ligand on Rh has been achieved with the aid of 2-(4-*tert*-butyl-phenyl)-8-methoxy-1,8-dimethyl-bicyclo[2.2.2]octa-2,5-diene, a chiral chelating diene ligand. The diene complex **3** containing an (*S*)-BIPHEP ligand was found to be configurationally stable in $CDCl_3$ solution at RT. Conversion of the diene complex **3** to a dicarbonyl Rh complex **4** that had a barrier of 25.0 kcal/mol for atropisomerization of the BIPHEP ligand. Preliminary studies of the use of the resolved complex **3** for catalysis are presented.

Keywords: Asymmetric catalysis; Resolution; Chiral induction

1. Introduction

Axially chiral C_2 symmetric ligands such as Bis(diphenylphosphino)-1,1′-binaphthyl (BINAP) and 2,2′-Dihydroxy-1,1′-binaphthyl (BINOL) have received much attention owing to their ability to induce high levels of enantioselectivity for a variety of catalytic reactions. These ligands, and others similar to them, maintain their chirality due to the large barrier to atropisomerization involving hindered rotation about the bond connecting the naphthalene moieties. The syntheses of enantiomerically pure binaphthyl derivatives usually entail a classical resolution of the two enantiomers [1–6]. Although methods exist for the asymmetric coupling of biaryl fragments, such methodology is far from being general in scope, or (in most cases) insufficiently enantioselective to produce the desired non-racemic biaryl in adequate enantiomeric purity for use as a chiral ligand [7–12].

Alternatively, several strategies exist for the use of racemic ligands in asymmetric catalysis [13,14]. One such approach is termed "chiral poisoning" [14–20]. This method involves the preparation of a chiral, racemic catalyst, followed by the addition of a substoichiometric amount of a "chiral poison". The chiral poison effectively serves to deactivate one enantiomer of the racemic catalyst, usually by strong, diastereoselective binding to a vacant site. This allows the non-poisoned enantiomer of the catalyst to carry out the asymmetric reaction. Excellent enantioselectivities have been obtained via this strategy in a number of cases [14,21,22].

Another strategy involves the selective activation of one enantiomer of a chiral, racemic metal complex, known as "asymmetric activation" [14,23–25]. This approach entails the addition of another enantiomerically pure chiral ligand to the racemic metal complex to create two diastereomeric species which catalyze the reaction in question at different rates. If this difference in rates is great, then the reaction will proceed selectively through only one of the diastereomeric catalysts, giving potentially high enantioselectivities.

An interesting variation of these strategies makes use of racemic atropisomeric ligands such as 2,2′-bis(diphenylphosphino)-1,1′-biphenyl (BIPHEP) [26]. The BIPHEP ligand exhibits hindered rotation about its biaryl axis, but the barrier to atropisomerization has been determined to be only 22 kcal/mol (Fig. 1) [27]. As such, the BIPHEP ligand racemizes slowly at room temperature, making it unresolvable by traditional methods. However, upon

* Corresponding author. Tel.: +1 203 432 3954; fax: +1 203 432 6144.
E-mail address: jack.faller@yale.edu (J.W. Faller).

0022-328X/$ - see front matter © 2005 Elsevier B.V. All rights reserved.
doi:10.1016/j.jorganchem.2005.09.050

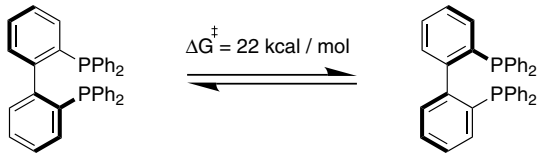
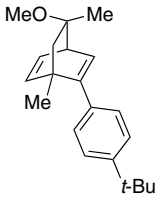


Fig. 1. Racemization of BIPHEP.



1

Fig. 2. A chiral diene developed by Carreira.

complexation to a transition metal, this barrier is raised, allowing the possibility of resolution. By using a second enantiopure chiral ligand as a chirality-controlling element, the BIPHEP ligand may be dynamically resolved on a particular transition metal. This chirality control stems from steric interactions between the racemic BIPHEP ligand and the added enantiopure ligand.

Several examples of the dynamic resolution of the BIPHEP ligand have been reported in the last decade. Mikami has performed dynamic resolutions of BIPHEP on Ru [28,29], Pd [30,31], and most recently Rh [32] with the use of enantiopure chiral diamine ligands. As the barriers to atropisomerization are generally higher when BIPHEP is complexed to a transition metal, these complexes can sometimes be used for asymmetric catalysis without significant erosion of the enantiopurity of the catalyst. For example, the epimerization of 3,3′-dimethyl-BIPHEP on Rh with (*R*)-2,2′-diamino-1,1′-binaphthyl (BINAM) as the chirality inducer took place at 80 °C within 5 h to give the diastereomerically pure (*R*,*R*) complex. The amine could then be removed by protonolysis at 5 °C, and the Rh complex was used as a catalyst for the asymmetric cycloisomerization of some 1,6-enynes with excellent enantioselectivity [32].

Gagné [33] has also studied the resolution of BIPHEP–Pt complexes with BINOL as the chiral controller. After performing a similar dynamic resolution as the above method, protonolysis of the BINOL ligand gave the (BIPHEP)Pt^{II} dication, which was used as a highly enantioselective Lewis acid catalyst in the asymmetric Diels–Alder reaction [34].

Herein, we present the resolution of the BIPHEP ligand on Rh with the aid of a chelating chiral diene auxiliary ligand [35]. The first example of a chelating chiral diene ligand used in asymmetric catalysis was Hayashi's C_2-symmetric diene based on the norbornadiene skeleton [36]. This ligand, though rather difficult to synthesize, proved to be very effective in the Rh-catalyzed 1,4-addition of arylboronic acids to α,β-unsaturated ketones. Hayashi has also developed C_2-symmetric chiral dienes based on the [2.2.2]-bicyclooctadiene skeleton, which are useful for the Rh-catalyzed asymmetric arylation of *N*-tosylarylimines [37] and the cyclization of alkynals [38], as well as the 1,4-addition reaction mentioned above [39].

Carreira has also prepared a chiral diene family based on the [2.2.2]-bicyclooctadiene skeleton for use in the Ir-catalyzed kinetic resolution of aryl-substituted allylic carbonates during the course of an asymmetric allylic etherification reaction (Fig. 2) [40]. Two important features of these ligands are that their chirality is derived from commercially available, inexpensive (−)-carvone, and that the syntheses of these ligands are facile. Carreira has also recently extended the utility of derivatives of these dienes to the Rh-catalyzed 1,4-addition of arylboronic acids to a wide variety α,β-unsaturated ketones with excellent ee [41].

2. Results and discussion

The diene, 2-(4-*tert*-butyl-phenyl)-8-methoxy-1,8-dimethyl-bicyclo[2.2.2]octa-2,5-diene **1** was synthesized in good overall yield according to the method of Careirra [40]. The strategy involved in our approach was to take advantage of the protruding *t*-BuC_6H_4 group on this particular chiral diene to exert control over the axial chirality of the BIPHEP ligand in a square-planar arrangement. As the *t*-BuC_6H_4 group is relatively large, and all other diene positions of **1** are unsubstituted, it would appear that the diphenylphosphino group of BIPHEP underneath this large group may preferentially orient itself away from it due to unfavorable steric interactions.

Rhodium dimer **2** was obtained in 97% yield from $[(cyclooctene)_2RhCl]_2$ (Fig. 3). Preparation of BIPHEP complex **3** was equally straightforward; abstraction of chloride ion from **2** in THF followed by slow addition of a BIPHEP solution produced the desired product in 95% yield. Initially, product **3** exists as a 3:1 diastereomeric ratio in $CDCl_3$ solution, where BIPHEP has adopted either an (*R*) or (*S*) configuration. Allowing this solution to stand for a period of 1 week at RT produced no change in the diastereomeric ratio.

All attempts at heating this mixture ($CHCl_3$, THF, toluene) resulted in extensive decomposition of the Rh complex; therefore, the two diastereomers of **3** could not be dynamically resolved utilizing this strategy. Fractional crystallization by slow evaporation of a rigorously degassed EtOH solution provided the major diastereomer, (*S*)-**3**, in 50% yield. An X-ray crystallographic study of this complex proved the structure of (*S*)-**3** unambiguously (Fig. 4). The remaining supernatant consisted of an approximately 1:1 ratio of the two diastereomers of **3**, along with a small amount of decomposition product. Unfortunately, further purification by crystallization of this mixture was not successful in our hands. Complex (*S*)-**3** was configurationally stable in $CDCl_3$ under N_2 for a period of 1 week, showing no trace of the other diastereomer. Thus, the chelation

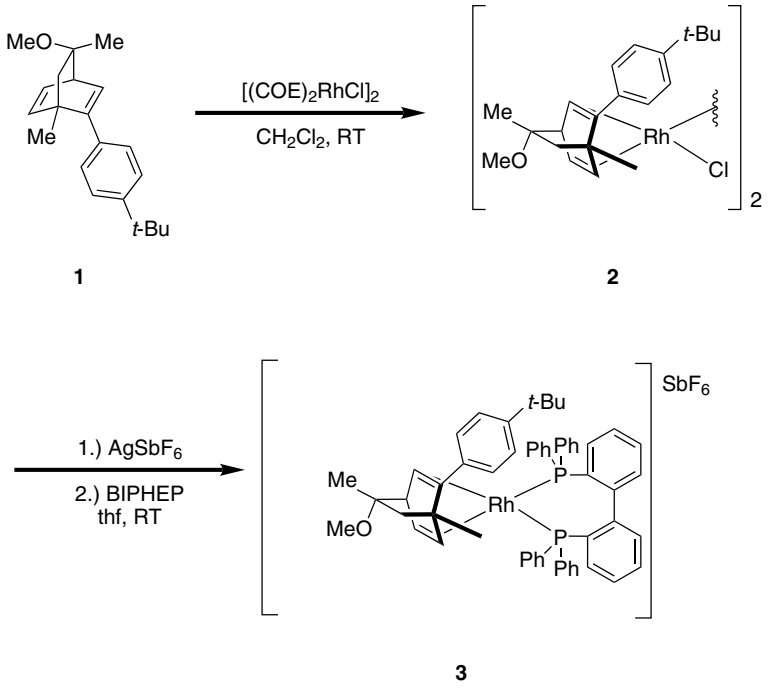


Fig. 3. Synthesis of **2** and **3**.

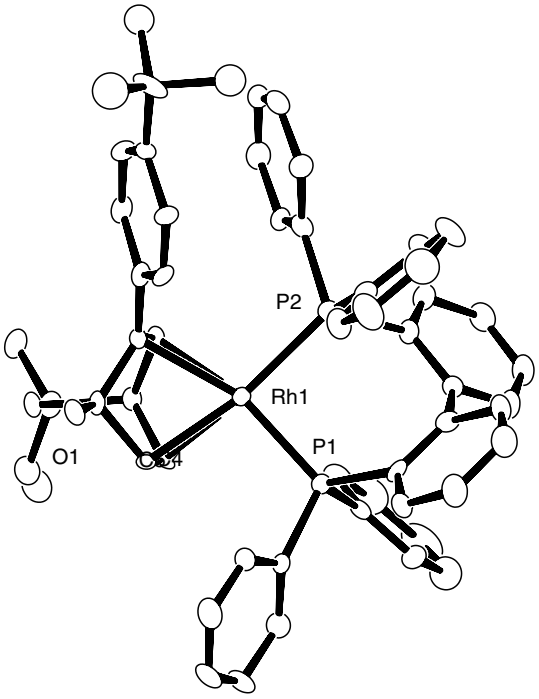


Fig. 4. An ORTEP diagram of (*S*)-**3** showing 30% probability ellipsoids.

and the diene ligand appear to be preventing the atropisomerization of the BIPHEP ligand.

To examine the atropisomerization of BIPHEP on Rh, it was necessary to remove the chirality inducer **1**. When CO was bubbled through a solution of (*S*)-**3**, the diene ligand was quickly replaced by carbonyls, giving complex **4** as a yellow solid (Fig. 5). Quick evaporation of the solvent followed by trituration of **4** with Et_2O provided the pure complex in optically active form. Though **4** may have racemized to some extent during its preparation (< 5 min), it retained sufficient optical activity to permit a study of its racemization using polarimetry. The rate constant for loss of optical rotation of **4** was determined to be 5.3×10^{-6} s^{-1}; therefore, the rate constant for atropisomerization of the BIPHEP ligand on Rh is $0.5k_{rac}$, or 2.6×10^{-6} s^{-1}. This rate constant for atropisomerization of the BIPHEP ligand in **4** corresponds to a $\Delta G^{\ddagger}$ value of 25.0 kcal/mol, which is ~ 3 kcal/mol higher than that of the free ligand.

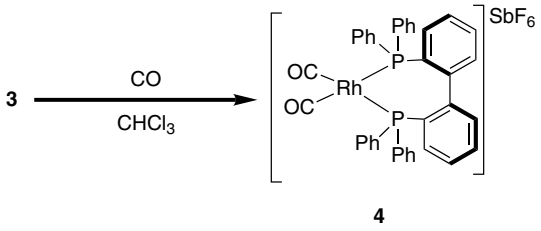


Fig. 5. Removal of diene **1**.

The relatively high barrier to atropisomerization of BIPHEP on Rh, in addition to the recent literature example [32], suggested that complex (*S*)-**3** may be used in asymmetric catalysis without substantial enantiomeric degradation of the catalyst. Initially, hydrogenation of prochiral olefins such as dimethyl itaconate was attempted with (*S*)-**3**. Interestingly, complex (*S*)-**3** showed no activity towards H_2 at 1 atm. Presumably, this lack of activity is derived from the difficulty of the hydrogenation of a trisubstituted olefin. An attempted hydrosilylation of dimethyl itaconate with diphenylsilane unfortunately led to the hydrogenated product in racemic form (Fig. 6). Although this type of reaction

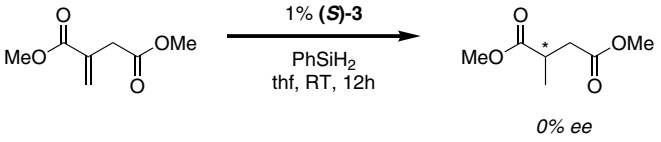


Fig. 6. Hydrosilylation of dimethyl itaconate with (*S*)-**3**.

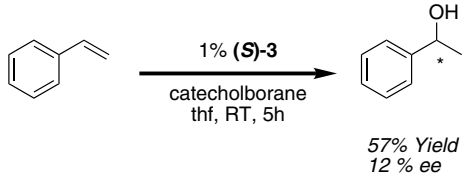


Fig. 7. Hydroboration of styrene with (*S*)-**3**.

with diphenylsilane is unusual, it has been previously documented [42–44]. This complex was also tested in the hydroboration of styrene with catecholborane. Although complex **3** was catalytically active, the product alcohol was produced in only 12% ee at ambient temperature (Fig. 7).

Upon lowering the temperature to −78 °C, complex (*S*)-**3** showed no catalytic activity. When adding catecholborane to complex (*S*)-**3** at RT, followed by cooling to −78 °C and then adding styrene, the reaction proceeded only to <5% conversion.

In conclusion, we have developed a novel method for the resolution of the atropisomerically chiral BIPHEP ligand on Rh with the aid of Carreira's chiral diene ligand **1**. Complex (*S*)-**3** is configurationally stable for over one week in $CDCl_3$ solution at RT. The barrier to atropisomerization of BIPHEP on Rh was found to be 25.0 kcal/mol after removal of **1** with CO. Thus, the barrier is dependent upon the other ligands, as well as chelation. Other nonracemic Rh(BIPHEP) catalysts should be available by displacement of the diene. Though a successful catalytic asymmetric reaction was not realized with this particular complex in our hands, the modularity of these chiral diene ligands should allow for sufficient tuning to make this a possibility in future studies.

Future work will focus on expanding this strategy to the resolution of other atropisomerically flexible chiral ligands analogous to BIPHEP.

3. Experimental

3.1. General

All synthetic manipulations were carried out under inert gas using standard Schlenk techniques. Dichloromethane was distilled from CaH_2 prior to use. THF was distilled over sodium metal before use. Ethanol was dried over 4 Å molecular sieves and subjected to three freeze–pump–thaw cycles prior to use. BIPHEP was purchased from Strem Chemicals and used without further purification. Chiral diene **1** was prepared as previously described [40]. $[(cyclooctene)_2RhCl]_2$ was prepared via a published procedure [45]. Optical rotations were measured on a Perkin–Elmer model 341 polarimeter at 589 nm and 25 °C, using a 1 dm path length. Enantiomeric excesses of *sec*-phenethyl alcohol were measured by the ^1H NMR integration of appropriate resonances of the Mosher ester.

3.2. Preparation of 2

To a stirred solution of $[(cyclooctene)_2RhCl]_2$ (204 mg, 0.28 mmol) in CH_2Cl_2 (8 mL) was added diene **1** (196 mg, 0.66 mmol). The resulting orange-red solution was stirred at RT for 16 h. The mixture was filtered through a pad of Celite and the solvent was removed under reduced pressure to leave a crude solid. This was subjected to column chromatography over silica gel, eluting first with pentane to remove displaced cyclooctene, then with Et_2O to elute an orange-yellow band. Removal of the solvent gave pure **2** as an orange-yellow powder (240 mg, 97%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.8 (4H, br s); 7.35 (4H, br s); 4.33 (2H, br t, $J = 6.0$ Hz); 3.87 (2H, br t, $J = 6.0$ Hz); 3.62 (2H, br s); 3.48 (2H, br s); 3.20 (6H, s, –OMe); 1.79 (6H, s, Me); 1.34 (18 H, s, *t*-Bu); 1.21 (6H, s, Me); 1.12 (2H, d, $J = 14.0$ Hz); 0.92 (2H, d, $J = 14.0$ Hz). ^{13}C NMR (125 MHz, $CDCl_3$): δ 149.9, 134.3, 130.2, 124.0, 80.3, 71.4, 56.5, 53.1, 53.0, 49.8, 49.5, 49.0, 48.7, 34.5, 31.3, 21.8, 21.7. Anal. Calc. for $C_{42}H_{56}Cl_2O_2Rh_2$: C, 58.01; H, 6.49. Found: C, 57.63; H, 6.70.

3.3. Preparation of 3

To compound **2** (25 mg, 28.8 μmol) and $AgSbF_6$ (18 mg, 52.4 μmol) in a Schlenk flask equipped with an addition funnel was added 5 mL THF. The resulting cloudy, light orange solution was stirred at RT for 0.5 h. A solution of BIPHEP (27.4 mg, 52.4 μmol) in 10 mL THF was transferred to the addition funnel via cannula, and was added dropwise to the reaction mixture. The solution quickly turned deep orange in color. After addition was complete, the reaction mixture was stirred at RT for an additional 0.5 h, then filtered through a pad of Celite and concentrated. Trituration of the resulting orange solid with Et_2O followed by filtration gave crude **3** as a 3:1 diastereomeric mixture (56 mg, 93%). The major diastereomer (*S*)-**3** could be isolated as red crystals by slow evaporation of a solution of crude **3** in carefully degassed EtOH under argon in 50% yield.

Major diastereomer (S)-**3**. ^1H NMR (400 MHz, $CDCl_3$): δ 7.82–6.87 (28H, BIPHEP aryl region); 6.86 (2H, $J = 8.2$ Hz); 6.74 (2H, $J = 8.2$ Hz); 5.04 (1H, t, $J = 5.9$ Hz); 4.85 (1H, br s); 4.75 (1H, br s); 3.48 (1H, br s); 3.16 (3H, OMe, s); 1.62 (3H, Me, s); 1.14 (3H, Me, s); 1.12 (9H, *t*-Bu, s); 0.97 (1H, d, $J = 13.6$ Hz); 0.89 (1H, d, $J = 13.6$ Hz); ^{31}P NMR (162 MHz, $CDCl_3$); δ 30.6 (dd, $J_{Rh-P} = 162.7$ Hz, $J_{PP} = 43.7$ Hz); 25.6 (dd, $J_{Rh-P} = 162.7$ Hz, $J_{PP} = 43.7$ Hz). ^{13}C NMR (125 MHz, $CDCl_3$):

δ 151.6–124.9 (aryl region, 42C); 108.2, 93.0, 90.5, 87.1, 83.7, 52.9, 51.5, 51.3, 50.6, 34.7, 31.4, 22.3, 22.2. $[\alpha]^{25}$ (589 nm) = −34.0 ($c = 0.0025$, $CHCl_3$). Anal. Calc. for $C_{57}H_{56}F_6OP_2RhSb$: C, 59.14; H, 4.88. Found: C, 59.21; H, 4.96.

3.4. Preparation of **4**

Carbon monoxide was vigorously passed through an orange solution of **3** (15 mg, 13 μmol) in chloroform (1 mL). Within 5 min, the solution became bright yellow, and the solvent was removed in vacuo. The resulting light yellow powder was triturated with Et_2O to remove displaced **1**, filtered, and dried under vacuum to leave the desired compound (11.2 mg, 95%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.72–7.35 (20H, BIPHEP aryl region); 7.14–7.00 (6H, BIPHEP aryl region); 6.58 (2H, BIPHEP aryl region). ^{31}P NMR (162 MHz, $CDCl_3$): δ 22.1 (d, $J_{Rh-P} = 123$ Hz). ^{13}C NMR (125 MHz, $CDCl_3$, carbonyl only): δ 143.3 (dd, $J = 7.2$, 13.7 Hz). IR (thin film solid): 2098 (ν_{CO}), 2054(ν_{CO}). $[\alpha]^{25}$ (589 nm) = +65.6 ($c = 0.0032$, $CHCl_3$). Anal. Calc. for $C_{38}H_{28}F_6O_2P_2RhSb$: C, 49.76; H, 3.08. Found: C, 49.44; H, 3.52.

3.5. Racemization rate of **4**

The optical rotation was measured over 40,000 s with a Perkin–Elmer model 341 polarimeter at 589 nm and 25 °C, using a 1 dm path length. The first order decay was fit using Kaleidograph 3.1 (Synergy Software) using all observed points to obtain a value for k based on the initial rate.The ln [α] decreases ~5% over this time period, so that there is not a significant variation in the accuracy of the measurements over the duration of the experiment.

3.6. Structure determination and refinement

Orange crystals were obtained by slow evaporation of an EtOH solution of complex **3**. Data were collected on a $0.1 \times 0.1 \times 0.2$ crystal at −100 °C on a Nonius KappaCCD (Mo Kα radiation) diffractometer and were not specifically corrected for absorption other than the inherent corrections provided by SCALEPACK [46]. The structure was solved by direct methods (SIR 92) [47] and refined on F for all reflections [48]. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms were included at calculated positions. An asymmetric unit containing two independent $[C_{57}H_{56}OP_2Rh][SbF_6]$ moieties was located in a primitive monoclinic cell with $Z = 4$ and lattice parameters of: $a = 12.322(2)$ Å; $b = 21.0771(4)$ Å; $c = 19.9387(3)$ Å; $\beta = 99.004(2)°$; $V = 5114.6(5)$ Å^3. The space group was $P2_1$ (#4). The total data collected was 21717 reflections of which 13036 were unique ($R_{int} = 0.055$). A rotational disorder in the orientation of the t-butyl groups was modeled as a 67:33 occupancy. The refinement converged with residuals: $R = 0.0529$ and $R_w = 0.0456$. The absolute configuration was determined by reference to the diene and by inverting the coordinates which yielded: $R = 0.0569$ and $R_w = 0.0511$. Detailed information is provided in the supporting information.

Appendix A. Supplementary data

Crystallographic data for the structural analysis has been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 278397 for compound **3**. Copies of this information may be obtained free of charge from: The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ UK; fax (int code): +44 (1223) 336 033; or e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk. The metrical parameters are also available in the supporting Information. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2005.09.050.

References

[1] H. Takaya, K. Mashima, K. Koyano, M. Yagi, H. Kumobayashi, T. Taketomi, S. Akutagawa, J. Org. Chem. 51 (1986) 629.
[2] A. Miyashita, H. Takaya, T. Souchi, R. Noyori, Tetrahedron 40 (1984) 1245.
[3] E.P. Kyba, G.W. Gokel, F. Dejong, K. Koga, L.R. Sousa, M.G. Siegel, L. Kaplan, G.D.Y. Sogah, D.J. Cram, J. Org. Chem. 42 (1977) 4173.
[4] J. Jacques, C. Fouquey, Tetrahedron Lett. (1971) 4617.
[5] M. Kawashima, A. Hirayama, Chem. Lett. (1990) 2299.
[6] F. Toda, K. Tanaka, J. Org. Chem. 53 (1988) 3607.
[7] K. Mikami, T. Miyamoto, M. Hatano, Chem. Commun. (2004) 2082.
[8] Y.H. Cho, A. Kina, T. Shimada, T. Hayashi, J. Org. Chem. 69 (2004) 3811.
[9] A.N. Cammidge, K.V.L. Crepy, Tetrahedron 60 (2004) 4377.
[10] T. Hayashi, J. Organometal. Chem. 653 (2002) 41.
[11] J.J. Yin, S.L. Buchwald, J. Am. Chem. Soc. 122 (2000) 12051.
[12] M. Nakajima, I. Miyoshi, K. Kanayama, S. Hashimoto, M. Noji, K. Koga, J. Org. Chem. 64 (1999) 2264.
[13] P.J. Walsh, A.E. Lurain, J. Balsells, Chem. Rev. 103 (2003) 3297.
[14] J.W. Faller, A.R. Lavoie, J. Parr, Chem. Rev. 103 (2003) 3345.
[15] J.W. Faller, J. Parr, J. Am. Chem. Soc. 115 (1993) 804.
[16] J.W. Faller, M. Tokunaga, Tetrahedron Lett. 34 (1993) 7359.
[17] J.W. Faller, M.R. Mazzieri, J.T. Nguyen, J. Parr, M. Tokunaga, Pure Appl. Chem. 66 (1994) 1463.
[18] R. Sablong, J.A. Osborn, J.W. Faller, J. Organometal. Chem. 527 (1997) 65.
[19] J.H. Koh, A.O. Larsen, P.S. White, M.R. Gagne, Organometallics 21 (2002) 7.
[20] J.W. Faller, X. Liu, Tetrahedron Lett. 37 (1996) 3449.
[21] J.W. Faller, D.W.I. Sams, X. Liu, J. Am. Chem. Soc. 118 (1996) 1217.
[22] J.W. Faller, A.R. Lavoie, B.J. Grimmond, Organometallics 21 (2002) 1662.
[23] K. Mikami, M. Yamanaka, Chem. Rev. 103 (2003) 3369.
[24] K. Mikami, M. Terada, T. Korenaga, Y. Matsumoto, M. Ueki, R. Angelaud, Angew. Chem., Int. Ed. 39 (2000) 3532.
[25] T. Ohkuma, H. Doucet, T. Pham, K. Mikami, T. Korenaga, M. Terada, R. Noyori, J. Am. Chem. Soc. 120 (1998) 1086.
[26] K. Mikami, K. Aikawa, Y. Yusa, J.J. Jodry, M. Yamanaka, Synlett (2002) 1561.
[27] O. Desponds, M. Schlosser, Tetrahedron Lett. 37 (1996) 47.
[28] K. Mikami, T. Korenaga, M. Terada, T. Ohkuma, T. Pham, R. Noyori, Angew. Chem., Int. Ed. 38 (1999) 495.

[29] T. Korenaga, K. Aikawa, M. Terada, S. Kawauchi, K. Mikami, Adv. Synth. Catal. 343 (2001) 284.
[30] K. Mikami, K. Aikawa, Y. Yusa, Org. Lett. 4 (2002) 95.
[31] K. Mikami, K. Aikawa, Y. Yusa, M. Hatano, Org. Lett. 4 (2002) 91.
[32] K. Mikami, S. Kataoka, Y. Yusa, K. Aikawa, Org. Lett. 6 (2004) 3699.
[33] M.D. Tudor, J.J. Becker, P.S. White, M.R. Gagne, Organometallics 19 (2000) 4376.
[34] J.J. Becker, P.S. White, M.R. Gagne, J. Am. Chem. Soc. 123 (2001) 9478.
[35] F. Glorius, Angew. Chem., Int. Ed. 43 (2004) 3364.
[36] T. Hayashi, K. Ueyama, N. Tokunaga, K. Yoshida, J. Am. Chem. Soc. 125 (2003) 11508.
[37] N. Tokunaga, Y. Otomaru, K. Okamoto, K. Ueyama, R. Shintani, T. Hayashi, J. Am. Chem. Soc. 126 (2004) 13584.
[38] R. Shintani, K. Okamoto, Y. Otomaru, K. Ueyama, T. Hayashi, J. Am. Chem. Soc. 127 (2005) 54.
[39] T. Hayashi, Y. Otomaru, K. Okamoto, R. Shintani, J. Org. Chem. 70 (2005) 2503.
[40] C. Fischer, C. Defieber, T. Suzuki, E.M. Carreira, J. Am. Chem. Soc. 126 (2004) 1628.
[41] C. Defieber, J.F. Paquin, S. Serna, E.M. Carreira, Org. Lett. 6 (2004) 3873.
[42] D. Milstein, R. Goikhman, Chem. Eur. J. 11 (2005) 2983.
[43] C.H. Jun, R.H. Crabtree, J. Organometal. Chem. 447 (1993) 177.
[44] G. Rivera, R.H. Crabtree, J. Mol. Catal. A: Chem. 222 (2004) 59.
[45] A. Van der Ent, A.L. Onderdelinden, Inorg. Synth. 28 (1990) 90.
[46] Z. Otwinowski, W. Minor, in: M.G. Rossman, E. Arnold (Eds.), International Tables for Crystallography, vol. F, 2001.
[47] A. Altomare, G. Cascarano, C. Giacovazzo, A. Guagliardi, J. Appl. Crystallogr. 26 (1993) 343.
[48] TEXSAN for Windows version 1.06, Crystal Structure Analysis Package, Molecular Structure Corporation (1997-9), 1999.