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Journal of Molecular Catalysis A: Chemical 214 (2004) 147–154
JOURNAL OF MOLECULAR CATALYSIS A: CHEMICAL
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Ruthenium-catalyzed tandem olefin migration—aldol- and Mannich-type reactions in ionic liquid

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Received 31 July 2003; accepted 16 December 2003

Abstract

In the presence of a catalytic amount of $RuCl_2(PPh_3)_3$, a cross-coupling of 3-buten-2-ol (2) with aldehydes and imines was developed via a tandem olefin migration—aldol–Mannich reaction in bmim[PF_6]. With $In(OAc)_3$ as a co-catalyst, α-vinylbenzyl alcohol (4) and aldehydes underwent similar coupling reactions. Compared with aqueous and other organic solvents, complementary diastereoselectivity was observed with ionic liquid as the solvent. The ionic liquid/catalyst system could be reused at least five times without loss of activity.

Keywords: Aldol- and Mannich-type reactions; Ionic liquid; Ruthenium catalyst; Catalyst recycling

1. Introduction

Aldol and Mannich-type reactions are among the most important C–C bond forming reactions in organic synthesis. However, under the classical aldol reaction conditions, side reactions such as dimerization, polymerization, and self-condensation also occur [1]. The classical Mannich reaction also has its limitations [2]. An important improvement over the classical aldol and Mannich reactions is the Lewis acid-catalyzed condensation of enol silyl ether with carbonyl compounds (Mukaiyama aldol reaction) [3] and imines [4]. More recently, Trost reported a highly efficient formation of aldol-type products via vanadium-catalyzed coupling of propargyl alcohols with aldehydes [5]. Other recent developments include hydrometalation-aldol [6] and C–H activation-aldol reactions of carbonyl compounds [7]. Alternatively, Motherwell developed the Rh- and Ni-catalyzed isomerization of allylic lithium alkoxide to lithium enolate that undergoes further aldol reaction [8]. Grée recently reported the isomerization of allyl alcohol to enol catalyzed by $Fe(CO)_5$ under photolytic conditions, which then reacted with aldehydes to give aldol products (together with other by-products) [9a]. They also found that the combination of $RhCl(PPh_3)_3$/BuLi is an effective catalyst for the coupling of allyl alcohols with aldehydes. However, no reaction was observed without the lithium co-reagent [9b].

Recently, interest in the development of organic reactions in environmentally friendly media, i.e. “green solvents,” has been growing [10]. For aqueous aldol-type condensations, Chan developed the tin and zinc-mediated cross-couplings of halo-ketones with aldehydes [11]. Lubineau [12] Kobayashi [13], Loh and co-workers [14,15] developed aqueous Mukaiyama-type reactions. Also, aqueous Mannich-type reactions were realized by Kobayashi [16a] and Akiyama [16b] using silyl enol ethers or silyl ketene acetals.

Another group of promising green solvents that have gained much attention recently are “ionic liquids” [17a,b]. Their nonvolatile nature gives them a significant advantage in minimizing solvent consumption. Their polarity renders them good solvents for transition metal catalysis and therefore good reaction media for homogeneous catalysis [17c]. Because of their unique solubility properties, i.e. a miscibility gap between water and organic solvents, they have become interesting candidates for separation processes by simple liquid–liquid extraction with either aqueous or conventional organic solvents [18].

Previously, during our studies of developing air- and water-tolerant catalysis, we reported that, in the presence of

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doi:10.1016/j.molcata.2003.12.031

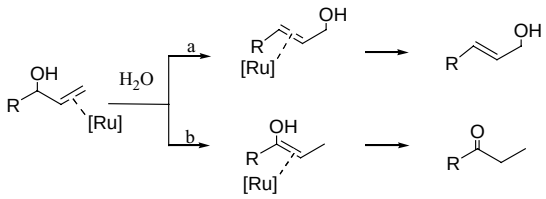


Fig. 1. Ruthenium-catalyzed olefin migration in water.

a catalytic amount of $RuCl_2(PPh_3)_3$, functional groups of homoallyl alcohols and allyl alcohols underwent rearrangement in air and water (Fig. 1, path a). A side product of the isomerization is the formation of a ketone (Fig. 1, path b) [19]. The formation of this product was rationalized by the competing process involving the break of the allylic C–H bond (instead of the C–O bond) to form a ruthenium-enol complex, which then tautomerised to give the ketone [20]. We postulated that in the presence of an aldehyde, such a ruthenium-enol complex might be captured [21]. Our previous work reported the successful capture of the intermediate by aldehydes to afford aldol-type products in water [22]. In addition, the cross-coupling of imines with allyl alcohols generated Mannich-type reaction products efficiently under similar conditions in methanol [23a]. Although these reactions were successful, an organic co-solvent was sometimes required. It would be more desirable to avoid such organic co-solvents. Recently, we reported our prelimnary studies of a tandem olefin migration-aldol reaction in ionic liquid [23b]. Herein, we described the detailed results of the reaction between aldehydes and imines with allyl alcohol catalyzed by $RuCl_2(PPh_3)_3$ in ionic liquid.

2. Results and discussion

In many applications ionic liquids with weakly coordinating anions, such as BF_4^- and PF_6^-, together with suitably substituted cations, often result in an altered chemical reactivity of the dissolved catalyst and therefore improve both reactivity and selectivity [17]. Among the commonly used ionic liquids, 1-butyl-3-methylimidazoliumhexafluorophosphate ([bmim]PF_6) is a popular reaction medium for a wide variety of transition metal catalyzed organic transformations such as hydrogenation, [24] oxidation, [25] hydroformylation, [26] oligomerization, [27] etc.

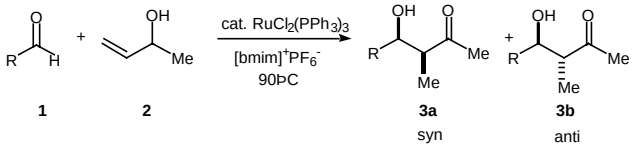
In initial explorations, when 3-buten-2-ol was stirred with benzaldehyde and a catalytic amount of $RuCl_2(PPh_3)_3$ (5 mol%) in [bmim]PF_6 for 24 h at room temperature, no desired product was obtained. When the mixture was heated at 90 °C in an oil bath, the reaction mixture became homogeneous and a smooth reaction occurred to give the desired aldol product (with a *syn*:*anti* ratio of 71:29, stereochemistry determined by comparison with literature data) 13 in 81% isolated yield. Decomposition of the desired product was observed after a prolonged reaction time, e.g. overnight, possibly due to elimination of the hydroxy group to give an α, β-unsaturated ketone. A small amount of 2-butanone was detected by GC, although exact quantification was difficult to achieve because of the volatility of the ketone. A control reaction of methyl ethyl ketone with benzaldehyde under the same reaction condition was carried out and only starting material was recovered after 24 h. Other popular Ru and Rh catalysts were also examined without much success (Table 1) Addition of either acid or base decreased the reactivity of the catalytic system (Table 1, Entries 9 and 10). Subsequently, the cross-coupling reactions between a variety of aldehydes/imines and allyl alcohols were examined under similar reaction conditions.

Table 1
Screening of Ru and Rh catalysts (R=Ph)

Entry	Catalyst (5 mol%)	Time (h)	Solvent	Yield (%)
1	$Ru_3(CO)_{12}$	5	I.L.	0
2	$RuCp_2$	5	I.L.	0
3	$Ru(CO)(H_2)(PPh_3)_3$	5	I.L.	0
4	$Ru(COD)_2{\cdot}BF_4$	5	I.L.	0
5	$Rh(PPh_3)_3Cl$	5	I.L.	0
6	$[Rh(COD)Cl]_2$	5	I.L.	0
7	$RuCl_2(PPh_3)_3$	1.5	I.L.	81
8	$RuCl_2(PPh_3)_3$	5	I.L.+Et_3N(3eq.)	12
9	$RuCl_2(PPh_3)_3$	5	I.L. +MsOH(3eq.)	26

2.1. Cross-coupling between aldehydes and allyl alcohols

A variety of aromatic aldehydes were examined under similar reaction conditions (Scheme 1, Table 2). In most cases, the desired aldol products were obtained in good yields even with less reactive aldehydes such as 2-naphthaldehyde or *p*-anisaldehyde. The only exception was 4-hydroxybenzaldehyde, with which a very low yield was achieved. One plausible explanation was the hydroxy group on the benzene ring coordinating with and deactivating the ruthenium catalyst. Compared with other solvents such as water and water–organic solvent mixture (entries **3**, **12**, and **13**, Table 2), ionic liquid provided higher activity from the catalyst, with improved yield and shortened reaction time. Moreover, the results exhibit a "reverse" trend in the reactivity of aldehydes as compared to aldol condensation in organic solvents and aqueous solutions (entries **7** and **14**, Table 2), in which electron-deficient aldehydes give better yields and electron-rich substrates normally result in low yields or no reaction. Although a detailed explanation is unavailable at this moment, this trend could be a result of the unique solvation properties of the ionic liquid, where more polarized



Scheme 1.

Table 2
Coupling of aldehydes with allylic alcohol in ionic liquid

Entry	Aldehyde (R)	Solvent	Time (h)	Yield (%)[a]	*Syn/anti*[b]
1	Ph	Ionic liquid	1.5	81	71/29
2	*m*-F-C_6H_4	Ionic liquid	1.5	68	68/32
3	*p*-Cl-C_6H_4	Ionic liquid	2	75	71/29
4	*p*-Br-C_6H_4	Ionic liquid	1	79	75/25
5	*p*-MeO-C_6H_4	Ionic liquid	1	80	72/28
6	*p*-Ph-C_6H_4	Ionic liquid	1	84	71/29
7	2-Naphthyl	Ionic liquid	2	94	69/31
8	*n*-C_7H_{15}	Ionic liquid	1	67	37/63
9	Diphenylmethyl	Ionic liquid	2	46	16/84
10	2-Phenylethyl	Ionic liquid	2	52	33/67
11	Cyclohexyl	Ionic liquid	2	65	42/58
12	*p*-Cl-C_6H_4	H_2O	5	35	60/40
13	*p*-Cl-C_6H_4	H_2O–toluene (4:1)	5	70	76/26
14	2-Naphthyl	H_2O–toluene (4:1)	5	44	63/37

[a] Isolated yields were reported.
[b] The diastereoselectivity was based on ^{1}H NMR analysis of the crude products.

electron-deficient aldehydes tend to be more readily solvated by anions and cations of the solvent and therefore are less likely to be approached by catalyst-activated enols. Nevertheless, the reaction provides good substitution for other aldol reactions when electron-rich aromatic aldehydes are desired reactants. The diastereoselectivity remains relatively consistent, favoring the *syn* product.

The reaction of 3-buten-2-ol with aliphatic aldehyde in [bmim]PF_6 was also examined (Scheme 1, Table 2). Unlike the aqueous condition, the reaction went smoothly in ionic liquid with all aliphatic aldehydes, although the yields were considerably lower than for aromatic substrates. It was also observed that unlike aromatic aldehydes, which gave a relatively constant *syn-anti* ratio, the diastereoselectivity in the condensation of aliphatic aldehydes was substrate-dependent, favoring the *anti* product. However, when this catalytic system was applied to ketones, neither aromatic nor aliphatic substrates gave satisfactory yields. Work is in progress to increase the conversion rate of ketone condensation and subsequently broaden the application of this methodology.

To improve the limitation of the substrate in our previous investigation, [23b] aldol reactions between α-vinylbenzyl alcohol (**4**) and aromatic aldehydes were examined (Scheme 2 and Table 2). However, under the same conditions, reaction of benzaldehyde with **4** gave the desired aldol product with low yield. Most of the α-vinylbenzyl alcohol **4** was converted into propiophenone, which resulted from the olefin migration through the path b as described in Fig. 1.

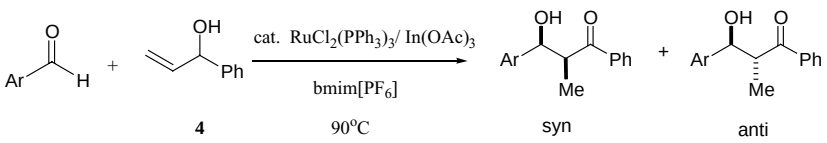
Table 3
Screening of Lewis acid co-catalyst

Entry	Co-Catalyst (10 mol%)	Time (h)	Yield (%)
1	$CuBr_2$	4	8
2	$Cu(OTf)_2$	4	11
3	$Sc(OTf)_3$	4	10
4	$Zn(OTf)_2$	4	0
5	$Yb(OTf)_2$	4	0
6	$In(OAc)_3$	3	83

We speculated that in order to decrease the production of propiophenone and increase the yield of aldol product, a Lewis acid was needed to activate the aldehyde. Through screening (Table 3) of common Lewis acids, $In(OAc)_3$ was found to be an excellent cocatalyst (Table 3, entry 6) [23]. Using the catalyst system [$RuCl_2(PPh_3)_3$ (5 mol%) + $In(OAc)_3$ (10 mol%)], the yield of the aldol product was increased dramatically to 83% (entries **1** versus **2** in Table 2). However, under the same conditions, no reaction occurred between propiophenone and benzaldehyde, which ruled out a mechanism involving a ketone formation-aldol reaction. The cross-coupling of allyl alcohol **4** with other aldehydes gave the desired aldol-type products in similar yields (45–83%) (entry **2**–**6** in Table 4). Unlike aqueous and other organic solvents, the *anti* product was preferred regardless of the substrate, although the diastereoselectivity was not very significant. Moreover, unlike the cross-coupling with allyl alcohol **2**, the electron-rich aldehydes gave a lower yield than electron-deficient substrates. When benzaldehyde was treated with 4-phenyl-2-hydroxy-3-butene, elimination of water from the alcohol took place, which indicates that the migration of the double bonds could become difficult by stabilizing it with conjugated aromatic systems.

2.2. Cross-coupling between imines and allyl alcohols

Our success with the aldol-type reaction led us to explore related coupling between allyl alcohols and imines to generate Mannich-type reaction products (Scheme 3, Table 5). As a model study, imines were generated in situ from aldehydes and *p*-anisidine, and the reactions between imines and 3-buten-2-ol (**2**) were carried out at 90 °C (oil bath temperature) using $RuCl_2(PPh_3)_3$ as a catalyst. The desired Mannich products were obtained efficiently (Scheme 3, Table 5). Compared with other solvents, ionic liquids resulted in a faster reaction with a higher yield (entries **3**, **8** and **9**). Unlike in the aqueous medium, where aldol products were also formed as a result of decomposition of imine, only Mannich products were detected in ionic liquids.

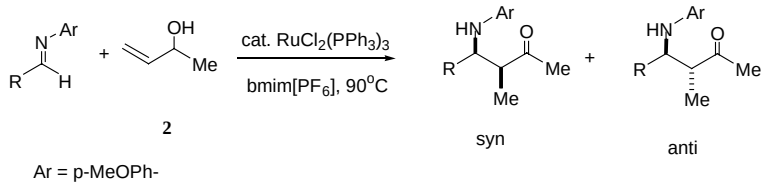


Scheme 2.

Table 4
Aldol-type reactions between allyl alcohols **4** and aldehydes **1** in ionic liquid

Entry	Substrate Ar	Catalyst	Product Ar	Yield (%)[a]	*Syn*/*anti*[b]
1	Ph	$RuCl_2(PPh_3)_3$ (5 mol%)	Ph	7	45/55
2	Ph	$RuCl_2(PPh_3)_3$ (5 mol%) + $In(OAc)_3$ (10 mol%)	Ph	83	43/57
3	*p*-Cl-Ph	$RuCl_2(PPh_3)_3$ (5 mol%) + $In(OAc)_3$ (10 mol%)	*p*-Cl-Ph	80	47/53
4	*p*-Br-Ph	$RuCl_2(PPh_3)_3$ (5 mol%) + $In(OAc)_3$ (10 mol%)	*p*-Br-Ph	75	44/56
5	2-Naphthyl	$RuCl_2(PPh_3)_3$ (5 mol%) + $In(OAc)_3$ (10 mol%)	2-Naphthyl	45	46/54
6	*p*-Me-Ph	$RuCl_2(PPh_3)_3$ (5 mol%) + $In(OAc)_3$ (10 mol%)	*p*-Me-Ph	70	38/62

[a] Isolated yields were referred.
[b] *Syn*/*anti* were determined by the ^1H NMR of the product mixture.



Scheme 3.

Table 5
Coupling of imines with allyl alcohol in ionic liquid

Entry	Imine (R)	Solvent	Time (h)	Yield (%)[a]	*Syn*/*anti*[b]
1	Ph	Ionic liquid	3	77	56/44
2	*m*-F-C_6H_4	Ionic liquid	3	61	61/39
3	*p*-Cl-C_6H_4	Ionic liquid	2	79	58/42
4	*p*-Br-C_6H_4	Ionic liquid	2.5	72	50/50
5	*p*-MeO-C_6H_4	Ionic liquid	3	84	53/47
6	*o*-Me-C_6H_4	Ionic liquid	3.5	70	43/57
7	2-Naphthyl	Ionic liquid	3	75	54/46
8	*p*-Cl-C_6H_4	MeOH	10	68	59/41
9	*p*-Cl-C_6H_4	H_2O–toluene (1:4)	10	38	55/45

[a] Isolated yields were reported.
[b] Diastereoselectivity was based on ^1H NMR analysis of crude products.

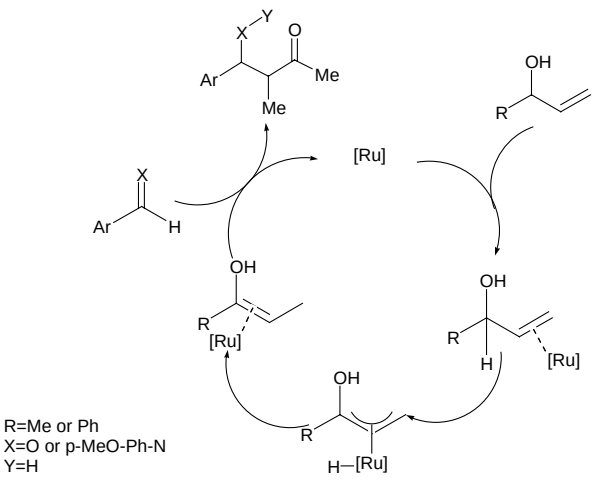


Fig. 2. Tentative mechanism for the catalytic tandem reaction.

Table 6
Recycling studies of ru-ionic liquid catalytic system

Cycle	Yield (%)	*Syn*/*anti*	Time (h)
1	92	72/28	2
2	89	71/29	2
3	90	70/30	2.5
4	93	70/30	3
5	88	70/30	4

A tentative mechanism for the product formation is depicted in Fig. 2. The ruthenium complex isomerizes the allyl alcohol **2** or **4** to an enol that is coordinated with the ruthenium catalyst; an in situ coupling between the enol-ruthenium complex with the aldehyde or imine generates the aldol or Mannich-type product. The formation of a primary *syn* isomer (Tables 1–3) is consistent with previous studies on aldol- or Mannich-type reaction with silyl enol ether [16,22].

2.3. Recycling of the ionic liquid/catalyst system

Having established the viability of this reaction, attention was focused on recycling the Ru catalyst in [bmim]PF_6. 2-Naphthaldehyde was used as a substrate in view of the high conversion rate. After ether extraction of the product, the ionic phase was dried in air, and the catalytic system was reloaded with 2-naphthaldehyde and 3-buten-2-ol for the next run. As shown in Table 6, the solvent/catalyst system could be reused at least five times with essentially no loss of activity. It remains to be established whether this high recycling efficiency can be achieved with other aromatic and aliphatic aldehydes.

3. Conclusions

In summary, we have developed an aldol-type and a Mannich-type reaction via the cross-coupling of aldehydes and imines with allylic alcohols catalyzed by $RuCl_2(PPh_3)_3$ in ionic liquid. The solvent/catalyst system is very reactive and can be reused at least five times without loss of activity. The scope, mechanism, and synthetic applications of the reactions are currently under investigation.

4. Experimental

4.1. General

Commercially available chemicals were used directly as received. The flash chromatography employed E.Merck silica gel (Kiesegel 60, 230–400 mesh) purchased from Scientific Adsorbents. High-resolution mass spectrometry was performed at the Institute of Chemistry, the Chinese Academy of Sciences.

4.2. Cross-coupling between aldehydes and allyl alcohols

A mixture of the aldehyde (0.1 mmol), allyl alcohol (0.25 mmol) and $RuCl_2(PPh_3)_3$ (0.005 mmol, 5 mol%) in bmim[PF_6] (0.3 ml) was stirred at 90 °C (oil bath temperature) in a sealed reaction vial for 1–2 h until the aldehyde was completely consumed (monitored by TLC). Then, the reaction mixture was cooled to room temperature and extracted with diethyl ether (3 × 2 ml). The combined ether phase was washed with brine and dried over anhydrous Na_2SO_4. The mixture was then concentrated under a reduced pressure. The residue was purified by flash chromatography on silica gel to afford the aldol-type product.

4.3. Cross-coupling between imines and allyl alcohols

A mixture of the aldehyde (0.1 mmol) and *p*-anisidine (0.1 mmol) in bmim[PF_6] (0.3 ml) was heated in an open reaction vial at 100 °C (oil bath temperature). Evaporation of water was observed immediately. After 1 h, allyl alcohol (0.25 mmol) and $RuCl_2(PPh_3)_3$ (0.005 mmol, 5 mmol%) was added and the reaction vial was sealed and heated at 90 °C for 2–3.5 h. Then, the reaction mixture was cooled to room temperature and extracted with diethyl ether (3 × 2 ml). The combined ether phase was washed with brine and dried over anhydrous Na_2SO_4. The mixture was then concentrated under a reduced pressure. The residue was purified by flash chromatography on silica gel to afford the Mannich-type product.

4.4. Characterization of new compounds

4.4.1. 1-(3-Fluorophenyl)-1-hydroxy-2-methyl-3-butanone (syn)

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.24–7.31 (m, 1H), 7.03–7.08 (m, 2H), 6.90–6.96 (m, 1H), 5.12 (d, 1H, J = 3.6 Hz), 3.20 (brs, 1H), 2.79 (dq, 1H, J = 3.6, 8.0 Hz), 2.17 (s, 3H), 1.04 (d, 3H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 213.9, 164.3, 161.9, 144.6(2), 130.2(2), 121.6(2), 114.5, 114.3, 113.3, 113.1, 72.2(2), 53.0, 29.5, 10.0; IR (film, cm^{-1}) 3436, 2937, 1708, 1591, 1449, 1359, 1245, 1179, 1133, 1028; HRMS *m/z* calcd. for $C_{11}H_{13}O_2F$: 219.0792(M + Na), 197.0972(M + H); found 219.0793(M + Na), 197.0970(M + H) (*anti*): ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.26–7.33 (m, 1H), 7.02–7.09 (m, 2H), 6.94–7.00 (m, 1H), 4.73 (dd, 1H, J = 3.6, 8.0 Hz), 2.97 (d, 1H, J= 3.6 Hz), 2.83–2.92 (m, 1H), 2.20 (s, 3H), 0.95 (d, 3H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 213.5, 164.4, 161.9, 144.8(2), 130.3, 130.2, 122.5(2), 115.2, 115.0, 113.8, 113.6, 76.0, 53.6, 30.3, 14.3; IR (film, cm^{-1}) 3426, 2924, 1708, 1591, 1451, 1359, 1246, 1170, 1023; HRMS *m/z* calcd. for $C_{11}H_{13}O_2F$: 219.0792(M + Na), 197.0972(M + H); found 219.0793(M + Na), 197.0970(M + H).

4.4.2. 1-(4-Methoxyphenyl)-1-hydroxy-2-methyl-3-butanone (syn)

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.19–7.24 (m, 2H), 6.83–6.87 (m, 2H), 4.99 (d, 1H, J = 4.0 Hz), 3.77 (s, 3H), 2.91 (brs, 1H), 2.79 (dq, 1H, J = 4.0, 8.0 Hz), 2.10 (s, 3H), 1.08 (d, 3H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 213.9, 159.1, 134.1, 127.3, 113.9, 73.1, 55.5, 53.5, 29.8, 10.7; IR (film, cm^{-1}) 3435, 2936, 1707, 1611, 1513, 1357, 1248, 1176, 1031 (*anti*): ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.19–7.25 (m, 2H), 6.83–6.88 (m, 2H), 4.67 (d, 1H, J = 8.4 Hz), 3.78 (s, 3H), 2.83–2.91 (m, 1H), 2.75 (brs, 1H), 2.20 (s, 3H), 0.88 (d, 3H, J = 8.4 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 213.8, 159.5, 134.3, 128.1, 114.1, 76.3, 55.5, 54.0, 30.2, 14.3; IR (film, cm^{-1}) 3428, 2934, 1706, 1611, 1513, 1249, 1176, 1032; HRMS *m/z* calcd. for $C_{12}H_{16}O_3$: 208.1094, 231.0992(M + Na); found 208.1095, 231.0991(M + Na).

4.4.3. 1-(4-Phenylphenyl)-1-hydroxy-2-methyl-3-butanone (syn)

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.54–7.59 (m, 4H), 7.30–7.45 (m, 5H), 5.15 (d, 1H, J = 3.6 Hz), 3.08 (brs, 1H), 2.86 (dq, 1H, J = 3.6, 8.0), 2.17 (s, 3H), 1.11 (d, 3H, 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 214.0, 141.0, 140.4, 129.0, 127.5, 127.3, 127.2, 126.6, 72.9, 53.3, 29.6, 10.4; IR (film, cm^{-1}) 3439, 2926, 1703, 1486, 1358, 1178, 1008; HRMS *m/z* calcd. for $C_{17}H_{18}O_2$: 254.1301; found 254.1308 (*anti*): ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.55–7.59 (m, 4H), 7.31–7.45 (m, 5H), 4.78 (d, 1H, J = 8.0 Hz), 2.91–3.00 (m, 1H), 2.85 (brs, 1H), 2.85 (brs, 1H), 2.23(s, 3H), 0.97 (d, 3H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 213.7, 141.1(2), 141.0, 129.0, 127.6, 127.5, 127.3(2), 76.5, 53.8, 30.3, 14.4; IR (film, cm^{-1}) 3426, 2926, 1708, 1485, 1357, 1241, 1170, 1008; HRMS *m/z* calcd. for $C_{17}H_{18}O_2$: 254.1301; found 254.1308.

4.4.4. 1-(2-Naphthyl)-1-hydroxy-2-methyl-3-butanone (syn)

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.79–7.84 (m, 4H), 7.42–7.50 (m, 2H), 7.35–7.39 (m, 1H), 5.26 (d,

1H, J = 3.6 Hz), 3.25 (brs, 1H), 2.92 (dq, 1H, J = 3.6, 8.0 Hz), 2.16 (s, 3H), 1.09 (d, 3H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 214.0, 139.3, 133.4, 133.0, 128.2, 127.9, 126.4, 126.1, 125.0, 124.1, 73.2, 53.2, 29.6, 10.3; IR (film, cm^{-1}) 3429, 2980, 1709, 1413, 1358, 1177, 1022; HRMS *m/z* calcd. for $C_{15}H_{16}O_2$: 228.1145, 251.1042(M + Na); found 228.1144, 251.1039(M + Na) (*anti*): ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.79–7.85(m, 3H), 7.73–7.76(m, 1H), 7.44–7.50(m, 3H), 4.90(d, 1H, J = 8.0 Hz), 2.98–3.07(m, 1H), 2.95(brs, 1H), 2.22(s, 3H), 0.94(d, 3H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 213.7, 139.5, 133.4, 133.3, 128.7, 128.2, 127.9, 126.5, 126.3, 126.1, 124.4, 53.7, 30.4, 14.4; IR (film, cm^{-1}) 3415, 2929, 1707, 1358, 1239, 1172, 1021; HRMS *m/z* calcd. for $C_{15}H_{16}O_2$: 228.1145, 251.1042(M + Na); found 228.1144, 251.1039(M + Na).

4.4.5. 1-Phenyl-1-(p-methoxyphenylamino)-2-methyl-3-butanone (syn)

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.18–7.32 (m, 5H), 6.62–6.67 (m, 2H), 6.41–6.48 (m, 2H), 4.65 (d, 1H, J = 5.2 Hz), 3.66 (s, 3H), 2.95–3.07 (m, 1H), 2.09 (s, 3H), 1.06 (d, 3H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 211.0, 152.3, 141.6, 141.5, 128.8, 127.5, 127.2, 115.1, 114.9, 55.9, 53.2, 29.6, 15.5, 11.3; IR (film, cm^{-1}) 3390, 2933, 1707, 1512, 1453, 1243, 1178, 1037; HRMS *m/z* calcd. for $C_{18}H_{21}O_2N$: 284.1645(M + H); Found 284.1647 (*anti*): ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.17–7.33 (m, 5H), 6.61–6.66 (m, 2H), 6.40–6.47 (m, 2H), 4.38 (d, 1H, J = 8.0 Hz), 3.65 (s, 3H), 2.87–2.95 (m, 1H), 2.01 (s, 3H), 1.07 (d, 3H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 212.8, 152.3, 141.4, 141.3, 128.9, 127.8, 127.3, 115.0, 114.9, 59.8, 55.9, 54.0, 29.2, 15.3; IR (film, cm^{-1}) 3390.0, 2931.4, 1707.2, 1623.9, 1506.8, 1453.4, 1288.6, 1246.3, 1191.1, 1034.1; HRMS *m/z* calcd. for $C_{18}H_{21}O_2N$: 284.1645(M + H); Found 284.1647.

4.4.6. 1-(3-Fluorophenyl)-1-(p-methoxyphenylamino)-2-methyl-3-butanone (syn)

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.25–7.30 (m, 1H), 6.99–7.10 (m, 2H), 6.87–6.94 (m, 1H), 6.63–6.69 (m, 2H), 6.38–6.46 (m, 2H), 4.64 (d, 1H, J = 5.2 Hz), 3.67 (s, 3H), 2.93–3.01 (m, 1H), 2.11 (s, 3H), 1.06 (d, 3H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 210.6, 164.6, 162.1, 152.5, 144.6, 140.9, 130.4, 130.3, 122.8, 122.7, 115.0, 114.9, 114.6, 114.4, 114.2, 113.9, 59.4, 55.9, 53.0, 29.6, 11.0; IR (film, cm^{-1}) 3387, 2932, 1708, 1590, 1513, 1446, 1241, 1038; HRMS *m/z* calcd. for $C_{18}H_{20}O_2NF$: 301.1472; found 301.1472 (*anti*): ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.22–7.29 (m, 1H), 7.03–7.07 (m, 1H), 6.95–7.00 (m, 1H), 6.87–6.93 (m, 1H), 6.63–6.68 (m, 2H), 6.42–6.47 (m, 2H), 4.38 (d, 1H, J = 7.6 Hz), 3.66 (s, 3H), 2.86–2.94 (m, 1H), 2.02 (s, 3H), 1.09 (d, 3H, J = 6.8 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 212.2, 164.6, 152.4, 144.7, 140.8, 130.4(2), 122.8(2), 115.0(2), 114.7, 114.5, 114.0, 113.8, 61.3, 55.9, 53.6, 29.4, 15.3; IR (film, cm^{-1}) 3360, 2931, 1707, 1590, 1513, 1449, 1241, 1037; HRMS *m/z* calcd. for $C_{18}H_{20}O_2NF$: 301.1472; found 301.1472.

4.4.7. 1-(4-Chlorophenyl)-1-(p-methoxyphenylamino)-2-methyl-3-butanone (syn)

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.12–7.23 (m, 5H), 6.56–6.63 (m, 2H), 6.32–6.40 (m, 2H), 4.56 (d, 1H, J = 5.6 Hz), 3.61 (s, 3H), 2.85–2.93 (m, 1H), 2.04 (s, 3H), 1.00 (d, 3H, J = 7.2); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 209.5, 151.3, 139.7, 139.1, 131.8, 127.8, 127.2, 113.8, 113.7, 58.0, 54.6, 52.4, 28.1, 14.1; IR (film, cm^{-1}) 3396, 2932, 1709, 1622, 1510, 1245, 1178, 1091, 1035; HRMS *m/z* calcd. for $C_{18}H_{20}O_2NCl$: 317.1177; found 317.1178 (*anti*): ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.13–7.24 (m, 5H), 6.55–6.62 (m, 2H), 6.31–6.40 (m, 2H), 4.32 (d, 1H, J = 7.6 Hz), 3.61 (s, 3H), 2.84–2.94 (m, 1H), 2.00 (s, 3H), 1.03 (d, 3H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 211.0, 151.2, 139.6, 138.8, 132.0, 127.8, 127.3, 113.8, 113.7, 59.8, 51.8, 28.4, 9.9; IR (film, cm^{-1}) 3423, 2931, 1709, 1621, 1507, 1253, 1093, 1032; HRMS *m/z* calcd. for $C_{18}H_{20}O_2NCl$: 317.1177; found 317.1178.

4.4.8. 1-(4-Bromophenyl)-1-(p-methoxyphenylamino)-2-methyl-3-butanone (syn)

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.32–7.39 (m, 2H), 7.10–7.15 (m, 2H), 6.58–6.64 (m, 2H), 6.39–6.47 (m, 2H), 4.60 (d, 1H, J = 5.6 Hz), 3.66 (s, 3H), 2.90–2.99 (m, 1H), 2.10 (s, 3H), 1.05 (d, 3H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 212.3, 152.5, 140.9, 140.6, 132.0, 129.0, 121.4, 115.5, 115.0, 59.3, 55.8, 53.6, 29.7, 11; IR (film, cm^{-1}) 3393, 2932, 1707, 1512, 1358, 1243, 1177, 1036, 1009; HRMS *m/z* calcd. for $C_{18}H_{20}O_2NBr$: 362.0750 (M + H); found 362.0749 (*anti*): ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.33–7.39 (m, 2H), 7.09–7.14 (m, 2H), 6.57–6.64 (m, 2H), 6.38–6.48 (m, 2H), 4.30 (d, 1H, J = 7.6 Hz), 3.61 (s, 3H), 2.85–3.00 (m, 1H), 2.01 (s, 3H), 1.03 (d, 3H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 210.7, 152.4, 140.9, 140.8, 132.0, 129.1, 121.6, 115.0, 114.9, 61.2, 55.9, 53.3, 29.4, 15.3; IR (film, cm^{-1}) 3364, 2931, 1705, 1512, 1357, 1239, 1176, 1036, 1009; HRMS *m/z* calcd. for $C_{18}H_{20}O_2NBr$: 362.0750 (M + H); found 362.0749.

4.4.9. 1-(4-Methoxyphenyl)-1-(p-methoxyphenylamino)-2-methyl-3-butanone (syn)

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.17–7.21 (m, 2H), 6.80–6.84 (m, 2H), 6.62–6.67 (m, 2H), 6.41–6.46 (m, 2H), 4.58 (d, 1H, J = 5.6 Hz), 3.76 (s, 3H), 3.66 (s, 3H), 2.90–3.00 (m, 1H), 2.06 (s, 3H), 1.06(d, 3H, J = 6.8 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 211.3, 158.9, 152.3, 141.3, 133.3, 128.1, 115.0, 114.9, 114.2, 59.4, 55.4, 53.5, 29.9, 29.7, 11.4; IR (film, cm^{-1}) 3383, 2929, 1707, 1607, 1511, 1247, 1177, 1031; HRMS *m/z* calcd. for $C_{19}H_{23}O_3N$:. 313.1672; found 313.1671 (*anti*): ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.09–7.15 (m, 2H), 6.74–6.79 (m, 2H), 6.56–6.62 (m, 2H), 6.38–6.43 (m, 2H), 4.28 (d, 1H, J = 8.0 Hz), 3.70 (s, 3H), 3.61 (s, 3H), 2.77–2.86 (m, 1H),

1.97 (s, 3H), 1.05 (d, 3H, $J = 6.8$ Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 212.8, 159.0, 152.3, 141.1, 133.5, 128.1, 115.1, 114.9, 114.2, 61.1, 55.9, 54.2, 29.9, 29.1, 15.3; IR (film, cm^{-1}) 3433, 2923, 1709, 1622, 1511, 1364, 1251, 1029; HRMS *m/z* calcd. for $C_{19}H_{23}O_3N$:. 313.1672; found 313.1671.

4.4.10. 1-(o-Methyl)-1-(p-methoxyphenylamino)-2-methyl-3-butanone (syn)

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.28–7.32 (m, 1H), 7.08–7.18 (m, 3H), 6.61–6.66 (m, 2H), 6.34–6.38 (m, 2H), 4.88 (d, 1H, $J = 4.4$ Hz), 3.65 (s, 3H), 2.92–3.01 (m, 1H), 2.47 (s, 3H), 2.17 (s, 3H), 1.04 (d, 3H, $J = 7.2$ Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 210.7, 152.4, 141.4, 139.1, 134.6, 131.2, 127.3, 126.8, 126.5, 114.9, 114.8, 55.9, 55.7, 50.7, 29.4, 19.4, 10.4; IR (film, cm^{-1}) 3396, 2930, 1708, 1512, 1463, 1357, 1243, 1179, 1037; HRMS *m/z* calcd. for $C_{19}H_{23}O_2N$: Calcd. 297.1723; found 297.1722 (*anti*): ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.18–7.23 (m, 1H), 7.07–7.15 (m, 3H), 6.61–6.66 (m, 2H), 6.37–6.43 (m, 2H), 4.63 (d, 1H, $J = 7.2$ Hz), 3.65 (s, 3H), 2.93–3.01 (m, 1H), 2.43 (s, 3H), 1.97 (s, 3H), 1.10 (d, 3H, $J = 7.2$ Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 213.1, 152.3, 141.3, 139.1, 135.4, 130.9, 127.3, 126.8, 125.9, 115.2, 114.9, 57.9, 55.9, 52.5, 30.3, 29.9, 19.7, 15.1; IR (film, cm^{-1}) 3405, 2926, 1708, 1620, 1506, 1461, 1244, 1035; HRMS *m/z* calcd. for $C_{19}H_{23}O_2N$: Calcd. 297.1723; found 297.1722.

4.4.11. 1-(2-Naphthyl)-1-(p-methoxyphenylamino)-2-methyl-3-butanone (syn)

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.74–7.82 (m, 4H), 7.41–7.47 (m, 3H), 6.60–6.66 (m, 2H), 6.44–6.49 (m, 2H), 4.82 (d, 1H, $J = 5.2$ Hz), 3.64 (s, 3H), 3.05–3.14 (m, 1H), 2.12 (s, 3H), 1.09 (d, 3H, $J = 7.2$ Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 211.0, 152.4, 141.3, 139.0, 133.6, 133.0, 128.7, 128.1, 127.9, 126.4, 126.1, 126.0, 125.0, 115.1, 114.9, 60.0, 55.9, 53.2,29.6, 11.1; IR (film, cm^{-1}) 3396, 2932, 1709, 1511, 1359, 1243, 1178, 1036; HRMS *m/z* calcd. for $C_{22}H_{23}O_2N$: 334.1801 (M + H); Found 334.1799 (*anti*): ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 7.71–7.81 (m, 4H), 7.38–7.47 (m, 3H), 6.59–6.65 (m, 2H), 6.47–6.52 (m, 2H), 4.54 (d, 1H, $J = 7.6$ Hz), 3.63 (s, 3H), 2.98–3.06 (m, 1H), 2.03 (s, 3H), 1.10 (d, 3H, $J = 7.2$ Hz); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 212.6, 152.4, 141.1, 139.1, 128.9, 128.1, 127.9, 126.4, 126.3, 126.1, 124.7, 115.2, 114.9, 62.0, 55.9, 53.8, 29.3, 15.4; IR (film, cm^{-1}) 2390, 2935, 2832, 1697, 1613, 1504, 1453, 1350, 1228, 1170, 1029; HRMS *m/z* calcd. for $C_{22}H_{23}O_2N$: 334.1801 (M + H); Found 334.1799.

Acknowledgements

We thank the NSF (0092001) and NSF-EPA Joint Program for a Sustainable Environment for partial support of our research. We are also grateful to Dr. Luc Moens of National Renewable Energy Laboratory for providing us with part of the ionic liquid.

References

[1] March Advanced Organic Chemistry: Reactions, Mechanisms and Structure, fourth ed., Wiley-Interscience, New York, 1992.
[2] R. Bloch, Chem. Rev. 98 (1998) 1407, and references cited therein.
[3] (a) T. Mukaiyama, Org. React. 28 (1982) 203;
(b) T.K. Hollis, B.J. Bosnich, Am. Chem. Soc. 117 (1995) 4570, for the mechanism of Mukaiyama-aldol reaction, see;
S.E. Denmark, W.J. Lee, Org. Chem. 59 (1994) 707.
[4] (a) S. Kobayashi, H. Ishitani, Chem. Rev. 99 (1999) 1069, and references cited therein;
(b) K. Ishihara, M. Miyata, K. Hattori, T. Tada, J. Yamamoto, J. Am. Chem. Soc. 116 (1994) 10520;
(c) S.J. Danishefsky, S. Chackalamannil, P. Harrision, M. Silvestri, P.J. Cole, Am. Chem. Soc. 107 (1985) 2474;
(d) P.G. Cozzi, B. Di Simone, A. Umani-Ronichi, Tetrahedron Lett. 37 (1996) 1691;
(e) G. Cainelli, M. Contento, A. Drusiani, M. Panunzio, L.J. Plessi, Chem. Soc., Chem. Commun. (1985) 240.
[5] B.M. Trost, S.J. Oi, Am. Chem. Soc. 123 (2001) 1230.
[6] (a) S. Sato, I. Matsuda, M.J. Shibata, Organomet. Chem. 377 (1989) 347;
(b) I. Matsuda, M. Shibata, S.J. Sato, Organomet. Chem. 340 (1988) C5. For use of Rh-enolate intermediates in aldol reactions, see;
(c) G.A. Slough, R.G. Bergman, C.H.J. Heathcock, Am. Chem. Soc. 111 (1989) 938;
(d) M.T. Reetz, A.E. Vougioukas, Tetrahedron Lett. 28 (1987) 793;
(e) S. Sato, I. Matsuda, Y. Izumi, Tetrahedron Lett. 27 (1986) 5517.
[7] (a) B.M. Trost, E.R. Silcoff, H. Ito, Org. Lett. 3 (2001) 2497. For examples, see;
(b) S. Murahashi, H. Takaya, Acc. Chem. Res. 33 (2000) 225;
(c) Y.R. Lin, X.T. Zhou, L.X. Dai, J.J. Sun, Org. Chem. 62 (1997) 1799;
(d) S.I. Murahashi, T. Naota, H. Taki, M. Mizuno, H. Takaya, S. Komiya, Y. Mizuho, N. Oyasato, M. Hiraoka, M. Hirano, A.J. Fukuoka, Am. Chem. Soc. 117 (1995) 12436;
(e) M. Picquet, C. Bruneau, P.M. Dixneuf, Tetrahedron 55 (1999) 3937;
(f) E. Gómez-Bengoa, J.M. Cuerva, C. Mateo, A.M. Echavarren, J. Am. Chem. Soc. 118 (1996) 8553.
[8] L.J. Gazzard, W.B. Motherwell, D.A.J. Sandham, Chem. Soc., Perkin Trans. 1 (1999) 979.
[9] (a) C. Crévisy, M. Wietrich, V. Le Boulaire, R. Umea, R. Grée, Tetrahedron Lett. 42 (2001) 395;
(b) R. Uma, M. Davies, C. Crévisy, R. Grée, Tetrahedron Lett. 42 (2001) 3069.
[10] (a) M.A. Abraham, L. Moens, (Eds.), Clean Solvents: Alternative Media for Chemical Reactions and Processing, ACS Symposium Series 819, American Chemical Society, Washington, DC, 2002;
(b) C.J. Li, T.H. Chan, Organic Reactions in Aqueous Media, Wiley, New York, 1997;
(c) P.A. Grieco (Ed.), Organic Synthesis in Water, Blackie Academic and Professional, Glasgow, 1998.
[11] T.H. Chan, C.J. Li, Z.Y. Wei, J. Chem. Soc., Chem. Commun. (1990) 505.
[12] A.J. Lubineau, Org. Chem. 51 (1986) 2142.
[13] S. Kobayashi, I.J. Hachiya, Org. Chem. 59 (1994) 3590.
[14] T.P. Loh, J. Pei, G.Q. Cao, J. Chem. Soc., Chem. Commun. (1996) 1819.
[15] H.Y. Tian, Y.J. Chen, D. Wang, C.C. Zeng, C.J. Li, Tetrahedron Lett. 41 (2000) 2529.

[16] (a) S. Kobayashi, K. Manabe, Acc. Chem. Res. 35 (2002) 209, and references cited therein;
(b) T. Akiyama, J. Takaya, H. Kagoshima, Tetrahedron Lett. 42 (2001) 4025.
[17] (a) R.D. Rogers, K.R. Seddon (Eds.), Ionic Liquids: Industrial Application for Green Chemistry, in: Proceedings of a Symposium, San Diego, CA, 1–5 April 2001, ACS Symp. Ser. 2002, p. 474;
(b) T. Welton, Chem Rev. 100 (2000) 2071;
(c) P. Wasserscheid, W. Keim, Angew. Chem. Int. Ed. Engl. 39 (2000) 3772.
[18] J.G. Huddleston, H.D. Willauer, R.P. Swatloski, A.E. Visser, R.D. Rogers, Chem. Commun. 1998, p. 1765.
[19] (a) C.J. Li, D. Wang, D.L. Chen, J. Am. Chem. Soc. 117 (1995) 12867;
(b) D. Wang, D.L. Chen, J.X. Haberman, C.J. Li, Tetrahedron 54 (1998) 5129;
(c) D. Wang, C.J. Li, Synth. Commun. 28 (1998) 507.
[20] (a) J.F. Hartwig, R.G. Bergman, R.A. Anderson, Organometallics 10 (1991) 3326. For other examples of Ru enolates, see;
(b) B.T. Rasley, M. Rapta, R.J. Kulawiec, Organometallics 15 (1996) 2852;
(c) S. Chang, Y. Na, E. Choi, S. Kim, Org. Lett. 3 (2001) 2089;
(d) B.M. Trost, F.D. Toste, A.B. Pinkerton, Chem. Rev. 101 (2001) 2067. For a review of ruthenium-catalyzed non-metathesis reactions, see.
[21] (a) B.M. Trost, A.B.J. Pinkerton, Am. Chem. Soc. 122 (2000) 8081. Previously, Ru_enol intermediates have been captured by aldehydes in a Michael addition reaction;
(b) O.J. Fujimura, Am. Chem. Soc. 120 (1998) 10032. For capturing of Pd and Pt enolates by aldehydes, see;
(c) M. Sodeoka, K. Ohrai, M.J. Shibasaki, Org. Chem. 60 (1995) 2648.
[22] M. Wang, C.J. Li, Tetrahedron Lett. 43 (2002) 3589.
[23] (a) Wang, M., Yang, X.-F., Li, C.-J., Eur. J. Org. Chem. 2003, 998.;
(b) X.-F. Yang, M. Wang, C.-J. Li, Org. Lett. 5 (2003) 657.
[24] S. Steines, B. Drieben-Holscher, P.J. Wasserscheid, Prakt. Chem. 342 (2000) 348.
[25] Song, C.E., Roh, E.J., Chem. Commun. 2000, 837.
[26] Chauvin, Y., Oliver, H., Mubamann, L. FR 95/14,147, 1995 Chem. Abstr. 1997, 127, P341298k.
[27] W. Chen, L. Xu, C. Chatterton, J. Xiao, Chem. Commun. (1999) 1247.