CHEMISTRY
A EUROPEAN JOURNAL

DOI: 10.1002/chem.201003662

Stereodivergent Ruthenium-Catalyzed Transfer Semihydrogenation of Diaryl Alkynes

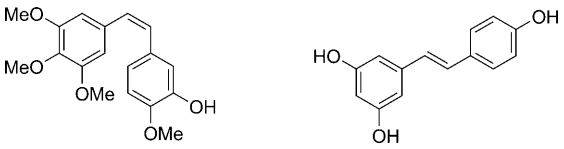
Jie Li and Ruimao Hua*[a]

Abstract: $[Ru_3(CO)_{12}]$-catalyzed transfer semihydrogenation of various functionalized diaryl alkynes with *N*,*N*-dimethylformamide (DMF) and water as hydrogen source affords *cis*- and *trans*-stilbenes. The stereodivergent approach can be switched by the use of acetic (HOAc) or trifluoroacetic (TFA) acid as additives. The catalytic processes can be applied to the synthesis of analogues of natural products such as *cis*-combretastatin A-4 and *trans*-resveratrol.

Keywords: alkynes · alkenes · hydrogenation · ruthenium · stereoselectivity

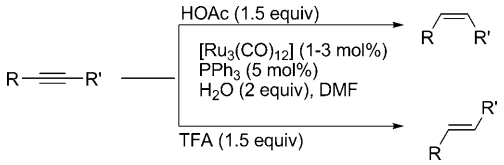
Introduction

Alkenes are not only a very important and fundamental class of compounds for organic transformations, but also occur in many natural products. As exemplified in Scheme 1, *cis*-combretastatin A-4[1] and *trans*-resveratrol,[2]



Scheme 1. Structures of *cis*-combretastatin A-4 (left) and *trans*-resveratrol (right).

as well as their analogues, have been reported to have a variety of interesting physiological and biological properties. Although there are various methods to access to *trans*- or *cis*-alkenes, the semihydrogenation of internal alkynes is the simplest and most straightforward approach, because the transfer semihydrogenation of internal alkynes through the use of hydrogen donors has clear advantages for safe operation and high control of the chemoselective formation of alkenes. Thus, many attempts have been made to develop efficient catalyst systems that can achieve such transfer semihydrogenation reactions.[3] Recently, we have developed an efficient transfer semihydrogenation of internal alkynes catalyzed by $Pd(OAc)_2$ through the use of *N*,*N*-dimethylformamide (DMF) and potassium hydroxide as hydrogen source to afford *cis*-alkenes in high yields with excellent stereoselectivity.[4] With the aim of developing stereocontrollable semihydrogenation of internal alkynes, we herein report our new finding of a novel and practical $[Ru_3(CO)_{12}]$-catalyzed transfer semihydrogenation of internal alkynes using DMF/H_2O as hydrogen source under acidic conditions to afford *trans*- and *cis*-alkenes in a stereochemically controlled manner in which the geometry of the product can be switched by the use of acetic (HOAc) or trifluoroacetic acid (TFA) (Scheme 2).



Scheme 2. Stereodivergent ruthenium-catalyzed transfer semihydrogenation of diarylalkyne.

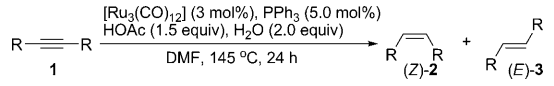
Results and Discussion

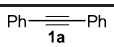
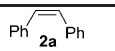
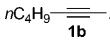
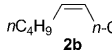
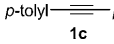
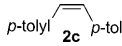
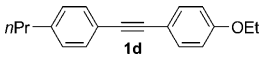
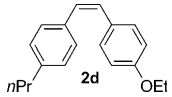
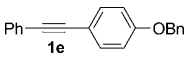
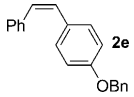
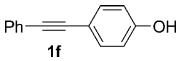
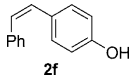
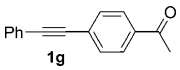
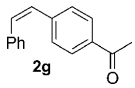
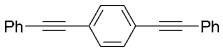
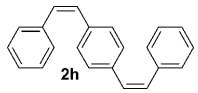
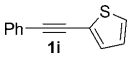
The choice of $[Ru_3(CO)_{12}]$ as catalyst for the transfer semihydrogenation of internal alkynes was based on the known ability of low-valent ruthenium complexes to catalyze transfer hydrogenation of carbonyl compounds,[5] alkenes,[6] and imines.[7] After screening the reaction conditions for the transfer semihydrogenation of diphenylacetylene (**1a**), it was found that $[Ru_3(CO)_{12}]$ (3.0 mol%) showed high catalytic activity in the presence of triphenylphosphane (PPh_3; 5 mol%), HOAc (1.5 equiv), and H_2O (2.0 equiv) in DMF. After 24 h heating at 145 °C, stilbenes were isolated in 93% yield with a *Z*/*E* ratio of 96:4, implying that the transfer semihydrogenation occurred with excellent stereoselectivity for the formation of the *cis*-isomer (**2a**; Table 1, entry 1). In the absence of either PPh_3 (conv. 72%, *Z*/*E* ratio 90:10) or

[a] J. Li, Prof. R. Hua
Department of Chemistry, Tsinghua University
Key Laboratory of Organic Optoelectronics
& Molecular Engineering of Ministry of Education
Beijing 100084 (P. R. of China)
Fax: (+86) 10-62771149
E-mail: ruimao@mail.tsinghua.edu.cn

Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/chem.201003662. It contains the general experimental procedure, characterization data, and copies of ^1H and ^{13}C NMR spectra of all products, including those of the intermediate $[Ru_2(CO)_4(\mu\text{-}CH_3COO^-)_2(PCy_3)_2]$.

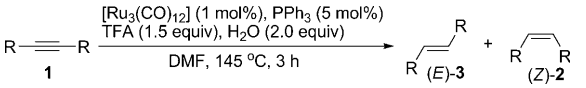
Table 1. $[Ru_3(CO)_{12}]$-catalyzed transfer semihydrogenation of diarylalkynes affording (*Z*)-stilbenes.[a]

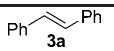
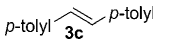
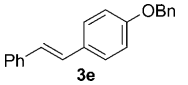
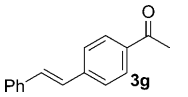
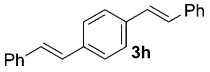
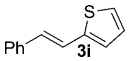
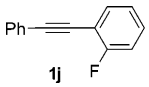
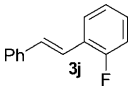


	Alkyne	Alkene	Yield [%][b]	*Z*/*E*[c]
1	 **1a**	 **2a**	93	96:4
2[d]	 **1b**	 **2b**	40	>99:1
3[e]	 **1c**	 **2c**	84	97:3
4	 **1d**	 **2d**	92	>99:1
5	 **1e**	 **2e**	93	>99:1
6	 **1f**	 **2f**	95	>99:1
7	 **1g**	 **2g**	92	95:5
8	 **1h**	 **2h**	96	99:1
9	 **1i**	 **2i**	98	75:25

[a] Reaction conditions: alkyne (0.5 mmol), HOAc (0.75 mmol), H_2O (1.0 mmol), PPh_3 (0.025 mmol), $[Ru_3(CO)_{12}]$ (0.015 mmol), DMF (1.0 mL), sealed tube, 145 °C, 24 h, under N_2. [b] Total isolated yield. [c] Determined by GC analysis of the reaction mixture. [d] Conversion was 44 % (GC analysis). [e] Conversion was 87 % (GC analysis).

Table 2. $[Ru_3(CO)_{12}]$-catalyzed transfer semihydrogenation of diarylalkynes affording (*E*)-stilbenes.[a]



	Alkyne	Stilbene	Yield [%][b]	*E*/*Z*[c]
1	**1a**	 **3a**	91	>99:1
2	**1c**	 **3c**	93	>99:1
3	**1e**	 **3e**	88	>99:1
4	**1g**	 **3g**	87	97:3
5	**1h**	 **3h**	85	–[d]
6[e]	**1i**	 **3i**	86	98:2
7	 **1j**	 **3j**	89	>99:1

[a] Reaction conditions: alkyne (0.5 mmol), TFA (0.75 mmol), H_2O (1.0 mmol), PPh_3 (0.025 mmol), $[Ru_3(CO)_{12}]$ (0.005 mmol), DMF (1.0 mL), sealed tube, 145 °C, 3 h, under N_2. [b] Total isolated yield. [c] Determined by GC analysis of the reaction mixture. [d] No *Z* product was isolated. [e] Reaction time was 6 h.

HOAc (conv. 39 %, *Z*/*E* ratio 77:23), both the conversion of **1a** and the stereoselectivity of the transfer semihydrogenation were unsatisfactory.

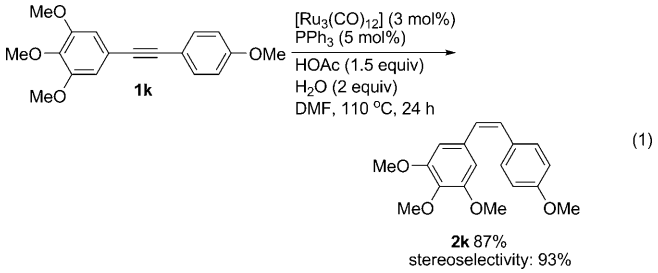
With the aim of extending the transfer semihydrogenation to other internal alkynes, various substrates were examined under the same reaction conditions. As shown in Table 1, although the reductive reaction of 5-decyne (**1b**) produced *cis*-5-decene in only 40 % isolated yield with 44 % conversion of **1b** (Table 1, entry 2), diaryl alkynes with a variety of substituents on the aromatic rings underwent the transfer semihydrogenation smoothly to afford the corresponding stilbenes in excellent yields (Table 1, entries 3–9). With the exception of **1h**, which showed relative low stereoselectivity, probably due to the existence of the coordinative thienyl group (Table 1, entry 9), all diaryl alkynes underwent transfer semihydrogenation with excellent stereoselectivity to afford *cis*-isomers with ratios higher than 96 %.

When the effect of organic acids on the catalytic activity was examined, it was surprising to find that the use of TFA instead of HOAc not only resulted in an acceleration in the rate of transfer semihydrogenation, but also switched the stereochemical behavior to afford the *trans*-isomer predominantly. As shown in Table 2, in the presence of $[Ru_3(CO)_{12}]$ (1.0 mol %), PPh_3 (5.0 mol %), TFA (1.5 equiv), and H_2O (2.0 equiv), **1a** underwent transfer semihydrogenation at 145 °C completely within only 3 h (Table 2, entry 1). Different diarylated alkenes could be obtained in high yield under similar reaction conditions with excellent *trans*-stereoselectivity (Table 2, entries 2–7).

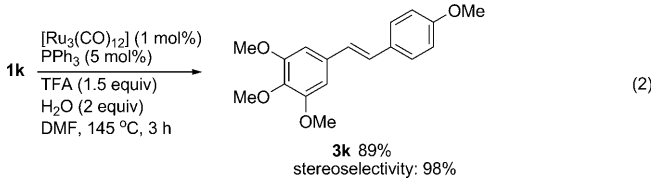
As summarized in Tables 1 and 2, the present stereodivergent ruthenium-catalyzed transfer semihydrogenation of internal alkynes occurs with excellent chemoselectivity. For example, the C–O bond of the benzyl ether in **1e** was not reductively cleaved, nor was concomitant double bond shift isomerization observed in **2b**. Furthermore, the carbonyl group in **1g** was tolerated. In the case of 1,4-bis(phenylethynyl)benzene (**1h**), both the *Z*,*Z* isomer (**2h**; Table 1, entry 8) and the *E*,*E* isomer (**3h**; Table 2, entry 5) were stereoselectively formed in excellent yield.

The synthetic usefulness of the present stereodivergent transfer semihydrogenation was further developed in the efficient synthesis of analogues of *cis*-combretastatin A-4 and *trans*-resveratrol. As shown in Equations (1) and (2), under two different standard reaction conditions, the transfer semihydrogenation of 1-(4-methoxyphenyl)-2-(3,4,5- trimethoxyphenyl)acetylene (**1k**) afforded the corresponding alkenes in high yields with the formation of *cis*-isomer **2k** with 93 % selectivity, and *trans*-isomer **3k** in 98 % isomeric purity, respectively.

Moreover, because the *p*-cyclohexyl-substituted stilbenes are potential liquid crystalline compounds,[8] the transfer semihydrogenation of 1-[4-(4-*trans*-*n*-propylcyclohexyl)-

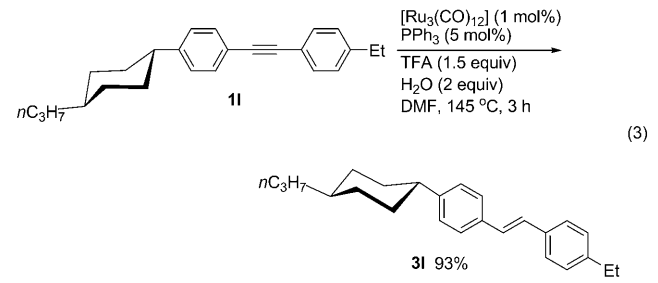


(1)



(2)

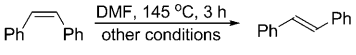
phenyl]-2-(4-ethyl)acetylene (**1l**) under the reaction conditions given in Table 2 was carried out; the reaction afforded the expected *trans*-isomer (**3l**) in 93% isolated yield [Eq. (3)].



(3)

DMF is not only a commonly used polar solvent, it has also been used as a versatile precursor under different reaction conditions in organic transformations.[9] In the present transfer semihydrogenation, formic acid (HCOOH), generated in situ from the hydrolysis of DMF,[10] is considered to be the hydrogen source.[11] The reaction of **1a** under similar reaction conditions to those given in Table 1 but in the absence of water, resulted in low conversion of **1a** (26% by GC), indicating the presence of water is crucial for the semihydrogenation. In addition, use of $[Ru(CO)_2(PPh_3)_3]$ (5 mol% under the reaction conditions given in Table 1), which is a mononuclear Ru^0 complex that is possibly formed under the reaction conditions, as an appropriate precatalyst showed low catalytic activity and stereoselectivity, affording stilbenes in 37% GC yield with a **2a**/**3a** ratio of 74:26. Therefore, attempts were made to isolate the active catalytic species from the reaction mixtures. Fortunately, a diruthenium bis(μ-acetate) species, $[Ru_2(CO)_4(\mu\text{-}CH_3COO^-)_2(PCy_3)_2]$, was isolated from the catalytic reaction mixture when PCy_3 was used instead of PPh_3; as expected, this diruthenium complex showed catalytic activity for the transfer semihydrogenation of **1a**.[12] Although the formation of hydrogen from the reaction of HCOOH with $[Ru_2(CO)_4(\mu\text{-}HCOO^-)_2(PPh_3)_2]$ was recently reported by Wills and coworkers,[11c] the mechanism of the present stereodivergent transfer semihydrogenation of diaryl alkynes requires further clarification.

The selective formation of *trans*-stilbenes through the use of TFA was then considered in more detail. According to the following experimental results, the formation of *trans*-stilbenes could arise from isomerization of *cis*-stilbenes under suitable reaction conditions: 1) In a separate experiment, the transfer semihydrogenation of **1a** under similar reaction conditions to those indicated in entry 1 of Table 2 for 1 h, resulted in 50% conversion of **1a** and a **2a**/**3a** ratio of 3:1, however, the final selectivity for **3a** increased to more than 99% after 3 h (Table 2, entry 1), indicating that isomerization of **2a** to **3a** also occurred during the transfer semihydrogenation of **1a**; 2) Product **2a** could be converted into **3a** when subjected to the reaction conditions given for entry 1 of Table 2 [Eq. (4)]; 3) As shown in Equation (4), in the absence of $[Ru_3(CO)_{12}]$, TFA did not promote the isomerization of **2a** to **3a** at 145 °C within 3 h.



(*Z*) - **2a** (*E*) - **3a**

Other conditions	Yield of **3a**
TFA (1.5 equiv)/H_2O (2.0 equiv)	< 5%
$[Ru_3(CO)_{12}]$ (1.0 mol%)/H_2O (2.0 equiv)	95%
under conditions indicated in Table 2	93%

(4)

It should be noted that although there were some reports on the *cis*-to-*trans* isomerization of stilbene under acidic conditions, such as in the presence of acidic zeolites[13] or H_2SO_4 (50–60%),[14] a series of careful experiments confirmed that, in DMF at 145 °C, the isomerization of **2a** to **3a** did not occur in the presence of TFA in the absence of $[Ru_3(CO)_{12}]$. The isomerization of *cis*- to *trans*-alkenes catalyzed by a ruthenium complex has been also reported recently by Cho et al.[15]

Conclusion

We have developed an efficient $[Ru_3(CO)_{12}]$-catalyzed transfer semihydrogenation of diaryl alkynes with DMF/H_2O as a hydrogen source. The stereoselectivity of *cis*- and *trans*-stilbenes can be easily achieved simply by the use of HOAc or TFA as additives. Application of HOAc selectively favors the production of *cis*-alkenes, and the use of TFA favors the formation of *trans*-alkenes. The observation that *cis*-stilbene forms in a higher ratio during the early stages of the semihydrogenation in the $[Ru_3(CO)_{12}]$/TFA catalytic system, and that the isomerization of *cis*- to *trans*-stilbene occurs in the presence of $[Ru_3(CO)_{12}]$ or $[Ru_3(CO)_{12}]$/TFA, suggests that the formation of *trans*-stilbene arises from the isomerization of *cis*-stilbene. It is very clear that the $[Ru_3(CO)_{12}]$/HOAc

catalytic system cannot promote the isomerization to give *cis*-stilbene even after an extended reaction time (24 h, Table 1). The present catalytic processes have also been applied to the preparation of analogues of *cis*-combretastatin A-4 and *trans*-resveratrol.

Experimental Section

Transfer semihydrogenation of diphenylacetylene (1a) to *cis*-stilbene (2a) (Table 1, entry 1)

Typical procedure: Diphenylacetylene (**1a**; 89.0 mg, 0.5 mmol), $[Ru_3(CO)_{12}]$ (9.6 mg, 0.015 mmol), PPh_3 (6.6 mg, 0.025 mmol), HOAc (46.0 mg, 0.75 mmol), H_2O (18.0 mg, 1.0 mmol), and DMF (1.0 mL) were placed in a 25 mL thick-walled Pyrex screw-cap tube under a flow of nitrogen. The tube was capped and the mixture was heated in oil bath at 145 °C with stirring for 24 h. The reaction mixture was cooled to RT and diluted with CH_2Cl_2 (4.0 mL) for GC and GC-MS analyses. After removal of volatiles under reduced pressure, the residue was then subjected to silica gel column chromatography (eluting with cyclohexane) to afford **2a** and **3a** (84.0 mg, 0.46 mmol, 93.0 %) as a colorless viscous oil. GC analysis of the reaction mixture revealed the formation of **2a** and **3a** in a ratio of 96:4.

When TFA employed, the reaction was carried out for 3 h, and the reaction mixture was worked up as above to afford **3a** in 91 % yield with more than 99 % selectivity.

Acknowledgements

This project (20573061) was supported by the National Natural Science Foundation of China.

[1] For selected reviews, see: a) N.-H. Nam, *Curr. Med. Chem.* **2003**, *10*, 1697–1722; b) A. Cirla, J. Mann, *Nat. Prod. Rep.* **2003**, *20*, 558–564. For selected recent reports, see: c) H. P. Hsieh, J. P. Liou, N. Mahindroo, *Curr. Pharm. Des.* **2005**, *11*, 1655–1677; d) G. R. Pettit, M. R. Rhodes, D. L. Herald, E. Hamel, J. M. Schmidt, R. K. Pettit, *J. Med. Chem.* **2005**, *48*, 4087–4099; e) G. C. Tron, T. Pirali, G. Sorba, F. Pagliai, S. Busacca, A. A. Genazzani, *J. Med. Chem.* **2006**, *49*, 3033–3044; f) A. Gosslau, S. Pabbaraja, S. Knapp, K. Y. Chen, *Eur. J. Pharmacol.* **2008**, *587*, 25–34; g) J. J. Hall, M. Sriram, T. E. Strecker, J. K. Tidmore, C. J. Jelinek, G. D. K. Kumar, M. B. Hadimani, G. R. Pettit, D. J. Chaplin, M. L. Trawick, K. G. Pinney, *Bioorg. Med. Chem. Lett.* **2008**, *18*, 5146–5149; h) R. Fürst, I. Zupkó, A. Berényi, G. F. Ecker, U. Rinner, *Bioorg. Med. Chem. Lett.* **2009**, *19*, 6948–6951.

[2] For selected reviews, see: a) J. A. Baur, D. A. Sinclair, *Nat. Rev. Drug Discovery* **2006**, *5*, 493–506; b) M. Athar, J. H. Back, X. Tang, K. H. Kim, L. Kopelovich, D. R. Bickers, A. L. Kim, *Toxicol. Appl. Pharmacol.* **2007**, *224*, 274–283; c) E. L. Robb, J. A. Stuart, *Molecules* **2010**, *15*, 1196–1212. For selected recent reports, see: d) M. Jang, L. Cai, G. O. Udeani, K. V. Slowing, C. F. Thomas, C. W. W. Beecher, H. H. S. Fong, N. R. Farnsworth, A. D. Kinghorn, R. G. Mehta, R. C. Moon, J. M. Pezzuto, *Science* **1997**, *275*, 218–220; e) B. D. Gehm, J. M. Mcandrews, P.-Y. Chien, J. L. Jameson, *Proc. Natl. Acad. Sci. USA* **1997**, *94*, 14138–14143; f) M. Holmes-McNary, A. S. Baldwin Jr. , *Cancer Res.* **2000**, *60*, 3477–3483; g) M. H. Azizi, R. Kumar, N. Ahmad, *Int. J. Oncol.* **2003**, *23*, 17–28; h) L. Whyte, Y.-Y. Huang, K. Torres, R. G. Mehta, *Cancer Res.* **2007**, *67*, 12007–12017; i) T. M. Bass, D. Weinkove, K. Houthoofd, D. Gems, L. Partridge, *Mech. Ageing Dev.* **2007**, *128*, 546–552.

[3] a) K. Tani, N. Ono, S. Okamoto, F. Sato, *J. Chem. Soc. Chem. Commun.* **1993**, 386–387; b) N. M. Yoon, K. B. Park, H. J. Lee, J. Choi, *Tetrahedron Lett.* **1996**, *37*, 8527–8528; c) K. Tani, A. Iseki, T. Yamagata, *Chem. Commun.* **1999**, 1821–1822; d) F. Alonso, I. Osante, M. Yus, *Adv. Synth. Catal.* **2006**, *348*, 305–308; e) H. Sajiki, S. Mori, T. Ohkubo, T. Ikawa, A. Kume, T. Maegawa, Y. Monguchi, *Chem. Eur. J.* **2008**, *14*, 5109–5111; f) P. Hauwert, G. Maestri, J. W. Sprengers, M. Catellani, C. J. Elsevier, *Angew. Chem.* **2008**, *120*, 3267–3270; *Angew. Chem. Int. Ed.* **2008**, *47*, 3223–3226; g) R. Barrios-Francisco, J. J. García, *Inorg. Chem.* **2009**, *48*, 386–393.

[4] J. Li, R. Hua, T. Liu, *J. Org. Chem.* **2010**, *75*, 2966–2970.

[5] For selected reports, see: a) Y. R. S. Laxmi, J.-E. Bäckvall, *Chem. Commun.* **2000**, 611–612; b) Ch. S. Cho, B. T. Kim, T.-J. Kim, S. C. Shim, *J. Org. Chem.* **2001**, *66*, 9020–9022; c) C. S. Yi, Z. He, I. A. Guzei, *Organometallics* **2001**, *20*, 3641–3643; d) V. Cadierno, P. Crochet, J. Diez, J. García-Álvarez, S. E. García-Garrido, S. García-Granda, J. Gimeno, M. A. Rodríguez, *Dalton Trans.* **2003**, 3240–3249; e) M. Yiğit, B. Yiğit, Í. Özdemir, E. Cetinkaya, B. Cetinkaya, *Appl. Organomet. Chem.* **2006**, *20*, 322–327; f) S. Zeror, J. Collin, J.-C. Fiaud, L. A. Zouioueche, *Adv. Synth. Catal.* **2008**, *350*, 197–204.

[6] For selected reports, see: a) H. Imai, T. Nishiguchi, M. Kobayashi, K. Fukuzumi, *Bull. Chem. Soc. Jpn.* **1975**, *48*, 1585–1589; b) M. A. Bennett, T.-N. Huang, A. K. Smith, T. W. Turney, *J. Chem. Soc. Chem. Commun.* **1978**, 582–583; c) J. M. Brown, H. Brunner, W. Leitner, M. Rose, *Tetrahedron: Asymmetry* **1991**, *2*, 331–334; d) S. Naskar, M. Bhattacharjee, *Tetrahedron Lett.* **2007**, *48*, 465–467.

[7] For selected reports, see: a) A. Basu, S. Bhaduri, K. Sharma, P. G. Jones, *J. Chem. Soc. Chem. Commun.* **1987**, 1126–1127; b) G.-Z. Wang, J.-E. Bäckvall, *J. Chem. Soc. Chem. Commun.* **1992**, 980–982; c) N. Uematsu, A. Fujii, S. Hashiguchi, T. Ikariya, R. Noyori, *J. Am. Chem. Soc.* **1996**, *118*, 4916–4917; d) E. Mizushima, M. Yamaguchi, T. Yamagishi, *Chem. Lett.* **1997**, 237–238; e) M. Kitamura, D. Lee, S. Hayashi, S. Tanaka, M. Yoshimura, *J. Org. Chem.* **2002**, *67*, 8685–8687; f) R. Kadyrov, T. H. Riermeier, *Angew. Chem.* **2003**, *115*, 5630–5632; *Angew. Chem. Int. Ed.* **2003**, *42*, 5472–5474.

[8] J. C. Liang, L. Chen, *Mol. Cryst. Liq. Cryst.* **1989**, *167*, 253–258.

[9] J. Muzart, *Tetrahedron* **2009**, *65*, 8313–8323.

[10] a) A. A. Pletnev, Q. Tian, R. C. Larock, *J. Org. Chem.* **2002**, *67*, 9276–9287; b) S. Liu, C. Wang, H. Zhai, D. Li, *J. Mol. Struct.* **2003**, *654*, 215–221.

[11] a) Y. Gao, J. Kuncheria, G. P. A. Yap, R. J. Puddephantt, *Chem. Commun.* **1998**, 2365–2366; b) A. F. Hill, *Angew. Chem.* **2000**, *112*, 134–138; *Angew. Chem. Int. Ed.* **2000**, *39*, 130–133; c) D. J. Morris, G. J. Clarkson, M. Wills, *Organometallics* **2009**, *28*, 4133–4140.

[12] When PCy_3 was used instead of PPh_3 under the reaction conditions given in Table 1, the transfer semihydrogenation of **1a** also occurred smoothly to afford *cis*-**2a** and *trans*-**3a** in 97 % yield with a ratio of 87:13. After the reaction mixture was cooled to room temperature, $[Ru_2(CO)_4(\mu\text{-}CH_3COO^-)_2(PCy_3)_2]$ could be obtained as crystals in 56 % yield (relative to ruthenium). This compound showed comparable catalytic activity to that obtained with the combination of $[Ru_3(CO)_{12}]$ and PCy_3, to give *cis*-**2a** and *trans*-**3a** in 93 % yield with a ratio of 89:11, when used in 5 mol % relative to **1a**. CCDC-798851 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif..

[13] M. Kojima, H. Takeya, Y. Kuriyama, S. Oishi, *Chem. Lett.* **1997**, 997.

[14] D. S. Noyce, D. R. Hartter, F. B. Miles, *J. Am. Chem. Soc.* **1964**, *86*, 3583–3584.

[15] C. S. Cho, D. T. Kim, S. C. Shim, *Bull. Korean Chem. Soc.* **2009**, *30*, 1931–1932.

Received: December 20, 2010
Revised: March 31, 2011
Published online: June 8, 2011

