of this material by flash silica gel chromatography (20 mm × 16 cm column using 2:1 hexane–EtOAc as eluant) afforded 88 mg of pure **6b** (90%): R_f 0.44 (1:1 hexane–EtOAc); ^{1}H NMR (250 MHz, $CDCl_3$) δ 5.87 (d, 1 H, J = 3.6 Hz, H_4), 5.55 (br dd, 1 H, J = 1.2, 5.9 Hz, H_{10}), 4.76 (t, 1 H, J = 2.9 Hz, THP), 4.30 (br d, 1 H, J = 5.9 Hz, H_{11}), 4.25 (dd, 1 H, J = 3.6, 4.7 Hz, H_3), 4.00 (m, 1 H, THP), 3.83 and 3.64 (AB, 2 H, J = 12.6 Hz, H_{15a}, H_{15b}), 3.75 (d, 1 H, J = 4.8, H_2), 3.01 and 2.74 (AB, 2 H, J = 3.9 Hz, H_{13}), 2.02 (s, 3 H, OAc), 1.70 (s, 3 H, H_{16}), 0.75 (s, 3 H, H_{14}); IR ($CHCl_3$) 3620, 3480 (br), 2940, 1720, 1435, 1370, 1210 (br), 1120, 1070, 1020, 970 cm^{-1}.

4β-Acetoxyscirpene-3α,15-diol (2). To a solution of THP ether **6a** (57 mg, 0.14 mmol) in 0.25 mL of THF was added 0.25 mL of H_2O and 0.5 mL of glacial HOAc. The reaction was stirred for 5 days at ambient temperature. The reaction was coevaporated with heptane (3 × 50 mL) to remove HOAc and H_2O. The resulting crude product (67 mg) was purified by flash silica gel chromatography (using a 10 mm × 14 cm column, 1:1 hexane–EtOAc as eluant) to afford 42 mg of **2** (90% yield). The yield of **2** from **6b** was comparable (75% of **2** plus 15% of recovered **6b** after a 3 day reaction period). Attempts to crystallize **2** were unsuccessful: $[\alpha]_D^{22}$ +10.0° (*c* 1.2, 99.7%, acetone), lit.[4] $[\alpha]_D^{22}$ +10.3° (*c* 1, acetone); R_f 0.20 (1:1 hexane–EtOAc), 0.48 (2:1 benzene–acetone); NMR (250 MHz, $CDCl_3$) δ 5.56 (br d, 1 H, J = 5.7 Hz, H_{10}), 5.50 (d, 1 H, J = 3.4 Hz, H_4), 4.24 (dd, 1 H, J = 4.6, 8.1 Hz, H_3), 4.18 (d, 1 H, J = 4.8 Hz, H_{11}), 3.8 (br d, 1 H, J = 10.7 Hz, H_{15a}), 3.65 (d, 1 H, J = 4.9 Hz, H_2), 3.61 (br d, 1 H, J = 10 Hz, H_{15b}), 3.05 and 2.76 (AB, 2 H, J = 4.0 Hz, H_{13}), 2.82 (br d, 1 H, J = 3.9 Hz, OH), 2.18 (s, 3 H, OAc), 1.72 (br s, 3 H, H_{16}), 0.65 (s, 3 H, H_{14}); IR ($CHCl_3$) 3600–3300 (br), 2940, 1720, 1370, 1250, 1070, 950 cm^{-1}; mass spectrum m/e 306 ($M^+ - H_2O$); CI mass spectrum of the bis(trimethylsilyl) derivative, m/e 469 (MH^+), 453 ($M^+ - CH_3$), 409 (M^+ - OAc); high resolution mass spectrum, calcd for $C_{17}H_{22}O_5$ ($M^+ - H_2O$) 306.1467), found 306.1492 ± 0.0007.

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Cyanoketenes. Cycloadditions of Chlorocyanoketene to Alkynes. Generation of Vinylogous Cyanoketenes

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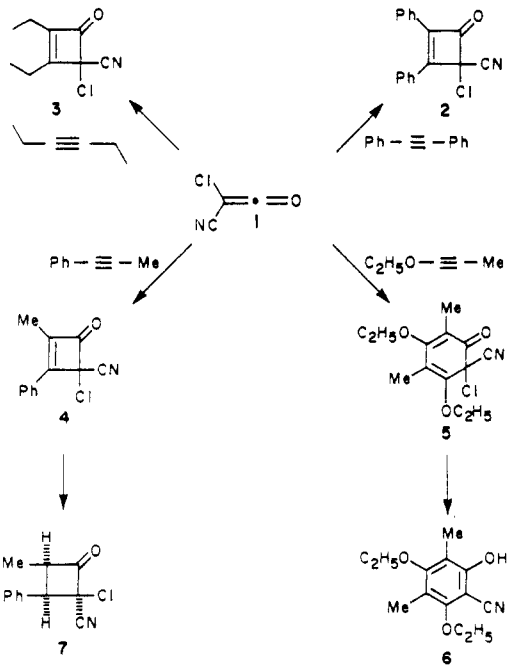
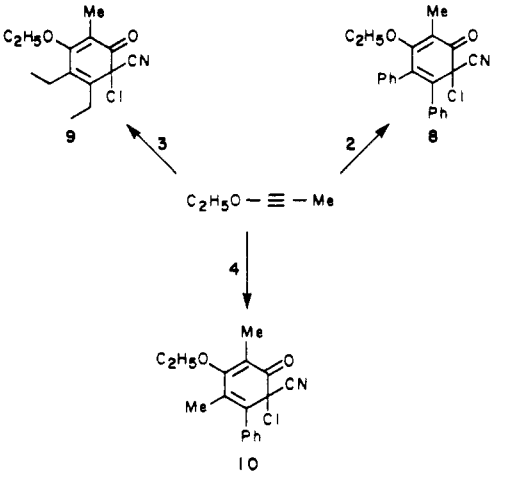
Chlorocyanoketene (CCK), an exceptionally reactive electrophilic ketene, has previously been shown to undergo facile cycloadditions to imines,[1] formimidates,[2] aryl aldehydes,[3] sulfurdimides,[4] and alkenes.[5] Its ability to cycloadd to selected alkynes is reported in this manuscript. Also reported is the observation that the resulting cyclobutenones function as precursors to vinylketenes. Indeed, the 4-chloro-4-cyanocyclobutenones arising from the initial cycloadditions are in equilibrium with their respective vinylketenes at ambient temperature, and these reactive intermediates can be intercepted by other ketenophiles. The details of these results are given below.

Scheme I



Scheme II



The cycloadditions were carried out in toluene (103 °C) by generating approximately 3 equiv of CCK in the presence of 1 equiv of the aklyne. The alkynes employed were 1-hexyne, phenylacetylene, diphenylacetylene, 3-hexyne, 1-phenylpropyne, and 1-ethoxypropyne. The reactions of the terminal alkynes with CCK gave complex mixtures from which no pure product was obtained. On the other hand, the internal alkynes, diphenylacetylene, 3-hexyne, and 1-phenylpropyne gave, respectively the cyclobutenones **2** (77%), **3** (61%), and **4** (84%) (Scheme I). The formation of **4** deserves further comment since only the indicated regioisomer was observed. Its structure was shown to be 4-chloro-4-cyano-2-methyl-3-phenylcyclobutenone as established by its conversion (H_2, Pd–C, 40%) to the corresponding cyclobutanone, **7**, which was independently prepared from the cycloaddition of CCK to (*Z*)-1-phenylpropene.[5]

A most interesting transformation took place when 1-ethoxypropyne was treated with CCK under the above reaction conditions. No cyclobutenone was observed. Rather, the cyclohexadienone, **5**, was obtained as a yellow crystalline solid in 56% yield. The structure of **5** is based upon its spectral properties, as well as upon the observation that it undergoes facile reductive elimination (Zn,

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(5) The cycloaddition of CCK to alkenes has previously been reported. See: Fishbein, P. L.; Moore, H. W. *J. Org. Chem.* **1984**, *49*, 2190.

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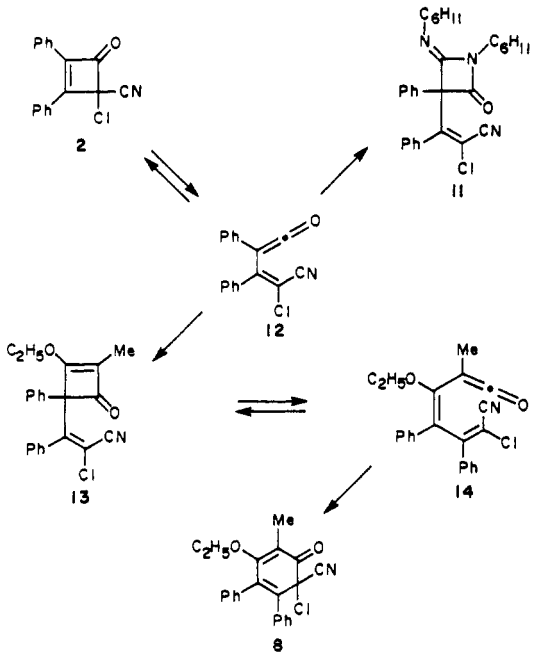
Scheme III



CH_3CO_2H) to 2-cyano-3,5-diethoxy-4,6-dimethylphenol (**6**) (85%).

Related highly functionalized phenols were obtained in a series of experiments in which the cyclobutenones **2**, **3**, and **4** were treated with 1-ethoxypropyne. In each case, an immediate reaction took place, even at ambient temperature, to give respectively the cyclohexadienones **8** (69%), **9** (85%), and **10** (33%) (Scheme II). The formation of these, along with **5**, most likely proceeds by a series of electrocyclic reactions involving a cyclobutenone/vinylketene equilibration. Evidence for this equilibration was clearly obtained by starting with the cyclobutenone **2**. Thus, when a benzene solution containing equimolar amounts of **2** and dicyclohexylcarbodiimide was allowed to stand for 16 h at ambient temperature, the 2-azetidinone **11** was realized in 76% yield. This observation that the vinylketene **12** is present in benzene at ambient temperature resonably suggests that **8** arises via the mechanistic sequence outlined in Scheme III, i.e., **2** → **12** → **13** → **14** → **8**.[6]

Experimental Section

Representative Procedure for the Cycloaddition of Chlorocyanoketene to Alkynes. A solution of 1.87 equiv of the alkyne in 130 mL toluene was heated to 103 °C. To this was added 1.00 g (5.28 mmol) of 4-azido-3-chloro-5-methoxy-2(5H)-furanone in 10 mL of toluene over a 5-min period. The reaction solution was maintained at 103 °C for 1.75 h, cooled to room temperature, and concentrated to give the crude product.

4-Chloro-4-cyano-2,3-diphenylcyclobutenone (2). CCK cycloaddition to diphenylacetylene gave 2.33 g of a light yellow crystalline solid which was recrystallized from hexanes/diethyl ether to give 1.14 g (77%) of **2**: light yellow plates, mp 118.0 °C; IR 1811, 1620; ^{1}H NMR δ 7.60 (m); MS (EI), m/e (relative intensity) 279 (M^+, 35), 216 (100), 189 (20); MS, m/e (relative intensity) (CI) 280 (M + 1, 100), 246 (67).

Anal. Calcd for $C_{17}H_{10}ClNO$: C, 73.00; H, 3.60. Found: C, 72.96; H, 3.50.

4-Chloro-4-cyano-2,3-diethylcyclobutenone (3). Cycloaddition of CCK to 3-hexyne gave 1.01 g of an orange oil which was purified by Kugelrohr distillation (oven temperature 35 °C, 0.005 mmHg) to give 0.59 g (61%) of **3** as a light yellow oil: IR 2187, 1800, 1637; ^{1}H NMR δ 2.74 (q of d, J = 7.9 Hz, J' = 0.8 Hz, 2), 2.30 (q of d, J = 7.6 Hz, J' = 0.8 Hz, 2), 1.39 (t, J = 7.6 Hz, 3), 1.18 (t, J = 7.6 Hz, 3); MS (EI), m/e (relative intensity) 183 (M^+, 100), 140 (52), 120 (96), 93 (81); MS (CI), m/e (relative intensity) 184 (M + 1, 100).

Anal. Calcd for $C_9H_{10}ClNO$: C, 58.87; H, 5.49. Found: C, 58.66, H, 5.50.

4-Chloro-4-cyano-2-methyl-3-phenylcyclobutenone (4). Cycloaddition of CCK to 1-phenyl-1-propyne gave 1.44 g of a light yellow solid. This was purified by recrystallization from hexanes/diethyl ether to give 0.97 g (84%) of **4** as light yellow needles, mp 96.3–97.3 °C; IR 1782, 1602; ^{1}H NMR δ 7.73 (m, 5), 2.20 (s, 3); MS (EI), m/e (relative intensity) 217 (M^+, 19), 182 (6), 154 (100), 127 (34), 77 (19); MS (CI), m/e (relative intensity) 218 (M + 1, 100), 182 (24).

Anal. Calcd for $C_{12}H_8ClNO$: C, 66.22; H, 3.70. Found: C, 66.45; H, 3.74.

2-Chloro-2-cyano-4-methyl-3-phenylcyclobutanone (7). CCK cycloaddition to 1.25 equiv of (*Z*)-1-phenylpropene gave 1.45 g of a light yellow solid. This was recrystallized from hexanes in a dry ice/carbon tetrachloride bath to give 1.00 g (86%) of the title compound as a white powder (92% pure as determined by GC): mp 73.5–77.0 °C; IR 1814; ^{1}H NMR δ 7.35 (m, 3), 7.07 (m, 2), 4.36 (m, 2), 1.06 (d, J = 6.5 Hz); ^{13}C NMR 191.62, 131.35, 130.31, 128.56, 128.36, 115.72, 63.83, 56.76, 48.34, 9.25 ppm; MS (EI), m/e (relative intensity) 219 (M^+, 0.4), 191 (5), 183 (8), 163 (100), 156 (10), 128 (42), 118 (39); MS (CI), m/e (relative intensity) 220 (M + 1, 100), 184 (31), 158 (64); exact mass calcd for $C_{12}H_{10}ClNO$ 219.0451, found 219.0459.

The cyclobutanone **7** was also prepared from cyclobutenone **4** as follows. A mixture of 0.20 g (2.3 mmol) of **4**, 0.05 g of 10% Pd–C, and 10 mL of ethyl acetate was vigorously stirred under an atmosphere of hydrogen for 2 days. The mixture was filtered and concentrated to leave 0.28 g of an orange solid. This was purified as above to give 0.20 g (39%) of **7** as a white powder, mp 73.0–77.0 °C. The spectral properties of this product were identical with those obtained from the cyclobutanone described above.

6-Chloro-6-cyano-3,5-diethoxy-2,4-dimethyl-2,4-cyclohexadienone (5). A solution of 1.07 mL (0.89 g, 10.6 mmol) of 1-ethoxy-1-propyne in 130 mL of toluene was heated to 103 °C. This was then treated with 1.00 g (5.28 mmol) of 4-azido-3-chloro-5-methoxy-2(5H)-furanone in 10 mL of toluene. After 2 h at 103 °C the solution was cooled to room temperature and concentrated to give 1.38 g of a bright yellow solid. This was purified by recrystallization from hexanes/diethyl ether to give 0.80 g (56%) of **5** as bright yellow needles, mp 78.5–80.0 °C: UV (ethanol) 362 (2694); IR 2222, 1690, 1654; ^{1}H NMR δ 4.89 (q J = 6.8 Hz, 2), 4.20 (q, J = 7.0 Hz, 2), 1.89 (s, 3), 1.81 (s, 3), 1.54 (t, J = 7.0 Hz, 3); ^{13}C NMR 188.44, 176,26, 164.48, 115.20, 114.57, 85.09, 72.03, 70.09, 62.80, 23.20, 15.38, 14.75, 10.24; MS (EI), m/e (relative intensity) 269 (M^+, 8), 235 (72), 207 (26), 179 (100), 150 (37); MS (CI), m/e (relative intensity) 270 (M + 1, 58), 236 (100).

Anal. Calcd for $C_{13}H_{16}ClNO_3$: C, 57.89; H, 5.98. Found: C, 57.62, H, 6.05.

6-Chloro-6-cyano-4,5-diphenyl-3-ethoxy-2-methyl-2,4-cyclohexadienone (8). A solution of 0.28 g (1.0 mmol) of **2** and 2 mL 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 solution was maintained at 40 °C for 8 h. The solution was concentrated and the resulting 0.36 g of orange crystalline solid was recrystallized from hexanes/diethyl ether to give 0.25 g (69%) of **8** as bright orange plates, mp 144.8–146.0 °C dec; UV (ethanol) 396 (400); IR, 2228, 1692, 1612; ^{1}H NMR, 7.00 (m, 10), 3.47 (q of d, J = 7.0 Hz, J' = 1.6 Hz, 2), 1.98 (s, 3), 1.07 (t, J = 7.0 Hz, 3); ^{13}C NMR 187.91, 168.28, 165.69, 135.44, 133.39, 130.98, 129.47, 128.17, 128.01, 127.70, 118.30, 114.46, 105.26, 71.95, 63.91, 22.87, 15.28 ppm; MS (EI), m/e (relative intensity) 363 (M^+, 7), 329 (100), 301 (66); MS (CI), m/e (relative intensity) 364 (M + 1, 13), 330 (100).

Anal. Calcd for $C_{22}H_{18}ClNO$: C, 72.62;, H, 4.99. Found: C, 72.60; H, 5.15.

6-Chloro-6-cyano-4,5-diethyl-3-ethoxy-2-methyl-2,4-cyclohexadienone (9). A solution of 0.18 g (1.0 mmol) of **3** in 2 mL

(6) This suggested "electrocyclic cascade" is in direct analogy to previously reported reactions of cyclobutenones with alkynes. See, for example: England, D. C.; Krespan, C. G. *J. Org. Chem.* **1970**, *35*, 3308. Danheiser, R. L.; Gee, S. K *Ibid.* **1984**, *49*, 1672.

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.

Acknowledgment. We thank the National Science Foundation (CHE-8025507) and the National Institutes of Health (AI-15651) for financial support of this work.

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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