duplication of results was obtained. Mass recoveries of all reactions varied from 85% to 100%. Microautoclaves[19] composed of 316 stainless-steel (12-mL volume) were used. Pyrex glass liners (5-mL volume) were made (0.9 cm × 11.5 cm) to fit into the 12-mL microautoclave reactor tubes. The reaction times and temperatures were varied, but the heat-up time was maintained at 2 min and the cool-down time at 30 s, respectively. At the end of the reactions, gases were vented while the microautoclaves were maintained at dry ice/acetone temperature to ensure minimal loss of volatile liquid products. The workup involved addition of an internal standard and the solvent diethyl ether. The internal standard used most frequently was 1-bromonaphthalene while *n*-decanol was used for the bibenzyl reactions. Between reactions the reactor tubes were cleaned with soapy water, rinsed with distilled water, treated with concentrated nitric acid for 30–60 min, brushed with steel wool, and finally rinsed with acetone.

Product analyses for the microautoclave reactions were done by gas–liquid chromatography with Aerograph Autoprep 700 (Varian) and Varian 2100 instruments. The following column materials were used: 5% OV-17 on 80/100-mesh Chromosorb W (0.25 in. × 6 ft) and 3% OV-17 on Suplecoport A (0.25 in. × 6 ft). The following compounds were identified as indicated. Benzoic acid: GLC retention time; mass spectrum (70 eV), *m/e* (relative intensity) 122 (94), 105 (100), 78 (10), 77 (61), 74 (7), 51 (35), 50 (21), 39 (12), which compares favorably with data for authentic benzoic acid. Toluic acid: GLPC retention time; mp 179–179.5 °C (lit.[20] mp 182 °C); mmp 178–179.5 °C. *p*-Xylene: GLPC retention time; ^{1}H NMR ($CDCl_3$) δ 2.29 (s, 6 H), 6.94 (s, 4 H); IR (neat) 2941, 2857, 1887, 1786, 1639, 1492, 1428, 1389, 1205, 1111, 1020, 775 cm^{-1}; these data compare favorably to the NMR and IR of authentic *p*-xylene.

Gas analyses were done at the University of North Dakota Engineering Experimental Station. Analyses were carried out on a Hewlett-Packard F&M Scientific 700 laboratory chromatograph fitted with a fixed-volume gas sample injection loop.

Molar Ratios. For reaction 1, the molar ratio was 1:9:9:9 diphenyl sulfide/CO/H_2/H_2O. The rest were as follows: reaction 2, diphenyl sulfide/CO_2/H_2/H_2O, 1:9:9:9; reaction 3, *p*-thiocresol/formic acid, 1:18; reaction 4, *p*-thiocresol/formic acid/CO/H_2O, 1:2:3.8:1.5; reaction 5, thiophenol/CO/H_2O, 1:18:18; reaction 6, thiophenol/CO, 1:18; reaction 7, diphenyl disulfide/tetralin, 1:9; reaction 8, diphenyl disulfide/CO/H_2O, 1:18:18.

Gas Analyses. The composition of the products formed on heating formic acid for 5 min at 450 °C in a stainless-steel microautoclave were as follows: CO, 37.5%; H_2, 12.5%; CO_2, 12.5%; the balance being water.

CO, H_2O, CO_2, and H_2 Reactions (Reactions 11 and 12, Table V). The percentages of CO, H_2O, CO_2, and H_2 were the same as those given by the thermal decomposition of formic acid at 5 min and 450 °C. The overall molar ratio of the gas mixture to diphenyl sulfide was 18:1.

Dibenzoyl Peroxide and Diphenyl Sulfide Reactions. The molar ratio of dibenzoyl peroxide to diphenyl sulfide was 1:1.

Formic Acid, Temperature and Time Decomposition. The extent of formic acid decomposition was obtained by titration with a potassium phthalate standardized NaOH solution. Phenolphthalein was used as the indicator.

Acknowledgment. We are grateful for the financial assistance from the United States Department of Energy via Contract No. DE-AB18-78FC02101. We also thank the University of North Dakota Engineering Experimental Station for carrying out the gas analysis. R.V.B. expresses appreciation for research traineeships from the Mining and Mineral Resources Institute (G5106005) and the National Science Foundation (SMI76-10492).

Registry No. Diphenyl sulfide, 139-66-2; formic acid, 64-18-6; thioanisole, 100-68-5; benzothiophene, 95-15-8; dibenzothiophene, 132-65-0; *N,N*-dimethylaniline, 121-69-7; diphenyl ether, 101-84-8; diphenylmethane, 101-81-5; bibenzyl, 103-29-7.

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Structure of Altertoxin I, a Mycotoxin from *Alternaria*

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The structure of altertoxin I, a mycotoxin produced by *Alternaria* molds, was established by ^{1}H, two-dimensional, *J*-resolved and ^{13}C polarization transfer NMR spectroscopy. This compound was found to be an unusual perylene derivative, 1,2,7,8,12b-pentahydro-1,4,6b,10-tetrahydroxyperylene-3,9-dione. Decomposition products of this material were tentatively identified by mass spectrometry as mono- and didehydration products.

The *Alternaria* are a group of molds that cause numerous plant diseases and are responsible for significant amounts of food spoilage. They produce a wide array of secondary metabolites, including many with toxic properties.[3] One metabolite, altertoxin I, $C_{20}H_{16}O_6$ (ATX-I), has been reported as toxic to bacteria, HeLa cells, and mice,[4] as well as being mutagenic by the Ames test.[5] In this paper we present the characterization of altertoxin I, **1**, a novel perylene metabolite.

Perylenes previously have been reported as fungal metabolites. *Daldinia concentrica*, a mushroom parasitic on ash trees,[6] and *Bulgaria inquinans* (Fries), a fungus on oak bark,[7] have been reported to produce 4,9-dihydroxyperylene-3,10-quinone, **2**.

Aspergilline, the black pigment of *Aspergillus niger* spores, also contains **2** as a peptide conjugate.[8]

Elsinochromes A, B, and C, **3**, which also contain the perylene quinone system, are produced by the *Elsinoe*, a fungal genus parasitic on leaves and stems of angiosperms.[9]

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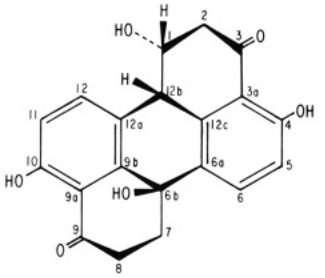

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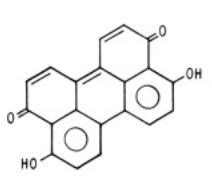

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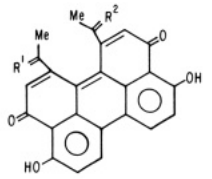

3a $R^1 = R^2 = O$ elsinochrome A

3b $R^1 = O$, $R^2 = H$, OH elsinochrome B

3c $R^1 = R^2 = H$, OH elsinochrome C

Elsinochrome A is identical with phycaron, which is produced by *Phyllosticta caryae*, the imperfect form of *Elsinoe randii*.[10] The *Cercospora*, fungi closely allied to the *Alternaria*, also produced perylenequinones (cercosporin and neocercosporin).[11]

The structure of ATX-I, 1, is unique, since the two aromatic centers in the molecule are not present as the conjugated biphenyl system but are completely isolated from each other by σ bonds. The structure of ATX-I was established on the basis of the spectroscopic analyses presented below.

Mass spectral analysis of altertoxin I revealed a molecular ion of M^+ 352.09561 (calculated for $C_{20}H_{16}O_6$, 352.0946), which decomposed to produce $C_{20}H_{14}O_3$ ($M^+ - H_2O$) and $C_{20}H_{12}O_4$ ($M^+ - 2H_2O$). The IR spectra showed carbonyl absorption at 1646 cm^{-1}, indicative of hydrogen bonding of carbonyls to proximate phenolic OH's. The IR spectrum of ATX-I in CD_3CN solution showed hydroxyl bands at 3627 and 3542 cm^{-1}. The UV spectral curve showed strong bands at 214, 257, 286, 296 (sh), and 355 cm^{-1}, similar in all respects to published UV data of ATX-I,[5] and had the major UV absorption peaks of *o*-hydroxyacetophenone.[13]

The proposed structure for ATX-I was arrived at through extensive ^{13}C and ^1H NMR studies including two-dimensional, *J*-resolved[14] and polarization transfer spectroscopy.[15] The consistency of the structural assignment was confirmed with the aid of CONGEN.[16] The following is a description of the spectral information that supports the assigned structure.

The 270-MHz, two-dimensional, *J*-resolved upfield spectrum of ATX-I is shown in Figure 1. Complete "isolation" and resolution of each individual resonance multiplet was made by selecting slices of the arrayed two-dimensional spectra and rotating them through an angle of 90° (see Figure 1d). This technique allows for the analysis of the normally overlapping resonances, as de-

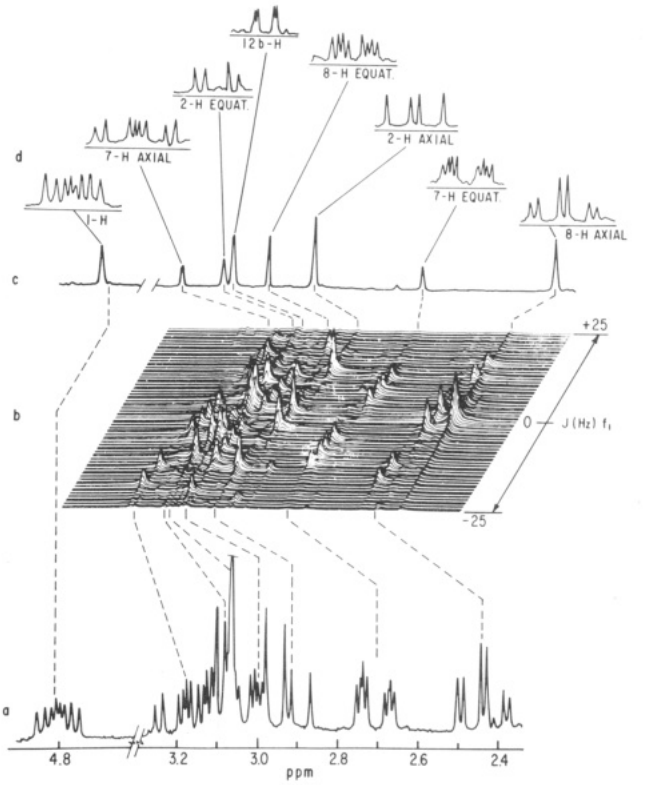

Figure 1. Upfield portion of the ^1H spectrum of I measured in $CDCl_3$ at 270 MHz. (a) Conventional NMR spectrum, (b) two-dimensional *J* spectrum (130 traces are shown; the methine H-1 at δ 4.77 has been omitted from this plot), (c) projected spectrum of b onto the horizontal (f_2) axis, (d) *J* spectra (cross sections of the stacked plot).

Table I. ^1H Chemical Shift Assignments[a] and Coupling Constants of I Measured at 270 MHz in $CDCl_3$

assignment	δ[b]	multiplicity	*J* values, Hz
H-1	4.77	8	$J_{1,2} = 11.8$, $J_{1,2} = 4.6$, $J_{1,12b} = 9.0$
H-2 axial	2.92	4	$J_{2,2} = 16.5$, $J_{1,2} = 11.8$
H-2 equatorial	3.10	4	$J_{2,2} = 16.5$, $J_{1,2} = 4.6$
OH-4[c]	12.34	1	
H-5,11	7.82	2	$J_{5,6} = J_{11,12} = 8.5$
H-6	7.08	2	$J_{5,6} = 8.5$
H-7 axial	3.18	8	$J_{7,7} = 16.5$, $J_{7,8} = 14.5$, $J_{7,8} = 5.5$
H-7 equatorial	2.70	8	$J_{7,7} = 16.5$, $J_{7,8} = 4.0$, $J_{7,8} = 3.9$
H-8 axial	2.43	6	$J_{8,8} = 14.5$, $J_{7,8} = 14.5$, $J_{7,8} = 3.9$
H-8 equatorial	3.02	8	$J_{8,8} = 14.5$, $J_{7,8} = 5.5$, $J_{7,8} = 4.0$
OH-10[c]	12.71	1	
H-12	7.03	4	$J_{11,12} = 8.5$, $J_{12,12b} = 1.0$
H-12b	3.07	4	$J_{1,12b} = 9.0$, $J_{12,12b} = 1.0$
OH-1			
OH-6b	1.90	1	

[a] Coupling relationships were established by homonuclear decoupling experiments at 360 MHz. [b] All shifts measured relative to internal Me_4Si. [c] Identification verified by exchange with D_2O.

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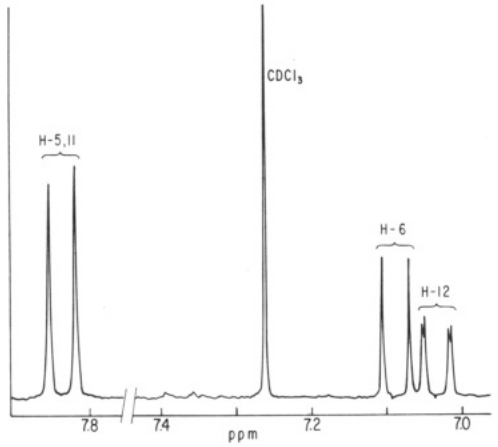

Figure 2. Downfield aromatic portion of the ^1H spectrum of I measured in $CDCl_3$ at 270 MHz; the two low-field phenolic OH singlets at δ 12.34 and 12.71 have been omitted.

picted in Figure 1a, before J resolution. Table I lists the chemical shifts and coupling constants for the resolved proton resonances depicted in Figure 1. The asymmetry of the molecule was immediately apparent from the observed shift pattern of the aromatic proton resonances. Although protons 5 and 11 had the same chemical shift, the shifts of protons 6 and 12 were clearly different due to the asymmetric environments produced by C-1, 12b and C-7, 6b. Furthermore, mutual long-range coupling of protons 12b and 12 (1 Hz) clearly established the sites of the OH groups 6b and 1 (see Figure 2 for details of the aromatic region of the spectrum). A clear indication of the equatorial stereochemistry of OH-1 was given by the 9-Hz (axial–axial) coupling between protons 1 and 12b (dihedral angle ~20°). This relationship requires that C-2 be puckered down in a half-chair conformation, which is consistent with the large, 11.8 Hz and small, 4.6 Hz couplings observed between the H-2 and H-1 protons. We have assigned the lower field resonance at δ 3.10 to the H-2 equatorial proton since it falls within the deshielding zone of the adjacent C-3 carbonyl. Similarly, we assigned the resonance at δ 3.02 to the deshielded equatorial proton at C-8. The stereochemistry of the conformation shown, with carbon-8 puckered up in the half-chair conformation, causes C-9 carbonyl to be moved closer to the hydrogen bonding plane with respect to OH-10 than is C-3 carbonyl with respect to OH-4. This gives rise to the lower field chemical shift of OH-10 (δ 12.71) relative to OH-4 (δ 12.34). Although there is a difference in planarity, the chemical shifts indicate that both phenolic protons are still subject to hydrogen bonding.

This coupling could also be consistent with a structure in which OH-1 would be equatorial and protons 12b and 1 would both be in the axial conformation. This would give rise to a dihedral angle between 12b and 1 of 180°. However, in order to accommodate this structure, carbon-2 would have to be puckered up with respect to C-1 (in order to give rise to two distinctly different coupling constants J_{12}(axial), J_{12}(equatorial)). We do, in fact, observe two distinctly different coupling constants J_{12}(axial), J_{12}(equatorial) of 11.8 and 4.6 Hz, respectively, which are consistent with dihedral angles of ~180° and 50°. The alternate suggestion necessitates an angle approximately 70° between H_1(axial) and H_2(equatorial), which would correspond to a coupling close to 0. This is contrary to our observation. All of the coupling relationships were verified by homonuclear decoupling experiments carried out at 360 MHz.

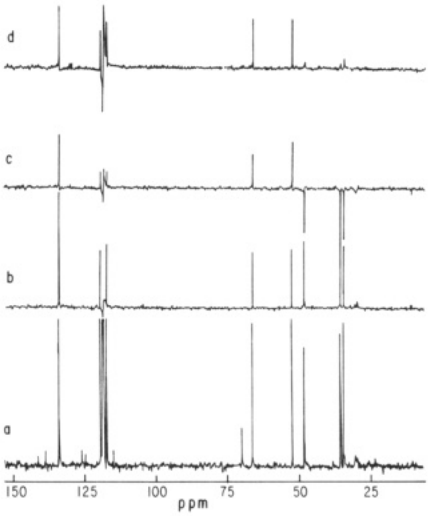

Figure 3. (a) Normal upfield portion of the 67.2-MHz ^{13}C NMR spectrum of I in CD_3CN, (b) polarization transfer spectrum with delay = $1/4J$, (c) delay = $3/4J$, (d) delay = $2/4J$.

Table II. ^{13}C NMR Resonance Positions and Assignments for I Measured at 67.2 MHz in Deuterioacetonitrile

positions	δ [a,b]
C-1	66.2
C-2	48.2
C-3	207.0 (204.5)
C-4	162.0 (162.5)
C-5	117.0 (119.2)
C-6	133.6
C-6b	69.8
C-7	34.5
C-8	35.7
C-9	204.5 (207.0)
C-10	162.5 (162.0)
C-11	119.2 (117.0)
C-12	133.6
C-9a (3a)	114.8
C-3a (9a)	117.8
C-12a (6a)	124.5
C-6a (12a)	125.8
C-12b	52.5
C-12c (9b)	138.4
C-9b (12c)	141.1

[a] Shifts are measured relative to internal Me_4Si. [b] Parenthetical values indicate definite assignment is not possible.

The complete assignment of the carbon spectrum of 1 was achieved by both single-frequency homonuclear proton decoupling as well as polarization transfer spectroscopy.[15] In the latter case, Figure 3, the identity of each proton-decoupled carbon resonance was made by combining the pulse sequence used to induce ^1H–^{13}C polarization transfer[18] with appropriate delay times.[15] Thus, if we select $1/4J$ (1.7 ms; $J \simeq 140$ Hz), for the time delay, the spectrum (Figure 3b) contains all resonances but those of the quaternary carbons. For $3/4J$ (5.3 ms), the CH and CH_3 resonances were observed in the positive direction (Figure 3c), while the CH_2 resonances were inverted. To differentiate between CH and CH_3 resonances, we ran the spectrum (Figure 3d) with a delay of $2/4J$ (3.5 ms), which suppresses all resonances but the CH type. Figure 3a shows the normal ^{13}C FT spectrum. The lower field section containing the quaternary phenolic carbon resonances at

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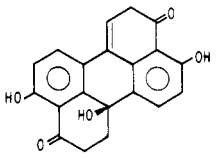
162.0 and 162.5 ppm and the carbonyl resonances of 204.5 and 207.0 ppm have been omitted. Table II lists the positions and ^{13}C resonance assignments for I. The assignments for the quaternary aromatic carbon resonances have been made with analogy to *o*-hydroxyacetophenone.[17]

Harvan and Pero[3] suggested that altertoxin I was closely related to another compound also present, $C_{20}H_{14}O_6$, which they considered the dehydro derivative of altertoxin I. This compound, which was not fluorescent, was named altertoxin II. We observed decomposition of ATX-I in organic solvents but did not find any evidence for the presence of a compound with this property or composition among the decomposition products isolated from our highly purified ATX-I. Detailed study revealed that ATX-I was relatively stable in toluene, with formation of only a trace amount of a yellow fluorescing monodehydration product (4) after 2 months at room temperature. This product was quite mobile under our TLC method (R_f 0.77), appearing considerably above ATX-I (R_f 0.42). This compound was formed slightly more rapidly in EtOH but still in minor quantities. Chloroform provided the greatest amount of decomposition, producing this compound as well as didehydration product **5** that fluoresced yellow under long-wave (366 nm) UV light (R_f 0.67), a blue fluorescing compound (R_f 0.56), and a brown precipitate not mobile in this solvent system but soluble in Me_2SO. Liquid chromatography provided sufficient amounts of **4** and **5** for MS: **4**, *m/e* 334 (ATX-I – H_2O); **5**, *m/e* 316 (ATX-I – $2H_2O$).



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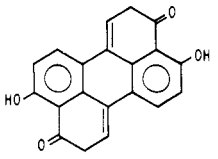

5

Preliminary ^1H NMR spectra of $CDCl_3$/altertoxin I solution in which the dehydration products **4** and **5** were found have disclosed the formation of minor new allylic (δ 3.45) and olefinic (δ 5.97) peaks having areas in the ratio of 2:1. This evidence is consistent with the structures shown and also with a monodehydration product involving 6b, 7.

Experimental Section

Altertoxin I was produced from *Alternaria alternata* RL-8442-2,[12] obtained from the American Type Culture Collection (Rockville, MD) as culture 34957. Plugs cut from 4-day-old cultures on V-8 agar media were used to innoculate twenty 500-mL flasks, each containing 200 g of the sterilized media (130-g long grain rice and 70 mL of H_2O). After 23 days, the contents of each flask was extracted with $CHCl_3$/ethanol (4:1, v/v). The combined extracts were concentrated in vacuo to a viscous brown syrup, 112 g. This was diluted with 500 mL of $CHCl_3$, which caused formation of a precipitate.

The soluble portion was divided into four portions. Each portion was chromatographed on a silicic acid column (silica gel 60, 70–230 mesh, EM Reagents), 7 cm i.d. and 25 cm deep with a CH_2Cl_2/MeOH stepwise gradient elution beginning with 2 L of CH_2Cl_2 followed by 2 L each, in succession, of 1%, 2%, 4%, and 8% MeOH in CH_2Cl_2. A series of fluorescent bands were observed to move down the column. These were collected separately and analyzed by TLC (silica gel GF plates (Analtech), 6:3:1 toluene/ethyl acetate/88% formic acid). The yellow-orange fluorescent band containing ATX-I was eluted in the 2% MeOH fraction. The crude ATX-I fractions were combined and evaporated in vacuo to 2.16 g. This material was further purified by silica gel column chromatography, followed by preparative TLC (500-μm silica gel plates), and finally by HPLC (Waters 6000 A pump; u6 K injector (maximum capacity, 2.5 mL); Model 450 variable-wavelength detector at 340 nm; μ-Porasil column; isocratic elution with toluene/ethyl acetate/formic acid/water (800:200:3 88% HCO_2H:H_2O to 50% saturation)). Particulate matter was eliminated with a disposable silicic acid column ("Sep-Pak", Waters).

The final yield of ATX-I was 52 mg, which represented an overall yield of 0.0013% from the original material: yellow waxy solid; UV (H_2O, pH 3.5) λ_{max} 257 nm (log ϵ 4.12); IR (KBr) ν_{max} 3436, 1643, 1457, 1369, 1336 (sh), 1232 cm^{-1}; high-resolution EI mass spectrum, *m/e* 352.09561 (calcd for $C_{20}H_{16}O_6$, 352.09468); CI mass spectrum, 352 (M^+), 334 ($M^+ - H_2O$), 316 ($M^+ - 2H_2O$), 291 ($M^+ - H_2O - C_2H_3O$), 275 ($M^+ - 2H_2O - C_2HO$).

Isolation of Decomposition Products 4 and 5. Compounds **4** and **5** were isolated by HPLC. The molecular weights of these compounds were determined by CI mass spectrometry, using methane as the reacting gas. CI mass spectrum for **4**: *m/e* 334 (ATX-I – H_2O). CI mass spectrum for **5**: *m/e* 316 (ATX-I – $2H_2O$). ^1H NMR of $CDCl_3$ solution containing both **4** and **5**: δ 3.45 and 5.97. Ratio of areas = 2:1.

NMR Spectra. ^1H NMR spectra were obtained on a JEOL FX-270 NMR spectrometer with a sample of 5 mg/0.5 mL of $CDCl_3$. All shifts were references to internal Me_4Si. Each spectrum was obtained with 16 transients, spectral width 4000 Hz, and 16K data points. A pulse delay of 5 s was used between pulses. In the 2-D experiments 130 scans were obtained for the arrayed plots. The resolution in the f_2 dimension was 2.7 Hz. The f_1 dimension spectrum was obtained with a 128 × 4K matrix from a total 512K data set, which gives 0.4-Hz resolution. Each displayed shift segment was obtained with a 50-Hz spectral width. Homonuclear decoupling experiments were performed on a Brüker WH-360 NMR spectrometer. All the conditions for these experiments were the same as indicated above.

^{13}C NMR spectra were obtained on a JEOL FX-270 NMR spectrometer operating at 67.8 MHz with a sample of 40 mg/0.5 mL of CD_3CN. Each spectrum was obtained with 1000 transients, a spectral width of 15 000 Hz, and 16K data points. To obtain a normal spectrum and optimize the response of the quaternary carbon resonances, we used a 20° pulse width and a 6-s pulse delay. The polarization-transfer spectra were obtained with the pulse sequence described by Doddrell et al.[15]

Spectrophotometric Data. Spectral absorbtion data were obtained with a Beckman Model 25 instrument for the UV absorption and a Nicolet 7199 FTIR instrument with a TGS detector and GE/KBr beam splitter for the IR. The mass spectral data was obtained with a Varian Mat 311 A mass spectrometer with an Incos-Finnegan 3000 data system.

Acknowledgment. We thank Dr. A. Evans, JEOL, Inc., for his help in obtaining the 2-D spectra, Eric Albert for his work on the CONGEN Structure Analysis, and Michael Byler for infrared and Calvin Dooley for mass spectral analysis. All 360-MHz spectra were recorded at the Middle Atlantic Regional NMR facility (supported by NIH Grant RR542, The University of Pennsylvania).

Registry No. 1, 56258-32-3; 4, 82752-82-7; 5, 82752-83-8.