as the carrier and CI reagent gas. The GC flow rate was adjusted to provide a source pressure of 500 μm. The mass spectrometer was scanned from 75 to 375 amu at a rate of 6 s per scan.

Chemicals. GC materials were purchased from Supelco. Pure ethyl alcohol (dehydrated U.S.P.) from U.S. Industrial Chemicals Co. and "Baker Analyzed Reagent" grade pyridine were dried over Type 4A molecular sieves. Anhydrous sulfur dioxide was purchased from Matheson with a minimum purity of 99.98%. Sulfur dioxide, enriched with 70 atom % of oxygen-18, was special ordered from U.S. Services, Inc. Biphenyl internal standard was obtained from Eastman Kodak Co. Solvents were purchased from J. T. Baker Co.

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Registry No. A, 108834-99-7; B, 88149-99-9; C, 70124-99-1; D, 623-81-4; E, 42761-68-2; F_2CHO-p-$C_6H_4CH(CH(CH_3)_2)C(O)Cl$, 70125-00-7; SO_2, 7446-09-5; EtOH, 64-17-5; thionyl chloride, 7719-09-7; pyridine, 110-86-1; N-sulfinopyridinium hydroxide inner salt, 42824-17-9.

Lewis Acid Promoter Reaction of Pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane-8,11-dione with Ethyl Diazoacetate: A Synthetic Entry into the Pentacyclo[6.5.0.0^{4,12}.0^{5,10}.0^{9,13}]tridecane Ring System

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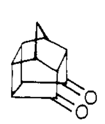
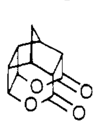
It has recently been reported[1] that pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane-8,11-dione (**1**) undergoes Baeyer–Villiger oxidation to afford a single, symmetrical dilactone, **2**. As part of a program designed to explore the synthesis and chemistry of novel, functionalized pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecanes,[2] we have examined the corresponding reaction of **1** with ethyl diazoacetate in the presence of boron trifluoride etherate as catalyst (Scheme I).



1



2

In our hands, the boron trifluoride etherate catalyzed reaction of **1** with ethyl diazoacetate (2 equiv) afforded a single 2:1 adduct (**3**, 45% yield). The ^{13}C NMR spectrum of **3** displayed only ten signals, a result which suggests that **3** possesses twofold symmetry. Of the three possible 2:1 adducts, only two (**3a** and **3b**, Scheme I) possess the required symmetry. The fact that the material which we isolated possesses structure **3a** rather than **3b** was confirmed unambiguously via single-crystal X-ray structural analysis. Interestingly, analysis of the infrared spectrum and of the ^{1}H and ^{13}C NMR spectra of **3a** suggests that this material exists virtually exclusively in its (hydrogen-bonded) enol form (see Experimental Section). The exclusive migration of the C(1)–C(11) and C(7)–C(8) bonds accompanying Baeyer–Villiger oxidation of **1** (that leads to the formation of a single dilactone, **2**)[1] is mirrored in the boron trifluoride catalyzed reaction of **1** with 2 equiv of ethyl diazoacetate.

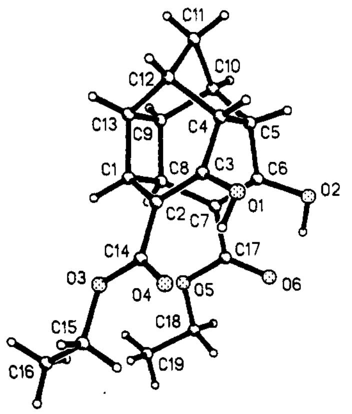
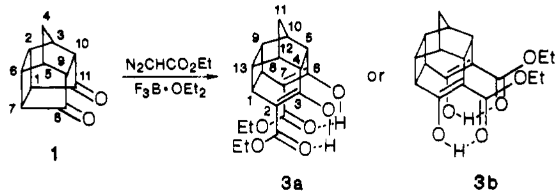


Figure 1. Diagram of **3a** as determined by X-ray diffraction. Only one of the two molecules in the asymmetric unit is shown.

Scheme I



Compound **3a**, diethyl 3,6-dioxopentacyclo[6.5.0.0^{4,12}.0^{5,10}.0^{9,13}]tridecane-2,7-dicarboxylate ($C_{19}H_{22}O_6$, Figure 1) crystallizes in the monoclinic space group $P2_1/c$ with unit cell dimensions a = 11.028 (4) Å, b ; 20.429 (6) Å, c = 16.015 (5) Å, β = 105.28 (3)°, and Z = 8 (two molecules per asymmetric unit). The volume of the cell is 3480.5 (2) Å^3, the molecular formula weight is 346.38, and the calculated crystal density is 1.32 g cm^{-3}.

Bond lengths and angles for the two molecules in the asymmetric unit are in good mutual agreement, and both molecules display the same conformation. All bond lengths fall into normal ranges except for the C(1)–C(8) [1.575 (5), 1.572 (5) Å] and C(4)–C(5) [1.582 (5), 1.586 (5) Å] bonds, which are longer than normal, unstrained sp^3–sp^3 carbon–carbon σ-bonds. Bond angles in **3a** also fall into normal ranges with the exception of the C(10)–C(11)–C(12) angle (94.7 (3)°, 94.8 (3)°] which is much smaller than a normal tetrahedral C–C–C bond angle (109.4°). These deviations result from internal strain in the cage and from the effects of nonbonded interactions between groups in each ketoester moiety. The six-membered rings defined by C(1)–C(2)–C(3)–C(4)–C(12)–C(13) and by C(5)–C(6)–C(7)–C(8)–C(9)–C(10) bend away from one another wherever they are not constrained by cross-cage bonds [i.e., C(1)–C(8), C(4)–C(5), and C(9)–C(13)]. The nonbonded C(3)···C(6) contact is only 2.87 Å, which is much less than

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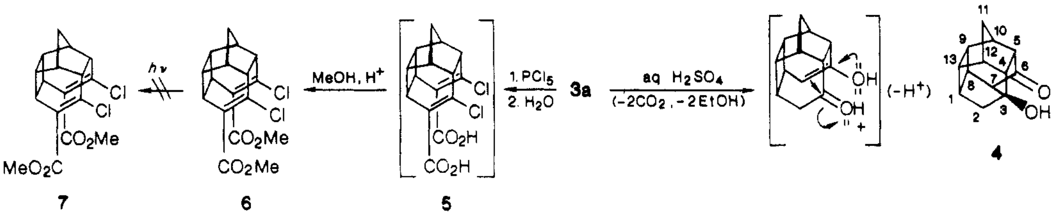
0022-3263/87/1952-3455$01.50/0

Scheme II



the usual nonbonded distance range of 3.2–3.4 Å seen in graphite and in numerous other aromatic structures. There are two intramolecular hydrogen bonds in each molecule, O(1)–H···O(4) and O(2)–H···O(6). In the O(1)–H···O(4) hydrogen bond, the O···O distance is 2.58 Å (2.58 Å) and the O–H···O angle is 151.2° (159.6°). In the case of the O(2)–H···O(6) bond, the O···O distance is 2.56 Å (2.57 Å) and the O–H···O angle is 154.7° (147.6°).

When **3a** was refluxed with aqueous sulfuric acid, a single product was obtained as a colorless, high-melting solid in 70% yield. This same material could be obtained in nearly quantitative yield via reaction of **3a** with sodium chloride in dimethyl sulfoxide.[3] We suggest structure **4** (Scheme II) for this product upon the basis of analysis of its infrared spectrum and of its ^{1}H and ^{13}C NMR spectra. Compound **4** most likely results via decarboxylation of the dicarboxylic acid derived via hydrolysis of diester **3a** (or via NaCl-promoted decarboalkoxylation of diester **3a**) followed by intramolecular aldol condensation (Scheme II).

Compound **3a** could be converted into the corresponding bis(vinyl chloride), **6**, via the reaction sequence shown in Scheme II. Compound **6** is of interest as a potential precursor to a substituted homohexaprismane (**7**). However, our attempts to promote intramolecular [2 + 2] photocyclization of **6** to afford **7** met with repeated failure.[4]

Experimental Section

Melting points are uncorrected.

Diethyl 3,6-Dioxopentacyclo[6.5.0.0^{4,12}.0^{5,10}.0^{9,13}]tridecane-2,7-dicarboxylate (3a). Compound **1** (3.0 g, 17 mmol) was dissolved in anhydrous ether (100 mL) and cooled to –78 °C via application of an external dry ice–acetone bath. Boron trifluoride etherate (9.5 g, 68 mmol) was added slowly with stirring during 10 min. After all of the boron trifluoride etherate had been added, ethyl diazoacetate (3.95 g, 34 mmol) was then added slowly at such rate that a slow, steady stream of nitrogen was evolved during the addition. The reaction mixture was stirred for 1 h after the addition of ethyl diazoacetate had been completed. The temperature of the reaction vessel was increased to –40 °C, and stirring was continued for an additional 1 h. The reaction mixture was then allowed to warm slowly to room temperature. The mixture was then transferred into a separatory funnel that contained water (300 mL). The layers were separated, and the ether layer was washed sequentially with 10% aqueous sodium bicarbonate solution (2 × 100 mL) and with water (2 × 100 mL), dried (anhydrous magnesium sulfate), and filtered. The filtrate was concentrated in vacuo, thereby affording a yellow oil. This material was heated in vacuo (0.1 mmHg) at 45–50 °C for ca. 4 h. The resulting material was primarily an oil that contained suspended solid. The solid was isolated by filtration, and the residue was washed with 25% ether–hexane (30 mL). Recrystallization of the solid residue from 20% ether–hexane afforded **3a** as colorless needles (1.25 g, 21%): mp 108.5–109.0 °C; IR (KBr) 2960 (s), 2943 (m), 2918 (m), 2865 (m), 1630 (s), 1598 (s), 1462 (m), 1404 (s), 1271 (s), 1212 (s), 1075 (m), 1060 cm^{-1} (m); ^{1}H NMR (CDCl$_3$) δ 1.24 (t, J = 6.9 Hz, 6 H), 1.51 (AB, J_{AB} = 10.6 Hz, 1 H), 1.62 (AB, J_{AB} = 10.6 Hz, 1 H), 2.38 (m, 2 H), 2.75 (m, 2 H), 2.94 (br s, 2 H), 3.76 (m, 2 H), 4.13 (q, J = 6.9 Hz, 4 H), 12.01 (br s, 2 H); ^{13}C NMR (CDCl$_3$) δ 14.5 (q), 33.9 (d), 36.2 (d), 37.8 (t), 40.8 (d), 46.9 (d), 60.1 (t), 97.8 (s), 171.1 (s), 175.7 (s); mass spectrum (70 eV), m/z (relative intensity) [no molecular ion] 149.0 (100).

Anal. Calcd for $C_{19}H_{22}O_6$: C, 65.88; H, 6.40. Found: C, 66.02; H, 6.47.

The filtrate was concentrated in vacuo, and the residue, which consisted primarily of unreacted starting material, was recycled by using the procedure described above. The product obtained after workup was purified via column chromatography on silica gel (25% ethyl acetate–hexane eluent). An additional 1.40 g of pure **3a** was thereby obtained; the combined yield of **3a** was 2.65 g (45%).

Single-Crystal X-ray Structural Analysis of 3a. Cell dimensions were obtained from a least-squares fit of the coordinates of 25 centered reflections with 2θ values between 18° and 31° by using Mo Kα radiation (λ = 0.71069 Å). The 4796 independent reflections were collected out to $2\theta_{max}$ = 55° on a Nicolet R3M automatic diffractometer with an incident beam graphite monochromator. The structure was solved by direct methods[5] and was refined by full-matrix least-squares refinement techniques in which the function minimized was $\sum w(|F_o| - |F_c|)^2$ where $w = 1/[\sigma^2|F_o| + g(F_o)^2$ is included to account for random instrumental errors (g is estimated to be 0.000 23). The data were corrected for isotropic sencondary extinction, p = 0.000 31, $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin 2\theta]^{0.25}$. A total of 476 parameters were refined, including atomic coordinates and anisotropic thermal parameters for all non-hydrogen atoms and coordinates only for the four hydroxyl hydrogen atoms. All other hydrogen atoms were allowed to ride on their covalently bonded atoms, and isotropic thermal parameters for all hydrogens were fixed. The final residuals for the 3172 observed reflections ($|F_o| > 3\sigma|F_o|$) used in the refinement were R = 0.065 and R_w = 0.056. In this final Fourier difference map, the ripple density ranged from –0.23 to +0.26 eÅ^{-3}. The programs used for structure solution and refinement were part of the MicroVAX version of the SHELX[6] system.

3-Hydroxyhexacyclo[6.5.0.0^{3,7}.0^{4,12}.0^{5,10}.0^{9,13}]tridecan-6-one (4). Method A. Compound **3a** (2.1 g, 6.1 mmol) was suspended in a mixture of 20% aqueous sulfuric acid solution (30 mL) and ethanol (6.0 mL), and the resulting mixture was refluxed for 48 h. The reaction mixture was cooled to room temperature and diluted with water (100 mL). The resulting mixture was extracted with methylene chloride (3 × 50 mL), and the combined organic layers were washed with water, dried (anhydrous magnesium sulfate), and filtered. The filtrate was concentrated in vacuo, thereby affording an oil, which upon trituration with benzene afforded crude **4** as a cream-colored solid. Chromatography (silica gel stationary phase, 20% ethyl acetate–hexane eluent) afforded pure **4** as a colorless microcrystalline solid (859 mg, 70%): mp 239–240 °C; IR (KBr) 3490 (br, vs), 2974 (s), 2885 (m), 1732 (s), 1318 (m), 1280 cm^{-1} (m); ^{1}H NMR CDCl$_3$) δ 1.42 (AB, J_{AB} = 10.3 Hz, 1 H), 1.52 (AB, J_{AB} = 10.3 Hz, 1 H), 1.74 (s, 2 H), 1.86 (d, J = 11.9 Hz, 1 H), 2.28 (m, 1 H), 2.54 (m, 3 H), 2.72 (m, 2 H), 2.94 (m, 2 H), 3.38 (m, 1 H); ^{13}C NMR (CDCl$_3$) δ 32.0 (d), 34.2 (d), 38.6 (d), 38.8 (t), 42.1 (d), 43.1 (t), 49.8 (d), 50.5 (d), 54.2 (d),

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[3] Stevens, R. V.; Lee, A. W. M. *J. Am. Chem. Soc.* **1979**, *101*, 7032 and references cited therein.

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58.5 (d), 83.6 (s), 224.4 (s); mass spectrum (70 eV), *m/z* (relative intensity) 202 (molecular ion, 12.6), 109 (8.9), 108 (100), 107 (31.8), 95 (22.0), 91 (12.2), 79 (16.5), 77 (19.6).

Anal. Calcd for $C_{13}H_{14}O_2$: C, 77.20; H, 6.98. Found: C, 77.47; H, 7.00.

Method B. Compound **3a** (500 mg, 1.44 mmol) and sodium chloride (300 mg, excess) were suspended in dimethyl sulfoxide (DMSO, 30 mL). The reaction mixture was heated at 150 °C for 1.5 h; slight darkening of the reaction mixture occurred, and a gas was evolved slowly during this period. The reaction mixture was then cooled to room temperature and water (150 mL) was added. The resulting mixture was extracted with methylene chloride (3 × 25 mL), and the combined organic layers were washed with water (3 × 30 mL), dried (anhydrous magnesium sulfate), and filtered. The filtrate was concentrated in vacuo to afford crude 4 as a white solid. Vacuum sublimation of this material [150 °C (0.05 mmHg)] afforded pure 4 (285 mg, 97%).

Dimethyl 3,6-Dichloropentacyclo[6.5.0.0^{4,12}.0^{5,10}.0^{9,13}]-trideca-2,6-diene-2,7-dicarboxylate (6). A suspension of **3a** (1.0 g, 2.9 mmol) in phosphorus trichloride (6 mL) was cooled via application of an external ice bath. To this stirred suspension was added phosphorus pentachloride (3.00 g, 14.3 mmol) in small portions during 30 min. The reaction mixture was stirred for 1 h after all of the PCl_5 had been added. The reaction mixture was then refluxed overnight. The resulting mixture was cooled to room temperature and poured over crushed ice (100 g). The resulting suspension wax extracted with methylene chloride (3 × 50 mL), and the combined organic layers were washed with water (3 × 50 mL), dried (anhydrous magnesium sulfate), and filtered. The filtrate was concentrated in vacuo to afford crude diacid **5** as a light brown microcrystalline solid. This material was dissolved in methanol (10 mL) that contained concentrated sulfuric acid (2 drops), and the resulting solution was refluxed overnight. The reaction mixture was cooled and diluted with water (60 mL), and the resulting mixture was extracted with methylene chloride (3 × 30 mL). The combined organic layers were washed with water (4 × 50 mL), dried (anhydrous magnesium sulfate), and filtered, and the filtrate was concentrated in vacuo. Crude **6** was thereby obtained as a light brown microcrystalline solid. This material was recrystallized from ether to afford pure **6** as colorless prisms (369 mg, 36%): mp 138–139 °C; IR (KBr) 2949 (s), 2890 (s), 2849 (m), 1722 (s), 1698 (s), 1626 (s), 1610 (m), 1440 (s), 1255 cm^{-1} (s); ^1H NMR ($CDCl_3$) δ 1.51 (AB, J_{AB} = 10.5 Hz, 1 H), 1.60 AB, J_{AB} = 10.5 Hz, 1 H), 2.42 (m, 2 H), 2.80 (m, 2 H), 3.15 (m, 2 H), 3.70 (s, 6 H), 3.78 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 35.2 (d), 37.0 (t), 39.7 (d), 40.5 (d), 51.9 (d), 53.2 (q), 126.9 (s), 141.5 (s), 165.8 (s); mass spectrum (70 eV), *m/z* (relative intensity) [no molecular ion] 184.0 (100.0), 124.9 (93.4).

Anal. Calcd for $C_{17}H_{16}Cl_2O_4$: C, 57.48; H, 4.54. Found: C, 57.39; H, 4.37.

Attempted Intramolecular [2 + 2] Photocyclization of 6. A solution of **6** (200 mg) in degassed ethyl acetate (250 mL) was irradiated with a 450-W Hanovia medium-pressure mercury lamp (Pyrex filter) for 24 h. Removal of solvent in vacuo afforded only unchanged starting material (194 mg). No reaction occurred even when considerably longer irradiation times were employed (i.e., 48 and 72 h, respectively).

Acknowledgment. Financial support of this study by the Air Force Office of Scientific Research (Grant AFO SR-84-0085), The Robert A. Welch Foundation (Grant B-963), and the North Texas State University Faculty Research Committee is gratefully acknowledged. The X-ray crystallographic structure determination of **3a** was supported in part by the Office of Naval Research Mechanics Division.

Registry No. 1, 2958-72-7; **3a**, 108744-07-6; 4, 108744-08-7; 5, 108744-09-8; 6, 108744-10-1; ethyl diazoacetate, 623-73-4.

Supplementary Material Available: A list of atomic coordinates and equivalent isotropic displacement parameters, bond lengths, bond angles, anisotropic displacement parameters, and H-atom coordinates and isotropic displacement parameters for **3a** (5 pages). Ordering information is given on any current masthead page.

Intramolecular [2 + 2] Cycloadditions of Ketenes to Carbonyl Groups. A Novel Synthesis of Substituted Benzofurans

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There is considerable current interest in synthetic applications of intramolecular [2 + 2] cycloaddition reactions of ketenes with carbon–carbon double bonds[1] and with ketone and aldehyde carbonyl groups.[2] We recently reported that (*o*-carbonylphenoxy)acetyl chlorides can be readily dehydrochlorinated to afford the corresponding ketenes, which then undergo facile intramolecular [2 + 2] cycloadditions to form the corresponding tricyclic β-lactones. Decarboxylation of these lactones then occurs spontaneously, thereby affording the corresponding benzofuran in 53–82% yield (Scheme I).[2] In order to further delineate the scope and limitations of this novel synthesis of substituted benzofurans, we have explored new methods to generate ketene intermediates for use in intramolecular cycloaddition reactions. We now report the results of our study.

The method utilized for the synthesis of the (*o*-carbonylphenoxy)acetic acids that were employed as substrates in this study is illustrated in Scheme II for the preparation of [(*o*-formylphenoxy)phenyl]acetic acid. Williamson ether syntheses were carried out via reaction of appropriately substituted phenoxide anions with substituted α-bromoacetic acids (see Experimental Section). On occasions when oily (*o*-acylphenoxy)acetic acids were prepared that could not be recrystallized, it proved expedient to purify them via the corresponding dicyclohexylammonium salt.[3] These salts, prepared conveniently by reaction of each of the crude acids with dicyclohexylamine, could be purified simply by washing with ligroin. The salts thereby obtained proved to be suitable for direct utilization in the one-pot ketene cycloaddition procedure (method B) without the necessity of prior conversion to the free acid. Three different methods were then utilized to generate the corresponding ketenes from these (*o*-carbonylphenoxy)acetic acids, as follows.

Method A. Base-promoted elimination of hydrogen chloride from acid chlorides of the type $R_2CHC(O)Cl$ is one of the classical methods by which ketenes have been generated traditionally.[4] Thus, the (*o*-carbonylphenoxy)acetic acids were converted into the corresponding acid chlorides via reaction with 5–8 equiv of oxalyl chloride in benzene at ambient temperature for 4–8 h. Excess oxalyl chloride was removed in vacuo, and the crude acid chloride was then added very slowly to a refluxing solution of triethylamine (3 equiv) in benzene. The fact that dehydro-

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