intensity) 236 (M^+, 46), 220 (14), 205 (14).

2,4-Dihydroxy-6-methyl-3-(3-methyl-2-butenyl)benzaldehyde (12): method B; colorless needles, mp 148 °C (from ether–hexane); UV max (MeOH) 238, 250, 338; IR (CH_2Cl_2) 3550, 3300, 3960, 3920, 1620 cm^{-1}; NMR (CD_2Cl_2, 250 MHz) δ 1.75 (d, 3, J = 1.5 Hz), 1.82 (s, 3), 2.45 (s, 3), 3.35 (d, 2, J = 7.5 Hz), 5.27 (dt, 1, J = 1.5 Hz), 6.20 (s, 1), 6.33 (s, 1, OH), 10.04 (s, 1), 12.66 (s, 1, OH); mass spectrum, m/e (relative intensity) 220 (M^+, 56), 205 (28), 177 (27), 166 (10), 165 (100), 163 (18), 136 (11); high-resolution mass spectrum calcd for $C_{13}H_{16}O_3$ 220.1099, found 220.1098.

3-[[4-(Benzyloxy)-2-methoxyphenyl]methylene]-5-hydroxy-6-(3-methyl-2-butenyl)-2(3*H*)-benzofuranone (16): method A; yellow needles mp 113 °C (from ether–hexane); UV max (MeOH); IR (CH_2Cl_2) 3550, 3025, 2925, 1760 (sh), 1600 cm^{-1}; NMR ($CDCl_3$, 250 MHz) δ 1.76 (s, 3), 1.78 (s, 3), 3.35 (d, 1, J = 6.8 Hz), 3.84 (s, 2.4), 3.88 (s, 0.6), 4.99 (s, 0.8), 5.05 (s, 0.2), 5.13 (s, 0.2), 5.25 (br, t, 1 J = 6.8 Hz), 6.57 (br, s, 0.2), 6.60 (br, s, 0.8), 6.64 (d, 1, J = 8.0 Hz), 6.87 (s, 0.2), 6.90 (s, 0.8), 7.00 (s, 0.2), 7.16 (s, 0.8), 7.70 (d, 0.8, J = 8.0 Hz), 7.82 (d, 0.2, J = 8.0 Hz), 7.94 (s, 1); mass spectrum, m/e (relative intensity) 442 (M^+, 45), 387 (5), 351 (12), 91 (100); high-resolution mass spectrum calcd for $C_{28}H_{26}O_5$ 442.1778, found 442.1785.

5-Chloro-2,4-dihydroxy-6-methyl-3-(3-methyl-2-butenyl)benzaldehyde (18): method B: colorless needles, mp 150 °C (from ether–hexane), UV max (MeOH) 263, 350; IR (CH_2Cl_2) 3675, 3500, 3100, 2925, 1618; NMR ($CDCl_3$, 250 MHz) δ 1.60 (s, 3), 1.80 (s, 3), 2.61 (s, 3), 3.42 (d, 2, J = 7.0 Hz), 5.24 (br, t, 1, J = 7.0 Hz), 6.45 (s, 1), 10.19 (s, 1), 12.71 (s, 1); mass spectrum, m/e (relative intensity) 254 (M^+, 13), 239 (22), 213 (5), 210 (16), 201 (32), 199 (100); high-resolution mass spectrum calcd for $C_{13}H_{15}ClO_3$ 254.0709, found 254.0709.

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Chemistry of the Adducts of *N,N'*-Diphenylformamidine with Oxalyl Chloride and Phosgene

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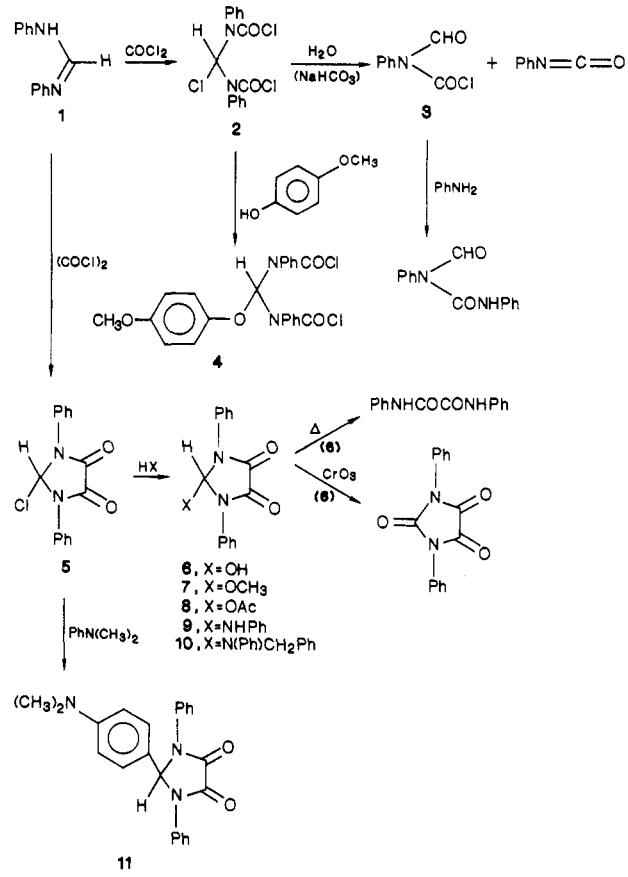
Reactions of N-substituted carboxylic acid amides with acylating agents such as acyl halides, phosphoryl chloride, thionyl chloride, and phosgene have been investigated in detail; the adduct formation between N,N-disubstituted formamides and $POCl_3$ has become the basis of the widely used Vilsmeyer–Haack synthesis of aldehydes.[1] Only few reactions involving substrates containing a C=N bond and acylating agents have been reported.[2] Heterocumulenes such as isocyanates, isothiocyanates, and carbodiimides have been shown to form a variety of adducts by reacting with phosgene or oxalyl chloride at the CN double bond of the N=C=X system.[3,4] We have now extended our investigations to include systems having a heteroatom conjugated to a CN double bond, and we chose *N,N'*-diphenylformamidine (1) as a candidate. The amidine 1 was reacted with both phosgene and oxalyl chloride in the hope of arriving at products or adducts useful in the synthesis of heterocyclic systems.

Scheme I



On adding 1 to a solution of excess phosgene in chlorobenzene at 0 °C and heating the resulting suspension to 70–75 °C with excess phosgene, a colorless product was formed in high yield that was identified as bis[(chlorocarbonyl)anilino]chloromethane (2). The ^{13}C NMR spectrum of 2 shows a signal at 80.6 ppm that was assigned to the central carbon atom. The bis(carbamoyl chloride) 2 was readily hydrolyzed under mild basic conditions to yield *N*-phenyl-*N*-formylcarbamoyl chloride (3) together with phenyl isocyanate.[5] This mode of hydrolysis, where presumably the chlorine on the central carbon of 2 is replaced by hydroxide followed by cleavage to 3, HCl, and phenyl isocyanate, shows that the chlorine on the central carbon is actually more labile than the carbamoyl chloride groups of 2. Compound 3 is thermally decomposed to

(1) For a review see: Jutz, C. In *Iminium Salts in Organic Chemistry*; Böhme, H., Viehe, H. G., Eds.; Wiley-Interscience: New York, 1976; p 225.

(2) Böhme, H.; Hartke, K. *Chem. Ber.* **1963**, *96*, 600. Böhme, H.; Ebel, S.; Hartke, K. *Ibid.* **1965**, *98*, 1463. Bose, A. K.; Spiegelman, G.; Manhas, M. S. *Tetrahedron Lett.* **1971**, 3167. See also: Speckamp, W. N.; Hiemstra, H. *Tetrahedron* **1985**, *41*, 4367.

(3) Ulrich, H.; Sayigh, A. A. R. *J. Org. Chem.* **1965**, *30*, 2781. Stachel, H. D. *Angew. Chem.* **1959**, *71*, 246.

(4) Richter, R.; Stuber, F. A.; Tucker, B. *J. Org. Chem.* **1984**, *49*, 3675. Richter, R.; Barsa, E. A. *J. Org. Chem.* **1986**, *51*, 417.

(5) Compound 3 is hydrolyzed further under these conditions, thus reducing the isolated yield of 3. The major products from the hydrolysis of 3 appear to be formanilide and unexpectedly *N*-formyl-*N,N'*-diphenylurea with a small amount of *N,N'*-diphenylurea.

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phenyl isocyanate. The structure of **3** was further verified by its conversion into the known *N*-formyl-*N*,*N*'-diphenylurea on treatment with aniline. The reactivity of chlorine on the central carbon of **2** is also shown by the reaction of **2** with *p*-methoxyphenol at room temperature in dichloromethane to produce bis[(chlorocarbonyl)anilino](*p*-methoxyphenoxy)methane (**4**). The potential of **2** as a building block for N-heterocycles will be further explored.

When **1** was added to a slight excess of an equimolar quantity of oxalyl chloride, 2-chloro-1,3-diphenylimidazolidine-4,5-dione (**5**) was obtained in virtually quantitative yield. The product was extremely moisture sensitive, which prevented the isolation of a pure analytical sample. The IR spectrum of **5** shows a strong carbonyl absorption at 1740 cm^{-1}, and the ^{13}C NMR spectrum exhibits a signal at 82.0 ppm for C-2, comparable to that for the central carbon of **2**. The reactivity of the chlorine in position 2 of compound **5** is manifested by a variety of exchange reactions (see Scheme I) with several nucleophiles. The 2-hydroxy derivative **6**, obtained in high yield on mild hydrolysis, was further oxidized to the known 1,3-diphenylimidazolidine-2,4,5-trione; the thermolysis of **6** produced oxanilide.

Experimental Section

Infrared spectra were recorded on a Beckman Acculab 8 spectrophotometer; ^1H NMR spectra were determined on a Varian EM360A and ^{13}C NMR spectra on a Varian CFT20 spectrophotometer with Me_4Si as internal standard; elemental analyses were performed by Galbraith Laboratories, Knoxville, TN; melting points are uncorrected.

Bis[*N*-(chlorocarbonyl)anilino]chloromethane (2). To a solution of 60 g (0.6 mol) of phosgene in 150 mL of chlorobenzene was added dropwise and with stirring a solution of 15.0 g (0.0765 mol) of *N*,*N*'-diphenylformamidine (**1**) in 140 mL of chlorobenzene while the temperature was maintained around 0 °C. A colorless precipitate was immediately formed consisting of the hydrochloride of **1**; the reaction temperature was increased to 40 °C at which point phosgene was passed through the suspension as the temperature was slowly increased to 75 °C. The phosgene treatment was continued until the solution was clear (approximately 20 min). Concentrating the reaction solution in vacuo after phosgene removal left a colorless solid that was recrystallized from carbon tetrachloride to yield 23.0 g (84%) of **2**: mp 115–125 °C dec; IR ($CHCl_3$) 1720 cm^{-1} (C=O); ^1H NMR ($DCCl_3$) δ 8.2 (s, 1 H), 7.6–6.2 (m, 10 H); ^{13}C NMR ($DCCl_3$) δ 149.6, 136.8, 130.5, 130.2, 129.5, 80.6. Anal. Calcd for $C_{15}H_{11}Cl_3N_2O_2$: C, 50.37; H, 3.10; N, 7.83; Cl, 29.74. Found: C, 50.41; H, 2.98; N, 7.80; Cl, 29.58.

***N*-Phenyl-*N*-formylcarbamoyl Chloride (3).** To a solution of 10.0 g (0.028 mol) of **2** in 80 mL of dichloromethane was added a solution of sodium bicarbonate (5 g in 80 mL of water) at 15 °C. The two-phase system was vigorously stirred until ^1H NMR monitoring showed that all **2** had reacted (approximately 2.5 h). The organic phase was dried ($MgSO_4$) and concentrated in vacuo at 30 °C (0.1 mm) to remove the phenyl isocyanate that formed (identified by IR and VPC). The residue was taken up in 30 mL of diethyl ether and filtered, and the solution was cooled to −70 °C. A colorless precipitate of **3** was collected, washed with hexane, and dried under vacuum at 25 °C: 3.3 g (62%); mp 59–60 °C dec; IR ($CHCl_3$) 1750, 1720 cm^{-1} (C=O); ^1H NMR ($CDCl_3$) δ 9.5 (s, 1 H), 7.6–7.1 (m, 5 H); ^{13}C NMR ($CDCl_3$) δ 161.1, 151.2, 134.7, 130.0, 129.7, 128.7. A GC–MS on a SPB-5 capillary column of **3** supported the identification.

***N*,*N*'-Diphenyl-*N*-formylurea** was obtained on treating a cold (−15 °C) solution of 0.8 g (8.6 mmol) of aniline in 20 mL of dichloromethane with a solution of 0.7 g (3.8 mmol) of **3** in 20 mL of the same solvent. Aniline hydrochloride precipitated from the reaction mixture; the suspension was warmed to 0 °C and washed with dilute hydrochloric acid and water. Concentration of the organic phase gave a colorless solid that was recrystallized from cyclohexane to afford 0.66 g (55%) of product: mp 108–110 °C (lit.[6] mp 116–118 °C); IR ($CHCl_3$) 1705 cm^{-1} (C=O); ^1H NMR (Me_2SO-d_6) δ 9.1 (br s, 1 H), 8.5 (s, 1 H), 7.0–6.2 (m, 10 H); ^{13}C NMR ($CDCl_3$) δ 164.3, 150.7, 137.1, 130.1, 129.8, 129.4, 129.1, 128.8, 124.6, 120.3. Anal. Calcd for $C_{14}H_{12}N_2O_2$: C, 69.98; H, 5.04; N, 11.66. Found: C, 69.76; H, 4.99; N, 11.53.

Bis[*N*-(chlorocarbonyl)anilino](*p*-methoxyphenoxy)methane (4). To 5.7 g (0.016 mol) of **2** was added 2 g (0.016 mol) of *p*-methoxyphenol and 50 mL of dichloromethane. The solution was stirred at room temperature for 16 h. The solvent was removed under vacuum and the residue dissolved in 50 mL of hot carbon tetrachloride. The hot solution was filtered and cooled to −10 °C. A 3.7-g (52%) sample of **4** was collected with more **4** remaining in the mother liquor: mp 131–134 °C; IR (CH_2Cl_2) 1750 cm^{-1} (C=O); ^1H NMR ($CDCl_3$) δ 7.8 (s, 1 H), 7.4–6.5 (m, 14 H), 3.7 (s, 3 H); ^{13}C NMR ($CDCl_3$) δ 156.0, 149.6, 148.5, 136.5, 130.3, 129.6, 129.2, 117.9, 114.9, 92.3, 55.6. Anal. Calcd for $C_{22}H_{18}N_2O_4Cl_2$: C, 59.34; H, 4.07; N, 6.29; Cl, 15.92. Found: C, 59.35; H, 4.00; N, 6.09; Cl, 16.02.

2-Chloro-1,3-diphenylimidazolidine-4,5-dione (5). To a chloroform solution (50 mL) of 5.5 g (0.043 mol) of oxalyl chloride was added dropwise and with stirring a dilute chloroform solution of 7.8 g (0.040 mol) of **1** (100 mL) while the reaction temperature was maintained around 0 °C. The resulting suspension was heated to reflux for 2 h, resulting in complete dissolution of the originally present hydrochloride of **1**. On concentration of the reaction solution in vacuo, a colorless solid of **5** was obtained in virtually quantitative yield: 11.4 g; mp 152–160 °C dec; IR ($CHCl_3$) 1740 cm^{-1} (C=O); ^1H NMR ($DCCl_3$) δ 7.8–7.2 (complex m); ^{13}C NMR ($DCCl_3$) δ 155.0, 133.2, 129.7, 128.3, 122.9, 82.0. Attempted recrystallization of **5** from chloroform/hexane caused partial hydrolysis as indicated by low chlorine values in the elemental analysis and the appearance of **6** in the ^{13}C NMR spectrum.

2-Hydroxy-1,3-diphenylimidazolidine-4,5-dione (6). A sample of **6** was obtained in 94% yield on treating a solution of crude **5** in chloroform (obtained from 7.8 g of **1** in 150 mL of $CHCl_3$) with 50 mL of a saturated aqueous potassium bicarbonate solution. The mixture was stirred at room temperature for 1 h, and the organic phase was concentrated to leave crude **6** (10.1 g). Recrystallization from methanol/water (3:1) gave colorless crystals of **6**: 3.6 g; mp 178–185 °C dec; IR (KBr) 1730 cm^{-1} (C=O); ^1H NMR (Me_2SO-d_6) δ 7.9–7.1 (complex m); ^{13}C NMR (Me_2SO-d_6) δ 155.6, 134.8, 129.0, 126.5, 122.3, 86.9. Anal. Calcd for $C_{15}H_{12}N_2O_3$: C, 67.15; H, 4.51; N, 10.44. Found: C, 66.93; H, 4.37; N, 10.33.

The oxidation of **6** (0.5 g in 10 mL of glacial acetic acid) with chromic acid (1 g of CrO_3 in the minimum amount of water/acetic acid) at 80 °C for 2 h gave on aqueous workup a colorless solid, mp 202–205 °C, identified to be 1,3-diphenylimidazolidine-2,4,5-trione (IR, mixture melting point with authentic material).

The thermolysis of **6** in refluxing *o*-dichlorobenzene for 1 h produced oxanilide in high yield, mp 246–250 °C, identical in IR comparison with authentic material.

2-Methoxy-1,3-diphenylimidazolidine-4,5-dione (7). A sample of crude **4** (obtained from 7.8 g of **1**) was heated to reflux for 1 h in a mixture of 8 g of methanol and 150 mL of chloroform. After the reaction solution was concentrated and the residue redissolved in dichloroethane to remove small amounts of insoluble material, the filtered solution was concentrated and the residue was recrystallized from toluene: 8.4 g (74%) of **7**; mp 150–151 °C; IR ($CHCl_3$) 1735 cm^{-1} (C=O); ^1H NMR ($CDCl_3$) δ 7.8–7.2 (m, 10 H), 6.95 (s, 1 H), 3.0 (s, 3 H); ^{13}C NMR ($CDCl_3$) δ 155.8, 134.4, 129.5, 127.1, 121.0, 90.3, 47.1. Anal. Calcd for $C_{16}H_{14}N_2O_3$: C, 68.07; H, 5.00; N, 9.92. Found: C, 68.32; H, 4.86; N, 9.98.

2-Acetoxy-1,3-diphenylimidazolidine-4,5-dione (8). Crude **5** (obtained from 7.8 g of **1**) was dissolved in 75 mL of acetic acid containing 3.3 g (0.04 mol) of sodium acetate, and the resulting mixture was stirred for 1 h at room temperature. Evaporation of the acetic acid left a solid residue that was taken up in 150 mL of chloroform, and the solution was washed with water and aqueous sodium bicarbonate solution. The solution was concentrated to yield crude **8** which was further purified by recrystallization from toluene: 8.4 g (68%) of **8**; mp 176–179 °C; IR ($CHCl_3$) 1745, 1700 cm^{-1} (weak sh); ^1H NMR ($CDCl_3$) δ 8.0 (s, 1 H), 7.5–7.2 (m, 10 H), 2.85 (s, 3 H); ^{13}C NMR ($CDCl_3$) δ 169.5,

(6) Logemann, W.; Artini, D. *Chem. Ber.* **1958**, *91*, 2574.

155.6, 133.1, 129.7, 127.9, 122.7, 84.5, 20.4. Anal. Calcd for $C_{17}H_{14}N_2O_4$: C, 65.80; H, 4.55; N, 9.03. Found: C, 65.60; H, 4.64; N, 8.98.

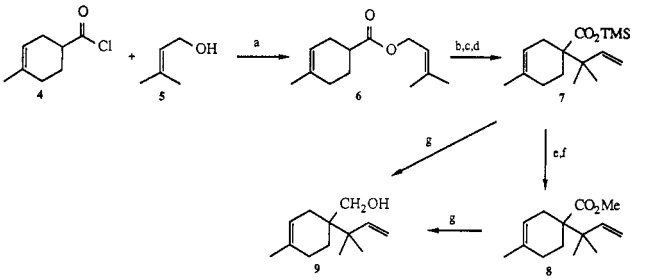
2-Anilino-1,3-diphenylimidazolidine-4,5-dione (9). To a solution of 7.5 g (0.08 mol) of aniline in 50 mL of chloroform was added dropwise a solution of crude **5** (obtained from 7.8 g of **1**) in 100 mL of chloroform at 0 °C. The solid product formed during the reaction was filtered off and thoroughly washed with methanol. Recrystallization from acetonitrile gave 9.3 g (68%) of **9**: colorless crystals; mp 230–233 °C; IR (KBr) 1710 cm^{-1} (C=O); ^1H NMR (Me_2SO-d_6) δ 7.7–6.5 (complex m); ^{13}C NMR (Me_2SO-d_6) δ 156.4, 143.5, 134.4, 129.0, 128.9, 126.9, 123.9, 118.9, 114.2, 78.0. Anal. Calcd for $C_{21}H_{17}N_3O_2$: C, 73.45; H, 4.99; N, 12.24. Found: C, 73.19; H, 5.04; N, 12.16.

2-(*N*-Phenyl-*N*-benzylamino)-1,3-diphenylimidazolidine-4,5-dione (10). A solution of crude **5** (obtained from 7.8 g of **1**) in 100 mL of chloroform was added dropwise to a cold solution of 15 g (0.082 mol) of *N*-phenyl-*N*-benzylamine in 75 mL of chloroform. The resulting reaction solution was stirred for 1 h at room temperature, washed with aqueous sodium hydroxide and water, and concentrated. The solid residue was recrystallized from toluene to give 12.0 g (71%) of **10**: mp 207–209 °C; IR ($CHCl_3$) 1730 cm^{-1} (C=O); ^1H NMR ($CDCl_3$) δ 7.3–6.4 (complex m, 20 H), 4.2 (s, 2 H); ^{13}C NMR ($CDCl_3$) δ 156.4, 145.0, 135.9, 133.9, 129.3, 129.0, 128.1, 127.6, 127.3, 126.8, 124.4, 122.5, 119.7, 85.0, 46.5. Anal. Calcd for $C_{28}H_{23}N_3O_2$: C, 77.58; H, 5.35; N, 9.69. Found: C, 77.61; H, 5.28; N, 9.75.

1,3-Diphenyl-2-[4-(dimethylamino)phenyl]imidazolidine-4,5-dione (11). A solution of crude **5** (obtained from 7.8 g of **1**) in 100 mL of chloroform was added dropwise to a cold solution of 9.7 g (0.08 mol) of *N,N*-dimethylaniline in 100 mL of chloroform. The resulting solution was heated to reflux for 3 h, cooled, diluted with 500 mL of chloroform, and washed with aqueous sodium hydroxide and water. Concentration of the dried ($MgSO_4$) solution left 9.4 g of a colorless residue which was recrystallized from acetonitrile to yield 7.6 g (51%) of **11**: mp 221–223 °C; IR (KBr) 1715 cm^{-1} (C=O); ^1H NMR (Me_2SO-d_6) δ 7.7–7.1 (m, 13 H), 6.5–6.3 (d, 8 Hz, 2 H), 2.7 (s, 6 H); ^{13}C NMR (Me_2SO-d_6) δ 156.0, 150.5, 134.8, 128.8, 126.5, 123.2, 119.4, 111.5, 71.9, 39.4. Anal. Calcd for $C_{23}H_{21}N_3O_2$: C, 74.37; H, 5.70; N, 11.31. Found: C, 74.10; H, 5.78; N, 11.20.

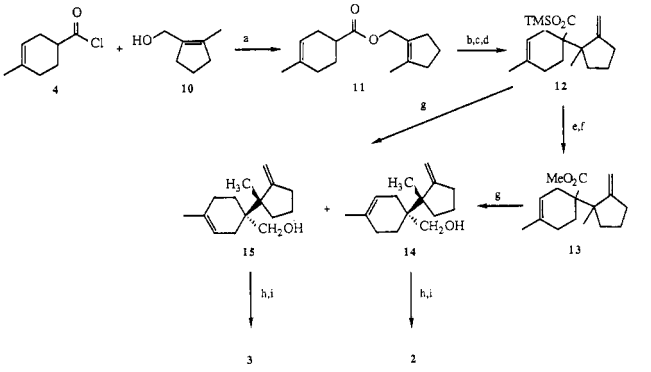
Registry No. **1**, 622-15-1; **2**, 104716-64-5; **3**, 104716-65-6; **4**, 104716-66-7; **5**, 104716-67-8; **6**, 104716-68-9; **7**, 104716-69-0; **8**, 104716-70-3; **9**, 104716-71-4; **10**, 104716-72-5; **11**, 104716-73-6; $COCl_2$, 75-44-5; PhN=C=O, 103-71-9; $PhNH_2$, 62-53-3; PhN(CHO)CONHPh, 92148-97-5; 4-$MeOC_6H_4OH$, 150-76-5; $PhNHCH_2Ph$, 103-32-3; $PhN(CH_3)_2$, 121-69-7; PhNHCOCONHPh, 620-81-5; oxalyl chloride, 79-37-8; 1,3-diphenylimidazolidine-2,4,5-trione, 6488-59-1.

Scheme I. Preparation, Rearrangement, and Reduction of Model Substrate 6[a]



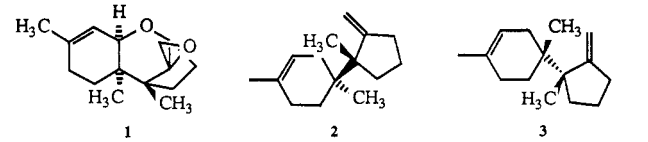
[a] (a) Pyridine; (b) LDA/−78 °C; (c) Me_3SiCl; (d) 25 °C; (e) *n*-Bu_4NF; (f) CH_2N_2; (g) $LiAlH_4$.

Scheme II. Preparation of Trichodiene (2) and Bazzanene (3)[a]



[a] (a) Pyridine/0 °C; (b) LDA/−78 °C; (c) Me_3SiCl; (d) reflux; (e) *n*-Bu_4NF; (f) CH_2N_2; (g) $LiAlH_4$; (h) *n*-BuLi, $(Me_2N)_2P(O)Cl$; (i) $EtNH_2$/Li/*t*-BuOH.

antifungal,[3] and cytotoxic[4] biological activity. Their biosynthetic precursor is the simple diene trichodiene (**2**).[5] Considerable effort has been expended in the synthesis of **2**, which is isolable in only trace quantities from natural sources,[6] because of its pivotal biosynthetic role and the synthetic challenge associated with the presence of the two contiguous quaternary centers in the molecule.



These efforts have borne fruit in that several total syntheses have previously been reported for racemic **2**[7] and its biogenetically divergent diastereomer bazzanene (**3**).[7bf,8]

As part of a program directed toward the total synthesis of trichothecenes, a route to **2** was developed to test the

Creation of Contiguous Quaternary Centers by way of [3,3] Sigmatropic Rearrangements: Synthesis of Trichodiene and Bazzanene

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The trichothecenes (**1**) are a class of almost four dozen fungal metabolites[1] that possess among them insecticidal,[2]

(1) Reviews: (a) Bamberg, J. R.; Strong, F. M. In *Microbial Toxins*; Kadis, S., Cieglek, A., Ajl, S. J., Eds.; Academic: New York, 1971; Vol. 7, Chapter 7. (b) Smalley, E. B.; Strong, F. M. In *Mycotoxins*; Elsevier Scientific: New York, 1974; Chapter 10. (c) Bamberg, J. R. *Adv. Chem. Ser.* **1976**, *No. 149*, 144. (d) Mirocha, C. J.; Pathre, S. V.; Christenson, C. M. In *Mycotoxic Fungi, Mycotoxins, Mycotoxicoses*; Marcel Dekker: New York, 1977; Vol. 1, pp 365–409. (e) *Mycotoxins Hum. Anim. Health, Proc. Conf.* **1976**, 187–337. (f) Pathre, S. V.; Mirocha, C. J. *J. Am. Oil Chem. Soc.* **1979**, *56*, 820.

(2) Grove, J. F.; Hasken, M. *Biochem. Pharmacol.* **1975**, *24*, 959.

(3) (a) Godtfredsen, W. O.; Vangedal, S. *Acta Chem. Scand.* **1965**, *19*, 1088. (b) Bogdanova, V. N.; Solovei, E. F. *Biol. Zashch. Rast.* **1976**, 9.

(4) Kupchan, S. M.; Jarvis, B. B.; Dailey, R. G.; Bright, W.; Bryan, R. F.; Shizuri, Y. *J. Am. Chem. Soc.* **1976**, *98*, 7092.

(5) (a) Nozoe, S.; Machida, Y. *Tetrahedron Lett.* **1970**, 2671. (b) Machida, Y.; Nozoe, Y. *Tetrahedron* **1972**, *28*, 5113. (c) Cane, D. E.; Ha, H.-J.; Pargellis, C. *Bioorg. Chem.* **1985**, *13*, 246.

(6) (a) Machida, Y.; Nozoe, S. *Tetrahedron Lett.* **1972**, 1969. (b) Evans, R.; Hanson, J. *J. Chem. Soc., Perkin Trans. 1* **1976**, 326.

(7) (a) Welch, S. C.; Rao, A. S. C. P.; Gibbs, C. G.; Wong, R. Y. *J. Org. Chem.* **1980**, *45*, 4077. (b) Suda, M. *Tetrahedron Lett.* **1982**, 427. (c) Schlessinger, R. H.; Schultz, J. A. *J. Org. Chem.* **1983**, *48*, 407. (d) Snowden, R. L.; Sonnay, P. *Ibid.* **1984**, *49*, 1464. (e) Harding, K. E.; Clement, K. S. *Ibid.* **1984**, *49*, 3871. (f) Gilbert, J. C.; Wiechman, B. E. *Ibid.* **1986**, *51*, 258. (g) Kraus, G. A.; Thomas, P. J. *Ibid.* **1986**, *51*, 503.

(8) Kogama, M.; Takahashi, T.; Kurihara, T.; Sho, I. *Tetrahedron Lett.* **1980**, 2811.

0022-3263/86/1951-4485$01.50/0