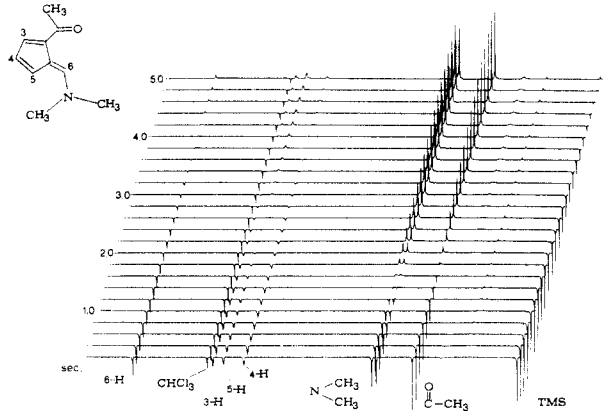

Figure 1. NMR spin-lattice relaxation spectra of 2-acetyl-6-(dimethylamino)fulvene (**3**).[5]

covered, and placed in a freezer (-24 °C) for 1 day. Filtration separated crude **9** which, after recrystallization from EtOH-H_2O, amounted to 0.095 g (65.1%) of **9** as golden yellow crystals: mp 103-104 °C; UV (hexanes) 256 nm (log ϵ 4.07), 336 (3.82), 398 (3.68); IR ($CHCl_3$) 3680 (NH), 3340 (NH), 1659 (C=O), 1590 cm^{-1} (C=C); NMR (acetone-d_6) δ 2.47 (s, 3, CH_3C=O), 6.26 (apparent t, 1, H-4, $J_{4,3}$ = 4.0 Hz, $J_{4,5}$ = 4.2 Hz), 7.10 (m, 7, H-5, H-3, and phenyl), 8.02 (d, 1, H-6, J = 9 Hz), 8.39 (m, 1, NH), 14.22 (br, 1, NH).

Anal. Calcd for $C_{14}H_{14}N_2O$: C, 74.31; H, 6.24; N, 12.38. Found: C, 74.36; H, 6.38; N, 12.30.

2-Acetyl-6-piperidylfulvene (10). A solution of 0.245 g (1.5 mmol) of 2-acetyl-6-(dimethylamino)fulvene (**3**) and 0.45 mL (0.387 g, 4.5 mmol) of piperidine in 20 mL of THF was stirred at room temperature for 2 days (TLC with 10:3 petroleum ether-EtOAc showed one major, yellow, UV-absorbing spot at R_f 0.31). Removal of the volatile components at 35 °C (reduced pressure) and chromatography (preparative TLC with 10:3 petroleum ether-EtOAc) of the brownish yellow oil gave three colored bands. The second band was extracted with THF and the solvent removed (vacuum pump). Recrystallization of the yellow solid residue from petroleum ether and drying under N_2 gave 0.235 g (77.1%) of **10** as yellow needles: mp 84-85 °C; UV (hexanes) 361 nm (log ϵ 4.33), 236 (4.12); IR ($CHCl_3$) 2950 (CH), 1630 C=O), 1602 cm^{-1} (C=C); NMR (CCl_4) δ 1.79 (br s, 6, CH_2), 2.36 (s, 3, CH_3C=O), 372 (br, 4, NCH_2), 6.20 (apparent t, 1, H-5, $J_{5,4}$ = 4.2 Hz, $J_{5,3}$ = 2.0 Hz), 6.89 (dd, 1, H-3, $J_{3,4}$ = 4.0 Hz, $J_{3,5}$ = 2.0 Hz), 8.79 (s, 1, H-6).

Anal. Calcd for $C_{13}H_{17}NO$: C, 76.81; H, 8.43; N, 6.89. Found: C, 76.94; H, 8.50; N, 6.79.

2-Acetyl-6-(diisopropylamino)fulvene (11). A mixture of 0.855 g (0.5 mmol) of 2-acetyl-6-(dimethylamino)fulvene (**3**), 2.1 mL (1.516 g, 1.5 mmol) of diisopropylamine, and 20 mL of absolute EtOH under an atmosphere of Ar was refluxed for 3.5 days (TLC with 4:1 EtOAc-petroleum ether showed spots for **3** at R_f 0.27 and for **11** at R_f 0.77). Removal of the volatile components under reduced pressure and preparative TLC of the brownish yellow residue with 4:1 EtOAc-petroleum ether gave two yellow bands. Extraction of the leading (R_f 0.77) band with THF and removal of the solvent left a yellow solid. Recrystallization from petroleum ether gave 0.0236 g (21.5%) of **11** as yellow crystals: mp 115-116 °C; UV (hexanes) 242 nm (log ϵ 3.99), 363 (4.03); IR ($CHCl_3$) 2920 (CH), 1620 (C=O), 1582 cm^{-1} (C=C); NMR ($CDCl_3$) δ 1.39 (d, 6, $(CH_3)_2C$, J = 6 Hz), 2.50 (s, 3, CH_3C=O), 3.85 (m, 1, Me_2CH), 4.89 (m, 1, Me_2CH), 6.45 (apparent t, 1, H-4, $J_{4,3}$ = 4.0 Hz, $J_{4,5}$ = 4.2 Hz), 6.88 (dd, 1, H-5, $J_{5,4}$ = 4.2 Hz, $J_{5,3}$ = 2.0 Hz), 7.15 (dd, 1, H-5, $J_{5,4}$ = 4.2 Hz, $J_{5,3}$ = 2.0 Hz), 7.15 (dd, 1, H-3, $J_{4,3}$ = 4.0 Hz, $J_{3,5}$ = 2.0 Hz), 9.33 (s, 1, H-6).

Anal. Calcd for $C_{14}H_{21}NO$: C, 76.67; H, 9.65; N, 6.39. Found: C, 76.46; H, 9.85; N, 6.45.

Registry No. (*E*)-**3**, 81158-08-9; **4**, 81158-09-0; **5**, 81158-10-3; **6**, 81158-11-4; **7**, 81158-12-5; **8**, 81158-13-6; **9**, 81158-14-7; **10**, 81158-15-8; **11**, 81158-16-9; hydroxylammonium chloride, 5470-11-1; 2-aminoethanol, 141-43-5; aniline, 62-53-3; *p*-(methylammonio)anilinium dichloride, 5395-70-0; *o*-aminoaniline, 95-54-5; phenylhydrazine, 100-63-0; piperidine, 110-89-4; diisopropylamine, 108-18-9.

Quinone Chemistry. Synthesis of a Masked Naphthazarin Synthon

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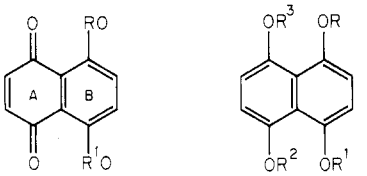
As part of a larger synthetic problem we required a naphthazarin (**1**) based synthon in which the B ring was



1, R = R^1 = H
2, R = R^1 = Me
3, R = H; R^1 = Me
4, R = Ac; R^1 = Me
5, R = Bz; R^1 = Me
6, R = H; R^1 = R^2 = R^3 = Me
7, R = R^2 = R^3 = Ac; R^1 = Me
8, R = Bz; R^1 = Me; R^2 = R^3 = H
9, R = Bz; R^1 = R^3 = Me; R^2 = H
10, R = Bz; R^1 = R^2 = R^3 = Me
11, R = Bz; R^1 = R^2 = Me; R^3 = H

Bz = COC_6H_5

monoprotected and the quinone system masked in such a way to increase the electron density of the B ring and yet permit facile regeneration of the quinone at a later stage of the synthesis. These requirements are fulfilled by the tetrasubstituted naphthalene **6**. In this paper we report the preparation of **6** from naphthazarin (**1**) which features the direct monoalkylation of **1**.

Several groups have reported the methylation of naphthazarin (**1**) with methyl *p*-toluenesulfonate or methyl iodide and silver oxide to afford the dimethyl ether **2** as the principal product.[1-4] The monomethyl ether **3** was reported as a side product in one of these preparations.[3] Subsequent to the beginning of our work a report[5] describing an efficient, albeit multistep, synthesis of a monoalkyl naphthazarin via a sequence involving monoacylation, alkylation, and deacylation appeared.

In our hands the reaction of **1** with methyl iodide and silver oxide in refluxing $CHCl_3$, as monitored by TLC, afforded an initial product more polar than **1** which was slowly consumed to afford a second still more polar product. After consumption of **1** as determined by TLC, the initial product could be isolated in 47% yield and was characterized as the monoethyl ether **3**. The second product, characterized as the dimethyl ether **2**, was also obtained in 10% yield. Upon prolonged reaction, **2** became the major reaction product.

The seemingly anomalous chromatographic mobilities of **1-3** can be explained by the strong intramolecular hydrogen bonds between the peri hydroxyl and carbonyl moieties, preventing the interaction of these polar groups with the absorbent. Methylation disrupts the hydrogen-bonded system, permitting interaction of the ether and

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carbonyl groups with the silica gel and thereby decreasing mobility.

Our planned protection of the quinone moiety involved acylation of **3** and reductive methylation of the quinone followed by deacylation. The quinone could be regenerated when needed via oxidative demethylation with silver(II) oxide[6] or ceric ammonium nitrate.[7] However, acylation (acetic anhydride, pyridine, reflux) of **3** proceeded with concomitant reduction of the quinone to afford the triacetate **7** as the major product instead of the expected **4**. Milder acylation conditions (benzoyl chloride, pyridine, 0 °C) did provide an acceptable monoacylated quinone **5** in 70% yield. Reduction of **5** with $SnCl_2$ afforded the dihydroquinone **8** in ca. 90% yield. The crude product was immediately methylated with dimethyl sulfate and K_2CO_3 in acetone at reflux. Again it was possible to isolate a partially methylated product, in this case **9**. Resubmission of **9** to the methylation conditions afforded the fully methylated product **10** in 43% yield from quinone **5**.

Characterization of **9** was based on its ^1H NMR spectrum in which a significant difference was observed in the chemical shifts, δ 4.05 and 3.45, of the methoxy groups, reflecting their different electronic environments in **9**. Greater similarity would be expected of these chemical shifts in the alternate structure **11**, in which neither methoxyl oxygen is hydrogen bonded. The observed regioselectivity can be rationalized as resulting from the stronger hydrogen bond of the phenolic proton peri to the methyl group vs. that peri to the benzoyloxy substitutient due to the greater electron density about the methoxyl oxygen. This would enhance the relative nucleophilicity of the latter phenol, making it more susceptible to methylation.

Deacylation of **10** was achieved by refluxing in ethanol in the presence of Dowex 2X-8 resin (OH^- form) to afford **6** in 73% yield. While **6** was sufficiently stable to permit complete characterization, upon storage it underwent decomposition to **2**. In practice it is best to stockpile material as **10** and generate **6** only as needed. Further investigations regarding the chemistry of **6** and its use as a masked naphthazarin synthon are underway and will be reported in due course.

Experimental Section

Naphthazarin was obtained from Tridom Chemical Inc. and was used without further purification. Ag_2O was freshly prepared immediately prior to use.[8,9] Solvent extracts of aqueous solutions were dried over anhydrous Na_2SO_4. Solutions were concentrated under reduced pressure by using a rotary evaporator. Melting points were determined with a Thomas-Hoover capillary melting point apparatus and are uncorrected. Specta were determined as follows: UV–visible, Cary 11 or Cary 14 recording spectrometers; IR (Nujol mull), Perkin-Elmer 137; ^{1}NMR ($CDCl_3$ solution, Me_4Si internal reference) measured by Mr. L. Garver using a Varian XL-100 spectrometer. Elemental microanalyses were provided by the microanalytical laboratory of Stanford University and by Galbraith Laboratories, Knoxville, TN. Thin-layer chromatograms (TLC) were obtained on silica gel GF 250-μm plates (Analtech). Preparative layer chromatograms (PLC) were obtained on 20 × 20 × 0.2 cm silica gel 60 F-254 plates (E. Merck).

5-Hydroxy-8-methoxy-1,4-naphthoquinone (3). To a solution of naphthazarin (3.0 g, 15.8 mmol) in $CHCl_3$ (120 mL) were added freshly prepared Ag_2O (4.5 g) and methyl iodide (5.0 mL) with stirring over 20 min, and the reaction mixture was stirred at 40 °C for 16 h. Additional Ag_2O (3.0 g) and MeI (5 mL) were added over 3 h, and stirring was continued at 40 °C for 5 h after the addition. The reaction mixture was filtered through Celite and the filtrate evaporated. The residue was recrystallized from acetone to afford 0.9 g of **3**. The mother liquors were evaporated, and the residue was chromatographed (PLC, silica gel 60, $CHCl_3$) to afford an additional 0.6 g of **3**: total yield 1.5 g (47%); mp 145 °C dec; IR 6.14 (C=O), 6.28, 6.43, 6.89, 7.03, 7.35, 7.53, 7.80, 8.08, 8.50, 8.67, 9.03, 9.18, 9.68, 10.63, 11.32, 11.70, 11.87, 12.04, 12.85, 13.82 μm; NMR δ 3.98 (s, 3, OMe), 6.87 (s, 2, 2- and 3-H's), 7.33 (m, 2, 6- and 7-H's), 12.40 (s, 1, OH); UV–vis (MeOH) λ_{max} 261 nm (ϵ 9700), 483 (4900); TLC R_f 0.2 ($CHCl_3$), 0.5 (1:1 hexane/EtOAc).

Anal. Calcd for $C_{11}H_8O_4$: C, 64.69; H, 3.97. Found: C, 64.61; H, 4.01.

Also isolated by PLC was 200 mg (10%) of **2**: mp 148–151 °C (lit.[1] mp 157 °C); IR 6.11 (C=O), 6.40, 6.92, 7.31, 7.58, 7.84, 8.05, 8.50, 9.10, 9.60, 9.82, 11.08, 11.63, 12.40, 13.85 μm; NMR δ 3.99 (s, 6, OMe), 6.80 (s, 2, 2- and 3-H's), 7.35 (s, 2, 6- and 7-H's); UV–vis (MeOH) λ_{max} 255–256 nm (ϵ 42900), 448–449 (13700); TLC (1:1 hexane/EtOAc) R_f 0.15.

5-(Benzoyloxy)-8-methoxy-1,4-naphthoquinone (5). To a solution of **3** (2.0 g, 1.0 mmol) in pyridine (50 mL) at 0 °C was added benzoyl chloride (3.0 mL), and the mixture was stirred at 0 °C for 40 min. H_2O (350 mL) was added slowly with stirring, and the yellow precipitate was collected. The precipitate was suspended in 5% Na_2CO_3 (5 mL), filtered, washed with water, and dried. The crude product from nine reactions on the above scale was recrystallized from $CHCl_3$/petroleum ether to afford **5**: 20.0 g (74%); mp 183–184 °C; IR 5.82 (ester C=O), 6.09 (quinone C=O) 6.20, 6.40, 6.95, 7.20, 7.32, 7.51, 7.80, 7.92, 8.02, 8.19, 8.45, 8.58, 9.18, 9.32, 9.45, 9.72, 9.80, 11.43, 11.80, 12.00, 12.60, 13.50, 14.25 μm; NMR δ 4.03 (s, 3, OMe), 6.84 (d, 1), 6.92 (d, 1), 7.3–7.8 (m, 5) 8.25 (dd, 2); UV (MeOH) λ_{max} 239–240 nm (ϵ 23100), 399 (3800); TLC (98:2 CH_2Cl_2/MeOH) R_f 0.45.

Anal. Calcd for $C_{18}H_{12}O_5$: C, 70.13; H, 3.92. Found: C 69.98; N, 3.99.

1-(Benzoyloxy)-4,5,8-trimethoxynaphthalene (10). To **5** (10.0 g, 32.4 mmoles) in 95% EtOH (1 L) was added a solution of concentrated HCl (600 mL) and $SnCl_2 \cdot 2H_2O$ (60 g) in H_2O (2 L). The solution was refluxed for 1 h, filtered, and cooled. The solution was extracted with $CHCl_3$ (5 × 500 mL). The extracts were combined, dried, and evaporated to afford 9 g of crude **8**. The residue (18 g) from two 10-g reactions was dissolved in acetone (1.5 L), dimethyl sulfate (160 mL) and anhydrous K_2CO_3 (240.0 g) were added, and the mixture was refluxed for 15 h. The reaction mixture, which contained **9** as the major product, was filtered. The filtrate was dried over anhydrous K_2CO_3 for 24 h and evaporated. The residual syrup was dissolved in acetone (800 mL), dimethyl sulfate (80 mL) and anhydrous K_2CO_3 (120 g) were added, and the mixture was refluxed for 24 h, cooled, filtered, and evaporated. The residual syrup was stirred with 2 M NH_4OH (500 mL) for 30 min to remove the excess dimethyl sulfate. The mixture was extracted with $CHCl_3$ (3 × 500 mL). The extracts were combined, dried, and evaporated. The residue was chromatographed (Prep 500, silica gel, CH_2Cl_2) to afford **10**: 11.5 g (52%); mp 185–187 °C; IR 5.82 (ester C=O), 6.30, 6.62, 6.95, 7.30, 7.60, 8.00, 8.20, 8.40, 8.60, 8.75, 8.90, 9.21, 9.40, 9.45, 9.73, 10.02, 11.55, 12.04, 12.30, 13.25, 13.80, 14.50 μm; NMR δ 3.54, (s, 3, 8-OMe), 3.94 and 4.00 (2 s, 6, 4- and 5-OMe's), 6.74 (d, 1, J = 8 Hz), 6.87 (d, 1, J = 8 Hz), 6.95 (d, 1, J = 8 Hz, 3-H), 7.16 (d, 1, J = 8 Hz, 2-H), 7.56 (m, 3), 8.25 (m, 2); UV (MeOH) λ_{max} 223 nm (ϵ 8200), 312 (1300); TLC (99:1 $CHCl_3$/MeOH) R_f 0.4.

Anal. Calcd for $C_{20}H_{18}O_5$: C, 71.00; H, 5.35. Found: C, 70.96; H, 5.57.

From an earlier run an analytical sample of **1-(benzoyloxy)-5-hydroxy-4,8-dimethoxynaphthalene (9)** was isolated: mp 186–187 °C; IR 3.00 (OH), 5.80 (ester C=O), 6.20, 6.60, 6.90, 8.00, 8.30, 8.45, 8.60, 8.70, 9.00, 9.25, 9.40, 9.65, 9.83, 11.50, 11.80, 12.10, 12.50, 13.10, 14.00 μm; NMR δ 3.54 (s, 3, 8-OMe), 4.05 (s, 3, 4-OMe), 6.62 (d, 1, J = 8 Hz), 6.75 (d, 1, J = 8 Hz), 6.89 (d, 1, J = 8 Hz, 3-H), 7.09 (d, 1, J = 8 Hz, 2-H), 7.50 (m, 3), 8.24 (dd, 1, J = 8, 2 Hz), 9.70 (s, 1, OH); UV (MeOH) λ_{max} 224 nm (ϵ 62600), 311–312 (9400), 334 (10200), 349.5 ′11144); TLC (99:1 $CHCl_3$/MeOH) R_f 0.5.

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(9) The use of commercial Ag_2O gave qualitatively similar results but resulted in a slower reaction and lower yields of product.

Anal. Calcd for $C_{19}H_{16}O_5$: C, 70.36; H, 4.97. Found: C, 70.11; H, 5.04.

1-Hydroxy-4,5,8-trimethoxynaphthalene (6). Compound 10 (200 mg, 0.59 mmol) and Dowex 2X-8 resin (200 mg, OH^- form) were placed in degassed absolute ethanol (20 mL) under N_2 and refluxed for 3 h. The reaction mixture was filtered to remove the resin and the filtrate cooled in an ice bath. The product crystallized from the filtrate and was collected to afford 88.0 mg of 6. The mother liquor was evaporated and the residue crystallized from $CHCl_3$/hexane to afford an additional 12.5 mg of 6: total yield 100.5 mg (73%); mp 148–149 °C; IR 2.95 (OH), 6.27, 6.60, 6.85, 6.95, 7.10, 7.28, 7.81, 8.00, 8.35, 8.78, 8.90, 9.38, 9.72, 9.98, 11.00, 12.15, 12.45, 13.40, 13.75, 14.70 μm; NMR δ 3.90, 3.92 (2 s, 6, 4- and 5-OMe's), 4.03 (s, 3, 8-OMe), 6.76 (s, 2, 6- and 7-H's), 6.85 (m, 2, 2- and 3-H's), 9.43 (s, 1, OH); UV (MeOH) λ_{max} 223 nm (ε 50400), 310–134 (8400), 334 (7800), 353 (8600); TLC ($CHCl_3$) R_f 0.6.

Anal. Calcd for $C_{13}H_{14}O_4$: C, 66.65; H, 6.02. Found: C, 66.62; H, 6.01.

Acknowledgment. This investigation was supported by PHS Grant CA awarded by the National Cancer Institute, DHHS.

Registry No. 1, 475-38-7; 2, 15013-16-8; 3, 4923-61-9; 4, 81194-55-0; 6, 81194-56-1; 7, 81194-57-2; 8, 81194-58-3; 9, 81194-59-4; 10, 81194-60-7.

Mechanism of Reaction of Oxetanes, Sodium, and Dimethyldichlorosilane. Synthesis of 1-Oxa-2-silacyclopentanes

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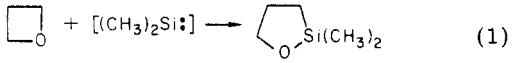
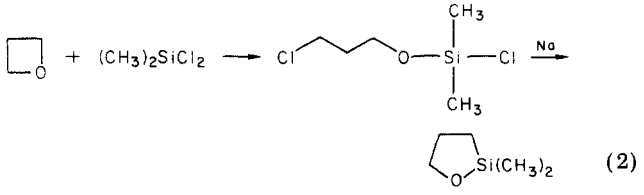
Reaction of dimethyldichlorosilane, sodium metal, and oxetane in refluxing toluene yields 2,2-dimethyl-1-oxa-2-silacyclopentane (42%, eq 1), while a similar reaction with 3,3-dimethyloxetane gives 2,2,4,4-tetramethyl-1-oxa-2-silacyclopentane (47%).



(1)

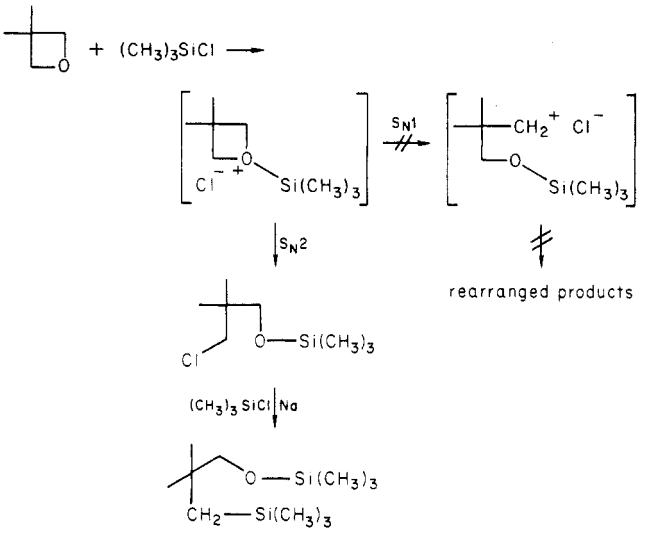
Since photochemically generated dimethylsilylene is known to react with oxetanes to yield similar products,[1] one might be led to propose that dimethyldichlorosilane undergoes reduction by sodium metal under these reaction conditions to form dimethylsilylene. While this explanation is economical, we are reasonably certain that it is not correct. Rather we have evidence that the reaction probably occurs by the following sequence of events. Initial reaction of dimethyldichlorosilane with oxetane (eq 2)



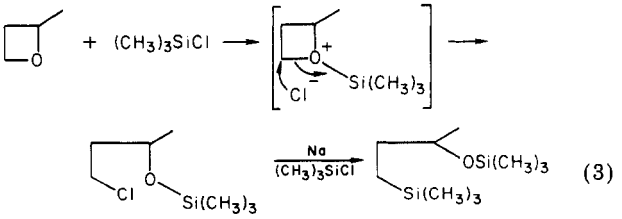
(2)

yields (3-chloropropoxy)dimethylchlorosilane[2] which then undergoes reductive cyclization with sodium to form 2,2-dimethyl-1-oxa-2-silacyclopentane. Independent experiments are consistent with this interpretation. Oxetane does react with dimethyldichlorosilane to give (3-chloropropoxy)dimethylchlorosilane in virtually quantitative yield. In addition, 2,2-dimethyl-1-oxa-2-silacyclopentane has been prepared by treatment of (3-chloropropoxy)dimethylchlorosilane with sodium metal.[3] The ethyl iodide catalyzed reaction of tetrahydrofuran with dimethyldichlorosilane and magnesium metal to yield 2,2-dimethyl-1-oxa-2-silacyclohexane is probably closely related.[4-6] In addition, we have found that oxetane reacts with trimethylchlorosilane and sodium metal in refluxing toluene to yield [3-(trimethylsiloxy)propyl]trimethylsilane (55%) and (3-chloropropoxy)trimethylsilane (8%). Similar reactions of 2-methyloxetane and 3,3-dimethyloxetane yield, respectively, [3-methyl-3-(trimethylsiloxy)propyl]trimethylsilane (52%) and [2,2-dimethyl-3-(trimethylsiloxy)propyl]trimethylsilane (48%).

Scheme I



These observations, we believe, provide insight into the mechanism of carbon–oxygen bond cleavage of oxetanes by chlorosilanes. The cleavage of epoxides by chlorosilanes has been thoroughly studied.[7-9] The following sequence of events is consistent with our data. Nucleophilic attack by oxygen lone pairs of electrons of the oxetane on the silyl center of the halosilane leads to a silyl-substituted oxonium ion/chloride ion pair (eq 3). Nucleophilic attack by



(3)

chloride on the least substituted alpha carbon of the silyl substituted oxonium ion results in carbon-oxygen bond

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