[8-(Benzyloxy)octyl]ethylamine (14). The amine 5 (1.8 g, 5 mmol) was hydrogenated under the conditions described in the preparation of 13 to yield after workup 1.35 g (100%) of a colorless, viscous liquid which showed a tendency to crystallize during storage: IR (neat) 3280 (N-H), 1454 (CH_2N), 1107 (C-O); ^1H NMR 1.12 (t, 3), 1.2-1.8 (m, 13), 2.5-2.8 (m, 4), 3.46 (t, 2), 4.48 (s, 2), 7.30 (s, 5). Anal. Calcd for $C_{17}H_{29}NO$: C, 77.51; H, 11.09. Found: C, 77.36; H, 11.28.

Benzyl *n*-Nonyl Ether (15). Sodium hydride (50% dispersion in mineral oil, 1.8 g, 75 mmol) was suspended in THF (10 mL) under nitrogen and a solution of 1-nonanol (7.2 g, 50 mmol) in THF (15 mL) was added dropwise. After stirring at 60 °C for 1 h, benzyl bromide (8.55 g, 50 mmol) was added, and the mixture was refluxed overnight. The solvent was removed in vacuo, water (20 mL) was added, and the mixture was acidified with 6 N HCl. Extraction with chloroform (3 × 10 mL), drying over $MgSO_4$ followed by filtration, and evaporation of the solvent in vacuo afforded the crude product which was vacuum distilled (bp 127-129 °C (0.2 mm)) to give 8.8 g (75%) of benzyl *n*-nonyl ether as a colorless liquid: IR (neat) 1103 (C-O); ^1H NMR 0.65-1.95 (m, 17 H), 3.43 (t, 2 H), 4.46 (s, 2 H), 7.27 (s, 5 H). Anal. Calcd for $C_{16}H_{26}O$: C, 81.99; H, 11.18. Found: C, 82.24; H, 11.16.

Benzyl Phenyl Ether (16). Phenol (2.35 g, 25 mmol), benzyl bromide (12.8 g, 75 mmol), and tri-*n*-butylhexadecylphosphonium bromide (1.3 g, 2.5 mmol) were dissolved in CH_2Cl_2 (125 mL) and a solution of NaOH (1.5 g, 37.5 mmol) in 125 mL of water was added. The mixture was stirred vigorously for 3 days at room temperature. The organic layer was separated and dried ($MgSO_4$). Following removal of the solvent in vacuo, the residue was dissolved in ether and passed through a short silica gel column to remove the catalyst. The solvent was evaporated in vacuo and the residue was vacuum distilled to give 4.15 g (90%) of a colorless viscous oil (bp 88 °C (0.25 mm)) which crystallized during storage, mp 39.5-40 °C (lit.[10-12] mp 38-39 °C).

11-(Benzyloxy)-1-undecene (17) was prepared by modifying a reported procedure.[13] Sodium hydride (50% dispersion in mineral oil, 9.6 g, 0.20 mol) was washed with *n*-pentane and suspended in THF (70 mL). A solution of 10-undecen-1-ol (25.0 g, 0.15 mol) in THF (40 mL) was added dropwise under nitrogen. After stirring at 60 °C for 1 h, a solution of benzyl bromide (34.2 g, 0.20 mol) in THF (40 mL) was added and the mixture was refluxed overnight. The solvent was then evaporated in vacuo and CH_2Cl_2 (50 mL) was added. After filtration and washing the filtered material with CH_2Cl_2, the solvent was evaporated in vacuo and the residue was vacuum distilled to give 34.7 g (91%) of the product, bp 119-121 °C (0.3 mm) (153-155 °C (1 mm)).[13]

Benzyl *n*-Undecyl Ether (18). Benzyl *n*-undecenyl ether (17) (0.52 g, 2.0 mmol) was dissolved in 95% ethanol (5 mL) and Pd/C (10%, 50 mg) and 3 drops of *n*-butylamine were added. The mixture was shaken under 45 psi of hydrogen at room temperature overnight. After filtration of the catalyst and evaporation of the solvent and the amine in vacuo, 18 (0.50 g, 95%) was obtained as a colorless liquid: IR (neat) 1103 (C-O); ^1H NMR 0.7-2.0 (m, 19), 3.47 (t, 2), 4.48 (s, 2), 7.28 (s, 5). Anal. Calcd for $C_{18}H_{30}O$: C, 82.38; H, 11.52. Found: C, 82.64; H, 11.65.

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Registry No. 4, 91842-55-6; 5, 91842-58-9; 6, 2050-25-1; 7, 91842-53-4; 8, 91842-54-5; 9, 51326-52-4; 10, 91842-56-7; 11, 31600-54-1; 12, 91842-57-8; 13, 91842-59-0; 14, 91842-60-3; 15, 91842-61-4; 16, 946-80-5; 17, 81518-75-4; 18, 91842-62-5; DHP, 110-87-2; $ClCH_2CO_2H$, 79-11-8; $PhCH_2NHEt$, 14321-27-8; $HO(CH_2)_8OH$, 629-41-4; $PhCh_2Br$, 100-39-0; Pd, 7440-05-3; 1-nonanol, 143-08-8; phenol, 108-95-2; tri-*n*-butylhexadecylphosphonium bromide, 14937-45-2; 10-undecen-1-ol, 112-43-6; *n*-butylamine, 109-73-9.

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Synthesis of 5,5,9,9-Tetranitropentacyclo[5.3.0.0^{2,6}.0^{3,10}.0^{4,8}]decane

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There is considerable current interest in the synthesis and chemistry of strained energetic compounds; polynitropolycyclic "cage" systems are potential members of this important class.[1-4] However, relatively few nitro-containing cage compounds have been synthesized.[1-4] We now report the synthesis of the title compound (1) in ten stereocontrolled steps (see Scheme I). To our knowledge, compound 1 is only the second polynitrobishomocubane to have been synthesized.[4]

The key intermediate in our synthesis of 1 is bishomocubanedione 7. This compound has been synthesized by Paquette and co-workers[5] via a multistep procedure, one step of which involves transoximation of *endo*-dicyclopentadienone dioxime.[6] However, we experienced some difficulty when we attempted to carry out the transoximation procedure. In our hands, only partial transoximation of *endo*-dicyclopentadienone dioxime occurred; accordingly, a gross mixture of several products was obtained. The desired *endo*-dicylopentadienone could be isolated via tedious column chromatographic separation from the product mixture in only 35% yield.

Our alternative approach to 7 is shown in Scheme I. An attempted shortcut to cage dioxime 8 by photocyclization of *endo*-dicyclopentadienone dioxime[6a] failed. Starting material could not be recovered, and no useful products resulted from photolyses attempted under a variety of conditions. Once cage dioxime 8 was in hand, we relied upon published procedures to effect conversion of the oxime functionalities first to nitro groups[7,8] and then to geminal dinitro groups.[9] The required tetranitrobishomocubane 1 proved to be accessible in good overall yield from readily available, inexpensive starting materials (i.e., cyclopentanone and ethylene glycol) by using the route indicated in Scheme I.

Experimental Section

Melting points and boiling points are uncorrected. Proton NMR spectra (60 MHz) were recorded on a Hitachi-Perkin-Elmer Model R-24B NMR spectrometer. ^{13}C NMR spectra were recorded on a JEOL FX-90Q NMR spectrometer. In all cases, signals are reported in parts per million (δ) downfield from internal tetramethylsilane. Infrared spectra were obtained with a Perkin-Elmer Model 1330 infrared spectrophotometer. Mass spectra were obtained with a Hewlett-Packard Model 5970A GC/MS system operating at 70 eV. Elemental microanalyses were per-

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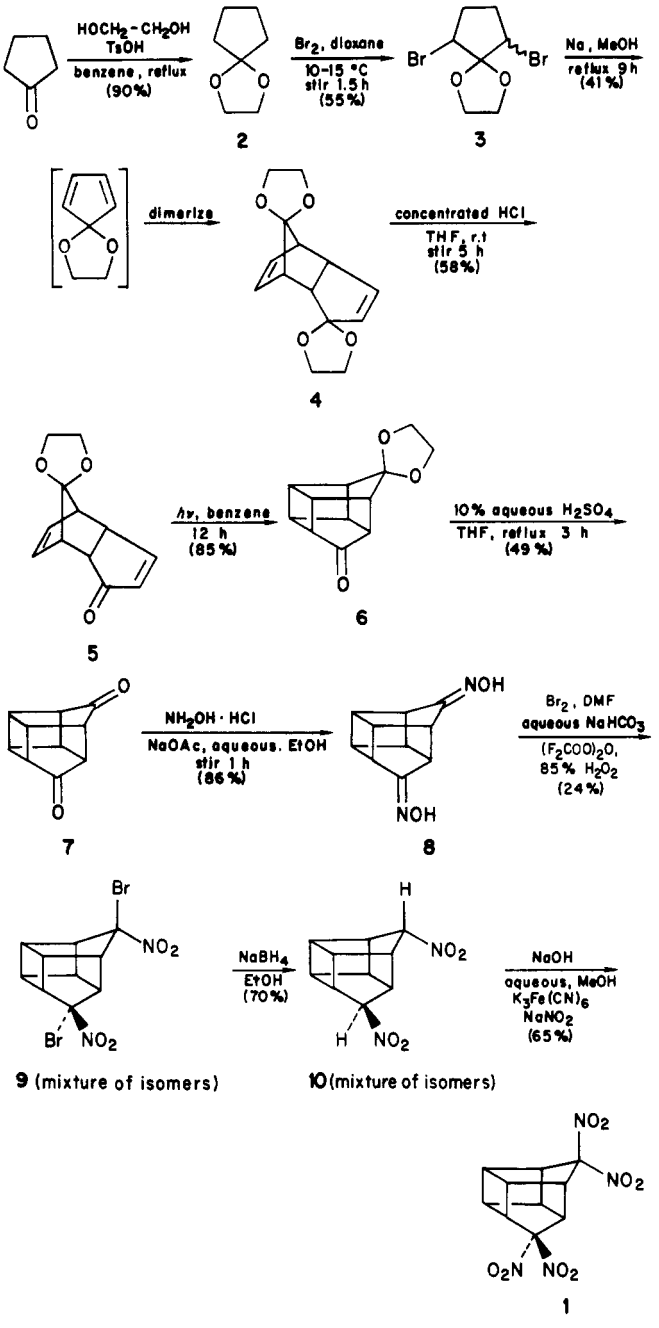
0022-3263/84/1949-4078$01.50/0

Scheme I



formed by Galbraith Laboratories, Inc., Knoxville, TN.

Pentacyclo[5.3.0.0^{2,6}.0^{3,10}.0^{4,8}]decane-5,9-dione (7). Compound 7 was synthesized by the following reaction sequence: 1,4-dioxaspiro[4.4]nonane, 2, bp 152–155 °C (lit. bp 153 °C)[10] was synthesized in 90% yield via reaction of cyclopentanone with ethylene glycol in the presence of *p*-toluenesulfonic acid (benzene solvent).[10] Bromination of 2 with bromine (2 equiv) in dry dioxane at 10–15 °C[11] afforded the corresponding dibromide 3 (55%), mp 62–64 °C (lit. mp 64 °C).[11] Reaction of 3 with sodium in methanol according to the procedure described by Chapman and coworkers[11] afforded *endo*-dicyclopentadiene-1,8-dione bis(ethylene ketal) (4, 41%), mp 91–92 °C (lit. mp 91–92 °C).[11] Partial hydrolysis of 4 to the corresponding 8-monoethylene ketal 5, (mp 93–94 °C, lit. mp 94 °C)[12] was performed in 58% yield by the method described by Vogel and Wyes.[12] Photocyclization[12] of 5 to pentacyclo[5.3.0.0^{2,6}.0^{3,10}.0^{4,8}]decane-5,9-dione monoethylene ketal (6, mp 58 °C, lit. mp 58–60 °C)[12] was effected in 85% yield with Pyrex-filtered light (benzene solvent).[12] Finally, acidic hydrolysis of 6 with 10% aqueous sulfuric acid[5] afforded 7 as a colorless microcrystalline solid (49%, mp 162 °C, lit. mp 162–163 °C):[5] ^{1}H NMR ($CDCl_3$) δ 2.86 (s, 2 H), 3.3–3.5 (m, 4 H); ^{13}C NMR ($CDCl_3$) δ 32.90 (d), 37.07 (d), 41.26 (d), 43.46 (d), 213.08 (s).

5,9-Dibromo-5,9-dinitropentacyclo[5.3.0.0^{2,6}.0^{3,10}.0^{4,8}]decane (9). Pentacyclo[5.3.0.0^{2,6}.0^{3,10}.0^{4,8}]decane-5,9-dione dioxime (8) was synthesized in 86% yield via reaction of 7 with hydroxylamine hydrochloride; the method described by Corey and co-workers was employed for this purpose.[13] To a stirred, ice-cold solution of dioxime 8 (1.90 g, 0.01 mol), sodium bicarbonate (4.2 g, 0.05 mol), dimethylformamide (20 mL), and water (100 mL) was added bromine (3.20 g, 0.02 mol) dropwise during 5 min. The mixture was then stirred for an additional 15 min after the bromine addition had been completed. The resulting blue solution was extracted with methylene chloride (3 × 25 mL), and the combined extracts were washed with water, dried (anhydrous magnesium sulfate), cooled in a refrigerator for 40 min, and then filtered. The cold filtrate was added dropwise during a 40-min period to a stirred solution of 90% hydrogen peroxide (15 mL) and trifluoroacetic anhydride (8 mL) in anhydrous methylene chloride (20 mL) at room temperature. The resulting mixture was concentrated in vacuo. The residue was then diluted with water and extracted with diethyl ether. The organic layer was washed successively with water and with brine, dried (anhydrous magnesium sulfate), and filtered, and the filtrate was then concentrated in vacuo to afford crude 9 (0.912 g, 24%). The crude product was recrystallized from ethyl acetate–hexane mixed solvent, thereby affording an analytical sample of 9 (mixture of isomeric compounds, exo and endo, 5,9-Br and 5,9-NO_2 substituents) as a colorless microcrystalline solid: mp 139–140 °C; ^{1}H NMR ($CDCl_3$) δ 2.76–4.0 (m); ^{13}C NMR ($CDCl_3$) δ 36.0 (d), 36.4 (d), 36.6 (d), 36.9 (d), 41.0 (d), 41.8 (d), 42.2 (d), 42.8 (d), 48.2 (d), 48.6 (d), 49.3 (d), 49.6 (d), 55.0 (d), 55.3 (d), 56.4 (d), 56.7 (d), 96.5 (s), 96.6 (s), 97.7 (s), 98.5 (s); IR (CCl_4) 1540 (vs) and 1375 cm^{-1} (m) (NO_2 absorptions); mass spectrum (70 eV) *m/e* (relative intensity) (no molecular ion), 144.1 (100).

Anal. Calcd for $C_{10}H_8Br_2N_2O_4$: C, 31.61; H, 2.12. Found: C, 31.64; H, 2.11.

5,9-Dinitropentacyclo[5.3.0.0^{2,6}.0^{3,10}.0^{4,8}]decane (10).[8] In a 250-mL flask was placed 8 (0.95 g, 5.0 mmol) along with a solution of sodium borohydride (1.6 g, 5.0 mmol) in 60% aqueous ethanol (45 mL). The resulting mixture was stirred magnetically at room temperature for 1 h, at which time the mixture was acidified with dilute aqueous acetic acid and concentrated in vacuo. The residue was diluted with water and extracted with methylene chloride (2 × 30 mL). The combined extracts were washed successively with water and with brine, dried (anhydrous magnesium sulfate), and filtered, and the filtrate was concentrated in vacuo. The residue was recrystallized from ethyl acetate–hexane mixed solvent to afford an analytical sample of 10 (mixture of isomeric compounds, exo and endo 5,9-NO_2 substituents) as a colorless microcrystalline solid (0.78 g, 70%): mp 70–73 °C; ^{1}H NMR ($CDCl_3$) δ 3.2–3.6 (m, 8 H), 4.62–4.91 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 36.48 (d), 36.59 (d), 39.03 (d), 39.03 (d), 40.22 (d), 41.46 (d), 41.46 (d), 41.68 (d), 41.90 (d), 42.55 (d), 42.76 (d), 43.09 (d), 48.13 (d), 48.13 (d), 49.26 (d), 49.64 (d), 90.98 (d), 91.41 (d), 91.63 (d), 91.74 (d); IR (CCl_4) 1540 (vs) and 1375 cm^{-1} (m) (NO_2 absorptions); mass spectrum (70 eV), *m/e* (relative intensity) (no molecular ion), 115.0 (100).

Anal. Calcd for $C_{10}H_{10}N_2O_4$: C, 54.06; H, 4.42. Found: C, 54.06; H, 4.53.

5,5,9,9-Tetranitropentacyclo[5.3.0.0^{2,6}.0^{3,10}.0^{4,8}]decane (1). To a rapidly stirred solution of sodium hydroxide (110 mg, 2.6 mmol) in 45.6% aqueous methanol (11 mL) was added 10 (222 mg, 1.0 mmol) under a nitrogen blanket. The resulting clear yellow solution was added dropwise under nitrogen to a vigorously stirred mixture of potassium ferricyanide (3.60 g, 11 mmol) and sodium nitrite (1.54 g, 22 mmol) in a two-phase solvent system consisting of water (100 mL) and pentane (200 mL). The reaction mixture was stirred for 1 h after the addition had been completed. The layers were then separated, and the aqueous layer was extracted with pentane (2 × 100 mL). The combined pentane extracts were

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washed with water, dried (anhydrous magnesium sulfate), and filtered, and the filtrate was concentrated in vacuo, affording crude 1 (202 mg, 65%). An analytical sample of 1 was obtained as a pale yellow microcrystalline solid, mp 155–156 °C, via careful recrystallization of the crude product from ethyl acetate–hexane mixed solvent: ^{1}H NMR ($CDCl_3$) δ 3.1–3.2 (br s, 2 H), 3.6–3.7 (m, 6 H); ^{13}C NMR ($CDCl_3$) δ 36.8 (d), 43.8 (d), 44.7 (d), 51.7 (d), 127.1 (s); IR (CCl_4) 1550 (vs), 1365 (m), 1330 (m), 1260 (m), 1220 (m), 1110 (m), 985 cm^{-1} (m); mass spectrum (70 eV), *m/e* (relative intensity) (no molecular ion), 144.0 (18.6), 132.0 (21.1), 131.0 (25.3), 128.0 (24.5), 127.0 (34.6), 126.0 (27.8), 117.9 (10.1), 116.0 (53.6), 115.0 (100), 114.0 (13.9), 105.0 (11.0), 104.0 (19.0), 103.0 (27.4), 102.0 (27.0), 101.0 (12.7), 92.1 (11.0), 91.0 (13.9), 90.0 (12.7), 89.0 (28.7), 79.9 (9.3), 78.0 (28.7), 77.0 (42.7), 76.0 (24.1), 74.9 (16.5), 74.0 (11.0), 64.9 (21.1), 63.0 (55.3), 62.0 (14.8), 54.9 (14.3), 52.9 (19.8), 52.0 (32.1), 51.0 (64.6), 50.0 (30.0), 45.9 (55.7), 43.9 (52.7).

Anal. Calcd for $C_{10}H_8N_4O_8$: C, 38.47; H, 2.58. Found: C, 38.50; H, 2.69.

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Registry No. 1, 91861-20-0; 2, 176-32-9; 3, 25834-57-5; 4, 4576-45-8; 5, 4576-44-7; 6, 4514-82-3; 7, 14725-77-0; 8, 91861-21-1; 9, 91861-22-2; 10, 91861-23-3; cyclopentanone, 120-92-3; ethylene glycol, 107-21-1.

(14) Research sponsored by Air Force Office of Scientific Research, Air Force Systems Command, USAF, under Grant Number AFOSR-84-0085.

Reversal of Attenuation in Substituent Effects. Influence of Substituent Angular Orientation

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The replacement of hydrogen by an electronegative atom such as fluorine, chlorine, or bromine on the hydrocarbon skeleton of a carboxylic acid usually produces the expected acidity enhancing effect.[1] As the number of carbons positioned between the dipole and the reaction site increases, the magnitude of the substituent effect normally diminishes.[2] This phenomenon has been interpreted in terms of either the Kirkwood–Westheimer electrostatic field model or in terms of diminishing polarizations of bonding electrons as the number of intervening carbons increases. We now report pK_a data which demonstrate a reversed distance attenuation, the origin of which is ascribed primarily to dipole angular orientation differences.

Results

During the course of a recent study[3] on elimination reactions, the two vinyl chlorides **3** and **4** were generated via base-induced eliminations from *syn*- and *anti-cis*-11,12-dichloro-9,10-dihydro-9,10-ethano-1-anthroic acids **1a** and **2a**. Isomeric assignments for **3** and **4** were made on the basis of the magnitude of the coupling of the C-9 and C-10 protons with the vinyl hydrogen. The C-9 proton (peri to the carboxylate group) absorbs at a lower field than does the C-10 proton.[4] Thus for **3** the lower field doublet lies at δ 6.43 (J = 7.0 Hz) and the higher field doublet at δ 5.32 (J = 2.1 Hz). For **4** essentially the reverse is true with the low-field doublet at δ 6.34 (J = 2.1 Hz) and the high-field doublet at δ 5.36 (J = 7.0 Hz). In each case the smaller J value corresponds to four-bond rather than three-bond coupling.

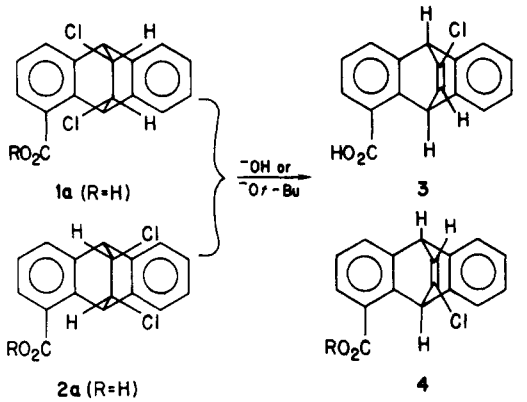

Table I. Apparent pK_a's of Several Bridged Anthracene-1-carboxylic Acids in 50% Aqueous Ethanol at 25 °C

acid	pK_a	acid	pK_a
3	5.72 ± 0.01	5	5.94 ± 0.01
4	5.90 ± 0.01	6	5.96 ± 0.01[5]

The apparent pK_a's of **3** and **4** were determined by potentiometric titration in 50% by volume aqueous ethanol. For comparison, 9,10-dihydro-9,10-etheno-1-anthroic acid (**5**) was prepared by zinc dechlorination of a mixture of **1b** and **2b** (R = CH_3) followed by hydrolysis. Its pK_a, that for the corresponding 9,10-ethano derivative **6**, and those for **3** and **4** are shown in Table I.

(1) Reversed (i.e., acid weakening) substituent effects have been observed in several systems possessing appropriate geometric orientations of the dipole and carboxylate group. See: Bowden, K.; Hojatti, M. *J. Chem. Soc., Chem. Commun.* **1982**, 273 and references therein.

(2) Fall-off factors in the range of 2–3 have been cited. See, for examples: Wilcox, C. F.; Leung, C. *J. Am. Chem. Soc.* **1968**, *90*, 336 and references therein.

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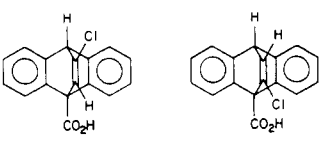
(4) The proton spectrum of 11-chloro-9,10-dihydro-9,10-etheno-anthracene (the "decarboxylated analog" of **3** or **4**) shows two bridgehead proton doublets centered at about 5.3 ppm. The two doublets have coupling constants of about 7 and 2 Hz, respectively. The assignments for **3** and **4** are supported by the observation of nearly identical coupling constants for related isomeric vinyl chlorides bearing as the only other substituent a methyl, a methoxyl, a nitro, or a carboxylic acid at C-9. Results obtained from the potassium *tert*-butoxide induced elimination of HCl from *cis*- or *trans*-11,12-dichloro-9,10-dihydro-9,10-ethano-9-anthroic acid (the analogues of **1a** and **2a** bearing CO_2H at C-9) are also in accord with the assignments made for **3** and **4**. From either of these substrates (bearing CO_2H at C-9) only one vinyl chloride is obtained, namely, the 11-chloro isomer (i). None of the isomeric vinyl halide ii was detected. Transition-state electrostatic calculations similar to those



successfully applied to eliminations from **1a**, **2a**, and the trans analogues[3] predicted a ratio i/ii = 97/3 from the 11,12-dichloro-9,10-dihydro-9,10-ethano-9-anthroic acids. The coupling constant involving the C-10 H of i is 2 Hz. That for ii obtained by another route is approximately 7 Hz. These data will be fully presented in a later publication dealing with the elimination reactions.

(5) Value reported by Golden, R.; Stock, L. M. *J. Am. Chem. Soc.* **1972**, *94*, 3080. See also footnote 5 in ref 6.

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