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Rhodium-catalyzed hydroaroylation of α,β-unsaturated esters using aroyl chlorides and Et_2MeSiH

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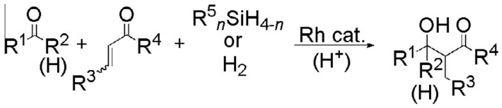
ABSTRACT

Hydroaroylation of methyl acrylate **2a** to give the α-aroyl esters **4** took place in the three-component reaction of **2a**, aroyl chlorides **1**, and Et_2MeSiH in the presence of 1 mol % of $[Rh(cod)(PR_3)_2]OTf$ (cod = 1,5-cyclooctadiene, OTf = OSO_2CF_3, R = Ph (**3a**), OPh (**3b**)) in CH_2Cl_2. GC and ^1H NMR investigation revealed that the rhodium-catalyzed hydroaroylation proceeds via two successive transformations, that is, hydrosilylation of **2a** to afford silyl enol ether **5a** followed by C–C bond formation between **5a** and **1a**.

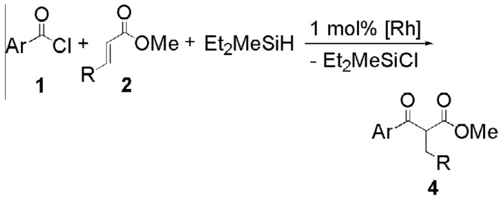
Introduction

Transition-metal catalyzed multicomponent reactions (MCRs) involving C–C bond formation have been recognized as versatile procedures for constructing the polyfunctional organic molecules.[1] Neutral and cationic rhodium(I) complexes with monodentate or bidentate phosphines as spectator ligands are widely investigated as the catalyst for MCRs.[2,3] In particular, $RhX(PR_3)_n$ and $[Rh(diene)(PR_3)_2]^+$ types of complexes have been used for catalytic, three-component reductive aldol condensations with aldehydes or ketones, α,β-unsaturated carbonyl compounds, and reducing reagents such as hydrosilanes or hydrogen gas (Scheme 1).[4,5] The rhodium-catalyzed reactions with hydrosilanes proceed via the in situ generation of enolate species which attacks aldehydes or ketones to give the C–C bond formation products. Thus, the three-component systems have significant advantages over the conventional two-component aldol reactions since, in the latter case, the enolate species should be prepared independently from carbonyl compounds using a stoichiometric amount of strong bases.

Development of the Rh-catalyzed systems applicable to a variety of electrophiles instead of aldehydes and ketones would provide efficient synthetic procedures for carbonyl compounds, however, such examples are limited to only an aldimine and a ketimine as the electrophiles.[6] We and co-workers have investigated on rhodium-catalyzed three-component couplings of electrophiles (allyl carbonates, aryl isocyanates, and aldimines), α,β-unsaturated carbonyl compounds, and hydrosilanes.[6a,7] In these reactions, functionalized carbonyl compounds, such as γ,δ-unsaturated-, α-amido-, and β-amino carbonyls, are selectively produced owing to preferential enolate generation followed by selective C–C bond formation with electrophiles. During the course of the study, we found that aroyl chloride **1** is applicable as an electrophile to the rhodium-catalyzed three-component reaction system. Aroyl chlorides have been known as efficient substrates for introducing a keto group to aromatic substrates electrophilically, that is, Lewis acid induced/catalyzed Friedel–Crafts acylation.[8] Aroyl chlorides have also been used for palladium-catalyzed cross-couplings with organometallic reagents, including organoboron, organotin, and organozinc reagents, to afford aromatic ketones.[9] However, the organometallic reagents required for the catalytic cross-coupling



Scheme 1. Catalytic reductive aldol condensations.



Scheme 2. Rh-catalyzed hydroaroylation of α,β-unsaturated esters **2** with aroyl chlorides **1** and Et_2MeSiH.

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systems are not readily available and also more than stoichiometric amounts of organometallic reagents should be employed to complete the reaction. Except for the aforementioned palladium-catalyzed reactions, only a few examples have been reported on the transition-metal catalyzed transformation using aroyl chlorides as a source of the keto functional group.[10] We report here the rhodium-catalyzed three-component reaction of aroyl chloride **1**, α,β-unsaturated carbonyl compound **2** and Et_2MeSiH to afford β-keto (α-aroyl) ester **4** in high yield (Scheme 2). This reaction can be categorized as a formal hydroaroylation of **2** in which the α-aroyl ester **4** is formed via regioselective introduction of hydride and aroyl groups to the β- and α-positions of **2**, respectively.

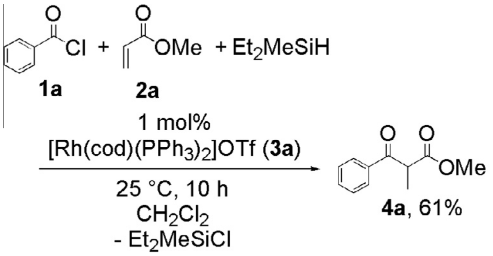
Table 1
Rhodium-catalyzed hydroaroylation of **2a** with **1a** and Et_2MeSiH[a]

Entry	Precatalyst	Temp.	Time	Yield of **4a**[b]
		°C	h	(%)
1	3a	25	10	61
2	3a	reflux	5	72
3	3b	25	10	74
4	3b	reflux	5	84

[a] A CH_2Cl_2 solution of benzoyl chloride **1a**, **2a**, Et_2MeSiH, and 1 mol% of **3a** or **3b** was stirred at 25 °C for 10 h or heated at reflux for 5 h.
[b] Isolated yield.

Results and discussion

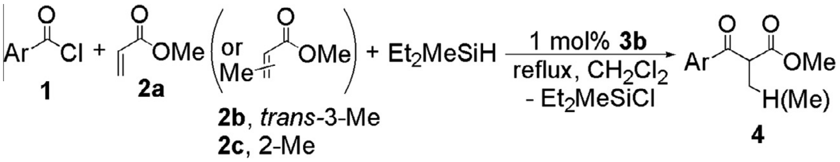
Treatment of benzoyl chloride **1a**, methyl acrylate **2a**, and Et_2MeSiH in CH_2Cl_2 in the presence of 1 mol % of $[Rh(cod)(PPh_3)_2]OTf$ (**3a**, cod = 1,5-cyclooctadiene, OTf = OSO_2CF_3) at 25 °C for 10 h afforded α-aroyl ester **4a**[11] in 61% yield with a concomitant formation of $Et_2MeSiCl$ (Eq. 1). The yield of **4a** slightly increased to 74% when the Rh complex was changed from **3a** to its $P(OPh)_3$ analogue, $[Rh(cod)\{P(OPh)_3\}_2]OTf$ (**3b**) (entry 3 in Table 1), and reached 84% under reflux for 5 h (entry 4 in Table 1).

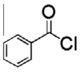
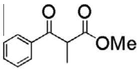
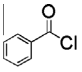
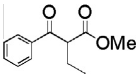
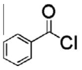
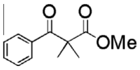
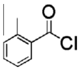
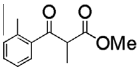
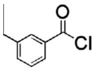
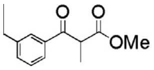
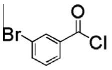
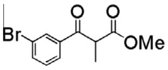
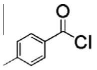
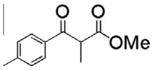
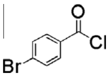
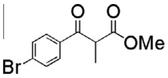
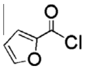
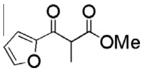
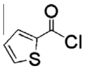
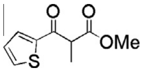


(1)

The scope of the hydroaroylation was explored using **3b** as catalyst, aroyl chloride **1**, α,β-unsaturated ester **2**, and Et_2MeSiH (Table 2). Hydroaroylation to methyl crotonate **2b** was sluggish owing to its β-Me substituent and the corresponding product

Table 2
Scope of the rhodium-catalyzed hydroaroylation[a]



Entry	ArCOCl (1)		2	Product 4		time h	yield[b] (%)
1		1a	2a		4a[11]	5	84
2		1a	2b		4a'[12]	20	35
3		1a	2c		4a''[13]	24	3
4		1b	2a		4b[11b]	6	50
5		1c	2a		4c[11b]	6 16	81 93
6		1d	2a		4d	3	86
7		1e	2a		4e[11b,14]	13	85
8		1f	2a		4f[11b]	5	86
9		1g	2a		4g[15]	6	67
10		1h	2a		4h[16]	10	86

[a] A CH_2Cl_2 solution of aroyl chloride **1**, **2a** (or **2b**), Et_2MeSiH, and 1 mol% of **3b** was refluxed for appropriate hours described in each entry.
[b] Isolated yield.

4a′[12] was formed in low yield after prolonged heating (35%, entry 2). Methyl group at the α-position in α,β-unsaturated ester hampered C–C bond formation. Thus the catalytic hydroaroylation with methyl methacrylate **2c** provided almost no coupling product (entry 3). Steric hindrance around the C(=O)Cl functionality in aroyl chloride **1** also influenced the progress of hydroaroylation. Catalytic reaction with *o*-toluoyl chloride **1b** gave hydroaroylation product **4b** in a modest yield after a mixture of **1b**, **2a**, and Et$_2$MeSiH with 1 mol % of **3b** was refluxed for 6 h (entry 4). In contrast to this result, aroyl chlorides with methyl and bromo substituents on *meta*- and *para*-positions **1c**–**1f** were successfully converted into corresponding α-aroyl esters **4c**–**4f** under usual conditions (entries 5–8). Hetero aroyl chlorides, 2-furoyl chloride **1g**, and 2-thioyl chloride **1h**, were also transformed into the corresponding α-aroyl esters **4g** and **4h**, respectively, in high yields (entries 9 and 10).

Rhodium-catalyzed reductive aldol reactions have been proposed to proceed via nucleophilic attack of the in situ generated Rh-enolates to aldehydes and ketones.[17] To explore the reaction mechanism, **3b**-catalyzed hydroaroylation of **2a** with benzoyl chloride **1a** and Et$_2$MeSiH was monitored by gas chromatography (GC) and ^{1}H NMR spectroscopy. The reaction profiles are summarized in Figure 1. Formation of the silyl enol ether **5a**,[7b] along with a small amount of the final product **4a**, was observed immediately after mixing of substrates and the catalyst. After 1 h, almost all of **2a** and Et$_2$MeSiH were converted into **5a**. The yield of **4a** gradually increased and reached 86% after 12 h along with consumption of **1a** and **5a**. Without the Rh complex, neither the reaction of the three components nor that between isolated **5a**[7b] and **1a** took place. These observations revealed that the Rh-catalyzed hydroaroylation proceeds via two successive transformations, i.e., Rh-catalyzed generation of the silyl enol ether **5a**[7b] by 1,4-hydrosilylation of **2a** with Et$_2$MeSiH followed by C–C bond formation between resulting silyl enol ether **5a** and **1** by the catalysis of the rhodium complex.

A possible reaction mechanism for Rh-catalyzed hydroaroylation of **2a** with aroyl chloride **1** and Et$_2$MeSiH is shown in Scheme 3. The reaction starts from the oxidative addition of Et$_2$MeSiH to [Rh] complex to give complex **A**. The insertion of **2a** to the Rh-H or/and Rh-SiEt$_2$Me bond in **A** results in the formation of **B** or/and **B′**. Isomerization to **C** or/and **C′** followed by reductive elimination of **5a** regenerates [Rh] species. It is well established that both silyl enol ethers and aroyl chlorides could be activated by rhodium complex.[18,19] Thus the [Rh] species activates both aroyl chloride **1** and silyl enol ether **5a** for the C–C bond forming reaction to take place, which affords **4** and Et$_2$MeSiCl as the final products.

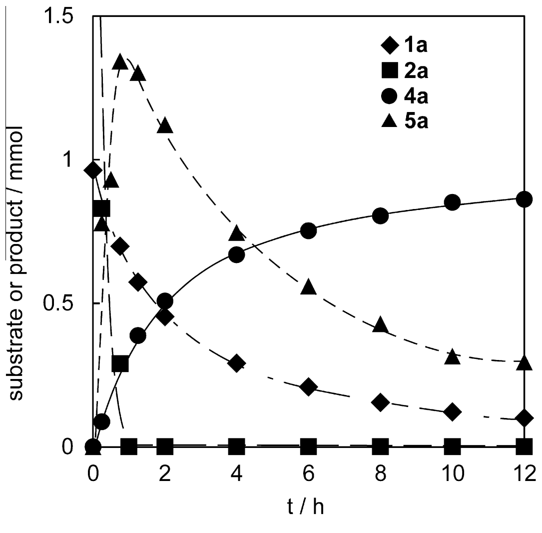
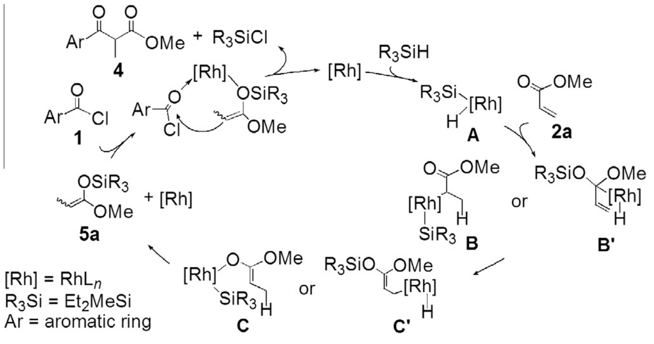


Figure 1. The reaction profile for the hydroaroylation of **2a** (2.1 mmol, ■) with **1a** (0.96 mmol, ◆) and Et$_2$MeSiH (2.0 mmol, not shown) in the presence of **3b** (0.0096 mmol, not shown) in CH$_2$Cl$_2$ under reflux condition. The yield of **4a** (●) reached 0.83 mmol (86%) via the initial formation of the intermediate **5a** (▲). The data of **2a** at 0 min were omitted for clarity.



Scheme 3. A possible reaction mechanism of Rh-catalyzed hydroaroylation.

Acknowledgment

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Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.tetlet.2013.06.009.

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2001, *3*, 1829; (f) Duffey, M. O.; LeTiran, A.; Morken, J. P. *J. Am. Chem. Soc.* **2003**, *125*, 1458; (g) Chiu, P. *Synthesis* **2004**, 2210; (h) Chiu, P.; Leung, S. K. *Chem. Commun.* **2004**, 2308; (i) Lam, H. W.; Joensuu, P. M. *Org. Lett.* **2005**, *7*, 4225; (j) Deschamp, J.; Chuzel, O.; Hannedouche, J.; Riant, O. *Angew. Chem., Int. Ed.* **2006**, *45*, 1292; (k) Chuzel, O.; Deschamp, J.; Chausteur, C.; Riant, O. *Org. Lett.* **2006**, *8*, 5943; (l) Welle, A.; Diez-Gonzalez, S.; Tinant, B.; Nolan, S. P.; Riant, O. *Org. Lett.* **2006**, *8*, 6059; (m) Lipshutz, B. H.; Amorelli, B.; Unger, J. B. *J. Am. Chem. Soc.* **2008**, *130*, 14378; (n) Deschamp, J.; Riant, O. *Org. Lett.* **2009**, *11*, 1217; (o) Kato, M.; Oki, H.; Ogata, K.; Fukuzawa, S. *Synlett* **2009**, 1299.

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