http://dx.doi.org/10.5012/bkcs.2012.33.4.1275

A Mild and Efficient Tris(pentafluorophenyl)borane-catalyzed Sakurai Allylation of *N*-Benzyloxycarbonylamino *p*-Tolylsulfone with Allyltrimethylsilane

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Received December 20, 2011, Accepted January 25, 2012

Tris(pentafluorophenyl)borane, $B(C_6F_5)_3$, was found to be an efficient catalyst for synthesis of *N*-Cbz-homoallylic amines using Sakurai allylation of *N*-benzyloxycarbonyl-amino *p*-tolylsulfones with allyltrimethylsilane.

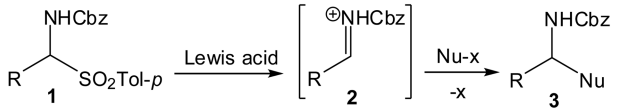
Key Words : *N*-Benzyloxycarbonylamino *p*-Tolylsulfone, Tris(pentafluorophenyl)borane, Allyltrimethylsilane, Allylation, *N*-Cbz-homoallylic amines

Introduction

The homoallylic amines are useful fundamental building blocks for the synthesis of many nitrogen containing natural products and bioactive molecules.[1,2] Mostly the acylated (Cbz or Boc) homoallylic amines are important synthons for many synthetic applications.[3] A variety of synthetic methods have been reported for the synthesis of protected homoallylic amines using various substrates.[4,5] The synthesis of protected homoallylic amines through *in situ* generations of *N*-acyliminum ion are highly attractive method. The *N*-acyliminum ion generation was observed when the *N*-benzyloxycarbonylamino *p*-tolylsulfone **1** was treated with a suitable Lewis acid. The *N*-acyliminium ions or *N*-benzyloxycarbonyliminium ions **2** are extremely versatile intermediates for the formation of carbon-carbon bonds in organic synthesis. These are highly reactive towards various nucleophiles in the addition reactions (Scheme 1).[6,7]

The development of new methods for the synthesis of protected homoallylic amines is an important task for chemists. In recent years, several Lewis acid catalysts such as $TiCl_4$, $SnCl_4$, $GaCl_3$, $InCl_3$, $Bi(OTf)_3$, and $In(OTf)_3$ have been employed for the synthesis of protected homoallylic amines.[8] $TiCl_4$ or $SnCl_4$ moisture sensitive and excess or stoichiometric quantity of the catalyst has been required. Some of these methods required long reaction time and showed low yields.

Tris(pentafluorophenyl)borane, $B(C_6F_5)_3$, have been highly attracted as a Lewis acid because of non-toxicity and thermal stablility.[9] Recently, various research groups have used $B(C_6F_5)_3$ in various organic transformations such as ring-opening of epoxides, *aza*-Ferrier glycosylation,[10-12] hydrosilylation of imines,[13] reduction of alcohols with silane,[14] and hydrogenation of imines.[15] $B(C_6F_5)_3$ was utilized in the regio- and stereo-selective cyclizations of unsaturated alkoxysilanes.[16] $B(C_6F_5)_3$ was also engaged as an efficient activator in the reduction of various functional groups by polymethylhydrosiloxane.[17,18] Very recently, we also reported Friedel-Crafts alkylation of activated arenes and heteroarenes using $B(C_6F_5)_3$.[19] However, tris(pentafluorophenyl)-borane has not been used for Sakurai allylation reaction of *N*-benzyloxycarbonylamino *p*-tolylsulfone. We report herein tris(pentafluorophenyl)borane-catalyzed Sakurai allylation reaction of *N*-benzyloxycarbonylamino *p*-tolylsulfone with allyltrimethylsilane.



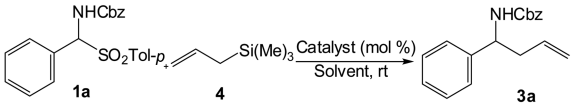
Scheme 1. Nucleophilic addition to *N*-acyliminium ion (**2**).

Results and Discussions

Initially, we screened the reaction conditions for Sakurai allylation of *N*-benzyloxycarbonylamino phenyl *p*-tolylsulfone **1a** with allyltrimethylsilane **4** using various catalysts and solvents (Table 1). The reaction of *N*-benzyloxycarbonylamino phenyl *p*-tolylsulfone **1a** and allyltrimethylsilane **4** in the absence of catalyst did not provide a desired product (entry 1). We searched for various Lewis acids which promoted the model reaction of *N*-benzyloxycarbonylamino phenyl *p*-tolylsulfone **1a**, and allyltrimethylsilane **4** (entries 2–6). Among all tested Lewis acids, tris(pentafluorophenyl)-borane was chosen to be the most effective one (entry 6). Interestingly, when the amount of $B(C_6F_5)_3$ was decrease to 5 mol %, the similar high yield (88%) was obtained in the reaction (entry 7). Further lowering of the amount of the catalyst lead to the decrease in yields (entry 8). Among the various solvents tested, the dichloromethane provided the desired product with a high yield (entries 7, 9-11). Thus, 5 mol % of $B(C_6F_5)_3$ and dichloromethane were selected as the optimized condition for the Sakurai allylation reaction (entry 7).

Under optimized reaction conditions as in Table 1, the scope of Sakurai allylation reactions have been studied using various *N*-benzyloxycarbonylamino *p*-tolylsulfone **1** and allyltrimethylsilane **4**. The results are summarized in Table 2.

Table 1. The optimized conditions for Sakurai allylation reaction of *N*-benzyloxycarbo-nylamino phenyl *p*-tolylsulfone with allyltrimethylsilane[a]



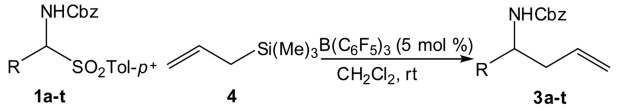
Entry	Catalyst (mol %)	Allylsilane (equiv)	Solvent	Reaction time (h)	Isolated yield (%)[b]
1	-	1.2	CH_2Cl_2	14	NR
2	$ZnCl_2$ (10)	1.2	CH_2Cl_2	14	15
3	$Zn(ClO_4)_2$ (10)	1.2	CH_2Cl_2	14	38
4	$Mg(ClO_4)_2$ (10)	1.2	CH_2Cl_2	14	27
5	$AgClO_4$ (10)	1.2	CH_2Cl_2	14	58
6	$B(C_6F_5)_3$ (10)	1.2	CH_2Cl_2	9	89
7	$B(C_6F_5)_3$ (5)	1.2	CH_2Cl_2	10	88
8	$B(C_6F_5)_3$ (2.5)	1.2	CH_2Cl_2	12	71
9	$B(C_6F_5)_3$ (5)	1.2	CH_3CN	12	68
10	$B(C_6F_5)_3$ (5)	1.2	THF	16	40
11	$B(C_6F_5)_3$ (5)	1.2	Et_2O	16	32

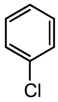
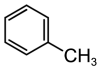
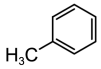
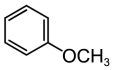
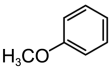
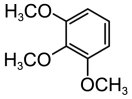
[a]Reaction condition: *N*-benzyloxycarbonylamino phenyl *p*-tolylsulfone **1a** (1.0 mmol) and allyltrimethylsilane **4** (1.2 mmol). [b]Isolated yield of product after column chromatography.

The reaction of *N*-benzyloxycarbonylamino phenyl *p*-tolylsulfone **1a** with allyltrimethylsilane **4** in presence of 5 mol % of $B(C_6F_5)_3$ produced the *N*-Cbz-homoallylic amine **3a** in 88% yield (entry a). The *N*-benzyloxycarbonylamino 2-naphthyl *p*-tolylsulfone **1b** reacted with allyltrimethylsilane **4** to afford the allylation product **3b** in 84% yield (entry b). The reactions of **1c**, **1d**, and **1e**, all with one chlorine substituent in the aromatic ring, gave the products in 81, 84 and 89% yield, respectively (entries c-e). Similarly, *N*-benzyloxycarbonylamino *p*-tolylsulfone derivatives containing other halogen group on phenyl ring proceeded smoothly to give the *N*-Cbz-homoallylic amines in 83, 90, and 80% yields, respectively (entries f-h). The *o*-isomers (entries 3, 6) provided the products in lower yield than the corresponding *p*-isomers, due to the steric and electronic effects of the substituent on the phenyl ring. The *N*-benzyloxycarbonylamino *p*-tolylsulfone compounds with electron-withdrawing substituents such as CN and NO_2 groups (**1i** and **1j**) brought about the corresponding allylation product in good yield (entries i and j). *N*-Benzyloxycarbonylamino *p*-tolylsulfones **1k-p** with electron-donating groups, such as Me, OMe and OPh reacted with allyltrimethylsilane **4** to produce the corresponding *N*-Cbz-homoallylic amines in high yield (entries k-p). The reactions of 2-cyclohexenyl and cyclohexyl *N*-benzyloxycarbonylamino *p*-tolylsulfone, **1q** and **1r** afforded the allylation derivatives in 81 and 78% yield, respectively (entries q and r). Similarly, acyclic aliphatic *N*-benzyloxycarbonyl-amino *p*-tolylsulfones **1s-t** were converted to the corresponding allylation product in moderate yield (entries s and t).

The results clearly indicate that there is a substitution effect of aromatic ring of the substrates on the reaction. The electron-donating substituted *N*-benzyloxycarbonylamino *p*-

Table 2. $B(C_6F_5)_3$-catalyzed Sakurai allylation of various *N*-benzyloxycarbonylamino *p*-tolylsulfone with allyltrimethylsilane[a]



Entry	R	Reaction time (h)	Isolated yield (%)[b]
a		10	88
b		10	84
c		10	81
d		10	84
e		10	89
f		10	83
g		10	90
h		10	80
i		10	80
j		10	81
k		9	86
l		9	90
m		8	91
n		9	90
o		10	87
p		8	89
q		9	81
r		9	78
s		10	75
t		10	76

[a]Reaction condition: *N*-benzyloxycarbonylamino *p*-tolylsulfone **1** (1.0 mmol), allyltrimethylsilane **4** (1.2 mmol) and $B(C_6F_5)_3$ (5 mol % with respective to *N*-benzyloxycarbonylamino *p*-tolylsulfone **1**) are used; [b]Isolated yield of product after column chromatography.

tolylsulfones provided the *N*-Cbz-homoallylic amines in higher yield than that of electron-withdrawing substituted *N*-benzyloxycarbonylamino *p*-tolylsulfones and *N*-benzyloxycarbonylamino aliphatic sulfones. This may be due to the resonance effect and/or inductive effect of the substituted aromatic ring and the similar substitution effect was observed in the other reactions.[8f,8h,8i]

Conclusion

We have described a mild and efficient Sakurai allylation reactions of *N*-benzyloxycarbonylamino *p*-tolylsulfone with allyltrimethylsilane using 5 mol % of tris(pentafluorophenyl)borane. The present method provides a facile and convenient route for the synthesis of *N*-Cbz-homoallylic amines. The major advantages of the present method are mild reaction conditions and broad applicability to various substrates with moderate to high yield.

Experimental Section

Anhydrous $B(C_6F_5)_3$ was purchased from Aldrich. The *N*-benzyloxycarbonylamino *p*-tolylsulfones were prepared from the corresponding aldehydes by the literature method.[1] Melting points were determined on a melting point apparatus (OptiMelt, Stanford Research System). IR analysis has been done with IR spectroscopy (model NICOLET 6700 FT-IR, Thermo electron corporation) with KBr pellet. The ^{1}H NMR (400 MHz) and ^{13}C NMR (50 MHz) spectra were recorded with Varian Gemini 200 or 400 MHz and 50 or 100 MHz spectrometer. Chemical shifts were reported in ppm in $CDCl_3$ with tetramethylsilane as the internal standard. Compounds have been also identified by HRMS (FAB/EI) with Jeol DMX 303.

General Experimental Procedure for the Allylation of *N*-Benzyloxycarbonylamino *p*-tolylsulfone with Allyltrimethylsilane: Allyltrimethylsilane (1.2 mmol) was added dropwise to a stirred solution of *N*-benzyloxycarbonylamino *p*-tolylsulfone (1 mmol) and $B(C_6F_5)_3$ (5 mol %) in CH_2Cl_2 (4 mL) under nitrogen atmosphere at rt., The reaction mixture is stirred at rt and monitored by TLC. After the completion of the reaction, the reaction mixture is filtered, and the residue is washed with CH_2Cl_2 (2 × 5 mL). The filtrate is dried over anhydrous Na_2SO_4 and concentrated under vacuum. The crude product is subject to column chromatography (silica gel, hexane-EtOAc, 90:10 or 95:5) to obtain the pure product.

The products were characterized by melting points, Mass, IR, ^{1}H and ^{13}C NMR data that are consistent with literature values.[4c,4d, 8f,8h,8i] The spectral data of the selected products are provided below.

(3a) *N*-Benzyloxycarbonyl-1-(phenyl)but-3-enylamine (Table 2, entry a): White solid, mp 67 °C; IR (KBr): ν_{max} 3367, 1685, 1530, 1257, 1028 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.22 (m, 10H), 5.70-5.59 (m, 1H), 5.20 (br s, 1H), 5.10-5.01 (m, 4H), 4.79 (br s, 1H), 2.51 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.7, 142.0, 136.5, 133.8, 128.6, 128.5, 128.1, 127.3, 126.3, 118.4, 66.8, 54.6, 41.1.[4c,8i]

(3d) *N*-Benzyloxycarbonyl-1-(3-chlorophenyl)but-3-enylamine (Table 2, entry d): White solid, mp 61-62 °C; IR (KBr): ν_{max} 3344, 1673, 1530, 1255, 1028 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.29 (m, 5H), 7.27-7.24 (m, 3H), 7.15 (d, J = 2.0 Hz, 1H), 5.73-5.60 (m, 1H), 5.26 (br s, 1H), 5.14-5.04 (m, 4H), 4.89 (br s, 1H), 2.50 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.6, 144.2, 136.2, 134.3, 133.1, 129.7, 128.4, 128.1, 127.4, 126.3, 124.4, 118.8, 66.8, 54.0, 40.8; HRMS-FAB (*m*/*z*) $[M+H]^+$ calcd for $C_{18}H_{19}ClNO_2$ 316.1104, found 316.1105.[8i]

(3h) *N*-Benzyloxycarbonyl-1-(3-fluorophenyl)but-3-enylamine (Table 2, entry h): Viscous mass; IR (KBr): ν_{max} 3320, 1684, 1530, 1243, 1028 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.22 (m, 6H), 7.03 (d, J = 7.0 Hz, 1H), 6.97-6.90 (m, 2H), 5.68-5.90 (m, 1H), 5.26 (d, J = 5.4 Hz, 1H), 5.11-5.02 (m, 4H), 4.79 (br s, 1H), 2.48 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 161.6, 155.6, 144.7, 136.2, 133.2, 130.0, 129.9, 128.4, 128.1, 121.8, 118.7, 114.2, 113.9, 113.2, 113.0, 66.8, 54.0, 40.8; HRMS-FAB (*m*/*z*) $[M+H]^+$ calcd for $C_{18}H_{19}FNO_2$ 300.1400, found 300.1398.[8i]

(3o) *N*-Benzyloxycarbonyl-1-(3-phenyloxyphenyl)but-3-enylamine (Table 2, entry o): White solid, mp 45-46 °C; IR (KBr): ν_{max} 3311, 1679, 1534, 1255, 1056 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.28 (m, 7H), 7.14-7.10 (m, 1H), 7.03-6.97 (m, 4H), 6.90 (d, J = 8.0 Hz, 2H), 5.73-5.64 (m, 1H), 5.19 (br s, 1H), 5.14-5.05 (m, 4H), 4.82 (br s, 1H), 2.48 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.3, 157.0, 155.6, 144.1, 136.3, 133.4, 129.8, 129.7, 128.4, 128.0, 123.2, 121.0, 118.8, 118.5, 117.4, 116.6, 66.7, 54.1, 40.9; HRMS-FAB (*m*/*z*) $[M+H]^+$ calcd for $C_{24}H_{24}NO_3$ 373.1756, found 374.1755.[8i]

(3s) *N*-Benzyloxycarbonyl-1-(hexyl)but-3-enylamine (Table 2, entry s): Viscous mass; IR (KBr): ν_{max} 3320, 1684, 1518, 1243, 1040 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.28 (m, 5H), 5.82-5.73 (m, 1H), 5.10-5.04 (m, 4H), 4.74 (br s, 1H), 3.71 (br s, 1H), 2.28-2.16 (m, 2H), 1.48-1.46 (m, 1H), 1.44-1.26 (m, 9H), 0.89 (t, J = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.9, 136.6, 134.2, 128.3, 128.0, 127.8, 117.6, 66.3, 50.6, 39.3, 34.5, 31.6, 29.0, 25.7, 22.4, 13.9; HRMS-FAB (*m*/*z*) $[M+H]^+$ calcd for $C_{18}H_{28}NO_2$ 286.1807, found 286.1806.[8i]

Acknowledgments. Authors thank the Department of Chemistry, Inha University. This was supported by grant of Inha University in Korea.

Supplementary Information. General procedures, preparation of *N*-benzyloxycarbonylamino *p*-tolylsulfone, IR, ^{1}H and ^{13}C NMR spectral data of the all products are available through the internet http://journal.kcsnet.or.kr.

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