yielded 1-methylbenzimidazole-2-carboxylic acid (7),[3] benzophenone, and traces of 1-methylbenzimidazole. Acid 7 is known to undergo decarboxylation readily even at room temperature.[4]

The above evidence establishes the structure of the hydrolysis product as ketone 4, and on the basis of this evidence, the structure of the adduct from the reaction of 1-methylbenzimidazole (1c) and diphenylketene should be 8 (1750 cm^{-1})[2] and not 2c.

The formation of vinyl ester 8 is related to the recent findings of Kohn, Gopichand, and Charumilind.[5]

Experimental Section[6]

Diphenylketene was prepared by either of two literature methods.[7a,b]

1-Methylbenzimidazole. The following method is a modification of that reported by Fisher[8] and Kimbrough.[1]

In a 1-L round-bottomed flask was dissolved benzimidazole (23.6 g) in methanol (150 mL). Methyl iodide (14.5 mL) was added, and the flask was tightly stoppered and swirled for 10 min. After the mixture had been allowed to stand at room temperature for 1 week, the brown solution was concentrated, and the residue was treated with chloroform (75 mL). The insoluble residue was filtered out. Evaporation of chloroform gave a solid which was dissolved in 10% sodium hydroxide. Addition of water to this solution resulted in two layers. Extraction with dichloromethane and evaporation of the dried dichloromethane gave an oily residue which was chromatographed on neutral alumina. 1-Methylbenzimidazole was eluted out in benzene. The product was hygroscopic and solidified at 0 °C: yield 6.97 g (26%); mp 60–61 °C (lit.[1] 60–61 °C); NMR δ 3.25 (s, 3 H), 6.80 (m, 3 H), 7.35 (m, 2 H); UV λ_{CCl_4} 281 nm (log ϵ 4.3), 274 (4.34), 266 (4.3); IR (neat) 1150, 1130, 1060, 1010, 890, 870, 750 cm^{-1}.

It was found that heating the reaction mixture increased the amount of side products at the expense of 1-methylbenzimidazole.

2-[1′-(Diphenylacetoxy)-2′,2′-diphenylethenyl]-1-methylbenzimidazole (8). The preparation and spectroscopic properties of 8 are found in ref 2 where the adduct was assigned structure 2c: UV λ_{EtOH} 303 nm (log ϵ 4.27), 262 (4.15). Anal. Calcd for $C_{36}H_{28}N_2O_2$: C, 83.0; H, 5.4; N, 5.4. Found: C, 82.7; H, 5.4; N, 5.2.

2-(Diphenylacetyl)-1-methylbenzimidazole (4). Benzimidazole 8 (1.5 g) was added to hot methanol (20 mL). Hydrazine (95%, 10 mL) was added, and the mixture was heated until the starting material dissolved. A saturated solution of sodium chloride was added. After the mixture was allowed to stand at room temperature for 1 h, the resulting solid was collected, washed with water, and dried. Diphenylacetylhydrazine precipitated from the mother liquor and was identified by comparison with an authentic sample[9] (mp 134 °C). Recrystallization of ketone 4 from methanol gave needles: mp 126–127 °C; 0.07 g (75%). Ketone 4 was obtained in 70% yield upon the heating of 8 in 10% methanolic potassium hydroxide for 1 h (1 g of 8 → 0.43 g of 4). Acidification of the basic mother liquor gave diphenylacetic acid (0.3 g). For 4: IR 1680, 1610, 1590, 1475, 1390, 1340, 1120, 1010, 1000, 750, 725, 700 cm^{-1}; NMR δ 3.88 (s, 3 H), 6.69 (s, 1 H), 6.85–7.25 (m, 13 H), 7.55 (m, 1 H); UV λ_{EtOH} 309 nm (log ϵ 3.95), 287 (4.02), 280 (4.03), 258 (4.06), 243 (4.09); mass spectrum, *m/e* 326, 194, 166, 152, 132, 131, 105, 77. Anal. Calcd for $C_{22}H_{18}N_2O$: C, 80.95; H, 5.56; N, 8.58. Found: C, 81.10; H, 5.63; N, 8.48.

2-(2′,2′-Diphenyl-1′-hydroxyethyl)-1-methylbenzimidazole (5) and Acetate 6. Ketone 4 (0.2 g) was dissolved in boiling methanol (10 mL), and sodium borohydride (0.2 g) was gradually added. The reaction mixture was allowed to stand at room temperature for 1 h, and the resulting solid was collected, washed with water, and recrystallized from methanol: yield 0.17 g (85%); mp 226–228 °C; IR 3100–3000 (broad band), 1620, 1600, 1450, 1400, 1287, 1220, 1095, 1080, 922, 750, 705 cm^{-1}; NMR δ 3.05 (s, 3 H), 4.47 (d, 1 H), 5.32 (d, 1 H), 6.6–7.1 (m, 14 H); UV λ_{EtOH} 284 nm (log ϵ 3.99), 277 (4.01), 270 (3.99), 257 (4.00); mass spectrum, *m/e* 328, 311, 222, 167, 165, 152, 134, 133, 132, 118, 92, 77. Anal. Calcd for $C_{22}H_{20}N_2O$: C, 80.30; H, 6.14; N, 8.53. Found: C, 80.30; H, 6.08; N, 8.33.

Acetylation of alcohol 5 (0.1 g) in pyridine–acetic anhydride (6:10) gave acetate 6: 71% yield (0.08 g); mp 165–166 °C; IR 1730, 1600, 1470, 1230, 1020, 920, 750, 710, 700 cm^{-1}; NMR δ 1.75 (s, 3 H), 3.32 (s, 3 H), 4.87 (d, 1 H), 6.31 (d, 1 H), 6.7–7.1 (m, 13 H), 7.4 (m, 1 H). Anal. Calcd for $C_{24}H_{22}N_2O_2$: C, 77.81; H, 5.99; N, 7.56. Found: C, 77.29; H, 5.91; N, 7.56. Acetate 6 (1.0 g) was hydrolyzed back to alcohol 5 (0.06 g) upon treatment with 10% methanolic potassium hydroxide.

Oxidation of Ketone 4 to 1-Methylbenzimidazole-2-carboxylic Acid. Ketone 4 (0.3 g) was dissolved in acetic acid (10 mL). A solution of *m*-chloroperbenzoic acid (0.48 g) in acetic acid (5 mL) was added. After 0.5 h at room temperature, 1-methylbenzimidazole-2-carboxylic acid precipitated out. It was collected and washed with chloroform. The product dissolves easily in water but not in $CHCl_3$: yield 0.35 g; mp 91–93 °C, with the loss of CO_2 (lit.[3,4,10] 90–93 °C dec); IR 3150–2700 (broad band), 1670, 1520, 1480, 1330, 1320, 845, 750 cm^{-1}; NMR (in D_2O) δ 3.45 (s, 3 H), 6.8–7.5 (m, 4 H). Acid 7 lost CO_2 on heating and gave 1-methylbenzimidazole which was identified by comparison with an authentic sample. The same effect was observed when the acid was allowed to stand at room temperature for 3 weeks. These findings confirm the observations of Tertov and Panchenko.[4] Moreover, the copper salt (complex?) of acid 7 was identical with the copper salt of 1-methylbenzimidazole-2-carboxylic acid prepared according to the method of Tertov and Koblik.[3]

The original acetic acid mother liquor was made basic with 10% sodium hydroxide and extracted four times with ether. Evaporation of ether gave benzophenone and 1-methylbenzimidazole which were identified by comparison with authentic samples.

Acknowledgment. The authors thank Professor Costas H. Issidorides for many helpful discussions.

Registry No. 1c, 13436-48-1; 2c, 40110-18-7; 4, 57301-77-6; 5, 57301-81-2; 6, 73286-44-9; 8, 57301-63-0; diphenylketene, 525-06-4; diphenylacetylhydrazine, 6636-02-8; diphenylacetic acid, 117-34-0.

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Phosphindolin-3-one. A Useful Intermediate for Phosphindole Synthesis

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Phosphindole 1-oxide (1a) and phosphindoline 1-oxide (1b) are little-studied ring systems.[1] This is particularly surprising since 1c is isoelectronic with indole. Reported syntheses of this ring system have been plagued by ex-

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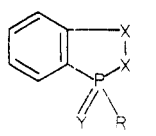
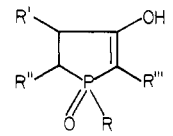


1a, X-X = CH=CH; Y = O
b, X-X = CH_2-CH_2; Y = O
c, X-X = CH=CH; Y = electron pair



2a, R = CH_3; R′ = R″ = R‴ = H
b, R = $C_6H_5CH_2$; R′ = R″ = CH_3; R‴ = C_6H_5

perimental difficulties and poor yields.[1] In addition, these methods are compound specific, in that any variation of substituents at phosphorus, C-2, or C-3 must be incorporated at the initial steps of the synthesis (C-2 is α to phosphorus). This note describes the simple high-yield synthesis of **3**, a compound that contains the desired basic ring system, and preliminary studies of reactions at positions 2 and 3.

Results

The synthesis of **3** is outlined in Scheme I. Mixed diester **4** is prepared in 95% yield via a $NiCl_2$-catalyzed Arbuzov reaction of diethyl methylphosphonite and ethyl 2-iodobenzoate at 170 °C. Adding **4** to 3 equiv of potassium *tert*-butoxide in ether effects cyclization to **3**. The reaction is complete within 10 min at room temperature and the yield is in excess of 90%. Other bases are less efficient for this cyclization. Lithium diisopropylamide gives inferior yields and sodium ethoxide in ether or ethanol gives no reaction at room temperature.

Compound **3** shows a notable preference for the keto form. The infrared spectrum of **3** in chloroform or Nujol shows a strong carbonyl band at 1718 cm^{-1} and no OH band. This is in sharp contrast to **2a** and **2b**. At high concentrations **2a** exists predominately in the enol form.[2] Berlin and Purdum[3] have proven **2b** to be the enol form in the solid state. An X-ray crystal structure of **3** clearly shows it to exist as the keto form in the solid state.[4] The C_2–C_3 (1.52 Å) and C_3–O (1.21 Å) bond distances correlate well with those expected for single and double bonds, respectively.[5] The strong intermolecular hydrogen bonding and possible p–dπ stabilization of the enol double bond advanced[2,3] to explain the structures of **2a** and **2b** are not important determinants of the structure of **3**.

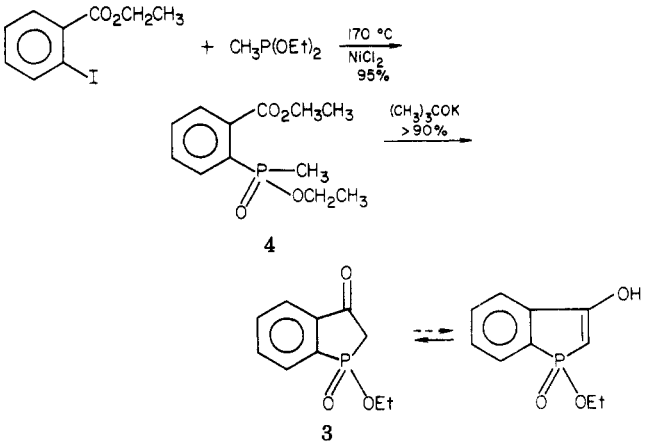
The ^1H NMR spectrum of **3** is in agreement with a ketonic structure. The methylene protons (equivalent at 60 MHz) appear as a doublet at δ 3.02 and integrate to two protons. In any of three solvents ($CDCl_3$, Me_2SO-d_6, and CF_3CO_2H) the integration remains the same. At 270 MHz[6] the methylene protons are resolved into an ABX pattern (Figure 1).

The methylene protons are quite acidic. Addition of 5 mol % of triethylamine causes the sharp methylene doublet to broaden nearly to base line. All P–H coupling is lost. Vigorous mixing of a $CDCl_3$ solution of **3** and D_2O does not exchange the methylene protons after a period of 1 h. On addition of a trace of NaOH, exchange is complete within 10 min.

The reactions of **3** with acid chlorides, isocyanates, and hydrazines have been explored and found to be site specific. In the presence of 1 equiv of triethylamine, acid chlorides react to give the 3-acyloxyphosphindoles **5a–c** in good yield. No C-acylation products are found. These enol esters slowly hydrolyze to **3** in moist air but are stable in an inert atmosphere. The vinyl protons (C-2) of **5a–c** appear as sharp doublets (J_{PH} = 16 Hz) at δ 6.17, 6.35, and 6.20 in the ^1H NMR spectra.

Isocyanates react slowly, if at all, with **3** in a solvent but rapidly on stirring neat with TEA. In contrast to acid

Scheme I



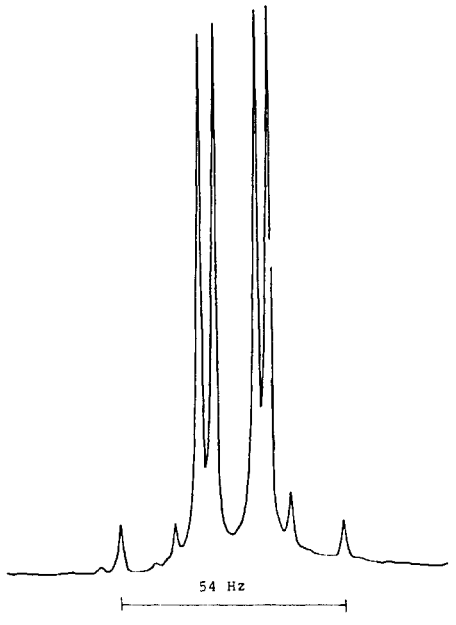


Figure 1. 270-MHz ^1H NMR spectrum of **3**, C-2 methylene protons only (ABX, J_{AB} = 18.8, J_{PA} = 13.5, J_{PB} = 12.8 Hz).

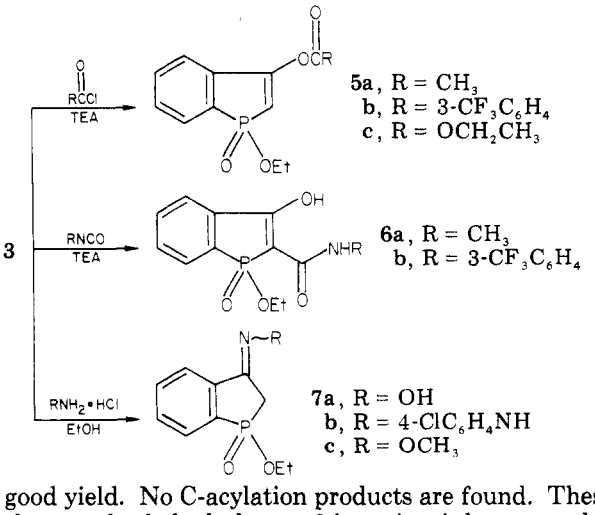


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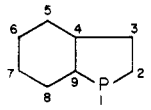
Table I. ^{13}C NMR Data for Phosphindolin-3-one 3 and Derivatives[a]



compd	C-2	C-3	C-4	C-9
3	35.7 (101)	192.5 (19)	141.2 (21)	139.1 (114)
5a	100.9 (132)	157.3 (42)	137.1 (27)	128.5 (131)
6a	88.8 (150)	178.7 (38)	136.1 (21)	130.1 (130)
7a[b]	25.0 (100)	148.4 (17)	139.9 (25)	133.0 (113)

[a] Chemical shifts in $CDCl_3$ downfield from $(CH_3)_4Si$. Values in parentheses are coupling constants to ^{31}P in hertz. [b] Me_2SO-d_6.

chlorides, isocyanates react exclusively on carbon to give **6**. The major features of the ^1H NMR spectrum of **6a** are an enol OH at δ 13.7, NH at δ 6.4, NCH_3 at δ 3.0, and no absorption for a vinyl C-2 proton. Isothiocyanates under varied conditions do not react with **3**.

Hydrazones and oximes **7a–c** are prepared from **3** and the appropriate hydrazine or hydroxylamine. The HCl salts are used since the free amines give inferior yields. Two doublets are found in the ^1H NMR spectra of **7a** (δ 2.95 and 3.04) for the methylene protons, indicating that both syn and anti isomers are formed.

The ^{13}C NMR chemical shifts for the carbon of the five-membered ring of **3**, **5**, **6**, and **7** are given in Table I. These data confirm the structural assignments above. The carbonyl group of **3** appears within the expected region.[7] No unique absorptions of olefinic carbons have been observed, even in a saturated solution.

Experimental Section

General. Melting points were determined on a Mel-Temp melting-point apparatus and are uncorrected. NMR spectra were obtained on Varian T-60 and JEOL FX-100 spectrometers. Chemical shifts are reported on the δ scale, parts per million downfield from a Me_4Si internal standard. Mass spectra were obtained on Varian MAT 311A and MAT CH 7 spectrometers. Elemental analyses were performed by Atlantic Microlabs, Atlanta, Ga.

Materials. Diethyl methylphosphonite[8] and mixed diester 4[9] were prepared by literature methods.

Phosphindolin-3-one (3). To a stirred mixture of potassium *tert*-butoxide (42.05 g, 374.7 mmol) in ether (1500 mL) was added diester **4** (32.0 g, 124.9 mmol). After the solution was stirred for 20 min, water (100 mL) and then 10% HCl (150 mL) were added. The water layer was separated and extracted with CH_2Cl_2 (3 × 150 mL). The organic layers were combined and dried ($MgSO_4$), and the solvent was removed to give a yellow oil which crystallized on standing (ca. 5 min). Recrystallization from ether gave 24.43 g (116.2 mmol, 93.05%) of **3** as a white solid: mp 79–81 °C; ^1H NMR ($CDCl_3$) δ 1.38 (t, J_{HH} = 7 Hz, 3, CH_2CH_3), 3.02 (d, J_{PH} = 13 Hz, 2, PCH_2), 4.30 (p, $J_{HH} = J_{PH}$ = 7 Hz, 2, CH_2CH_3), 7.63–8.18 (m, 4, Ar H); IR ($CHCl_3$) 1718 cm^{-1} (C=O); mass spectrum (high-resolution EI), calcd for $C_{10}H_{11}O_3P$ 210.0446, found 210.0451.

Anal. Calcd for $C_{10}H_{11}O_3P$: C, 57.14; H, 5.29. Found: C, 57.03; H, 5.31.

Acylation of 3. 3-Acetoxyphosphindole (5a). To a stirred CH_2Cl_2 (40 mL) solution of phosphindolin-3-one **3** (3.00 g, 14.27 mmol) was added triethylamine (1.01 mL, 14.27 mmol). Acetyl chloride (1.99 mL, 14.27 mmol) was added in ca. three equal portions over a 3-min period. After being stirred for 10 min, the mixture was poured into 80 mL of water. The CH_2Cl_2 layer was separated and the water extracted with CH_2Cl_2 (3 × 100 mL). The organic layers were combined and extracted with water (1 × 80 mL). The water was back extracted with CH_2Cl_2 (1 × 100 mL). All the organic layers were combined and dried ($MgSO_4$), and the solvent was removed to give a yellow solid. Recrystallization from ether gave 2.99 g (11.86 mmol, 83.13%) of **5a** as a white solid: mp 92–94 °C; ^1H NMR ($CDCl_3$) δ 1.36 (t, J_{HH} = 7 Hz, 3, CH_2CH_3), 2.37 (s, 3, $COCH_3$), 4.17 (p, $J_{HH} = J_{PH}$ = 7 Hz, 2, CH_3CH_2), 6.17 (d, J_{PH} = 16 Hz, 1, vinyl H), 7.33–7.87 (m, 4, Ar H); mass spectrum (high-resolution EI), calcd for $C_{12}H_{13}O_4P$ 252.0551, found 252.0534.

Anal. Calcd for $C_{12}H_{13}O_4P$: C, 57.14; H, 5.21. Found: C, 56.93; H, 5.29.

Phosphindoles **5b** and **5c** were prepared in an analogous manner. Physcial and spectral data are as follows.

5b: 82% as a white solid; mp 110–112 °C; ^1H NMR ($CDCl_3$) δ 1.38 (t, J_{HH} = 7 Hz, 3, CH_2CH_3), 4.21 (p, $J_{HH} = J_{PH}$ = 7 Hz, 2, OCH_2), 6.35 (d, J_{PH} = 14 Hz), 7.33–8.08 (m, 6), 8.38 (m, 2); mass spectrum (high-resolution EI), calcd for $C_{18}H_{14}F_3O_4P$ 382.0582, found 382.0558.

Anal. Calcd for $C_{18}H_{14}F_3O_4P$: C, 56.55; H, 3.70. Found: C, 56.52; H, 3.68.

5c: 85.8% as a white solid; mp 103–105 °C; ^1H NMR ($CDCl_3$) δ 1.35 (t, 3), 1.42 (t, 3), 4.18 (p, 2, $POCH_2$), 4.41 (q, 2, CO_2CH_2), 6.20 (d, 1, J_{PH} = 16 Hz, vinyl H), 7.38–7.90 (m, 4, Ar H); mass spectrum (EI, 90 eV), *m/e* 282 ($M^{+\cdot}$), 181 ($M^{+\cdot}$ - CO_2Et, C_2H_4).

Anal. Calcd for $C_{13}H_{15}O_5P$: C, 55.32; H, 5.37. Found: C, 55.01; H, 5.47.

Reaction of 3 with Isocyanates. *N*-Methyl Amide 6a. To phosphindolin-3-one **3** (2.0 g, 9.52 mmol) and triethylamine (1.33 mL, 9.52 mmol) was added methyl isocyanate. The mixture was stirred under a N_2 atm. After 48 h the solid mass was taken up in CH_2Cl_2 and extracted with 5% HCl. The solution was dried and the solvent removed to give a red semisolid. Crystallization from ether/CH_2Cl_2 (95:5) gave 1.15 g (4.30 mmol, 45.20%) of **6a**: mp 170–80 °C; ^1H NMR ($CDCl_3$) δ 1.28 (t, 3, J_{HH} = 7 Hz, CH_2CH_3), 2.97 (d, 3, J = 4.6 Hz, NCH_3), 4.02 (p, 2, OCH_2), 6.38 (br s, 1, NH), 7.49–7.88 (m, 4, Ar H), 13.70 (br s, 1, enol OH); mass spectrum, *m/e* 267 ($M^{+\cdot}$), 209 ($M^{+\cdot}$ - CH_3NHCO).

Anal. Calcd for $C_{12}H_{14}NO_4P$: C, 53.93; H, 5.29; N, 5.24. Found: C, 53.99; H, 5.33; N, 5.25.

Amide **6b** was prepared in an analogous manner. Physical and spectral data are as follows.

6b: 88.3% as a light yellow solid; mp 148–153 °C; ^1H NMR ($CDCl_3$) δ 1.32 (t, 3, J_{HH} = 7 Hz, CH_2CH_3), 4.13 (p, 2, $J_{HH} = J_{PH}$ = 7 Hz, OCH_2), 7.36–8.07 (m, 8), 8.18 (br s, 1, NH), 12.66 (br s, 1, enol OH); mass spectrum (EI, 100 eV), *m/e* 397 ($M^{+\cdot}$).

Anal. Calcd for $C_{18}H_{15}F_3NO_4P$: C, 54.41; H, 3.81; N, 3.53. Found: C, 54.15; H, 3.83; N, 3.48.

1-Ethoxyphosphindolin-3-one 1-Oxide Oxime (7a). To a solution of phosphindoline **3** (3.62 g, 17.22 mmol) in ethanol (150 mL) was added hydroxylamine hydrochloride (2.39 g, 33.45 mmol). The mixture was stirred for 24 h. Water (100 mL) was added and the ethanol removed on a rotary evaporator. The white solid that formed was collected and dried. Recrystallization from ether gave 3.42 g (15.19 mmol, 88.19%) of oxime **7a** (two isomers): mp 206–208 °C; ^1H NMR (Me_2SO-d_6) δ 1.22 (t, 3, J_{HH} = 6 Hz, CH_3), 2.95 and 3.04 (2 d, 2 total, J_{PH} = 13, J_{PH} = 12 Hz, PCH_2), 4.09 (p, 2, $J_{PH} = J_{HH}$ = 6 Hz, OCH_2), 7.51–8.08 (m, 4, Ar H).

Anal. Calcd for $C_{10}H_{12}NO_3P$: C, 53.33; H, 5.38; N, 6.22. Found: C, 53.27; H, 5.39; N, 6.18.

Hydrazone **7b** and oxime **7c** were prepared in an analogous manner. Physical and spectral data are as follows.

7b: 72.2% as a white solid; mp 178–179 °C; ^1H NMR ($CDCl_3$) δ 1.48 (t, J_{HH} = 8 Hz, 3, CH_2CH_3), 2.87 (d, 2, J_{PH} = 15 Hz, PCH_2), 4.13 (p, 2, $J_{HH} = J_{HP}$ = 8 Hz, OCH_2), 7.15–8.21 (m, 9); mass spectrum (EI, 90 eV), *m/e* 334 ($M^{+\cdot}$) ($M^{+\cdot}$ - C_2H_4).

Anal. Calcd for $C_{16}H_{16}ClN_2O_2P$: C, 57.40; H, 4.83; N, 8.37; Cl, 10.59. Found: C, 57.35; H, 4.85; N, 8.37; Cl, 10.51.

7c: 90.3% as a white solid; mp 106–108 °C; ^1H NMR ($CDCl_3$) δ 1.35 (t, 3, J_{HH} = 7 Hz, CH_2CH_3), 3.00 (2 d, 2, PCH_2, oxime isomers), 4.05 (s, 3, OCH_3), 4.22 (p, 2, $J_{PH} = J_{HH}$ = 7 Hz, OCH_2), 7.40–8.15 (m, 4, Ar H); mass spectrum (EI, 90 eV), *m/e* 239 ($M^{+\cdot}$), 211 ($M^{+\cdot}$ - C_2H_4).

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Anal. Calcd for $C_{11}H_{14}NO_3P$: C, 55.22; H, 5.91; N, 5.86. Found: C, 55.38; H, 6.00; N, 5.83.

Registry No. 3, 73466-84-9; 4, 57020-81-2; 5a, 73466-85-0; 5b, 73466-86-1; 5c, 73466-87-2; 6a, 73466-88-3; 6b, 73466-89-4; (*E*)-7a, 73481-48-8; (*Z*)-7a, 73466-90-7; 7b, 73466-91-8; 7c, 73466-92-9; CH_3COCl, 75-36-5; 3-$CF_3C_6H_4COCl$, 2251-65-2; $EtOC(O)Cl$, 541-41-3; CH_3NCO, 624-83-9; 3-$CF_3C_6H_4NCO$, 329-01-1; $HONH_2$·HCl, 5470-11-1; 4-$ClC_6H_4NHNH_2$·HCl, 1073-70-7; $MeONH_2$·HCl, 593-56-6.

Structures of Satratoxin F and Satratoxin G, Metabolites of *Stachybotrys atra*: Application of Proton and Carbon-13 Nuclear Magnetic Resonance Spectroscopy

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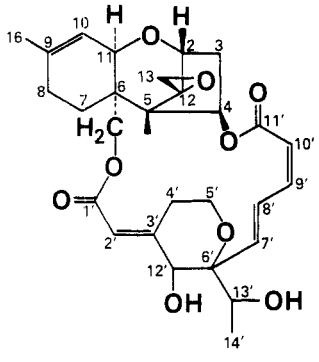
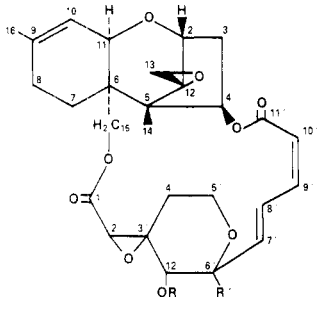
Recent work in our laboratories has demonstrated that two known trichothecenes of the roridin class[1] are produced by the mold *Stachybotrys atra*, viz., roridin E[2] and satratoxin H (1).[3] We now describe spectral studies which



show that the structures of two new *S. atra* metabolites, designated satratoxins F and G, are **3** and **2**, respectively.

MS, IR, and proton and carbon-13 NMR spectral data of **2** were observed to be quite similar to those of **1** (Tables



I and II). In particular, characteristic NMR signals due to (i) methyl protons at 0.83 and 1.74 ppm, (ii) epoxide methylene protons centered at 2.98 ppm, (iii) the methyl carbinol pendant group at 1.12 and 4.45 ppm, and (iv) the cis–trans diene group at 5.90, 5.93, 6.68, and 7.00 ppm clearly indicate that **2** is a trichothecene compound belonging to the roridin group and is closely related to **1**. Its molecular weight of 544 is 16 mass units greater than that of **1**, suggesting incorporation of an oxygen into the satratoxin H system.

Table I. ^1H NMR Data[a] of Satratoxins H (1), G (2), and F (3)

position	1	2	3
2	3.9 (m)	3.90 (d, 5)	3.85 (d, 5)
3A	2.45 (dd, 7.5, 15)	2.5 (m)	2.5 (m)
3B	2.20 (dt, 4,5, 15)	2.0 (m)	2.0 (m)
4	5.9 (m)	6.0 (m)	5.9 (m)
7	1.9 (m)	2.0 (m)	2.0 (m)
8	2.1 (m)	2.0 (m)	2.0 (m)
10	5.46 (d, 5)	5.46 (d, 5)	5.43 (d, 5)
11	3.62 (d, 5)	3.61 (d, 5)	3.59 (d, 5)
13	2.98[b] (AB, 4)	2.98[b] (AB, 4)	2.98[b] (AB, 4)
14	0.83	0.87	0.83
15	4.22[b] (AB, 12)	4.02[b] (AB, 12)	3.88[b] (AB, 12)
16	1.74	1.74	1.73
2′	5.85 (d, 2)	3.43	3.38
4′A	3.74 (dt, 3, 10)	2.5 (m)	2.5 (m)
4′B	2.6 (m)	2.5 (m)	2.5 (m)
5′	3.9 (m)	3.9 (m)	4.15 (m)
7′	6.09 (d, 17.5)	5.90 (d, 16.5)	5.60 (d, 15.5)
8′	7.36 (dd, 10.5, 17.5)	7.00 (dd, 7.5, 16.5)	6.81 (dd, 6, 15.5)
9′	6.63 (t, 10.5)	6.68 (dd, 7.5, 10.5)	6.57 (dd, 6, 10.5)
10′	5.91 (d, 10.5)	5.93 (d, 10.5)	5.92 (d, 10.5)
12′	3.97	4.35	4.24
13′	4.38 (q, 7)	4.45 (q, 7)	
14′	1.16 (d, 7)	1.12 (d, 7)	2.30

[a] In $CDCl_3$ in parts per million from Me_4Si; peak descriptions and J values (Hz) in parentheses. [b] Center of AB system.

Table II. ^{13}C NMR Data[a] of Satratoxins H (1), G (2), and F (3)

position	1	2	3
2	79.1 (d)	79.3 (d)	79.2 (d)
3	34.4 (t)	34.4 (t)	34.6 (t)
4	74.2 (d)	73.7 (d)	74.3 (d)
5	49.0	49.3	49.5
6	43.4	43.3	43.2
7	20.4 (t)	20.2 (t)	20.1 (t)
8	27.6 (t)	27.5 (t)	27.5 (t)
9	140.2	140.3	140.4
10	119.0 (d)	118.8 (d)	118.6 (d)
11	68.2 (d)	68.1 (d)	67.9 (d)
12	65.4	65.4	65.3
13	48.0 (t)	48.1 (t)	48.0 (t)
14	7.6 (q)	8.0 (q)	8.0 (q)
15	64.2 (t)	64.9 (t)	65.1 (t)
16	23.3 (q)	23.3 (q)	23.3 (q)
1′	166.2	166.9	166.1
2′	119.0 (d)	61.0 (d)	58.9 (d)
3′	155.1	65.4	63.9
4′	25.3 (dd)	22.7 (t)	22.7 (t)
5′	60.4 (t)	60.3 (t)	61.2 (t)
6′	81.4	81.5	87.1
7′	132.2 (d)	132.0 (d)	130.2 (d)
8′	134.2 (d)	131.5 (d)	130.5 (d)
9′	143.0 (d)	144.2 (d)	143.2 (d)
10′	120.4 (d)	120.0 (d)	121.2 (d)
11′	167.0	166.9	166.9
12′	73.7 (d)	72.6 (d)	73.7 (d)
13′	69.7 (d)	70.1 (d)	217.0
14′	15.7 (q)	16.1 (q)	29.7 (q)

[a] In $CDCl_3$ in parts per million from Me_4Si.

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