ORGANIC LETTERS

2007
Vol. 9, No. 6
1013−1016

N-Heterocyclic Carbene-Catalyzed Mukaiyama Aldol Reactions

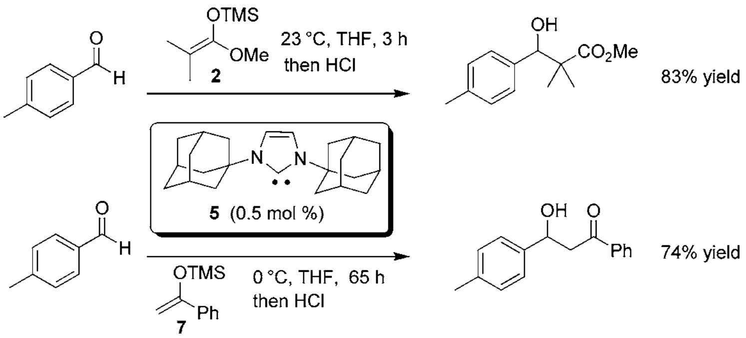
Jinhua J. Song,* Zhulin Tan, Jonathan T. Reeves, Nathan K. Yee, and Chris H. Senanayake

Department of Chemical Development, Boehringer Ingelheim Pharmaceuticals, Inc.,
900 Old Ridgebury Road/P.O. Box 368, Ridgefield, Connecticut 06877-0368

jsong@rdg.boehringer-ingelheim.com

Received December 17, 2006

ABSTRACT



N-Heterocyclic carbenes were shown to be highly effective catalysts to promote Mukaiyama aldol reactions. In the presence of only 0.5 mol % of N-heterocyclic carbene (5), various aldehydes and 2,2,2-trifluoroacetophenone underwent Mukaiyama aldol reactions in THF with trimethylsilyl ketene acetal (2) at 23 °C as well as with trimethylsilyl enol ether (7) at 0 °C to afford aldol adducts in good yields. These conditions are extremely mild and operationally simple and tolerate various functional groups.

The use of N-heterocyclic carbenes (NHCs) as organocatalysts has attracted considerable attention in recent years.[1] NHCs have been employed to catalyze organic reactions such as nucleophilic substitutions,[2] benzoin and Stetter-type reactions,[3] homoenolate formations,[4] Diels−Alder reactions,[5] formylcyclopropane ring opening,[6] and redox processes.[7] It is believed that the common step in these transformations involves initial reaction of the NHC with aldehydes to form the "Breslow intermediate" which can lead into various subsequent reaction pathways. NHCs were also used for trimerization of isocyanates[8] and transesterification reactions,[9] for which the intermediacy of an NHC−acyl species was hypothesized. For a reported NHC-catalyzed amide bond formation reaction, it was proposed that the NHC acted as a carbon-centered Brønsted base.[10] NHCs were also found to catalyze an intramolecular alkylation reaction.[11]

Recently, we have discovered a new mode of reactivity for NHCs where certain silicon−carbon bonds, such as in TMSCN and $TMSCF_3$, could be effectively activated by

---

(1) (a) Enders, D.; Balensiefer, T. *Acc. Chem. Res.* **2004**, *37*, 534. (b) Dalko, P. I.; Moisan, L. *Angew. Chem., Int. Ed.* **2004**, *43*, 5138. (c) Nair, V.; Bindu, S.; Sreekumar, V. *Angew. Chem., Int. Ed.* **2004**, *43*, 5130.

(2) (a) Suzuki, Y.; Toyota, T.; Imada, F.; Sato, M.; Miyashita, A. *Chem. Commun.* **2003**, 1314. (b) Miyashita, A.; Suzuki, Y.; Iwamoto, K.-I.; Oishi, E.; Higashino, T. *Heterocycles* **1998**, *49*, 405. (c) Miyashita, A.; Suzuki, Y.; Iwamoto, K.-I.; Higashino, T. *Chem. Pharm. Bull.* **1998**, *46*, 390.

(3) (a) Enders, D.; Niemeier, O.; Raabe, G. *Synlett* **2006**, 2431. (b) Kerr, M. S.; Read de Alaniz, J.; Rovis, T. *J. Am. Chem. Soc.* **2002**, *124*, 10298. (c) Enders, D.; Breuer, K.; Runsink, J.; Teles, J. H. *Helv. Chim. Acta* **1996**, *79*, 1899. (d) Enders, D.; Breuer, K.; Teles, J. H. *Helv. Chim. Acta* **1996**, *79*, 1217. (e) Teles, J. H.; Melder, J.-P.; Ebel, K.; Schneider, R.; Gehrer, E.; Harder, W.; Brode, S.; Enders, D.; Breuer, K.; Raabe, G. *Helv. Chim. Acta* **1996**, *79*, 61. (f) Myers, M. C.; Bharadwaj, A. R.; Milgram, B. C.; Scheidt, K. A. *J. Am. Chem. Soc.* **2005**, *127*, 14675. (g) Kerr, M. S.; Rovis, T. *J. Am. Chem. Soc.* **2004**, *126*, 8876. (h) Chow, K. Y. K.; Bode, J. W. *J. Am. Chem. Soc.* **2004**, *126*, 8126. (i) Mattson, A. E.; Bharadwaj, A. R.; Zuhl, A. M.; Scheidt, K. A. *J. Org. Chem.* **2006**, *71*, 5715. (j) Myers, M. C.; Bharadwaj, A. R.; Milgram, B. C.; Scheidt, K. A. *J. Am. Chem. Soc.* **2005**, *127*, 14675. (k) Enders, D.; Niemeier, O.; Balensiefer, T. *Angew. Chem., Int. Ed.* **2006**, *45*, 1463. (l) Enders, D.; Kallfass, U. *Angew. Chem., Int. Ed.* **2002**, *41*, 1743. (m) Moore, J. L.; Kerr, M. S.; Rovis, T. *Tetrahedron* **2006**, *62*, 11477. (n) Liu, Q.; Rovis, T. *J. Am. Chem. Soc.* **2006**, *128*, 2552. (o) Read de Alaniz, J.; Rovis, T. *J. Am. Chem. Soc.* **2005**, *127*, 6284.

10.1021/ol0630494 CCC: $37.00

NHCs for nucleophilic addition reactions. In this context, we have described an efficient NHC-catalyzed trifluoromethyl transfer reaction from TMSCF$_3$ to carbonyl compounds[12] as well as an NHC-catalyzed cyanation reaction between TMSCN and carbonyl compounds using very low catalyst loadings (0.01−1 mol %).[13] Analogous NHC-catalyzed cyanation reactions were also independently demonstrated by three other research groups.[14] More recently, an interesting NHC-catalyzed aziridine ring-opening reaction with silylated nucleophiles was reported.[15] As part of our ongoing research program on NHC catalysis, we now wish to report that NHCs are also capable of catalyzing Mukaiyama aldol reactions with as low as 0.05−0.5 mol % catalyst loadings.

The Mukaiyama aldol reaction, i.e., reaction of an enoxysilane with a carbonyl compound, is one of the most fundamental organic transformations and has been the subject of intensive investigations for the past three decades.[16] The Mukaiyama aldol reaction can be catalyzed either by Lewis acids via activation of the electrophiles (i.e., carbonyl compounds) or by Lewis bases via activation of the nucleophiles (i.e., enoxysilanes, Figure 1). For example, reactions between enoxysilanes and aldehydes could be catalyzed by fluoride,[17] phosphines,[18] CaCl$_2$,[19] lithium amides,[20] lithium acetate,[21] lithium alkoxides,[22] *N*-oxides,[23] or *N*-methylimidazole[24] with 5−20 mol % catalyst loadings. It was also reported that Mukaiyama aldol reactions could proceed without added catalysts in some highly polar solvents such as DMF, DMSO, ionic liquids, water, or DBU.[25] Denmark has shown that Mukaiyama aldol reactions using the more reactive enoxytrichlorosilanes could be effectively catalyzed by phosphoramides or *N*-oxides.[26] It is generally accepted that, for these Lewis base catalyzed Mukaiyama aldol reactions, the key step involves the activation of Si−O bonds through interaction of Si with Lewis basic heteroatom centers (e.g., N, O, or P, etc.).[27]

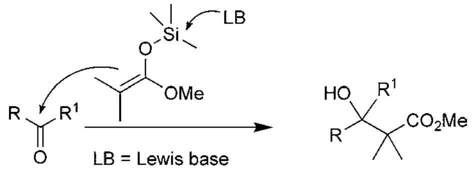


Figure 1. Lewis base catalyzed Mukaiyama aldol reactions.

As part of our efforts to expand the scope of NHC catalysis, we proposed that NHCs could potentially be utilized as carbon-centered nucleophilic catalysts for activating the Si−O bonds in enoxysilanes, thereby catalyzing Mukaiyama aldol reactions. As a test case, we carried out a series of reactions between 2-naphthaldehyde (**1a**) and trimethylsilyl ketene acetal **2** (1.2 equiv) in THF at 23 °C by using four readily available NHC catalysts (**3**, **4a**, **4b**, and **5**, Table 1). We were glad to find that, in the presence of 0.5 mol % of 1,3-di-*tert*-butylimidazol-2-ylidene (**3**), the desired aldol adduct **6a**[28] was obtained in 78% yield (after desilylation) within 3 h at 23 °C (entry 1). Aryl-substituted imidazol-2-ylidenes were found to be less effective for this reaction. For example, reaction using 1,3-bis(2,6-diisopro-

---

(4) (a) Sohn, S. S.; Rosen, E. L.; Bode, J. W. *J. Am. Chem. Soc*. **2004**, *126*, 14370. (b) Burstein, C.; Glorius, F. *Angew. Chem., Int. Ed*. **2004**, *43*, 6205. (c) Chan, A.; Scheidt, K. A. *Org. Lett*. **2005**, *7*, 905. (d) Burstein, C.; Tschan, S.; Xie, X.; Glorius, F. *Synthesis* **2006**, 2418. (e) Nair, V.; Vellalath, S.; Poonoth, M.; Mohan, R.; Suresh, E. *Org. Lett*. **2006**, *8*, 507. (f) Nair, V.; Vellalath, S.; Poonoth, M.; Suresh, E. *J. Am. Chem. Soc*. **2006**, *128*, 8736. (g) He, M.; Bode, J. W. *Org. Lett*. **2005**, *7*, 3131.

(5) (a) He, M.; Struble, J. R.; Bode, J. W. *J. Am. Chem. Soc*. **2006**, *128*, 8418. (b) He, M.; Uc, G. J.; Bode, J. W. *J. Am. Chem. Soc*. **2006**, *128*, 15088.

(6) Sohn, S. S.; Bode, J. W. *Angew. Chem., Int. Ed*. **2006**, *45*, 6021.

(7) (a) Chan, A.; Scheidt, K. A. *J. Am. Chem. Soc*. **2006**, *128*, 4558. (b) Reynolds, N. T.; Read de Alaniz, J.; Rovis, T. *J. Am. Chem. Soc*. **2004**, *126*, 9518. (c) Reynolds, N. T.; Rovis, T. *J. Am. Chem. Soc*. **2005**, *127*, 16406.

(8) Duong, H. A.; Cross, M. J.; Louie, J. *Org. Lett*. **2004**, *6*, 4679.

(9) (a) Grasa, G. A.; Guveli, T.; Singh, R.; Nolan, S. P. *J. Org. Chem*. **2003**, *68*, 2812. (b) Singh, R.; Kissling, R. M.; Letellier, M.-A.; Nolan, S. P. *J. Org. Chem*. **2004**, *69*, 209. (c) Grasa, G. A.; Kissling, R. M.; Nolan, S. P. *Org. Lett*. **2002**, *4*, 3583. (d) Nyce, G. W.; Lamboy, J. A.; Connor, E. F.; Waymouth, R. M.; Hedrick, J. L. *Org. Lett*. **2002**, *4*, 3587. (e) Nyce, G. W.; Glauser, T.; Connor, E. F.; Mock, A.; Waymouth, R. M.; Hedrick, J. L. *J. Am. Chem. Soc*. **2003**, *125*, 3046. (f) Connor, E. F.; Nyce, G. W.; Myers, M.; Mock, A.; Hedrick, J. L. *J. Am. Chem. Soc*. **2002**, *124*, 914. (g) Kano, T.; Sasaki, K.; Maruoka, K. *Org. Lett*. **2005**, *7*, 1347. (h) Singh, R.; Nolan, S. P. *Chem. Commun*. **2005**, *43*, 5456. (i) Suzuki, Y.; Yamauchi, K.; Muramatsu, K.; Sato, M. *Chem. Commun*. **2004**, 2770.

(10) Movassaghi, M.; Schmidt, M. A. *Org. Lett*. **2005**, *7*, 2453.

(11) Fischer, C.; Smith, S. W.; Powell, D. A.; Fu, G. C. *J. Am. Chem. Soc*. **2006**, *128*, 1472.

(12) Song, J. J.; Tan, Z.; Reeves, J. T.; Gallou, F.; Yee, N. K.; Senanayake, C. H. *Org. Lett*. **2005**, *7*, 2193.

(13) Song, J. J.; Gallou, F.; Reeves, J. T.; Tan, Z.; Yee, N. K.; Senanayake, C. H. *J. Org. Chem*. **2006**, *71*, 1273.

(14) (a) Fukuda, Y.; Maeda, Y.; Ishii, S.; Kondo, K.; Aoyama, T. *Synthesis* **2006**, 589. (b) Fukuda, Y.; Maeda, Y.; Kondo, K.; Aoyama, T. *Chem. Pharm. Bull*. **2006**, *54*, 397. (c) Fukuda, Y.; Maeda, Y.; Kondo, K.; Aoyama, T. *Synthesis* **2006**, 1937. (d) Fukuda, Y.; Kondo, K.; Aoyama, T. *Synthesis* **2006**, 2649. (e) Suzuki, Y.; Abu Bakar, M. D.; Muramatsu, K.; Sato, M. *Tetrahedron* **2006**, *62*, 4227. (f) Kano, T.; Sasaki, K.; Konishi, T.; Mii, H.; Maruoka, K. *Tetrahedron Lett*. **2006**, *47*, 4615.

(15) (a) Wu, J.; Sun, X.; Ye, S.; Sun, W. *Tetrahedron Lett*. **2006**, *47*, 4813. (b) For a related aziridine opening reaction with acid anhydride, see: Sun, X.; Ye, S.; Wu, J. *Eur. J. Org. Chem*. **2006**, 4787.

(16) For a recent review, see: Mukaiyama, T. *Angew. Chem., Int. Ed*. **2004**, *43*, 5590.

(17) (a) Noyori, R.; Nishida, I.; Sakata, J. *J. Am. Chem. Soc*. **1983**, *105*, 1598. (b) Nakamura, E.; Shimizu, M.; Kuwajima, I.; Sakata, J.; Yokoyama, K.; Noyori, R. *J. Org. Chem*. **1983**, *48*, 932.

(18) Matsukawa, S.; Okano, N.; Imamoto, T. *Tetrahedron Lett*. **2000**, *41*, 103.

(19) Miura, K.; Nakagawa, T.; Hosomi, A. *J. Am. Chem. Soc*. **2002**, *124*, 536.

(20) Mukaiyama, T.; Fujisawa, H.; Nakagawa, T. *Helv. Chim. Acta* **2002**, *85*, 4518.

(21) Nakagawa, T.; Fujisawa, H.; Nagata, Y.; Mukaiyama, T. *Bull. Chem. Soc. Jpn*. **2004**, *77*, 1555.

(22) Fujisawa, H.; Nakagawa, T.; Mukaiyama, T. *Adv. Synth. Catal*. **2004**, *346*, 1241.

(23) Hagiwara, H.; Inoguchi, H.; Fukushima, M.; Hoshi, T.; Suzuki, T. *Synlett* **2005**, 2388.

(24) Hagiwara, H.; Inoguchi, H.; Fukushima, M.; Hoshi, T.; Suzuki, T. *Tetrahedron Lett*. **2006**, *47*, 5371.

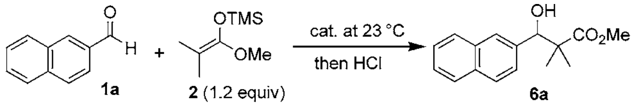
(25) (a) Loh, T.-P.; Feng, L.-C.; Wei, L.-L. *Tetrahedron* **2000**, *56*, 7309. (b) Chen, S.-L.; Ji, S.-J.; Loh, T.-P. *Tetrahedron Lett*. **2004**, *45*, 375. (c) Shen, Z.-L.; Ji, S.-J.; Loh, T.-P. *Tetrahedron Lett*. **2005**, *46*, 507. (d) Genisson, Y.; Gorrichon, L. *Tetrahedron Lett*. **2000**, *41*, 4881.

(26) (a) Denmark, S. E.; Winter, S. B. D.; Su, X.; Wong, K.-T. *J. Am. Chem. Soc*. **1996**, *118*, 7404. (b) Denmark, S. E.; Fan, Y. *J. Am. Chem. Soc*. **2002**, *124*, 4233. (c) For a review, see: Denmark, S. E.; Stavenger, R. A. *Acc. Chem. Res*. **2000**, *33*, 432.

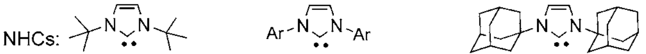
(27) (a) Denmark, S. E.; Lee, W. *J. Org. Chem*. **1994**, *59*, 707. (b) See refs 16, 18, 20, and 21.

(28) Fu, F.; Teo, Y.-C.; Loh, T. P. *Tetrahedron Lett*. **2006**, *47*, 4267.

Table 1. NHC-Catalyzed Mukaiyama Aldol Reactions



1a **2** (1.2 equiv) **6a**



3

Ar = 2,6-di-isopropylphenyl (**4a**),
Ar = 2,4,6-trimethylphenyl (**4b**)

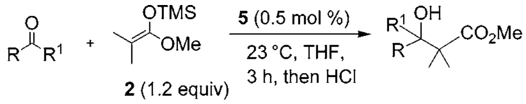
5

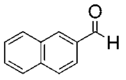
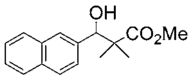
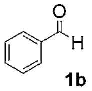
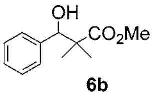
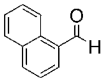
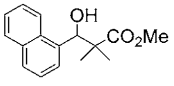
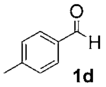
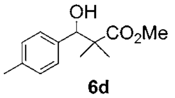
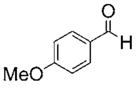
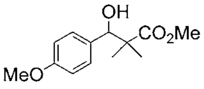
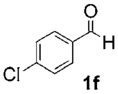
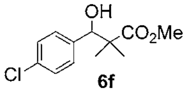
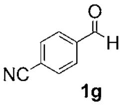
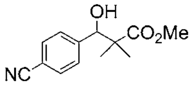
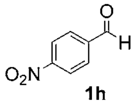
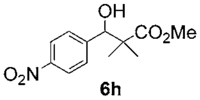
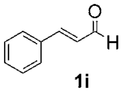
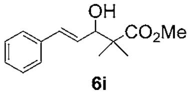
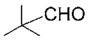
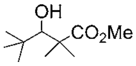
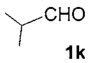
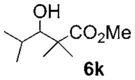
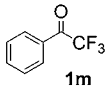
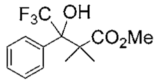
entry	solvent	cat. (loading)	time (h)	isolated yield
1	THF	**3** (0.5 mol %)	3	78%
2	THF	**4a** (0.5 mol %)	3	32%
3	THF	**4a** (0.5 mol %)	6.5	70%
4	THF	**4b** (0.5 mol %)	6	60%
5	THF	**5** (0.5 mol %)	3	77%
6	THF	**5** (0.05 mol %)	22.5	79%
7	THF	no catalyst	16	0%
8	Et_2O	**5** (0.5 mol %)	4.3	53% convn
9	toluene	**5** (0.5 mol %)	1	0%
10	THF	TTMPP (0.5 mol %)	22.5	36%
11	THF	*N*-methylimidazole (10 mol %)	21	0%
12	THF	pyridine oxide (10 mol %)	21	0%

pylphenyl)imidazol-2-ylidene (**4a**) gave 32% yield after 3 h (entry 2) and 70% yield after 6.5 h at 23 °C (entry 3). Similarly, the use of carbene **4b** (Ar = 2,4,6-trimethylphenyl)[9] gave 60% yield of aldol product after 6 h (entry 4). Bulky adamantyl-substituted carbene **5** exhibited good catalytic activity to give 77% yield of **6a** after 3 h at 23 °C (entry 5). NHC **5** was used for all subsequent experiments because it possesses good thermal stability[29] and is easy to handle in laboratories. Further optimization revealed that the catalyst loading could be lowered to 0.05 mol % without a decrease in yield, and the reaction did take longer (22.5 h) to complete (entry 6). Finally, it was shown in a control experiment that, without the addition of the carbene catalyst, no aldol product was formed in THF after 16 h at 23 °C (entry 7). A few solvents were briefly screened, and it was found that the reaction was slower in ether and that no reaction occurred in toluene (entries 8 and 9). As a comparison, we carried out the same Mukaiyama aldol reaction using some other charge-neutral Lewis base catalysts. It has been reported that tris(2,4,6-trimethoxyphenyl)-phosphine (TTMPP)[18] could be used to promote Mukaiyama aldol reactions with various aldehydes in THF at a 20 mol % catalyst loading level, giving aldol products in 48−93% yield after 3 h at room temperature. However, when a much lower catalyst loading (0.5 mol % of TTMPP) was used for the aldol reaction between **1a** and **2**, only 36% yield was obtained after 22.5 h at 23 °C (entry 10, cf. entries 5 and 6). Furthermore, reactions using 10 mol % of *N*-methylimidazole or pyridine oxide as catalysts gave no aldol addition products after 21 h at 23 °C in THF (entries 11 and 12).[30] These observations in our study have indicated that NHCs possess superior catalytic capability compared to these commonly employed nucleophilic catalysts.

The scope of the NHC-catalyzed Mukaiyama aldol reactions was explored using a range of substrates as illustrated in Table 2. Aromatic aldehydes with electron-donating or electron-withdrawing groups were well tolerated (entries

Table 2. Mukaiyama Aldol with Silyl Ketene Acetal Catalyzed by NHC



entry	carbonyl compound	product	isolated yield	lit. ref.
1	 **1a**	 **6a**	77%	28
2	 **1b**	 **6b**	76%	20
3	 **1c**	 **6c**	83%	20
4	 **1d**	 **6d**	83%	20
5	 **1e**	 **6e**	91%	20
6	 **1f**	 **6f**	81%	20
7	 **1g**	 **6g**	68%	20
8	 **1h**	 **6h**	75%	20
9	 **1i**	 **6i**	74%	20
10	 **1j**	 **6j**	61%	–
11	 **1k**	 **6k**	60%	31
12	 **1m**	 **6m**	87%	–

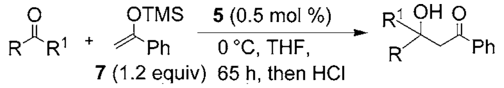
---

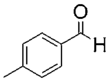
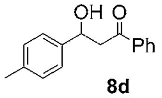
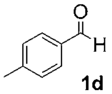
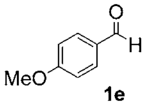
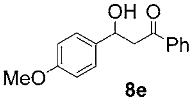
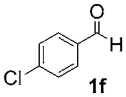
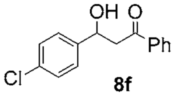
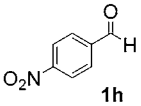
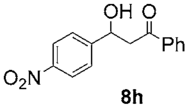
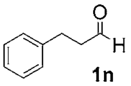
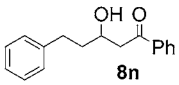
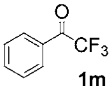
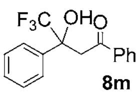
[29] (a) Arduengo, A. J., III; Harlow, R. L.; Kline, M. *J. Am. Chem. Soc.* **1991**, *113*, 361. (b) Arduengo, A. J., III; Bock, H.; Chen, H.; Denk, M.; Dixon, D. A.; Green, J. C.; Herrmann, W. A.; Jones, N. L.; Wagner, M.; West, R. *J. Am. Chem. Soc.* **1994**, *116*, 6641.

1−8), and they all gave good yields of aldol products[20,28] under our standard reaction conditions (3 h, 23 °C). Reaction with *trans*-cinnamaldehyde proceeded uneventfully to furnish β-hydroxy ester **6i** in 74% yield (entry 9).[20] The sterically demanding pivalaldehyde as well as enolizable isobutyraldehyde were found to be suitable substrates giving products in moderate yields (entries 10 and 11).[31] Attempts to effect Mukaiyama aldol addition to acetophenone were unsuccessful. However, it is interesting to note that reaction between **2** and 2,2,2-trifluoroacetophenone took place in 87% yield to afford compound **6m**, which contains two contiguous quarternary carbon centers (entry 12).

Next, we investigated Mukaiyama aldol reactions with trimethylsilyl enol ether **7** (Table 3). Because of the relatively low reactivity of silyl enol ethers compared to silyl ketene acetals, this reaction turned out to be quite challenging. When the reaction was initially carried out at 23 °C for ∼2 h, the desired aldol adduct was observed along with significant byproduct formation. Lowering the reaction temperature to 0 °C slowed down the aldol addition but effectively suppressed byproduct formation. Thus, reaction of *p*-tolualdehyde with trimethylsilyl enol ether **7** (1.2 equiv) at 0 °C for 65 h under the influence of 0.5 mol % of NHC **5** gave 74% yield of compound **8d** (entry 1).[32] Without the addition of the NHC catalyst, no aldol product formation was observed after 65 h at 0 °C (entry 2). Reaction of silyl enol ether **7** with the electron-rich *p*-anisaldehyde gave only 10% yield (entry 3).[33] On the other hand, electron-deficient substrates such as *p*-chlorobenzaldehyde and *p*-nitrobenzaldehyde proved to be much better aldol acceptors with 60% and 84% yields, respectively (entries 4 and 5).[33,34] Reaction with hydrocinnamaldehyde proceeded poorly giving only 25% yield (entry 6).[35] To our delight, 2,2,2-trifluoroacetophenone was found to be an excellent substrate for this reaction and compound **8m** was isolated in 95% yield (entry 7).[36]

Table 3. Mukaiyama Aldol with Silyl Enol Ether Catalyzed by NHC



entry	carbonyl compound	product	isolated yield	lit. ref.
1	 **1d**	 **8d**	74%	32
2	 **1d**	no catalyst	0%	–
3	 **1e**	 **8e**	10%	33
4	 **1f**	 **8f**	60%	33
5	 **1h**	 **8h**	84%	34
6	 **1n**	 **8n**	25%	35
7	 **1m**	 **8m**	95%	36

We propose that, analogous to other Lewis base catalyzed[12,13,27] Mukaiyama aldol reactions, NHC-catalyzed aldol additions proceed through a possible pentavalent silicon complex that is formed by initial attack of NHC on the Si atom in enoxysilanes.

In summary, we have demonstrated that NHCs can serve as highly efficient organocatalysts for Mukaiyama aldol reactions. In the presence of only 0.5 mol % of N-heterocyclic carbene **5**, a range of aldehydes underwent Mukaiyama aldol reactions in THF with trimethylsilyl ketene acetal **2** at 23 °C as well as with trimethylsilyl enol ether **7** at 0 °C to afford aldol adducts in good yields. 2,2,2-Trifluoroacetophenone was also found to be a suitable substrate for these reactions. These new conditions are metal-free, extremely mild, and operationally simple and tolerate various functional groups. Importantly, this methodology further illustrates the remarkable ability of NHCs to activate silicon-based reagents for nucleophilic C−C bond-forming reactions. Efforts to further explore potential NHC catalysis for other fundamental organic transformations are underway.

Supporting Information Available: Experimental procedures and data for compounds **6a−k**, **6m**, **8d−f**, **8h**, **8m**, and **8n**, including copies of ^{1}H and ^{13}C NMR spectra. This material is available free of charge via the Internet at http://pubs.acs.org.

OL0630494

---

(30) Both pyridine oxide and *N*-methylimidazole have been used to catalyze Mukaiyama aldol reactions in DMF, which is a much more polar solvent than THF. See refs 23 and 24.

(31) Wessjohann, L.; Gabriel, T. *J. Org. Chem.* **1997**, *62*, 3772.

(32) Komoto, I.; Kobayashi, S. *J. Org. Chem.* **2004**, *69*, 680.

(33) Jankowska, J.; Mlynarski, J. *J. Org. Chem.* **2006**, *71*, 1317.

(34) Wang, G.-W.; Zhang, Z.; Dong, Y.-W. *Org. Process Res. Dev.* **2004**, *8*, 18.

(35) Shimizu, M.; Kobayashi, F.; Hayakawa, R. *Tetrahedron* **2001**, *57*, 9591.

(36) Sokeirik, Y. S.; Sato, K.; Omote, M.; Ando, A.; Kumadaki, I. *Tetrahedron Lett.* **2006**, *47*, 2821.