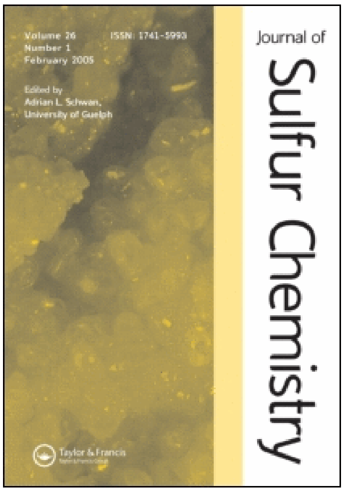

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Photolytic aziridination by thianthrene sulfilimine derivatives

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Photolytic aziridination by thianthrene sulfilimine derivatives

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Photolyses of thianthrene sulfilimine derivatives in the presence of several olefins were studied and found to afford the corresponding aziridines in good yields, particularly, in case of 2,8-dibromo-[5-(*N*-tosyl)imino]thianthrene. This reaction was considered to proceed via generation of nitrene by photolytic S–N bond cleavage of thianthrene sulfilimines. Further, photo-aziridination of olefins by thianthrene sulfilimine derivatives was intensively studied to make clear the ability as nitrene precursors.

Keywords: thianthrene; sulfilimine; photolysis; nitrene; aziridination

1. Introduction

Aziridine derivatives have been attractive and useful intermediate in organic syntheses. Many biologically important moieties, such as amino acids, β-lactam antibiotics, and alkaloids, have been derived from aziridines (*1*). Concerning the synthetic method of aziridines, it is well known that copper-catalyzed nitrogen atom transfer by the thermal decomposition of benzenesulfonyl azide in olefins probably via nitrenoid formation pathway (*2*). Aziridinations of olefins by [*N*-(*p*-toluenesulfonyl)imino]phenyliodinane (PhI=NTs) have been reported (*3*). Recently, sodium salt of *N*-chloro-*p*-toluenesulfonamide (Chloramine T) has been found to be useful as a nitrogen source for olefins in the presence of copper chloride (*4*), phenyltrimethylammonium tribromide (*5*) or iodine (*6*). Further, aziridination of olefins using sulfonamide and *t*-BuOI has been reported as a simple and efficient method (*7*). Recently, good examples of metal-catalyzed aziridination of olefins using nitrene precursor, such as PhI=NTs, several azides and amines in the presence of several ligands have been reported (*8*). The formation of aziridines by the addition of photochemically generated nitrenes to olefins is another well-known reaction. However, their utilities are limited due to the low yields by the competitive hydrogen abstraction and insertion process, and so on [for reviews, see (*9*)]. The photolysis of dimethyl and diphenyl sulfilimines were reported to form aziridines in the presence of cyclohexene in low yields (*10, 11*). Desikan *et al.* (*12*) recently reported generation of benzoylnitrene by photolysis of dibenzothiophene sulfilimine, and direct observation of the nitrene was achieved by time resolved infrared (TRIR) method.

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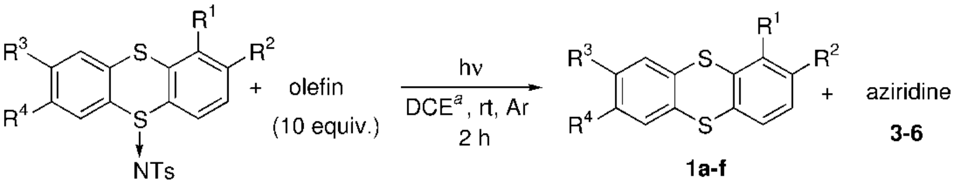
In thianthrene system, the authors have reported imino-transfer process to diphenyl sulfide in the photolysis of 5-(*N*-tosyl)iminothianthrene 10-monooxide or 10,10-dioxide in good yield via nitrene formation (*13*). In pursuing the efficient and convenient recyclable precursor for aziridination, photolysis of the various thianthrene sulfilimine derivatives, which was prepared by the same procedure as reported previously, in the presence of several olefins were studied to develop a new aziridination procedure.

2. Results and discussion

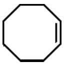
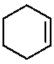
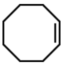
2.1. *Photolysis of substituted-[5-(N-tosyl)imino]thianthrene in the presence of several olefins*

The photolyses of 5-(*N*-tosyl)iminothianthrenes (**2a–2f**) (*13*) bearing a substituent (or substituents) on thianthrene benzene rings in the presence of several olefins were studied using a 400 W high-pressure mercury lamp with a Pyrex filter in 1,2-dichloroethane (DCE). The aziridination products were identified by the data reported elsewhere (*14, 15*). These results were summarized in Table 1.

Table 1. Effect of substituent on phenyl ring of the thianthrene in the photolysis of 5-(*N*-tosyl)iminothianthrene deivatives in the presence of cyclooctene.



2a (R^1=H, R^2=H, R^3=H, R^4=H) **2d** (R^1=H, R^2=Br, R^3=H, R^4=H)
2b (R^1=Me, R^2=H, R^3=H, R^4=H) **2e** (R^1=H, R^2=Br, R^3=H, R^4=Br)
2c (R^1=TMS, R^2=H, R^3=H, R^4=H) **2f** (R^1=H, R^2=Br, R^3=Br, R^4=H)

			Yield %[b]					
Entry	Compound	Olefin	1		3–6		7[c]	Recover
1	**2a**		12	**1a**	13	**3a**	Trace	87
2	**2b**		66	**1b**	27	**3a**	20	23
3	**2c**		72	**1c**	46	**3a**	20	13
4	**2d**		82	**1d**	64	**3a**	7	3
5	**2d**		81	**1d**	39	**4a**	20	2
6	**2d**		77	**1d**	41	**5a**	20	2
7	**2d**		78	**1d**	20	**6**	41	3
8	**2e**		85	**1e**	58	**3a**	24	0
9	**2f**		90	**1f**	70	**3a**	0	0

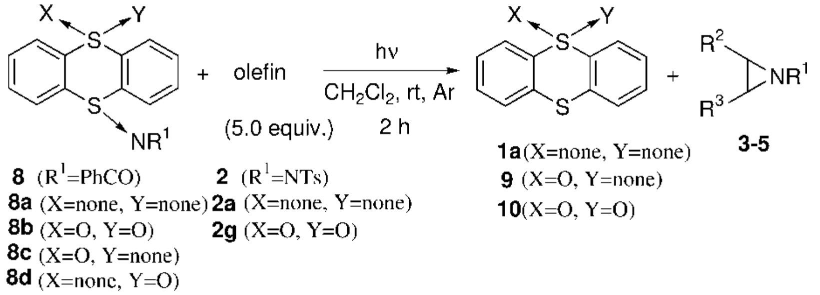
[a] 1,2-Dichloroethane; [b] Isolated yield, not optimized; [c] $TsNH_2$.

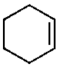
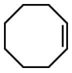
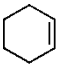
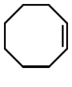
In the photolysis of the unsubstituted thianthrene *N*-tosyl sulfilimine, 5-(*N*-tosyl)iminothianthrene (**2a**), the yield of 9-tosyl-9-azabicyclo[6.1.0]nonane (**3a**) (*16*) was not high as 13%. In the case of **2b** and **2c** with methyl or trimethylsilyl (TMS) substituent(s), yields of **3a** were improved as 27 and 46%, respectively. Particularly, in the cases of **2d**–**2f** with bromine substituent(s), desired aziridine **3a** was obtained in good to high yield as 58–70% (entries 4, 8 and 9). The photolyses of **2d** in the presence of the other olefins, such as cyclohexene, cyclopentene, and 1-heptene, were also carried out. As shown in entries 5–7, the corresponding aziridines **4a**, **5a**, and **6** (*17,18*) were obtained in moderate yields. The effect of the bromine substitutuent(s) due to the electronic effect on the SN bond, however, is not clear and controversial, because **2e**, which have two bromines of apparently different electronic effect, exhibited yield improvement.

2.2. *Photolysis of 5-(N-benzoyl)iminothianthrene derivatives in the presence of several olefins*

The photolysis of 5-(*N*-benzoyl)iminothianthrene derivatives (**8a**–**8d**) (*14*) in the presence of several olefins was studied under the same conditions in CH_2Cl_2. The results were summarized in Table 2.

Table 2. Photolysis of **2**–**8** in the presence of several olefins.



Entry	Compound	Olefin	Yield %[a]				
			1a	**9**	**10**	**3–5**	Recover
1	**8a**		88			24 **5b**	5
2			77			24 **4b**	7
3			81			23 **3b**	9
4	**8b**		0	0	88	34 **4b**	10
5	**8c**		10	72		28	Trace[b]
6	**8d**		Trace	49		23	59
7[c]	**2a**		12			13 **3a**	87
8[c]	**2g**				27	18	49

[a]Isolated yield, not optimized; [b]*cis*-form; [c] DCE, 10 equiv. of cyclooctene.

The photolysis of compound **8a** in the presence of cyclopentene, cyclohexene, and cyclooctene revealed to afford the expected aziridines, 6-benzoyl-6-azabicyclo[3.1.0]hexane (**5b**), 7-benzoyl-7-azabicyclo[4.1.0]heptane (**4b**), and 9-benzoyl-9-azabicyclo[6.1.0]nonane (**3b**), in rather low yields of ca. 24%, respectively (entries 1–3) (*13,19*). In order to study the effect of the oxygen substituents at 10-position, photolyses of 5-(*N*-benzoyl)iminothianthrene 10-monooxide and 10,10-dioxide derivatives **8b–8d** in the presence of cyclohexene were carried out under the same conditions (entries 4–6), since these oxidized thianthrene sulfilimine derivatives enhanced the photo-imino-transfer reaction in the presence of diphenyl sulfide, as described later. However, the yield improvement of aziridine **4b** was not observed compared with **8a** (cf. entry 2).

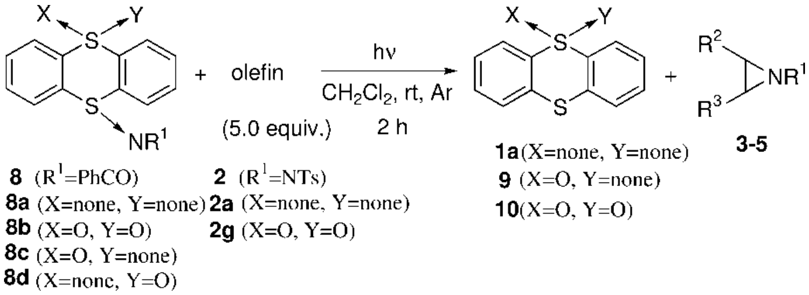
These results have revealed that the formation of aziridines by the photolysis of thianthrene sulfilimines in the presence of olefins is more effective compared with the previous Oae's work (*11*) using diphenyl sulfilimine derivatives. The aziridination by photolysis of 5-(*N*-tosyl)iminothianthrene 10,10-dioxide (**2g**) is also interesting and was carried out under the same conditions in DCE (entry 8), because, in the precedent paper by authors (*13*), 5-(*N*-tosyl)iminothianthrene 10,10-dioxide (**2g**) exhibited the great yield improvement for the photo-imino-transfer reaction toward diphenyl sulfide.

2.3. *The effect of substituents on phenyl ring in 5-[N-(p-substituted) benzoyl]iminothianthrene*

In order to study the photolyses of thianthrene sulfilimines in detail, the effect of several *para*-substituents on the phenyl ring of *N*-benzoylimino group was examined. The results were summarized in Table 3.

All the photolyses of **8a** and **11a–11c** in the presence of cyclohexene for 1 h found to decompose smoothly in 70–80% conversion. However, the formations of aziridines, *i.e.*, 7-benzoyl-7-azabicyclo[4.1.0]heptane (**4b**), 7-(*p*-methyl)benzoyl-7-azabicyclo[4.1.0]heptane (**4c**), 7-(*p*-methoxy)benzoyl-7-azabicyclo[4.1.0]heptane (**4d**) (*20*), and 7-(*p*-chloro)benzoyl-7-azabicyclo[4.1.0]heptane (**4e**) (*21*), were revealed to afford almost same yields as in around 17%, respectively, indicating practically no effect of *para*-substituents (entries 1–4).

Table 3. Photolysis of 5-[*N*-(*p*-substituted) benzoyl]iminothinthrenes **11a–11c** in the presence of cyclohexene in CH_2Cl_2.



Entry	X		Convesion %	Yield %[a]			
				1a	4		Recover
1	H	**8a**	76	65	17	**4b**	24
2	CH_3	**11a**	79	73	18	**4c**	21
3	OCH_3	**11b**	80	72	16	**4d**	20
4	Cl	**11c**	70	69	16	**4e**	30

[a] Isolated yield, not optimized.

3. Conclusion

The generations of nitrene by photolytic S-N bond cleavage of the 5-(*N*-benzoyl or *N*-tosyl)iminothianthrene derivatives were studied. In order to trap nitrenes, the photolyses were carried out in the presence of several olefins, such as cyclopentene, cyclohexene, cyclooctene, 1-hexene, and so on. 5-(*N*-Substituted-benzoyl)iminothianthrenes **8a** and **11a**–**11c** and 5-(*N*-benzoyl)iminothianthrene oxides **8b**–**8d** afforded the corresponding aziridines in poor yields (ca. 16–30%). Interestingly, in the cases of bromo-substituted-[5-(*N*-tosyl)imino]thianthrenes **2d**–**2f**, aziridines were formed in relatively high yields (ca. 58–70%), suggesting that these compounds are potent recyclable nitrene precursors for aziridine syntheses under the photolytic conditions.

4. Experimentals

4.1. *General*

All the melting points were uncorrected. The ^{1}H-NMR (400 MHz) and ^{13}C-NMR (100 MHz) spectra were recorded in $CDCl_3$ using TMS as an internal standard. The IR spectra were recorded on a HORIBA FT-71 spectrometer. The elemental analyses were performed at Microanalytical Laboratory of the Department of Material Systems Engineering and Life Science, University of Toyama. All the reactions were monitored with TLC using Silica Gel 60 F_{254} TLC plates and the products were separated by column chromatography using Silica Gel 60 and also by preparative layer chromatography using Silica Gel 60 PF_{254} with UV detection. All reagents were of the highest quality and were further purified by distillation or recrystallization. The solvents were further purified by general methods.

4.2. *General photolysis procedure*

A solution of thianthrene derivatives in a solvent was placed in a Pyrex tube equipped with a rubber septum. The solvent was used after removal of O_2 by passing Ar or N_2 bubbling for 30 min. Irradiation of samples was carried out using a 400 W high-pressure mercury lamp under Ar atmosphere at room temperature. The reaction progress was monitored by HPLC, TLC, or ^{1}H-NMR spectroscopy. After UV irradiation, the solvent was evaporated and the residue was purified by column chromatography or preparative thin-layer chromatography on silica gel, and the products were characterized by IR and NMR spectral data.

4.3. *General photolysis procedure of 5-iminothianthrene derivatives 2a–2g, 8a–8d, 11a–11c in the presence of olefins*

The solution of 5-iminothianthrene derivatives in CH_2Cl_2 and DCE (concentration = 2.5 mM) was placed in a Pyrex tube equipped with a rubber septum. The photolyses were performed under the same conditions as the general photolysis procedure after addition of 5 and 10 equiv. of olefins. The workup, separation, purification, and identification procedures were followed as the general photolysis procedure.

4.4. *General procedure of N-benzoylation of 5-iminothianthrene derivatives*

To a stirred solution of 5-iminothianthrene derivatives in CH_2Cl_2 (5 ml) was added 1.2 equiv. of triethylamine at rt. The solution was stirred for 10 min before the addition of 1.2 equiv.

of substituted benzoylchloride. After enough time by monitoring the reaction with TLC, the reaction mixture was washed with brine and water, and dried over anhydrous $MgSO_4$. After removal of solvent under reduced pressure, the residue was recrystallized from CH_2Cl_2-hexane to give 5-(*N*-benzoyl)iminothianthrene derivatives **8** and **11**.

4.5. *5-(N-benzoyl)iminothianthrene 10,10-dioxide (8b)*

Yield 89%; mp 186–188 °C (colorless crystals from CH_2Cl_2-hexane); ^{1}H-NMR ($CDCl_3$) δ 7.49–7.53 (m, 2H), 7.56–7.60 (m, 1H), 7.74–7.79 (m, 4H), 8.13–8.15 (m, 2H), 8.24–8.26 (m, 2H), 8.37–8.39 (m, 2H); ^{13}C-NMR ($CDCl_3$) δ 126.4, 127.0, 128.2, 129.4, 131.3, 132.0, 133.3, 135.4, 135.6, 138.4. IR (KBr) 1600, 1310, 1150 cm^{-1}. Anal. Calcd. for $C_{19}H_{13}NO_3S_2$: C, 62.11; H, 3.57; N, 3.81. Found: C, 62.10; H, 3.52; N, 3.84.

4.6. *trans-5-(N-benzoyl)iminothianthrene 10-monooxide (8c)*

Yield 80%; mp 177–180 °C (colorless crystals from CH_2Cl_2-hexane); ^{1}H-NMR ($CDCl_3$) δ 7.29–7.33 (m, 2H), 7.38–7.42 (m, 1H), 7.66 (dt, $J_1 = 7.6$ Hz, $J_2 = 1.2$ Hz, 2H), 7.78 (dt, $J_1 = 8.0$ Hz, $J_2 = 1.2$ Hz, 2H), 7.95–7.98 (m, 2H), 8.19 (dd, $J_1 = 7.6$ Hz, $J_2 = 1.2$ Hz, 2H), 8.26 (dd, $J_1 = 7.8$ Hz, $J_2 = 1.2$ Hz, 2H); ^{13}C-NMR ($CDCl_3$) δ 126.9, 127.9, 128.7, 129.0, 130.7, 131.2, 131.5, 132.6, 135.2, 146.8; IR (KBr) 1590, 1320, 1290, 1070 cm^{-1}. Anal. Calcd. for $C_{19}H_{13}NO_3S_2$: C, 64.93; H, 3.73; N, 9.10. Found: C, 65.05; H, 3.74; N, 9.21.

4.7. *cis-5-(N-benzoyl)iminothianthrene 10-monoxide (8d)*

Yield 89%; mp 202–204 °C (colorless crystals from CH_2Cl_2-hexane); ^{1}H-NMR ($CDCl_3$) δ 7.50–7.53 (m, 2H), 7.57–7.61 (m, 1H), 7.71 (dt, $J_1 = 7.8$ Hz, $J_2 = 1.2$ Hz, 2H), 7.78 (dt, $J_1 = 7.6$ Hz, $J_2 = 1.2$ Hz, 2H), 8.10–8.13 (m, 4H), 8.41–8.43 (m, 2H); ^{13}C-NMR ($CDCl_3$) δ 124.2, 125.7, 128.2, 128.8, 129.4, 131.1, 131.4, 131.9, 135.4, 140.2, 178.5; IR (KBr) 1590, 1320, 1290, 1180 cm^{-1}. Anal. Calcd. for $C_{19}H_{13}NO_3S_2$: C, 64.93; H, 3.73; N, 9.10. Found: C, 64.99; H, 3.74; N, 9.15.

4.8. *5-[N-(p-methyl)benzoyl]iminothianthrene (11a)*

Yield 89%; mp 248–249 °C (colorless crystals from CH_2Cl_2-hexane); ^{1}H-NMR ($CDCl_3$) δ 2.44 (s, 3H), 7.27–7.29 (m, 2H), 7.46–7.55 (m, 4H), 7.69–7.71 (m, 2H), 7.97–7.99 (m, 2H), 8.28–8.30 (m, 2H). ^{13}C-NMR ($CDCl_3$) δ 21.6, 126.8, 128.7, 129.3, 129.4, 130.6, 131.5, 133.4, 133.4, 141.7, 178.2. IR (KBr) 1600, 1320, 1280 cm^{-1}. Anal. Calcd. for $C_{20}H_{15}NOS_2$: C, 68.74; H, 4.33; N, 4.01. Found: C, 68.90; H, 4.41; N, 4.11.

4.9. *5-[N-(p-methoxy)benzoyl]iminothianthrene (11b)*

Yield 81%; mp 204–206 °C (colorless crystals from CH_2Cl_2-hexane); ^{1}H-NMR ($CDCl_3$) δ 3.89 (s, 3H), 6.98 (d, $J = 8.0$ Hz, 2H), 7.46–7.55 (m, 4H), 7.69–7.71 (m, 2H), 7.97–7.99 (m, 2H), 8.35–8.37 (m, 2H). ^{13}C-NMR ($CDCl_3$) δ 55.4, 113.1, 126.8, 128.7, 128.8, 129.4, 130.6, 131.1, 131.5, 133.5, 162.3, 177.8. IR (KBr) 1600, 1320, 1280 cm^{-1}. Anal. Calcd. for $C_{20}H_{15}NO_2S_2$: C, 65.73; H, 4.14; N, 3.83. Found: C, 65.90; H, 4.21; N, 4.01.

4.10. *5-[N-(p-chloro)benzoyl]iminothianthrene (11c)*

Yield 82%; mp 251–253 °C (colorless crystals from CH_2Cl_2-hexane); ^{1}H-NMR ($CDCl_3$) δ 7.44 (d, 2H), 7.48–7.57 (m, 4H), 7.72 (dd, $J_1 = 7.4$ Hz, $J_2 = 1.2$ Hz, 2H), 7.95–7.97 (m, 2H), 8.31–8.33 (m, 2H). ^{13}C-NMR ($CDCl_3$) δ 127.0, 128.2, 128.7, 129.5, 130.7, 130.8, 131.8, 132.8, 134.6, 137.5, 177.1. IR (KBr) 1600, 1300, 1280 cm^{-1}. Anal. Calcd. for $C_{19}H_{12}ClNOS_2$: C, 61.70; H, 3.27; N, 3.79. Found: C, 61.90; H, 3.50; N, 3.99.

4.11. *7-(p-Methyl)benzoyl-7-azabicyclo[4.1.0]heptane (4c)*

Yield 18%; Mp 77–80 °C (colorless crystals from CH_2Cl_2-hexane); ^{1}H-NMR ($CDCl_3$) δ 1.34–1.38 (m, 2H), 1.54–1.57 (m, 2H), 1.92 (m, 2H), 2.04–2.07 (m, 2H), 2.41 (s, 3H), 2.73–2.74 (m, 2H), 7.25–7.26 (m, 3H), 7.87–7.89 (d, 2H). ^{13}C-NMR ($CDCl_3$) δ 20.0, 21.6, 24.0, 37.0, 129.0, 129.1, 131.0, 143.0, 180.2. IR (KBr) 1670, 1320, 1300 cm^{-1}. Anal. Calcd. for $C_{14}H_{17}NO$: C, 78.10; H, 7.96; N, 6.51. Found: C, 78.15; H, 8.01; N, 6.55.

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