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Registry No. 1, 1679-51-2; *cis*-2, 91108-45-1; *trans*-2, 91108-46-2; 3, 10437-78-2; *cis*-4, 81370-42-5; *trans*-4, 81370-41-4; MCPBA, 937-14-4; tBHP, 75-91-2.

New Developments in Heterocyclic Silyl Enol Ether Chemistry: Synthesis and Lewis Acid Mediated Reactions with Carbon Electrophiles of 2,5-Bis(trimethylsiloxy)thiophene and 1-Methyl-2,5-bis(trimethylsiloxy)pyrrole

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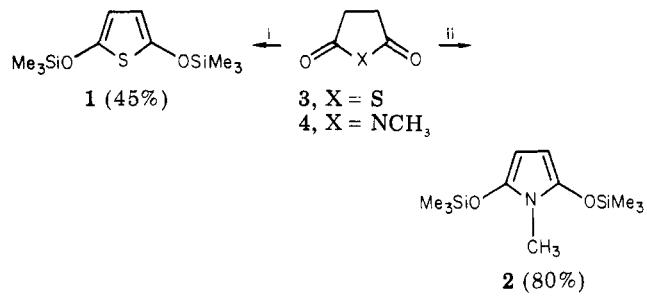
Although heterocyclic mono silyl enol ethers such as 2-(trimethylsiloxy)furan,[1a,b] -thiophene,[2] and -pyrrole[2] show reactivity with electrophiles, in acidic[1b,2] or basic[1a] conditions, predictable on the grounds of the behavior of aliphatic and aromatic silyl enol ethers,[3] the recently reported 2,5-bis(trimethylsiloxy)furan (**6**)[4a] exhibits several unexpected features with electrophiles and $TiCl_4$.[4b]

We now report the synthesis of the new compounds 2,5-bis(trimethylsiloxy)thiophene (**1**) and 1-methyl-2,5-bis(trimethylsiloxy)pyrrole (**2**) and a study of their regioselective functionalization with electrophiles in the presence of a Lewis acid, which shows how electrophilic addition in 2,5-bis(trimthylsiloxy) five-membered heterocyclic compounds changes with the nature of the ring heteroatom, leading to different reaction products of potential interest in synthetic organic chemistry.

The preparation of the title compounds, starting from the corresponding dicarbonyl compounds **3** and **4** is outlined in Scheme I. Product **1** was synthesized in satisfactory yields (45%) from the thioanhydride **3** by using $Et_3N/Me_3SiCl/ZnCl_2$ as silylating agent, whereas under the same reaction conditions the succinimide **4** did not react. **2** was obtained in very good yields by treatment of **4** with 2 equiv of LDA at −78 °C, followed by Me_3SiCl, and was purified by fractional distillation.[5]

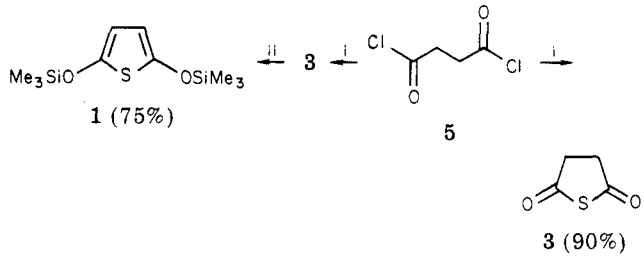
Serious limits to the synthetic approach for compound **1** lay in the low yields and the difficulties in the previously reported preparation of **3**.[6] We have developed a new method for the synthesis of this thioanhydride; starting from succinyl chloride **5** and $(Me_2Si)_2S$ in the presence of $ZnCl_2$, **3** was obtained in high yields (90%) almost pure. A further advantage of this procedure is that, by simply adding Et_3N to the reaction mixture, the bis-siloxy derivative **1** was obtained directly, in a one-pot reaction besides minor amounts of **3** (Scheme II).

Scheme I[a]



[a] (i) $ZnCl_2$, Et_3N, Me_3SiCl; (ii) LDA, Me_3SiCl.

Scheme II[a]



[a] (i) $(Me_3Si)_2S$, $ZnCl_2$; (ii) Et_3N.

Compounds **1** and **2** are yellow oils that could be kept indefinitely at room temperature under inert atmosphere; **2** solidifies at 0 °C.

Compounds **1** and **2**, as well as the oxygen analogue 2,5-bis(trimethylsiloxy)furan (**6**),[4b] present two reactive sites at C(3) and C(4) and undergo regioselective functionalization with carbon electrophiles (Scheme III).

When 2 equiv of $SnCl_4$ were added to a mixture of **6** and of acetone (**7a**), butyraldehyde (**7b**), or benzaldehyde (**7c**) as electrophiles in CH_2Cl_2 at −78 °C, after quenching with 10% NH_4Cl at room temperature, dilactones **8a–c** (entry 1 in Scheme III), analogues of naturally occurring lignans, were obtained in good yields, probably through intramolecular rearrangement of the products of electrophilic addition. This reaction had been previously described[4b] and occurred with the same features even though we used $SnCl_4$ instead of $TiCl_4$ as Lewis acid.

Under the same reaction conditions, 2,5-bis(trimethylsiloxy)thiophene (**1**) afforded a mixture containing comparable yields of the lignans **8a–c** and of the 3,4-disubstituted thioanhydrides **9a–c** (entry 2 in Scheme III), probably formed from the same intermediate,[4b] easily separated by column chromatography on silica gel.

1-Methyl-2,5-bis(trimethylsiloxy)pyrrole (**2**) on the other hand, gave only products **10a–c** coming from an electrophilic attack at C(3) and C(4) (entry 3 in Scheme III); the lower reactivity of the imidic carbonyl center in this case prevents intramolecular attack leading to the dilactone system.

2 undergoes also regioselective monofunctionalization with 1 equiv of the electrophile and of $SnCl_4$; compound **11** was thus obtained in high yields by employing **7a** as an electrophile. A differential functionalization at C(3) and C(4) was also performed in a one-pot reaction by simply adding to **2** a mixture of 1 equiv of acetone (**7a**) and of $SnCl_4$ in CH_2Cl_2 at −78 °C, followed by addition of 1 equiv

(1) (a) Fiorenza, M.; Ricci, A.; Romanelli, M. N.; Taddei, M.; Dembech, P.; Seconi, G. *Heterocycles* **1982**, *19*, 2327. (b) Asaoka, M.; Yanagida, N.; Ishibashi, K.; Takei, M. *Tetrahedron Lett.* **1981**, *22*, 4269.

(2) Fiorenza, M.; Reginato, G.; Ricci, A.; Taddei, M.; Dembech, P. *J. Org. Chem.* **1984**, *49*, 551.

(3) For a complete review on the argument, see: Brownbridge, P. *Synthesis* **1983**, 1.

(4) (a) Brownbridge, P.; Chan, T. H. *Tetrahedron Lett.* **1980**, *21*, 3423. (b) Brownbridge, P.; Chan, T. H. *Ibid.* **1980**, *21*, 3427.

(5) Attempts to use other silylating reagents such as Me_3SiNEt_2, DBU/Me_2SiI, *n*-BuLi/Me_3SiCl, or $NaN(SiMe_3)_2/Me_3SiCl$ did not give the same good results.

(6) To our knowledge a simple preparation of **3** is reported only in "Beilstein Handbuch der Organischen Chemie", Vol. 17, p 421, but we obtained only unsatisfactory yields by following it. On the other hand the more recent syntheses outline a tedious series of lithiations of halogenated thiophenes [Jakobsen, H. J.; Larsen, E. H.; Lawesson, S. O. *Tetrahedron* **1963**, *19*, 1867] or photochemical pathways [Saito, K.; Sato, T. *Bull. Chem. Soc. Jpn.* **1979**, *52*, 3601].

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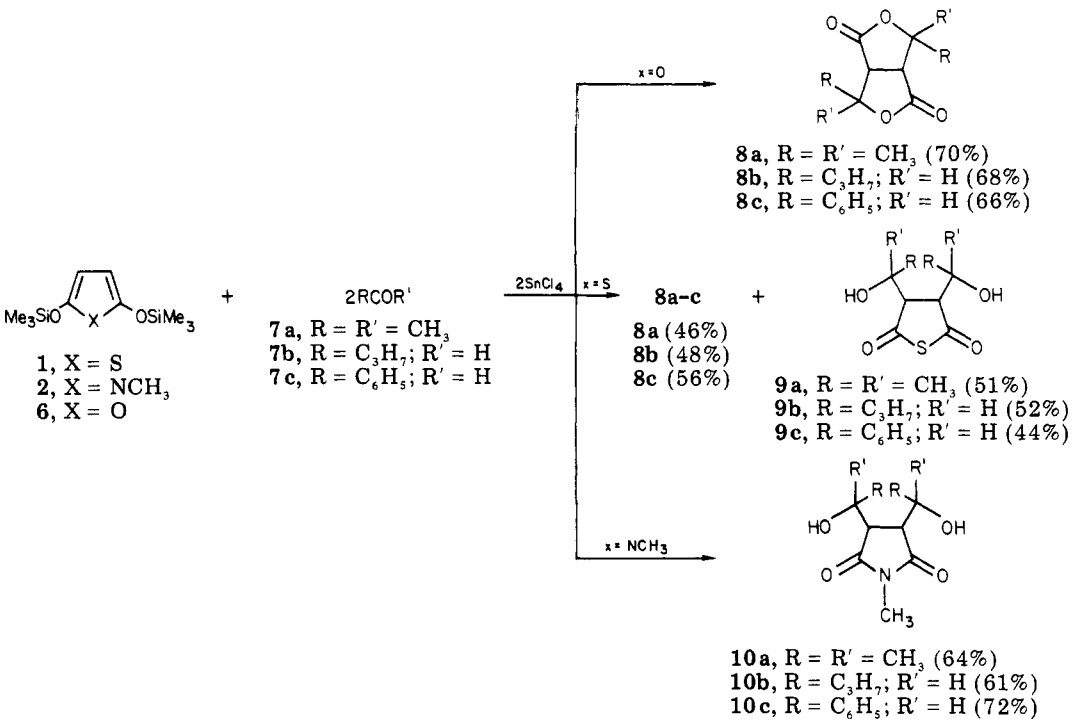
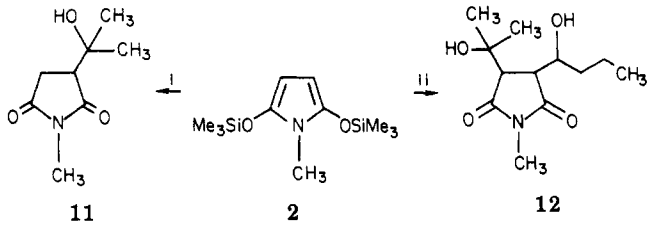
Scheme III



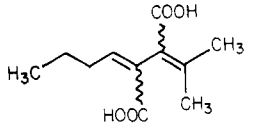
Scheme IV[a]



[a] (i) $1CH_3COCH_3/SnCl_4$; (ii) $1CH_3COCH_3/SnCl_4$; $1C_3H_7CHO/SnCl_4$.

of butyraldehyde (**7b**) and $SnCl_4$, leading, after the usual workup, to **12**[7] (Scheme IV).

The synthetic potential of the reactions so far described is not limited to the selective mono- or bisfunctionalization at five-membered heterocyclic rings: 10% NaOH hydrolysis of **12** leads in fact to the unsaturated dicarboxylic acid **13**, thus suggesting wider synthetic applications of these heterocyclic silyl enol ethers.



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Experimental Section

Boiling points are uncorrected and ^1H NMR spectra were obtained on a Perkin-Elmer R 32,90 MHz spectrometer; chemical shifts are reported in δ units downfield from internal Me_4Si. IR spectra were recorded as a liquid film with NaCl cells or in KBr pellets on a Perkin-Elmer 283 spectrophotometer. Mass spectra were determined on a Varian Matt 111 instrument, and GC/MS with a HP 5970A equipped with a OV 101 5% 12-m capillary column. Preparative TLC were carried out with E. Merk silica gel F plates and visualized by ultraviolet lights. Column chromatography was carried out with a 25-cm column filled with silica gel. Microanalyses were performed with a Perkin-Elmer 240-C analyzer. CH_2Cl_2 was used free from ethanol; THF was distilled over $LiAlH_4$. Compounds **6** and **8a-c** were prepared according to the procedure described by Chan[4a] and were presented the same spectroscopical features. $(Me_3Si)_2S$ is commercially available from Fluka A.G. chemicals. All reactions were performed under nitrogen.

Tetrahydrothiophene-2,5-dione (3). A solution of **5** (1.54 g, 10 mmol) and $(Me_3Si)_2S$ (2.14 g, 12 mmol) in dry CH_2Cl_2 (5 mL) was added dropwise at 0 °C and with vigorous stirring to anhydrous $ZnCl_2$ (2.72 g, 20 mmol), and the mixture was stirred overnight at room temperature. Et_2O (25 mL) was then added and the mixture washed 3 times with a 10% $NaHCO_3$ solution and then with water. After evaporation of the solvent **3** was purified by fractional distillation: 1.00 g (90% yield); bp 100 °C (1.75 mmHg); IR (neat) 2970, 1720 cm^{-1}; ^1H NMR ($CDCl_3$) δ 3.00 (s, 4 H); MS, *m/e* (relative intensity) 116 (30), 56 (32), 42 (12).

2,5-Bis(trimethylsiloxy)thiophene (1) from Tetrahydrothiophene-2,5-dione (3). A solution of **3** (1.16 g, 10 mmol) in dry CH_2Cl_2 (10 mL) was added dropwise, at 0 °C with vigorous stirring, to anhydrous $ZnCl_2$ (2.27 g, 20 mmol). A solution of Et_3N (2.82 g, 28 mmol) in CH_2Cl_2 (10 mL) was then added, followed by Me_3SiCl (3.03 g, 28 mmol). The reaction mixture was warmed to room temperature and stirred overnight. It was then poured into pentane (100 mL), the red brown solid was decanted, and, after evaporation of the solvent, fractional distillation gave **1**: 1.17 g (45% yield); bp 62–63 °C (0.75 mmHg); ^1H NMR δ 0.46 (s, 18 H, $SiCH_3$), 5.70 (s, 2 H, CH=); MS, *m/e* (relative intensity) 260 (100), 245 (8), 116 (8), 73 (81). Anal. Calcd for $C_{10}H_{20}O_2SSi_2$: C, 46.12; H, 7.68. Found: C, 46.41; H, 7.70.

2,5-Bis(trimethylsiloxy)thiophene (1) from Succinyl Chloride (5). A solution of **5** (1.54 g, 10 mmol) and $(Me_3Si)_2S$ (2.14 g, 12 mmol) in CH_2Cl_2 (5 mL) was added dropwise, at 0 °C and under vigorous stirring, to anhydrous $ZnCl_2$ (2.27 g, 20 mmol). The mixture was stirred overnight at room temperature; the total conversion to **3** was monitored by GC/MC analyses, and then, after cooling to −78 °C, Et_3N (2.02 g, 20 mmol) was added dropwise. The mixture was warmed to room temperature, and, after 8 h of additional stirring, the same procedure previously described was followed to give pure **1**: 1.95 g (75% yield).

1-Methyl-2,5-bis(trimethylsiloxy)pyrrole (2). To a solution of 2.5 equiv of LDA in 20 mL of THF was added **4** (1.13 g, 10 mmol) dissolved in THF (5 mL) dropwise at −78 °C. The temperature was allowed to rise to 0 °C, and then Me_3SiCl (3.25 g,

(7) Surprisingly all the attempts at monofunctionalization of **1** and **6** under the same reaction conditions failed since besides starting materials only small amounts of **8** and **9** were recovered.

30 mmol) was added slowly with a syringe. After 4 h of stirring at room temperature, the mixture was poured into pentane (100 mL), filtered, the solvent evaporated, and the residue treated again with pentane (50 mL). After the precipitate had settled and the solution was decanted, the solvent was evaporated to give **2** which was purified by fractional distillation: 2.05 g (80% yield); bp 84–85 °C (0.15 mmHg); ^{1}H NMR δ 0.35 (s, 18 H, $SiCH_3$), 3.13 (s, 3 H, NCH_3), 4.70 (s, 2 H, CH=); MS, *m/e* (relative intensity) 257 (30), 242 (8), 184 (9), 145 (100), 113 (10). Anal. Calcd for $C_{11}H_{23}NO_2Si_2$: C, 51.32; H, 8.94. Found: C, 51.78; H, 8.90.

Reaction of 1 and 2 with 2 Equiv of Electrophile and $SnCl_4$. General Procedure. 3,4-Bis(hydroxydimethylmethyl)tetrahydrothiophene-2,5-dione (9a). To a solution of **1** (200 mg, 0.8 mmol) and **7a** (90 mg, 1.6 mmol) in dry CH_2Cl_2 (2 mL) at −78 °C was added $SnCl_4$ (402 mg, 1.6 mmol) dropwise with a syringe. The mixture was allowed to warm to room temperature, then poured into 20 mL of Et_2O, and washed twice with 10 mL of a NH_4Cl saturated solution. The ethereal layer was separated, dried on $MgSO_4$, and analyzed by GC/MS, showing a mixture of 3% of **3**, 46% of **8a**, and 51% of **9a**. The solvent was evaporated and the crude residue eluted on a silica gel column with cyclohexane:acetone (2:1) from which **3** and **8a** were isolated. **8a**: 66 mg (41% yield); mp 105 °C; MS, *m/e* (relative intensity) 183 (9), 152 (60), 139 (27), 113 (23), 82 (100). Further elution with methanol led to **9a** as a dense yellowish oil. **9a**: 79 mg (42% yield); ^{1}H NMR ($CDCl_3$) δ 1.21 (s, 6 H, CH_3), 1.28 (s, 6 H, CH_3), 2.62 (s, 1 H, CH), 2.70 (s, 1 H, CH), 3.6 (broad, 2 H, OH); IR (neat) 3450, 2990, 1710, 1495, 1090 cm^{-1}; MS, *m/e* (relative intensity) 230 (9), 196 (15), 116 (25), 59 (100). Anal. Calcd for $C_{10}H_{16}O_4S$: C, 51.70; H, 6.94. Found: C, 51.35; H, 6.96.

3,4-Bis(hydroxy-*n*-propylmethyl)tetrahydrothiophene-2,5-dione (9b). GC/MS analyses of the reaction mixture showed 48% of **8b** and 52% of **9b**. Column chromatography on silica gel with cyclohexane:acetone (2:1) gave **8b**: 72 mg (40% yield); mp 189–190 °C; MS, *m/e* (relative intensity) 276 (1), 186 (20), 168 (4), 152 (20), 82 (100). Further elution with ethanol led to **9b** as a yellowish oil. **9b**: 98 mg (47% yield); IR (neat) 3300, 2980, 1705, 1105 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.2 (m, 6 H, CH_3), 1.5 (m, 8 H, CH_2), 2.8 (m, 2 H, CHC=O), 3.4 (m, 2 H, CHO), 4.0 (broad, 2 H, OH); MS, *m/e* (relative intensity) 258 (8), 224 (11), 116 (53), 59 (100). Anal. Calcd for $C_{12}H_{20}O_4S$: C, 55.36; H, 7.74. Found: C, 55.01; H, 7.70.

3,4-Bis(hydroxyphenylmethyl)tetrahydrothiophene-2,5-dione (9c). GC/MS analyses of the reaction mixture showed 56% of **8c** and 44% of **9c**. Column chromatography with cyclohexane:acetone (3:1) gave **8c**: 120 mg (51% yield); mp 183–184 °C; MS, *m/e* (relative intensity) 292 (3), 217 (4), 140 (25), 82 (100). Further elution with acetone led to **9c**: 91 mg (35% yield); mp 142–143 °C; IR (KBr) 3510, 3080, 3060, 2930, 1700, 1610, 1090, 745, 700 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 3.30 (d, 2 H, J = 1.8 Hz, CHC=O), 5.03 (d, 2 H, J = 1.8 Hz, CHO), 5.8 (broad, 2 H, OH), 7.1 (m, 10 H, Ar H); MS, *m/e* (relative intensity) 310 (2), 296 (40), 145 (100), 77 (46). Anal. Calcd for $C_{18}H_{16}O_4S$: C, 65.84; H, 4.91. Found: C, 65.41; H, 4.91.

1-Methyl-3,4-bis(hydroxydimethylmethyl)succinimide (10a). Preparative TLC of the crude with ethanol:chloroform (4:1) gave pure **10a** as a brown wax: 122 mg (64% yield); IR (CCl_4) 3480, 2990, 1700, 1480, 1120 cm^{-1}; ^{1}H NMR (CCl_4) δ 1.20 (s, 6 H, CH_3), 1.28 (s, 6 H, CH_3), 2.61 (s, 1 H, CHC=O), 2.68 (s, 1 H, CHC=O), 3.02 (s, 3 H, NCH_3), 4.6 (broad, 2 H, OH); MS, *m/e* (relative intensity) 227 (6), 210 (4), 196 (26), 113 (100). Anal. Calcd for $C_{11}H_{19}NO_4$: C, 57.62; H, 8.35. Found: C, 57.12; H, 8.35.

1-Methyl-3,4-bis(hydroxy-*n*-propylmethyl)succinimide (10b). Column chromatography of the crude with cyclohexane:acetone (2:1) gave **10b** as a yellow oil: 125 mg (61% yield); IR (neat) 3460, 2980, 1700, 1110 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.1 (m, 6 H, CH_3), 1.4–1.8 (broad, 8 H, CH_2), 2.8 (m, 2 H, CHC=O), 3.00 (s, 3 H, NCH_3), 3.5 (m, 2 H, CHO), 3.9 (broad, 2 H, OH); MS, *m/e* (relative intensity) 239 (9), 224 (8), 185 (26), 113 (100). Anal. Calcd for $C_{13}H_{23}NO_4$: C, 60.67; H, 9.01. Found: C, 60.47; H, 9.06.

1-Methyl-3,4-bis(hydroxyphenylmethyl)succinimide (10c). Column chromatography of the crude with cyclohexane:acetone (1:1) gave **10c**: 192 mg (72% yield); mp 62–63 °C; IR (KBr) 3480, 3085, 3040, 2920, 1700, 1605, 1140 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 3.02 (s, 3 H, NCH_3), 3.20 (d, 2 H, J = 1.9 Hz, CHC=O), 4.65 (d, 2 H, J = 1.9 Hz, CHO), 5.2 (broad, 2 H, OH), 7.3 (m, 10 H, Ar H); MS, *m/e* (relative intensity) 325 (1), 307 (5), 252 (10), 113 (100), 77 (55). Anal. Calcd for $C_{19}H_{19}NO_4$: C, 70.13; H, 5.88. Found: C, 70.29; H, 5.80.

1-Methyl-3-(hydroxydimethylmethyl)succinimide (11). **11** was prepared following the same general procedure of **10a–c**, simply adding dropwise, with a syringe, a solution of **7a** (46 mg, 0.8 mmol) and $SnCl_4$ (201 mg, 0.8 mmol) in CH_2Cl_2 (1 mL) to **2** (205 mg, 0.8 mmol) in CH_2Cl_2 (2 mL) at −78 °C. GC/MS analyses of the crude product showed 91% of **11** and 9% of **10a**. **11** was isolated by column chromatography with cyclohexane:acetone (2:1) as a yellow oil. **11**: 112 mg (82% yield); IR (neat) 3480, 2990, 1710, 1490, 1090 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.25 (s, 6 H, CH_3), 2.3 (m, 2 H, CH_2C=O), 2.8 (m, 1 H, CHC=O), 3.00 (s, 3 H, NCH_3), 3.4 (s, 1 H, OH); MS, *m/e* (relative intensity) 156 (21), 153 (17), 113 (100), 59 (54). Anal. Calcd for $C_8H_{13}NO_3$: C, 56.18; H, 7.65. Found: C, 56.05; H, 7.60.

1-Methyl-3-(hydroxy-*n*-propylmethyl)-4-(hydroxydimethylmethyl)succinimide (12). To a solution of **2** (200 mg, 0.8 mmol) in CH_2Cl_2 (2 mL) was added a mixture of **7a** (44 mg, 0.8 mmol) and $SnCl_4$ (201 mg, 0.8 mmol) in CH_2Cl_2 (0.5 mL) dropwise at −78 °C. After 1 h at this temperature, a mixture of **7b** (66 mg, 0.8 mmol) and $SnCl_4$ (201 mg, 0.8 mmol) in CH_2Cl_2 (0.5 mL) was added dropwise. After 8 h at −78 °C the mixture was warmed at room temperature, treated with 15 mL of Et_2O, and washed twice with 10 mL of a saturated solution of NH_4Cl. The ethereal layer was separed and dried on $MgSO_4$; GC/MS analyses of the crude showed the presence of 5% of **11**, 8% of **10b**, and 87% of **12** which was isolated by preparative TLC with CCl_4:ethanol (4:1) as a brown dense oil. **12**: 136 mg (73% yield); IR (CCl_4) 3470, 2980, 1700, 1110, 1085 cm^{-1}; ^{1}H NMR δ 1.0 (m, 3 H, CH_3), 1.24 (s, 3 H, CH_3CO), 1.26 (s, 3 H, CH_3CO), 1.2–1.6 (broad, 4 H, CH_2), 2.8 (m, 2 H, CHCO), 3.00 (s, 3 H, NCH_3), 3.9 (broad, 2 H, OH); MS, *m/e* (relative intensity) 210 (6), 192 (2), 182 (10), 113 (100), 59 (16), 43 (20). Anal. Calcd for $C_{12}H_{21}NO_4$: C, 59.24; H, 5.38. Found: C, 59.01; H, 5.34.

2-Methyl-3,4-dicarboxy-2,4-octadiene (13). **12** (100 mg, 0.4 mmol) was added to a 10% solution of NaOH (5 mL) and the mixture boiled until all the oil was dissolved; 8 mL of AcOH and 5 mL of Ac_2O were added, and the solution was maintained under stirring for 2 h at room temperature. The mixture was extracted twice with 10 mL of Et_2O and then washed with three portions of water (10 mL). The ethereal layer was separated, dried on $MgSO_4$ and, after evaporation of the solvent and high vacuum treatment for 3 h, **13** was obtained as a waxy material solidifying on standing. **13**: 63 mg (75% yield); IR (KBr) 3460, 2980, 1725, 1650, 1470 cm^{-1}; ^{1}H NMR (CD_3COCD_3) δ 0.9 (m, 3 H, CH_3), 1.3–1.7 (broad, 10 H, CH_2 and CH_3C=), 6.85 (t, 1 H, J = 6 Hz, CH=), 8.3 (broad, 2 H, OH); MS, *m/e* (relative intensity) 212 (3), 179 (9), 142 (100). Anal. Calcd for $C_{11}H_{10}O_4$: C, 62.26; H, 7.54. Found: C, 62.46; H, 7.50.

Registry No. **1**, 91210-72-9; **2**, 91210-73-0; **3**, 3194-60-3; **4**, 1121-07-9; **5**, 543-20-4; **7a**, 67-64-1; **7b**, 123-72-8; **7c**, 100-52-7; **8a**, 91210-74-1; **8b**, 91210-75-2; **8c**, 91278-68-1; **9a**, 91210-76-3; **9b**, 91210-77-4; **9c**, 91210-78-5; **10a**, 91210-79-6; **10b**, 91210-80-9; **10c**, 91210-81-0; **11**, 91210-82-1; **12**, 91210-83-2; **13**, 91228-88-5; $(Me_3Si)_2S$, 3385-94-2; Me_3SiCl, 75-77-4; $SnCl_4$, 7646-78-8.

A Convenient Method for Hydrazone Hydrolysis

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Protection of the carbon–oxygen double bonds in aldehydes and ketones as carbon–nitrogen double bonds has become a valuable strategy in organic synthesis.[1] The carbon–nitrogen double-bonded derivatives, compared with

(1) (a) "Protective Groups in Organic Chemistry", McOmie, J. F. W., Ed.; Plenum Press: London and New York, 1973. (b) Whitesell, J. K.; Whitesell, M. A. *Synthesis* **1983**, 517 and references therein.

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