JOC Article

Et_3B-Induced Radical Addition of *N*,*N*-Dichlorosulfonamide to Alkenes and Pyrrolidine Formation via Radical Annulation

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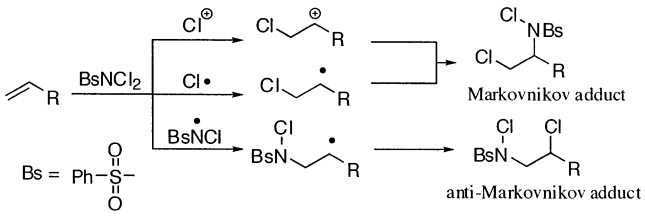
A highly regioselective radical addition of *N*,*N*-dichlorobenzenesulfonamide (dichloramine-B) to 1-alkenes is achieved at −78 °C by the use of triethylborane as a radical initiator. The reaction of 1,3-dienes with *N*,*N*-dichlorosulfonamide in the presence of Et_3B regioselectively provides *N*-chloro-*N*-allylamide derivatives. *N*-Chloro-*N*-allylamides thus obtained react with a variety of alkenes to furnish pyrrolidine derivatives in good yields. A radical annulation reaction among *N*,*N*-dichlorosulfonamide, 1,3-dienes, and alkenes has been developed.

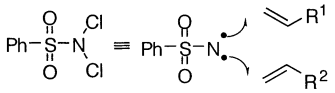
Introduction

N-Chlorosulfonamides and related compounds are inexpensive N1 sources that allow efficient introduction of nitrogen with a variety of unsaturated molecules, providing important compounds such as aziridines and hydroxylamine derivatives.[1] However, utility of *N*,*N*-dichlorosulfonamides in organic synthesis is limited so far. *N*,*N*-Dichlorosulfonamides are known to yield adducts readily in the reaction with alkenes.[2] The reaction with 1-alkenes generally affords a regioisomeric mixture of anti-Markovnikov and Markovnikov adducts.[2e,f] The failure in regiocontrol is mainly due to the mixed reaction pathways where both radical and cationic species such as Cl^+, $Cl^•$, and $^•N(Cl)SO_2R$ are involved (Scheme 1).[3] The thermally or photochemically induced addition increases the selectivity for the anti-Markovnikov adduct. However, the selectivity is not necessarily satisfactory. Furthermore, a prolonged reaction time leads to undesirable reactions such as rearrangement or further addition reactions of the initial 1:1 adducts. Thus, the efficient regioselective addition of *N*,*N*-dichlorosulfonamides to 1-alkenes has not been addressed so far.

SCHEME 1



SCHEME 2



We envisaged that *N*,*N*-dichlorobenzenesulfonamide (dichloramine-B)[4] would serve as a nitrogen diradical equivalent to allow two-directional and sequential carbon−nitrogen bond formations (Scheme 2). Thus, we have investigated the regiocontrol in the addition of *N*,*N*-dichlorobenzenesulfonamide (**1**) to 1-alkenes. Here we

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(1) (a) Ando T.; Minakata, S.; Ryu, I.; Komatsu, M. *Tetrahedron Lett.* **1998**, *39*, 309. (b) Ando, T.; Kano, D.; Minakata, S.; Ryu, I.; Komatsu, M. *Tetrahedron* **1998**, *54*, 13485. (c) Jeong, J. U.; Tao, B.; Sagasser, I.; Henniges, H.; Sharpless, K. B. *J. Am. Chem. Soc.* **1998**, *120*, 6844. (d) Albone, D. P.; Aujla, P. S.; Taylor, P. C.; Challenger, S.; Derrick, A. M. *J. Org. Chem.* **1998**, *63*, 9569. (e) Ali, S. I.; Nikalje, M. D.; Sudalai, A. *Org. Lett.* **1999**, *1*, 705. (f) Chanda, B. M.; Vyas, R.; Bedekar, A. V. *J. Org. Chem.* **2001**, *66*, 30. (g) Nishimura, M.; Minakata, S.; Takahashi, T.; Oderaotoshi, Y. Komatsu, M. *J. Org. Chem.* **2002**, *67*, 2101. (h) Sharpless, K. B.; Chong, A. O.; Oshima, K. *J. Org. Chem.* **1976**, *41*, 177. (i) Herranz, E.; Sharpless, K. B. *J. Org. Chem.* **1978**, *43*, 2544. (j) Li, G.; Chang, H.-T.; Sharpless, K. B. *Angew. Chem., Int. Ed. Engl.* **1996**, *35*, 451. (k) Rubin, A. E.; Sharpless, K. B. *Angew. Chem., Int. Ed. Engl.* **1997**, *36*, 2637. (l) Campbell, M. M.; Johnson, G. *Chem. Rev.* **1976**, *78*, 65.

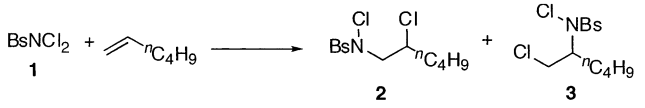
(2) (a) Ziegler, K.; Spath, A.; Schaat, E.; Schumann, W.; Winkelmann, E. *Justus Liebigs Ann. Chem.* **1942**, *551*, 80. (b) Thielacker, W.; Wessel, H. *Justus Liebigs Ann. Chem.* **1967**, *703*, 34. (c) Seden, T. P.; Turner, R. W. *J. Chem. Soc. C* **1968**, 876. (d) Ueno, Y.; Takemura, S.; Ando, Y.; Terauchi, H. *Chem. Pharm. Bull.* **1965**, *13*, 1369. (e) Ohashi, T.; Sugie, M.; Okahara, M.; Komori, S. *Tetrahedron Lett.* **1968**, *39*, 4195. (f) Ohashi, T.; Sugie, M.; Okahara, M.; Komori, S. *Tetrahedron* **1969**, *25*, 5349. (g) Matsarev, G. V.; Ushakov, A. A.; Inshakova, V. T.; Raskina, A. D.; Rozenberg, V. R.; Kolbasov, V. I. *Zh. Vses. Khim. O-va. im. D. I. Mendeleeva* **1986**, *31*, 467. (h) Markov, V. I.; Danileiko, D. A.; Doroshenko, V. A. Gella, I. M.; Polyakov, A. E. *Org. Soedin. Sery, 2* **1980**, 176. (i) Glushkova, N. E.; Kochina, T. A.; Kharitonov, N. P. *Zh. Org. Khim.* **1984**, *20*, 447. (j) Vorob'eva, I. S.; Stadnichuk, M. D. *Zh. Obshch.. Khim.* **1984**, *54*, 2731. (k) Daniher, F. A.; Melchior, M. T.; Butler, P. E. *J. Chem. Soc., Chem. Commun.* **1968**, 931. (l) Daniher, F. A.; Butler, P. E. *J. Org. Chem.* **1968**, *33*, 4336.

(3) For reviews on *N*-centered radicals, see: (a) Fallis, A. G.; Brinza, I. M. *Tetrahedron* **1997**, *53*, 17543. (b) Neale, R. S. *Synthesis*, **1971**, 1. (c) Stella, L. *Angew. Chem., Int. Ed. Engl.* **1983**, *22*, 337. (d) Zard, S. Z. *Synlett* **1996**, 1148. (e) Esker, J. L.; Newcomb, M. *Adv. Heterocycl. Chem.* **1993**, *58*, 1. (f) Stella, L. In *Radicals in Organic Synthesis*; Renaud, P., Sibi, M. P., Eds.; Wiley-VCH: Weinheim, 2001; Vol. 2, Chapter 5.1, p 407.

(4) *N*,*N*-Dichlorobenzenesulfonamide is commercially available from TCI.

10.1021/jo034043k CCC: $25.00
Published on Web 03/19/2003

TABLE 1. Addition of *N,N*-Dichlorobenzenesulfonamide to 1-Hexene



entry	equiv of **1**	equiv of 1-hexene	conditions	combined yield[a] (%)	**2**/**3**
1[b]	1.5	1.0	benzene rt	95	62/38
2[b]	1.5	1.0	benzene Et_3B (5 mol %) rt	96	71/29
3[b]	1.5	1.0	toluene −78 °C	45	62/38
4[b]	1.5	1.0	toluene Et_3B (5 mol %) −78 °C	71	87/13
5[c]	1.0	3.0	toluene Et_3B (5 mol %) −78 °C	98	>95/5

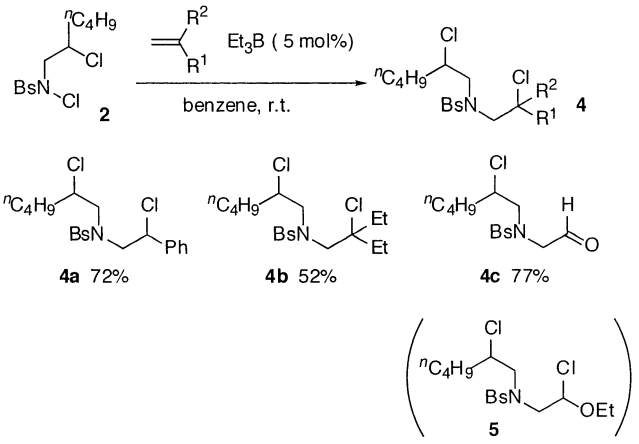
[a] NMR yield with dibenzyl ether as an internal standard. [b] Reaction conditions: **1** (1.5 mmol), 1-hexene (1.0 mmol), toluene or benzene (4 mL), 3 h. [c] Reaction conditions: **1** (1.0 mmol), 1-hexene (3.0 mmol), toluene (7 mL), 3 h.

wish to report a Et_3B-initiated regioselective addition of *N,N*-dichlorobenzenesulfonamide to various carbon−carbon double bonds. Radical annulation[5] of **1** with 1,3-dienes and alkenes to provide pyrrolidine derivatives is also described.

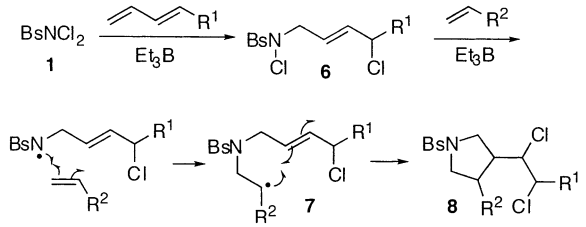
Results and Discussion

(1) Et_3B-Induced Addition of *N,N*-Dichlorobenzenesulfonamide to 1-Alkenes. The regioselectivity observed in the addition of *N,N*-dichlorobenzenesulfonamide (**1**) to 1-alkenes was examined with 1-hexene as the 1-alkene. The results under a variety of conditions are listed in Table 1. The use of 1.5 equiv of **1** to 1-hexene in benzene at room temperature provided a mixture of anti-Markovnikov **2** and Markovnikov adduct **3** along with other products (entries 1 and 2). The reaction in toluene at −78 °C proceeded cleanly and yielded only a mixture of **2** and **3** (entries 3 and 4). However, the regioselectivity was still not sufficient. Although the addition took place without Et_3B as a radical initiator, the presence of Et_3B enhanced the reactivity and the regioselectivity.[6] When an excess of 1-hexene (3.0 equiv) was employed in the presence of Et_3B in toluene at −78 °C, anti-Markovnikov adduct **2** was obtained in good yield as a sole product

SCHEME 3



SCHEME 4



(entry 5). The use of hexane as the solvent afforded a poor result because of the low solubility of **1** in hexane. The reaction in THF or ether resulted in formation of byproducts bearing a tetrahydrofuranyl or 1-ethoxyethyl groups, respectively. In these cases, an *N*-centered radical from **1** abstracts hydrogen at the α-position of the ether linkage.

Having optimized reaction conditions of the regioselective addition of **1**, we then investigated exploitation of the product **2**, which has still one N−Cl bond, as a nitrogen-centered radical precursor.[7] The reaction of **2** with styrene (2.0 equiv) and 2-ethyl-1-butene (9.0 equiv) in the presence of Et_3B furnished the corresponding adduct in good yields with high regioselectivity via a chlorine atom transfer radical process (Scheme 3).[8] Ethyl vinyl ether (2.0 equiv) afforded α-amino aldehyde **4c** in good yield, which probably resulted from hydrolysis of the initial adduct **5** (α-chloro ether). Although the hydrogen abstraction with a *N*-centered radical can be problematic in the intermolecular reaction, such side reactions are much slower than the addition reaction in these cases.

(2) Et_3B-Induced Addition of *N,N*-Dichlorobenzenesulfonamide to 1,3-Dienes. With an efficient protocol of **1** as a nitrogen diradical equivalent in hand, we then attempted to synthesize pyrrolidine derivatives via three-component radical coupling reaction. Recently,

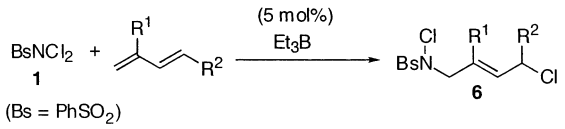
(5) For examples of radical annulation with carbon-centered radicals, see: (a) Cekovic, Z.; Saicic, R. *Tetrahedron Lett.* **1986**, *27*, 5893. (b) Barton, D. H. R.; Zard, S. Z.; da Silva, E. *J. Chem. Soc., Chem. Commun.* **1988**, 285. (c) Curran, D. P.; Chen, M.-H. *J. Am. Chem. Soc.* **1987**, *109*, 6558. (d) Curran, D. P.; Chen, M.-H.; Spletzer, E.; Seong, C. M.; Chang, C.-T. *J. Am. Chem. Soc.* **1987**, *111*, 8872. (e) Feldman, K. S.; Romanelli, A. L.; Ruckle, R. E., Jr.; Miller, R. F. *J. Am. Chem. Soc.* **1988**, *110*, 3300. (f) Miura, K.; Fugami, K.; Oshima, K.; Utimoto, K. *Tetrahedron Lett.* **1988**, *29*, 5135. (g) Curran, D. P.; van Elburg, P. A. *Tetrahedron Lett.* **1989**, *30*, 2501. (h) Kitagawa, O.; Yamada, Y. Fujiwara, H.; Taguchi, T. *J. Org. Chem.* **2002**, *67*, 922. (i) Kitagawa, O.; Yamada, Y. Sugawara, A.; Taguchi, T. *Org. Lett.* **2002**, *4*, 1011.

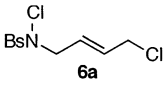
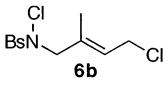
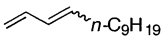
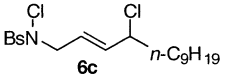
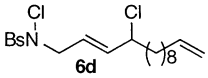
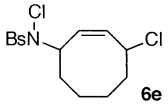
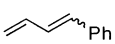
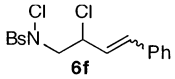
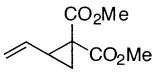
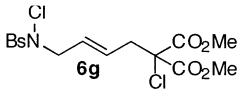
(6) For reviews on Et_3B as a radical initiator, see: (a) Ollivier, C.; Renaud, P. *Chem. Rev.* **2001**, *101*, 3415. (b) Yorimitsu, H.; Oshima, K. In *Radicals in Organic Synthesis*; Renaud, P., Sibi, M. P., Eds.; Wiley-VCH: Weinheim, 2001; Vol. 1, Chapter 1.2, p 11. (c) Yorimitsu, H.; Shinokubo, H.; Oshima, K. *Synlett* **2002**, 674.

(7) For intermolecular addition of *N*-centered radicals to alkenes, see: (a) Newcomb, M.; Kumar, M. U. *Tetrahedron Lett.* **1990**, *31*, 1675. (b) Nagao, Y.; Katagiri, S. *Chem. Lett.* **1992**, 2379. (c) Goosen, A.; McCleland, C. W.; Merrifield, A. J. *J. Chem. Soc., Perkin Trans. 1* **1992**, 627.

(8) For reviews on atom transfer radical reactions, see: (a) Byers, J. In *Radicals in Organic Synthesis*; Renaud, P., Sibi, M. P., Eds.; Wiley-VCH: Weinheim, 2001; Vol. 1, Chapter 1.5, p 72. (b) Curran, D. P.; Chen, M.-H.; Kim, D. *J. Am. Chem. Soc.* **1986**, *108*, 2489. (c) Curran, D. P. *Synthesis* **1988**, 417; 489.

TABLE 2. Radical Addition of Dichloramine to 1,3-Dienes[a]



entry	dienes	product	yield (%)
1		 **6a**	95
2		 **6b**	89
3		 **6c**	90
4		 **6d**	97
5		 **6e**	90
6		 **6f**	74
7[b]		 **6g**	39

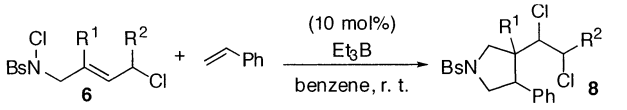
[a] *N*,*N*-Dichlorobenzenesulfonamide (1.0 mmol), 1,3-dienes (1.5−2.0 mmol), Et_3B (0.05 mmol), toluene, −78 °C, 30 min to 1 h. [b] Benzene, rt, 3 h.

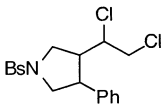
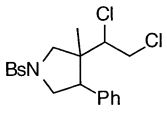
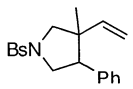
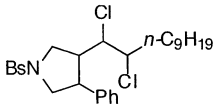
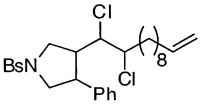
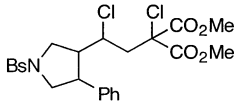
Taguchi et al. and we have independently reported radical [3 + 2] annulation strategy that exploits an *N*-allylsulfonamidyl radical as a key intermediate.[9] As depicted in Scheme 4, the reaction of 1,3-dienes with **1** in a 1,4-addition manner provides *N*-allyl-*N*-chlorobenzenesulfonamides **6**, which further react with alkenes to furnish pyrrolidine derivatives **8** via a radical addition−cyclization sequence. The *N*-centered radical from **6** undergoes the intermolecular addition toward an alkene to form an alkyl radical **7**. The subsequent 5-exo radical cyclization proceeds to yield the cyclized product **8** via a chlorine atom transfer process.

Daniher and Butler reported the predominant mode of the addition of *N*,*N*-dichlorosulfonamides toward 1,3-dienes is 1,4-fashion with a selectivity of >95%.[21] However, they examined only 1,3-butadiene and chloroprene as a substrate. Consequently, we reinvestigated the reaction of **1** with various 1,3-dienes with Et_3B as a radical initiator.

(9) (a) Tsuritani, T.; Shinokubo, H.; Oshima, K.*Org. Lett.* **2001**, *3*, 2709. (b) Kitagawa, O.; Yamada, Y.; Fujiwara, H.; Taguchi, T. *Angew. Chem., Int. Ed.* **2001**, *40*, 3865.

TABLE 3. Radical Annulation with Styrene[a]



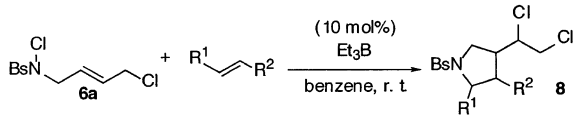
entry	**6**	product		yield (%)
1	**6a**		**8a**	96
2	**6b**		**8b**	52
			**9b**	9
3	**6c**		**8c**	72
4	**6d**		**8d**	70
5	**6e**	–	**8e**	–
6	**6g**		**8g**	87

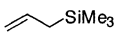
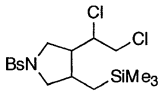
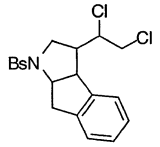
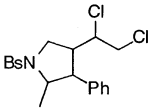
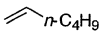
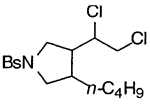
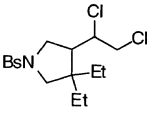
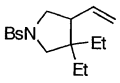
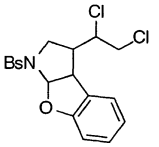
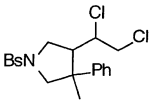
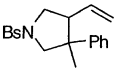
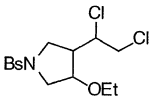
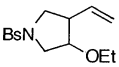
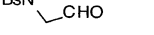
[a] *N*-Chlorosulfonamide (**6**, 0.5 mmol), styrene (1.0−1.5 mmol), Et_3B (0.05 mmol), benzene, rt, 3 h.

Treatment of 1,3-alkadienes with **1** exclusively provides the 1,4-adducts, 4-chloro-2-butenylamides **6**, in good yields (Table 2). None of the 1,2-addition products could be detected in the crude reaction mixture. However, the use of 1-phenyl-1,3-butadiene yielded the 1,2-adduct **6f** predominantly. The reaction tolerates the presence of nonconjugated alkenes in the substrate (entry 4). Cyclic dienes such as 1,3-cyclooctadiene also yields 1,4-adduct **6e** (entry 5). The use of a vinylcyclopropane as a substrate provided a ring opening adduct **6g** in moderate yield (entry 7).

(3) Radical Annulation with *N*-Chlorosulfonamide. The study on radical annulation was commenced with the reaction of **6** with styrene (Table 3). Treatment of a mixture of **6** and styrene with 10 mol % of Et_3B in benzene yielded the cyclized products **8** in modest to good yields at room temperature. A terminal alkenyl moiety of **6d** survived under the reaction conditions, and no addition of the *N*-centered radical to the alkene occurred (entry 4). Unfortunately, cyclic *N*-chloroamide **6e** provided none of the cyclized product, and only the reduction product, 4-chlorocyclooct-2-en-1-ylsulfonamide **8e**, was obtained (entry 5). *N*-Chloroamide **6g** derived via ring opening of cyclopropane also afforded annulation adduct **8g** in 87% yield. In the reaction with **6b**, dechlorination

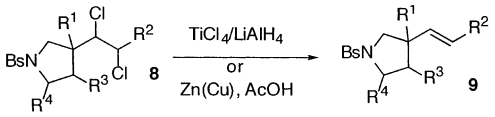
TABLE 4. Radical Annulation with Various Alkenes[a]

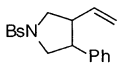
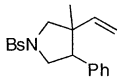
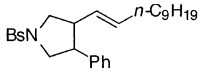
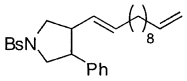
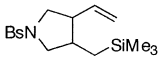
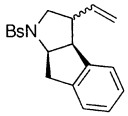
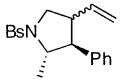
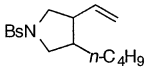
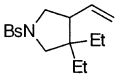
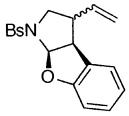
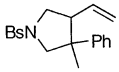
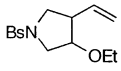


entry	alkene	product		yield (%)
1			**8h**	84
2			**8i**	90
3			**8j**	86
4[b]			**8k**	62
5[b]			**8l**	44
			**9l**	30
6			**8m**	80
7			**8n**	60
			**9n**	15
			**8o**	72
8[c]			**9o**	12
			**10o**	12

[a] Reactions employed **6a** (0.5 mmol) and alkene (1.0−1.5 mmol) in benzene (4.0 mL) in the presence of Et_3B (0.05 mmol) unless otherwise noted. The reaction mixture was stirred for 3 h at room temperature. [b] An excess amount of alkenes (9.0 equiv) was employed. [c] The reaction employed 25 mL of benzene under otherwise the same reaction conditions.

TABLE 5. Conversion of Vicinal Dichlorides to Alkenes[a]



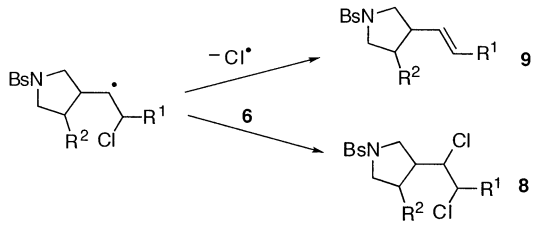
entry	dichloride	method	product		yield (%)	Ratio
1	**8a**	A		**9a**	81	74/26 [b,c]
2	**8b**	B		**9b**	54	87/13 [b,c]
3	**8c**	A		**9c**	71	>95/5
4	**8d**	A		**9d**	56	>95/5
5	**8h**	B		**9h**	86	53/47
6	**8i**	B		**9i**	84	56/44
7	**8j**	B		**9j**	74	77/23 [b,c]
8	**8k**	A		**9k**	66	N.D. [d]
9	**8l**	B		**9l**	89	–
10	**8m**	B		**9m**	66	56/44
11	**8n**	B		**9n**	68	72/28 [b,c]
12	**8o**	B		**9o**	85	78/22

[a] Reactions conditions: Conditions A: $TiCl_4$ (1.2−2.0 equiv), $LiAlH_4$ (1.2−2.0 equiv), THF reflux. Conditions B: Zn(Cu) (20 equiv), AcOH, reflux. [b] *trans*/*cis*. [c] The stereochemistry was assigned on the basis of NOE difference experiments. [d] The stereoselectivity could not be determined.

product **9b** was also obtained in 9% yield (entry 2). The formation of olefin **9** can be attributed to fragmentation of the cyclized radical via elimination of chlorine radical in the case of slow abstraction of chlorine from **6** due to steric reasons (Scheme 5).[10]

We then investigated the radical annulation reaction with a variety of alkenes (Table 4). Aromatic alkenes other than styrene yielded the corresponding pyrrolidine

SCHEME 5

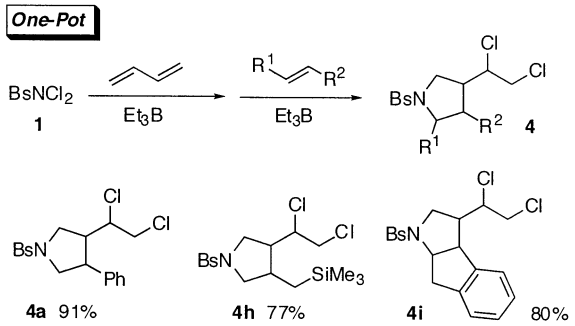


derivatives **8** in good to excellent yields with 2.0−3.0 equiv of alkenes. On the other hand, the reaction with aliphatic alkenes as a radical acceptor requires the use of a large excess (9.0 equiv) of the alkene. Although indene is a good hydrogen donor toward radical species, the radical addition-cyclization sequence is much faster than the hydrogen abstraction reaction with the *N*-centered radical derived from **6a** (entry 2). Ethyl vinyl ether was also a good partner in this radical annulation reaction (entry 8). Interestingly, cyclic alkenes such as indene and benzofuran provided the corresponding tricyclic compounds **8i** and **8m** in excellent yields (entries 2 and 6). A minor amount of alkenyl byproducts **9** was formed in addition to vicinal dichlorides when the reaction employed 1,1-disubstituted alkenes or vinyl ether as a substrate (entries 5, 7, and 8).

As a result of the presence of many stereocenters, analysis of the cyclized products **8** with NMR was quite difficult, and the isomeric ratio could not be determined. Conversion of vicinal dichloride **8** into alkenes **9** would simplify the spectrum of the products. Moreover, introduction of the alkenyl moiety is also beneficial for further functionalization of the pyrrolidine derivatives obtained with the present protocol. Dechlorination of **8** was successfully achieved upon treatment with either a low valent titanium reagent ($TiCl_4$−$LiAlH_4$) in THF (Method A) or a zinc−copper couple in refluxing acetic acid (Method B) (Table 5).[11]

Finally, we conducted this radical [2 + 2 + 1] annulation reaction among *N*,*N*-dichlorosulfonamide, 1,3-butadiene, and alkenes in a one-pot operation (Scheme 6). To a mixture of *N*,*N*-dichlorobenzenesulfonamide and 1,3-butadiene (2.0 equiv) in toluene was added a hexane solution of Et_3B at −78 °C. After stirring for 1 h at −78 °C, the reaction mixture was warmed to room temperature, and excess 1,3-butadiene was removed under reduced pressure. An addition of alkenes such as styrene, allyltrimethylsilane, or indene, followed by a solution of Et_3B at room temperature eventually furnished the desired pyrrolidine derivatives in excellent overall yields.

SCHEME 6



Conclusion

In the reaction of *N*,*N*-dichlorobenzenesulfonamide with 1-hexene, complete regioselection for anti-Markovnikov addition has achieved by using Et_3B as a radical initiator at −78 °C. The obtained product can be further utilized as a nitrogen-centered radical precursor. This protocol is applicable to construct pyrrolidine derivatives via a radical annulation reaction by taking advantage of regioselective 1,4-addition of *N*,*N*-dichlorosulfonamide to 1,3-dienes. This facile protocol provides an easy access to nitrogen heterocycles from 1,3-dienes and alkenes, demonstrating the utility of *N*,*N*-dichlorosulfonamide as a nitrogen diradical equivalent.

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Supporting Information Available: General procedures and spectral data for compounds. This material is available free of charge via the Internet at http://pubs.acs.org.

JO034043K

(10) *β*-Elimination of chlorine radical is significantly slower than chlorine abstraction. The addition of *N*,*N*-dichlorosulfonamide to allyl chloride was reported to provide a vicinal dichloride. See ref 2h.

(11) (a) Olah, G. A.; Prakash, G. K. S. *Synthesis* **1976**, 607. (b) Handa, S.; Earlam, G. J.; Geary, P. J.; Hawes, J. E.; Phillips, G. T.; Pryce, R. J.; Ryback, G. Shears, J. H. *J. Chem. Soc., Perkin Trans. 1* **1994**, 1885.