of benzene was treated with 0.11 mL (0.09 g, 1.1 mmol) of 1-ethoxy-1-propyne. This was stirred for 2 h at room temperature followed by 3 h at 40 °C. Another 0.11 mL of the alkyne was added and the reaction mixture was maintained at 40 °C for 8 h. The solution was concentrated to leave 0.25 g of a bright orange oil which was purified by column chromatography to give 0.23 g (85%) of **9** as a bright orange oil: UV (ethanol) 382 (1967); IR 2224, 1692, 1660; ^{1}H NMR δ 4.28 (d of q, J = 7.4 Hz, J' = 4.0 Hz, 2), 2.77 (q, J = 7.7 Hz, 2), 2.43 (q, J = 7.3 Hz, 2), 1.82 (s, 3), 1.45 (t, J = 6.2 Hz, 3), 1.29 (t, J = 6.2 Hz, 3), 1.12 (t, J = 7.4 Hz, 3); ^{13}C NMR 188.24, 172.96, 163.44, 123.35, 114.26, 105.82, 72.03, 62.96, 27.19, 22.58, 19.19, 15.77, 14.21, 13.77 ppm; MS (EI), m/e (relative intensity) 233 (54), 218 (40), 204 (15), 190 (100); MS (CI), m/e (relative intensity) 268 (M + 1, 59), 234 (100).

Anal. Calcd for $C_{14}H_{18}ClNO_2$: C, 62.80, H, 6.78. Found: C, 62.71; H, 7,06.

6-Chloro-6-cyano-2,4-dimethyl-3-ethoxy-5-phenyl-2,4-cyclohexadienone (10). A solution of 0.22 g (1.0 mmol) of **4** and 2 mL of benzene was treated with 0.11 mL (0.09 g, 1.1 mmol) of 1-ethoxy-1-propyne. This solution was maintained 2 h at room temperature followed by 3 h at 40 °C. Another 0.11 mL of the alkyne was added, and the reaction mixture was allowed to stir at 40 °C for 8 h. The solution was concentrated and the resulting 0.29 g of bright orange semisolid was purified by column chromatography to give 0.10 g (33%) of **48** as a bright orange semisolid: IR 2212, 1688, 1618; ^{1}H NMR δ 7.36 (m, 5), 4.26 (q of d, J = 7.0 Hz, J' = 1.3 Hz, 2), 1.89 (s, 3), 1.69 (s, 3), 1.45 (t, J = 7.0 Hz, 3); ^{13}C NMR 188.20, 168.43, 137.97, 129.17, 128.51, 127.00, 118.56, 116.19, 114.19, 72.30, 63.49, 22.54, 25.76, 15.63 ppm; MS (EI), m/e (relative intensity) 301 (M^+, 1), 267 (100), 239 (95); exact mass calcd for $C_{17}H_{16}ClNO_2$ 301.0869, found 301.0867.

3-(2-Chloro-2-cyano-1-phenyl-1-ethenyl)-1-cyclohexyl-4-(cyclohexylimino)-3-phenyl-2-azetidinone (11). A solution of 0.28 g (1.0 mmol) of **2**, 0.23 g (1.1 mmol) of 1,3-dicyclohexylcarbodiimide, and 2 mL of benzene was stirred for 16 h. The solvent was removed in vacuo to give 0.57 g of a light yellow filmy solid. This was purified by recrystallization from hexanes/ethyl acetate to give 0.37 g (76%) of **11** as white cubes, mp 156.4–157.4 °C: IR 2118, 1818, 1688; ^{1}H NMR δ 7.63 (m, 10), 3.38 (brs, 2), 2.00–0.75 (brm, 20); ^{13}C NMR 165.68, 153.14, 146.38, 135.10, 135.04, 133.55, 129.36, 129.31, 128.84, 128.68, 127.33, 112.74, 106.72, 77.64, 59.27, 51.84, 33.97, 33.76, 29.64, 29.56, 25.41, 24.95, 24.84, 24.37 ppm (The peaks at 59.27 and 51.84 ppm were clear doublets in an off-resonance decoupled spectrum.); MS (EI) m/e (relative intensity) 279 (80), 216 (100); MS (CI), m/e (relative intensity) 486 (M + 1, 100), 207 (41).

Anal. Calcd for $C_{30}H_{32}ClN_3O$: C, 74.13; H, 6.64. Found: C, 74.31; H, 6.87.

2-Cyano-3,5-diethoxy-4,6-dimethylphenol (6). To an open flask were added 0.27 g (1.0 mmol) of **5**, 6.8 mL of diethyl ether, 0.56 mL of glacial acetic acid, and 0.15 g (2.3 mmol) of zinc dust. The mixture was vigorously stirred for 2 h and then filtered. Dichloromethane (10 mL) was added to the filtrate, and it was then washed 4 times with 5-mL portions of brine. The organic layer was dried with magnesium sulfate and then concentrated to leave 0.22 g of an off-white solid. This was recrystallized from hexane to give 0.20 g (85%) of **6** as white needles, mp 107.8–108.3 °C; IR 3325, 2222, 1604; ^{1}H NMR δ 5.69 (brs, 1), 4.11 (q, J = 7.0 Hz, 2), 3.84 (q, J = 7.0 Hz, 2), 2.12 (s, 3), 2.10 (s, 3), 1.43 (t, J = 7.0 Hz, 3), 1.41 (t, J = 7.1 Hz, 3); MS (EI), m/e (relative intensity) 235 (M^+, 87), 207 (41), 179 100), 151 (43); MS (CI), 236 (M + 1, 100).

Anal. Calcd for $C_{13}H_{17}NO_3$: C, 66.36; H, 7.28. Found: C, 66.13; H, 7.31.

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Registry No. 1, 60010-89-1; **2**, 97315-61-2; **3**, 97315-62-3; **4**, 97315-63-4; **5**, 97315-65-6; **6**, 97336-06-6; **7**, 97315-64-5; **8**, 97315-66-7; **9**, 97315-67-8; **10**, 97315-68-9; **11**, 97315-69-0; **12**, 97315-70-3; PhC≡CPh, 501-65-5; $CH_3CH_2C{\equiv}CCH_2CH_3$, 928-49-4; PhC≡$CCH_3$, 673-32-5; CH≡$C(CH_2)_3CH_3$, 693-02-7; PhC≡CH, 536-74-3; (*Z*)-PhCH=$CHCH_3$, 766-90-5; EtOC≡CCH_3, 14273-06-4; dicyclohexylcarbodiimide, 538-75-0; 4-azido-3-chloro-5-methoxy-2(5*H*)-furanone, 60010-88-0.

The Formation of Thiiranes from Olefins in the Course of the Deoxygenation of Tertiary Amine *N*-Oxides by Carbon Disulfide

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In connection with our studies on oxygen transfer-mechanisms[1] we became interested in the analogous reactions of sulfur compounds. In this regard, we found that if tertiary amine *N*-oxides were deoxygenated by carbon disulfide[2,3] in the presence of olefins, the corresponding thiiranes were formed. This reaction could be synthetically valuable, since thiiranes,[4] which are not readily prepared directly from unsaturated compounds,[5-7] can be conveniently transformed to bifunctional derivatives.[8,9] We have, therefore, investigated the scope of this reaction.

Experimental Section

Materials and Methods. Analytical grade solvents were used without further purification. *N,N*-Dimethylaniline *N*-oxide,[10] tetramethylethylene sulfide,[11] and *cis*- and *trans*-stilbene sulfide[12] were made by the literature procedures. Cyclohexene sulfide, trimethylamine *N*-oxide dihydrate, and *N,N*-dimethylaniline were obtained from Aldrich. GLC analysis was performed on a Varian model 3700 equipped with FID detector and a HP 3392 A-integrator; the carrier gas was helium and a Varian WCOT capillary column (vit. silica, 18 m, 50 QC2/BPI-0.25) was used.

General Reaction Procedures. To 1.75 mL of a solution of *N,N*-dimethylaniline *N*-oxide (23 mmol/L) and olefin (2.3 mol/L) over 20 mg of anhydrous sodium sulfate, there was added with stirring, at 25–26 °C, 0.25 mL (4.15 mmol) of carbon disulfide. The reaction mixture was analyzed by GLC after 15 min. The products were identified by comparison with authentic samples, which were also used for the construction of calibration curves to determine the percentage yields.

Results Section

When employing acetonitrile as solvent, the reaction of *N,N*-dimethylaniline *N*-oxide (1) with 100-fold excess of carbon disulfide (2) yields *N,N*-dimethylaniline (DMA) in 90% and *N*-methylaniline (MMA) in 10% yield after a reaction time of 15 min. The demethylation reaction may

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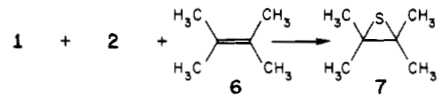
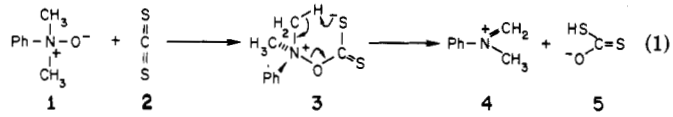
Table I. Influence of Reactant Concentrations on the Yield of 7



1:6:2	solv	% yield of 7
1:10:100	CH_3CN	20[a]
1:100:100	CH_3CN	38[a]
1:1200:120[b]	CH_3CN	52[a]
1:100:10[c]	CH_2Cl_2	34[a]
1:100:100	CH_2Cl_2	40[a]
1:100:1000	CH_2Cl_2	40[a]
4:1:17[d]	CH_3CN	6[e]

[a] Based on 1. [b] [1] = 3.3 mmol/L. [c] [1] = 10 mmol/L. [d] [1] = 88 mmol/L. [e] Yield based on 6.

be written as an intramolecular cyclic elimination reaction involving a covalent adduct (**3**)[3] of the *N*-oxide and CS_2 (eq 1). Alternatively, **3** may give way to DMA and OCS_2,



(1)

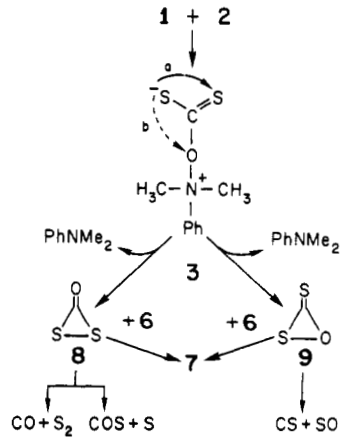
which undergoes an initial intermolecular one e^- or H^- transfer reaction, which leads eventually to MMA.

In the presence of 2,3-dimethyl-2-butene (**6**), the reaction of **1** with CS_2 provides tetramethylethylene sulfide (**7**) as a product (see equation accompanying Table I). The reaction is completed within 5 min as shown by the fact that yields are not changed when the reaction is sampled at longer times. Lowering the reaction temperature to −78 °C and gradually warming to room temperature did not noticeably alter the yield of **7**, nor did the presence or absence of anhydrous sodium sulfate as drying agent. Sodium sulfate was added, however, to all reaction solutions (Experimental Section) to maintain standard reaction conditions. The percentage yields of **7** obtained with varying ratios of reactants in acetonitrile solvent are given in Table I. Change of solvent, other reaction conditions remaining constant (**1**:**6**:CS_2 = 1:100:100), did not greatly alter the % yield of **7** (CH_2Cl_2, 40%; $CHCl_3$, 36%; C_6H_5CN, 40%; CH_3CN, 38%; CH_3OH, 31%), nor did the presence or absence of oxygen. No other products besides **7**, MMA, and DMA were observed by GLC. To probe the effect of moisture or of a reaction product on the yields of **7**, the thioepoxidation was performed in the presence of water or *N*,*N*-dimethylaniline. If a 180-fold excess of water was added to the reaction mixture (i.e., **1**:**6**:CS_2:H_2O = 1:100:100:180), the yield of **7** dropped to 29%. Adding a 100-fold excess of *N*,*N*-dimethylaniline also decreased its yield to 30%.

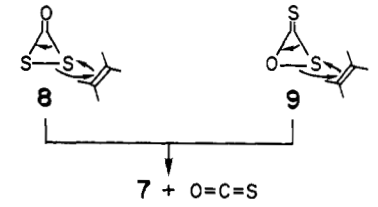
The thioexpoxidation of cyclohexene as well as *cis*-stilbene by **1** plus CS_2 was investigated in acetonitrile at 25 °C. Cyclohexene sulfide was obtained in 18% yield (as compared to the 38% yield with **7**) and the stereospecific episulfidation of *cis*-stilbene provided *cis*-stilbene sulfide in 1.8% yield.

It was of interest to ascertain if the reaction of CS_2 with other monooxygen transfer agents might be useful in the thioepoxidation of alkenes. A 35% yield of **7** was obtained when trimethylamine *N*-oxide dihydrate was employed in place of **1** with acetonitrile solvent. The use of iodosobenzene (acetonitrile), aqueous hydrogen peroxide (two-phase system with dichloromethane), and *m*-chloroperbenzoic acid (methanol/acetonitrile) with CS_2 and **6** did not give rise to detectable yields of **7**. In these experiments it cannot be excluded that thioepoxidation to some extent was followed by further oxidation of thio epoxide to a sulfone, which would escape GLC detection.

Scheme I



Scheme II



Discussion Section

Evidently, in the reaction of nonheteroaromatic tertiary amine *N*-oxides with carbon disulfide a reactive electrophilic sulfur species is generated, which can be trapped by electron-rich olefins. The conspicuous dependence of the yields of tetramethylethylene sulfide (**7**) upon the concentration of 2,3-dimethyl-2-butene (**6**) indicates that there are competing reaction pathways responsible for the disappearance of this species. Yoshimura et al.[3] postulated the formation of dithiiranone (**8**) by neighboring group assisted elimination of dimethylaniline from the covalent adduct **3**. Furthermore, **8** has been suggested as intermediate in the gas-phase reaction of CS_2 with oxygen atoms.[13] An alternative structure to be considered is oxathiiranethione (**9**) formed in an analogous fashion (Scheme I).

Both **8** and **9** could serve efficiently as sulfur-transfer agents to olefins, yielding carbon oxysulfide and thiirane (Scheme II). It has been proposed[3] that the main mode of decomposition for **8** is aqueous hydrolysis to generate CO_2 and H_2S_2. It has been shown, however, that H_2S_2 does not react with olefins to form thiiranes.[14] Furthermore, as demonstrated by our results, the yield of thiirane is little changed by the presence of water. CNDO/B calculations of the structures of **8** and **9** have been reported,[15] and monomolecular decomposition pathways have been proposed which are in agreement with experimental data from the reaction of CS_2 with atomic oxygen, the reaction products being carbon sulfide + sulfur oxide, carbon oxysulfide + sulfur and carbon monoxide + disulfur (Scheme I).[13,16] It must be noted that atomic sulfur which is formed in these reactions is known to add both in its ^3P and ^1D state to olefins to form thiiranes.[6,17] This reaction, however, proceeds at a diffusion-controlled rate with zero or negative activation energy[17,18] and the yields of thiirane

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should not show any dependence on the concentration of olefin. The absence of any effect of molecular oxygen, which should trap atomic sulfur competitively,[19] on the yield of the reaction also disfavors elemental sulfur as reactive agent in this reaction. These results strongly argue for either **8** or **9** as genuinely novel species, capable of episulfidation of olefins. Based on these results, we envision the following mechanism: Reactive **8** and/or **9** is formed in the rate-determining step from the *N*-oxide **1** and CS_2 via the covalent intermediate **3**;[3] the subsequent bimolecular reaction of **8** and/or **9** with the olefin then competes inefficiently with a rapid monomolecular decomposition of **8**/**9** with a low energy of activation,[15] thus giving only low yields of thiirane. Therefore, this reaction could only be synthetically useful if a large excess of olefin can be employed.

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Hydrolysis of 2,4-Dinitrophenyl Phosphate in Hydrophobic Ammonium Salts

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Spontaneous hydrolyses of dinitrophenyl phosphate dianions involve spontaneous breaking of the P–O bond, giving dinitrophenoxide and metaphosphate ion.[1] The metaphosphate ion reacts rapidly with nucleophiles, and this reaction occurs before the partner ions have completely separated.[2]

$$(NO_2)_2C_6H_3OPO_3^{2-} \rightarrow (NO_2)_2C_6H_3O^-PO_3^- \rightarrow P_i$$

Aqueous solutions of micellized cationic surfactants speed decomposition of dinitrophenyl phosphate dianions,[3] although the rate of the corresponding reaction of the monoanion is not affected. This micellar rate enhancement is akin to a submicroscopic solvent effect and in the absence of micelles reaction is speeded by organic solvents.[1] Rates of spontaneous dephosphorylations and decarboxylations[4] increase as the polarity of the solvent is decreased, and micellar polarities, as given by the apparent dielectric constant, or *Z* value, are lower than those of water.[5]

Hydrophobic quaternary ammonium ions containing the tri-*n*-octyl group do not form micelles, but they effectively speed attack by hydrophobic nucleophilic anions.[6,7] These hydrophobic ions can be functionalized with hydroxyl and oxime groups which are effective nucleophiles at high pH,[8] and the overall rate effects are similar to those of cationic micelles, although the magnitude of the rate increases may differ.

Table I. Interaction of 2,4-Dinitrophenoxide Ion with 2a

10^3[**2a**], M	A^a
	0.140
0.5	0.155
0.8	0.164
1.0	0.172
2.0	0.219

[a] Absorbance at 365 nm with 7×10^{-6} M 2,4-dinitrophenol at pH 10 in H_2O/MeCN (95:5 v/v).

Table II. Hydrolysis of 2,4-DNPP in 1[a]

10^3[1], M	10^4k_ψ, s^{-1}	10^3[1], M	10^4k_ψ, s^{-1}
	0.08[b]	3.0	3.2
0.5	4.5	5.0	5.0[c]
0.8	5.2	6.0	5.5[c]
1.0	3.1	7.0	3.8[c]
2.0	4.0	8.0	4.2[c]

[a] At 25.0 °C with 5×10^{-6} M 2,4-DNPP (lutidinium salt) and 10^{-3} M KOH. [b] Reference 1b. [c] Turbid solutions.

Decarboxylation of 5-nitrobenzisoxazole carboxylate ion is speeded by cationic micelles,[9] and (nonmicellizing) tri-*n*-octylmethylammonium chloride also markedly speeds decarboxylation.[10] In the present work we examined rates of spontaneous dephosphorylation of the dianion of 2,4-dinitrophenyl phosphate (2,4-DNPP) in solutions of hydrophobic ammonium ions and compared the reaction rates with those in cationic micelles.[3] The ammonium salts were tri-*n*-octylethylammonium mesylate (**1**) and tri-*n*-octyl(2-hydroxyethyl)ammonium mesylate and bromide (**2a** and **2b**, respectively) which give nucleophilic alkoxide zwitterion **3** at high pH.[8]

$$\underset{\mathbf{2}}{(C_8H_{17})_3N^+CH_2CH_2OH} \rightleftharpoons \underset{\mathbf{3}}{(C_8H_{17})_3{}^+NCH_2CH_2O^-} + H^+$$

These salts, which are also phase-transfer catalysts, are only sparingly soluble in water,[6,7] and it is sometimes desirable to increase their solubility by addition of acetonitrile.

Experimental Section

Materials. 2,4-Dinitrophenyl phosphate was prepared as the lutidinium salt, mp 141–142 °C (lit. mp 142 °C) by the method of Rawji and Milburn.[11] It was converted into the acid form by treatment with Dowex 50W-X 8 resin. The hydrophobic ammonium salts were prepared by methods described elsewhere.[7,8] Solvents were purified by standard methods.

Kinetics. Formation of 2,4-dinitrophenoxide ion was followed spectrophotometrically at 358 nm in Gilford or Beckman spectrophotometers at 25.0 °C following standard methods.[1,3] [Substrate] was ca. 7×10^{-6} M. The first-order rate constants, k_ψ, are in reciprocal seconds.

Interactions with Aryloxide Ions. The absorbance of 7×10^{-6} M 2,4-dinitrophenoxide ion in H_2O/MeCN (95:5 v/v) at pH 10 increase with increasing [**2a**] (Table I). There is no effect of **2a** if the water content of the solvent is less than 90%, cf. ref 6.

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