Preparation of *N*-(Arylsulfonyl)sulfoximines by Oxidation of *N*-(Arylsulfonyl)sulfilimines with Sodium Hypochlorite in a Two-Phase System

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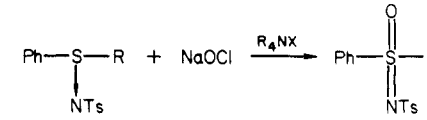
Sulfoximines are tetracoordinate organosulfur compounds isoelectronic to sulfones. The synthetic application of these attractive organosulfur compounds have not been fully explored.[1] Johnson et al. revealed that α-sodio derivatives of *N*-(arylsulfonyl)sulfoximines are useful nucleophilic alkylidene transfer reagents in the conversion of aldehydes and ketones to the corresponding oxiranes.[1] Several synthetic procedures for *N*-(arylsulfonyl)sulfoximines have been explored, namely, oxidation of sulfilimines,[2] oxidative imination of sulfoxides,[1,3] formation of sulfur–carbon linkages of sulfoximines,[4] and sulfonylation of unsubstituted sulfoximines.[5] However, apart from the sulfonylation of unsubstituted sulfoximines, other methods have only limited use in the preparation of various *S,S*-dialkyl-*N*-sulfonyl- and *S*-alkyl-*S*-arylsulfoximines.

The only unambiguous procedure to prepare *S,S*-diaryl-*N*-(arylsulfonyl)sulfoximines is the treatment of the N-unsubstituted compounds with arenesulfonyl chloride. N-Unsubstituted sulfoximines have been prepared by oxidation of N-unsubstituted sulfilimines[5] with NaOCl,[6] $KMnO_4$,[5,7] $NaIO_4$,[8] or *m*-chloroperbenzoic acid.[5] Swern et al. have reported the oxidation of *S,S*-diphenyl-*N*-tosylsulfilimine with *m*-chloroperbenzoate anion in CH_2Cl_2–H_2O.[9] Johnson et al. have shown that *S*-methyl-*S*-phenyl-*N*-tosylsulfilimine is oxidized to the corresponding sulfoximine in a good yield by treatment with H_2O_2 and NaOH.[4] However, they could not oxidize *N*-(arylsulfonyl)sulfilimines by treatment with NaOCl.[4] In contrast, we have found that NaOCl is an excellent reagent to oxidize N-unsubstituted sulfilimines to the corresponding sulfoximines in good yields in the reaction in MeOH–H_2O under alkaline condition at room temperature.[6]

We have now found a convenient preparation of *N*-(arylsulfonyl)sulfoximines by oxidation of the sulfilimines with NaOCl in a two-phase system. The oxidation is carried out by mixing a twofold excess of NaOCl solution into a vigorously stirred mixture of the sulfilimine and a quaternary ammonium salt in CH_2Cl_2–EtOAc at room temperature. Sulfilimines were consumed completely within a few hours as determined by HPLC or TLC. The results obtained are summarized in Tables I and II.

Table I. Preparation of *S,S*-Diaryl-*N*-tosylsulfoximines[a]



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sulfoximine no.	R	yield,[b] %	R_4NX[c]	mp (lit. mp), °C
1a	C_6H_5	98	a	142.5–143.0 (137–138[8])
	C_6H_5	100	b	
	C_6H_5	35	c	
	C_6H_5	43	d	
	C_6H_5	100	e	
	C_6H_5	8	f	
	C_6H_5	5	g	
	C_6H_5	trace	h	
	C_6H_5	trace	i	
	C_6H_5	trace	j	
1b	C_6H_4Cl-*p*	93	a	115.5–116.0
1c	$C_6H_4CH_3$-*p*	93	a	108.0–109.0
1d	$C_6H_4OCH_3$-*p*	90	a	89.5–90.5
1e	$C_6H_4NO_2$-*p*	94	a	150.0–150.5
1f	$C_6H_4CH_3$-*m*	90	a	95.0–96.5
1g	2-$C_{10}H_7$	95	a	163.0–164.5

[a] Satisfactory analyses (0.3% for C, H, N) and consistent infrared and NMR values were reported for **1a–g**. [b] Isolated yield. [c] a, Bu_4NBr; b, Bu_4NCl; c, Bu_4NClO_4; d, Bu_4NHSO_4; e, $CH_3(CH_2)_{15}N(CH_3)_3Br$; f, $PhCH_2NEt_3Cl$; g, $C_{12}H_{25}NMe_3Cl$; h, 18-crown-6 ether; i, dibenzo-18-crown-6 ether; j, 15-crown-5 ether.

Inspection of the results reveals the following characteristic features.

(1) All kinds of N-substituted sulfilimines, except *S*-(*o*-substituted phenyl (Me, Et, MeO, or NO_2))-*S*-phenyl- and *S*-methyl-*S*-(*o*-substituted phenyl)-*N*-substituted-sulfilimines can be converted to the corresponding sulfoximines in high yields with this reaction system. Electron-withdrawing substituents such as the *p*-NO_2 group on the phenyl ring of *S*-(*p*-substituted phenyl)-*S*-phenyl- or -*S*-methyl-*N*-tosylsulfilimines accelerated the reaction.

(2) The oxidation of *S,S*-diphenyl-*N*-tosylsulfilimine was found to depend strongly on both the catalyst and the solvent used. Quaternary ammonium salts such as Bu_4NCl, Bu_4NBr, Bu_4NHSO_4, and $CH_3(CH_2)_{15}N(CH_3)_3Br$ catalyzed the reaction, whereas generally accepted phase-transfer catalysts such as $PhCH_2NEt_3Cl$, 15-crown-5, 18-crown-6, and dibenzo-18-crown-6 did not. Ethyl acetate and CH_3CN are excellent solvents, but other common solvents such as MeOH, THF, and dioxane were ineffective. The rate of the reaction increased with increasing rate of stirring from 0 to 1000 rpm.

(3) Sulfoxides also reacted with NaOCl to give the corresponding sulfones under the same conditions. However, treatment of sulfides, e.g., diphenyl sulfide, gave neither the corresponding sulfoxides nor the sulfones.

(4) NaOCl is also known to be a chlorinating reagent. Therefore, under the condition indicated in Table II, *S*-benzyl-*N*-tosylsulfoximines once formed is immediately chlorinated at the benzylic carbon, affording eventually the α,α-dichloro sulfoximine. The sulfoximine obtained by oxidation of the *S*-benzyl derivative with *m*-chloroperbenzoic acid in CH_2Cl_2 was also chlorinated at the S-α-benzylic carbon under the same condition.

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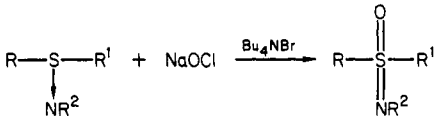
Table II. Preparation of *S*-Alkyl-*S*-aryl- or *S*,*S*-Dialkyl-*N*-substituted-sulfoximines in the Presence of Bu_4NBr[a]



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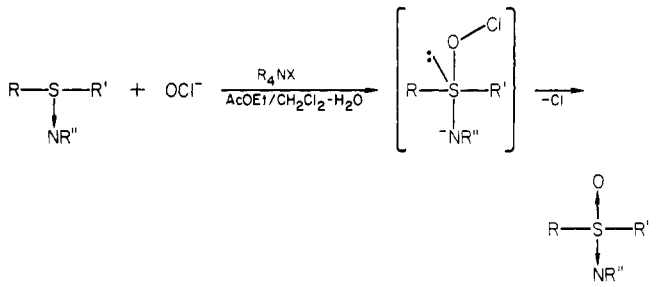
sulfoximine				yield,[b]	
no.	R	R^1	R^2	%	2, mp (lit. mp), °C
2a	C_6H_5	CH_3	$SO_2C_6H_4CH_3$-*p* (Ts)	92	110.0–110.5 (107–109[1])
2b	C_6H_5	CH_3	$SO_2C_6H_4NO_2$-*m*	85	142.0–143.0
2c	C_6H_5	CH_3	$SO_2C_6H_5$	90	91.0–92.0
2d	*p*-$CH_3C_6H_4$	CH_3	Ts	82	95.0–95.5 (140–141.5[11])
2e	*p*-$CH_3OC_6H_4$	CH_3	Ts	80	111.5–112.0 (101–102[11])
2f	*p*-ClC_6H_4	CH_3	Ts	86	126.0–126.5
2g	C_6H_5	C_2H_5	Ts	90	126.0–126.5 (123–125[2c])
2h	C_6H_5	cyclopropyl	Ts	71	115.5–117.0
2i	*p*-$CH_3C_6H_4$	cyclopropyl	Ts	80	101.5–102.5
2j	C_6H_5	cyclopentyl	Ts	78	147.0–148.0 (142.5–143.5[1])
2k	C_6H_5	cyclohexyl	Ts	73	151.0–151.5 (145.5–146.0[1])
2l	C_6H_5	cyclohexyl	$SO_2C_6H_4NO_2$-*m*	70	140.5–141.0
2m	$-(CH_2)_4-$		Ts	77	110.5–111.0 (109[12])
2n	$-(CH_2)_5-$		Ts	74	141.5–142.5 (136–137[13])
2o	CH_3	CH_3	Ts	12	169.0–170.0 (169–170[1])
	CH_3	CH_3	Ts	83[c]	
2p	C_6H_5	C_6H_5	Cl	92	150.0–151.0 (149–150[15])
2q	C_6H_5	$C_6H_5CH_2$	Ts	10[d]	151.0–152.0 (51–152[1])
	C_6H_5	$C_6H_5CH_2$	Ts	61[e]	120.5–121.5 (120–121[14])

[a] Satisfactory analyses (0.3% for C, H, N) and consistent infrared and NMR values were reported for **2b,c,f,h,i,l**. [b] Isolated yield. [c] Bu_4NHSO_4. [d] *S*-Benzyl-*S*-phenyl-*N*-tosylsulfoximine. [e] *S*-Phenyl-*S*-(α,α-dichlorobenzyl)-*N*-tosylsulfoxime.

Scheme I. Oxidation of Sulfilimines with NaOCl



These observations suggest that this oxidation involves an initial nucleophilic addition of hypochlorite anion on the *N*-(arylsulfonyl)sulfinimidoyl sulfur to form an incipient sulfurane as the intermediate and subsequent elimination of chloride anion from the sulfurane intermediate as shown in Scheme I.

Since both *N*-(arylsulfonyl)sulfilimine and NaOCl are readily in access, this reaction is an excellent general method for the synthesis of *N*-(arylsulfonyl)sulfoximines.

Experimental Section

General Procedures. Melting points of the products were measured by a Yanaco instrument and were uncorrected. IR spectra were obtained with a Hitachi 215 spectrophotometer. ^1H NMR spectra of all the compounds were obtained with a Hitachi Perkin-Elmer R-20 spectrometer in 20% $CDCl_3$ with Me_4Si as an internal standard. Liquid chromatographs were obtained by a Yanaco-L-1030 instrument using MeOH as an eluent. TLC was carried out on Merck DC-Plastikofolien Kieselgel 60F 254 Art. 5735 with fluorescent indicator with use of various solvents and mixed solvents. Development was followed with UV light or by I_2. Elemental analyses were carried out at the Chemical Analysis Center of this University. Oxidizing agent, 10% aqueous NaOCl, and phase-transfer catalysts as shown in Table I were obtained from Wako Pure Chemicals. Other chemicals were of reagent grade.

Preparation of *N*-Sulfonylsulfoximines. In a typical run, to a solution of *S*,*S*-diphenyl-*N*-tosylsulfilimine[10] (1.0 g, 2.8 mmol) and Bu_4NBr (0.4 g, 1.2 mmol) in 20 mL of AcOEt was added a twofold excess of aqueous NaOCl solution (4.2 g). The mixture was stirred vigorously at room temperature and the reaction was monitored by HPLC or TLC. After the reaction was complete, the mixture was separated upon addition of water and the organic layer was washed with H_2O. The solution was dried ($MgSO_4$) and filtered through silica gel. The resulting solution was concentrated in vacuo. The crude *S*,*S*-diphenyl-*N*-tosylsulfoximine was obtained (98%) as a white precipitate, which was then recrystallized from EtOH to afford colorless crystals: yield 98%; mp 142.5–413.0 °C (lit.[9] mp 137–138 °C); IR (KBr, cm^{-1}) 1290, 1235, 1140, 1105, 1080, 1060, 1005, 990; ^1H NMR ($CDCl_3$, δ) 2.45 (3 H, s, $C_6H_4CH_3$-*p*), 7.20–8.12 (14 H, m, aromatic protons).

Other sulfoximines listed in Tables I and II were prepared in the same manner.

Reaction of Diphenyl Sulfoxide. To a solution of diphenyl sulfoxide (1.0 g, 5.0 mmol) and Bu_4NCl (590 mg, 2.1 mmol) in 20 mL of AcOEt was added a twofold excess of aqueous NaOCl (7.4 g). The mixture was stirred vigorously at room temperature. After the sulfoxide was completely consumed, the mixture was treated as described above. Diphenyl sulfone was obtained in 98% yield.

Reaction of Diphenyl Sulfide. To a solution of diphenyl sulfide (500 mg, 2.7 mmol) and Bu_4NCl (330 mg, 1.2 mmol) in 15 mL of AcOEt was added a twofold excess of aqueous NaOCl (4.0 g). After the mixture was stirred for 24 h, HPLC and TLC showed neither formation of diphenyl sulfoxide nor diphenyl sulfone. After the usual workup, diphenyl sulfide was recovered quantitatively.

Reaction of *S*-Benzyl-*S*-phenyl-*N*-tosylsulfilimine. To a solution of *S*-benzyl-*S*-phenyl-*N*-tosylsulfilimine (1.0 g, 2.7 mmol) and Bu_4NBr (390 mg, 1.2 mmol) in 20 mL of AcOEt and 10 mL of CH_2Cl_2 was added an eightfold excess of aqueous NaOCl (16.0 g). The mixture was stirred vigorously at room temperature and the reaction was monitored by HPLC and TLC. After the sulfilimine was consumed, the mixture was quenched with H_2O and the organic layer was separated, washed with H_2O, and dried ($MgSO_4$). After the solvent was removed in vacuo, the residue

(10) For the preparation of *N*-tosylsulfilimines, see the following reviews: (a) Gilchrist, T. L.; Moody, C. J. *Chem. Rev.* **1977**, *77*, 409. (b) Oae, S.; Furukawa, N. "Sulfilimines and Related Derivatives"; American Chemical Society: Washington, DC, 1983; Monograph Series No. 179.

was chromatographed with silica gel column using $CHCl_3$ as an eluent. *S*-Phenyl-*S*-(α,α-dichlorobenzyl)-*N*-tosylsulfoximine and *S*-benzyl-*S*-phenyl-*N*-tosylsulfoximine were obtained in 61% and 10% yields, respectively. *S*-Phenyl-*S*-(α,α-dichlorobenzyl)-*N*-tosylsulfoximine, mp 120.5–121.5 °C (lit.[14] mp 120–121 °C); *S*-benzyl-*S*-phenyl-*N*-tosylsulfoximine, mp 151.0–152.0 °C (lit.[1] mp 151–152 °C).

Reaction of *S*-Benzyl-*S*-phenyl-*N*-tosylsulfoximine. The title sulfoximine was prepared by oxidation of the corresponding sulfilimine with *m*-chloroperbenzoic acid in CH_2Cl_2 (yield 90%). To a solution of *S*-benzyl-*S*-phenyl-*N*-tosylsulfoximine (400 mg, 1.0 mmol) and Bu_4NBr (150 mg, 0.5 mmol) in 10 mL of AcOEt and 10 mL of CH_2Cl_2 was added an eightfold excess of aqueous NaOCl solution (6.2 g). The mixture was stirred vigorously at room temperature and the reaction was monitored by HPLC and TLC. After the reaction was complete, the mixture was separated by addition of H_2O. The organic layer was separated and dried ($MgSO_4$). After the solvent was removed, the residue was chromatographed with silica gel column using $CHCl_3$ as an eluent. *S*-Phenyl-*S*-(α,α-dichlorobenzyl)-*N*-tosylsulfoximine was obtained in 86% yield.

Registry No. **1a**, 38764-58-8; **1a** (sulfilimine), 13150-76-0; **1b**, 80816-37-1; **1b** (sulfilimine), 24702-38-3; **1c**, 80816-38-2; **1c** (sulfilimine), 24702-37-2; **1d**, 80816-39-3; **1d** (sulfilimine), 80816-33-7; **1e**, 80816-40-6; **1e** (sulfilimine), 24698-06-4; **1f**, 80816-41-7; **1f** (sulfilimine), 80816-34-8; **1g**, 89923-37-5; **1g** (sulfilimine), 89923-38-6; **2a**, 42153-74-2; **2a** (sulfilimine), 10330-22-0; **2b**, 80816-43-9; **2b** (sulfilimine), 60121-20-2; **2c**, 80816-44-0; **2c** (sulfilimine), 38492-27-2; **2d**, 28832-82-8; **2d** (sulfilimine), 24702-26-9; **2e**, 38764-57-7; **2e** (sulfilimine), 15436-21-2; **2f**, 35539-97-0; **2f** (sulfilimine), 24702-28-1; **2g**, 69765-77-1; **2g** (sulfilimine), 10330-18-4; **2h**, 80816-45-1; **2h** (sulfilimine), 53799-63-6; **2i**, 80816-46-2; **2i** (sulfilimine), 58463-53-9; **2j**, 33332-99-9; **2j** (sulfilimine), 69765-76-0; **2k**, 33367-88-3; **25** (sulfilimine), 56561-39-8; **2l**, 80816-47-3; **2l** (sulfilimine), 80816-36-0; **2m**, 57872-24-9; **2m** (sulfilimine), 13553-70-3; **2n**, 35188-38-6; **2n** (sulfilimine), 13553-73-6; **2o**, 22236-45-9; **2o** (sulfilimine), 13150-75-9; **2p**, 70975-35-8; **2p** (sulfilimine), 42787-33-7; **2q**, 38764-59-9; **2q** (sulfilimine), 24702-30-5; **2q** (α,α-dichloro deriv.), 80824-64-2; Ph_2S, 139-66-2; Ph_2SO, 945-51-7; Ph_2SO_2, 127-63-9; Bu_4NBr, 1643-19-2; Bu_4NCl, 1112-67-0; Bu_4NClO_4, 1923-70-2; Bu_4NHSO_4, 32503-27-8; $CH_3(CH_2)_{15}N(CH_3)_3Br$, 57-09-0; $PhCH_2NEt_3Cl$, 56-37-1; $C_{12}H_{25}NMe_3Cl$, 112-00-5; 18-crown-6, 17455-13-9; dibenzo-18-crown-6, 14187-32-7; 15-crown-5, 33100-27-5.

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Bicyclic Dioxaphosphorane. 4.[1] A Kinetic Investigation of the Reactions of Trivalent Phosphorus Compounds with Bicyclic Endoperoxides

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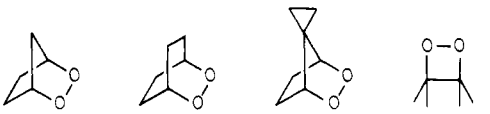
The deoxygenation of peroxides by trivalent phosphorus was first reported in 1927 by Challenger and Wilson.[2] Since that initial report, nucleophilic,[3] free-radical,[4] and biphilic[5] mechanisms have been suggested for these reactions.

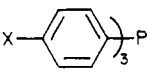
We[1] have recently reported the formation of several phosphoranes by insertion of trivalent phosphorus compounds into the oxygen–oxygen bond of 2,3-dioxabicyclo[2.2.1]heptane (1). We suggested a biphilic mechanism



1 2 3 4

for these reactions based upon the absence of a solvent effect on the rate of reaction of **1** with triphenylphosphine. We now report a more extensive kinetic study of this reaction and the reactions of bicyclic endoperoxides **2** and **3** with several phosphorus compounds. We will also compare these results to the most extensively studied peroxide, tetramethyldioxetane **4**,[6] and discuss their mechanistic implications.

The pseudo-first-order rates of reaction of **5a** with **3** were conveniently measured at five different temperatures by following the disappearance of the phosphine at 290 nm.



5a, X = H
b, X = CF_3
c, X = F
d, X = Cl
e, X = Me
f, X = OMe

The excellent linear correlations observed verify that the reactions are first order in phosphine. The second-order rate constants in Table I were determined by dividing the pseudo-first-order values by the concentrations of the peroxides. Verification that the reactions were also first order in peroxides was obtained by observing the fluctuation in the pseudo-first-order rate constants as a function of peroxide concentration. These results and the activation barriers for all four peroxides, which are typical of those observed for many bimolecular reactions,[7] are consistent with our earlier suggestion of a biphilic mechanism.

These reactions are visibly exothermic. Addition of 1 equiv of phosphine **5a** to a 0.2 M solution of **1** in a NMR tube generates enough heat to make it uncomfortable to hold the sample. This is not surprizing since a considerable amount of ring strain[8] present in the peroxides is relieved in the reaction and two stronger phosphorus–oxygen bonds (45 kcal/mol)[11] are formed at the expense of a weaker

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(8) We suggest that the relative strain energies of these peroxides parallel those of their hydrocarbon analogues.[9] The dihedral angles between the carbon–oxygen bonds[10] and consequently the lone-pair–lone-pair interactions do not change dramatically from compound to compound.
(9) (a) Goldfarb, V. I.; Belenkii, L. I. *Russ. Chem. Rev.* **1960**, *29*, 214. (b) Allinger, N. L. In "Advances in Physical Organic Chemistry"; Vol. 13. Academic Press: New York, 1978; p 48. (c) Martella, D. J.; Joner, M., Jr.; Scheleyer, P. v. R.; Maier, W. F. *J. Am. Chem. Soc.* **1979**, *101*, 7634. Supplementary material.
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