of pentane was followed by sequential extraction with water, 1 N HCl, and $NaHCO_3$ solution, and the organic phase was dried and evaporated to give 0.56 g (74%) of **14**, which VPC (180 °C) showed to be greater than 95% one component: ^1H NMR δ 0.00 (s, 9 H), 0.86 (m, 3 H), 0.91 (*ABX* pattern, 2 H), 1.23 (s, 16 H), 1.5 (m, 2 H, CH_2CO), 1.99 (s, 3 H), 4.96 (AB*X* pattern, 1 H); IR 1735, 1250, 840 cm^{-1}. Anal. Calcd for $C_{17}H_{36}O_2Si$: C, 67.94; H, 12.08. Found: C, 68.00; H, 12.11.

Ethyl 2-(Trimethylsilyl)dodecanoate (15). A solution of 1.1 g (1.5 mL, 11 mmol) of diisopropylamine in 10 mL of THF was treated at -78 °C with 3.5 mL (11 mmol) of 3.15 N *n*-BuLi in hexane. After 10 min, 1.6 g (10 mmol) of ethyl (trimethylsilyl)acetate in 2 mL of THF was added dropwise. HMPT (0.9 g, 5 mmol) was added 30 min later, followed by 2.7 g (10 mmol) of 1-iododecane. The reaction mixture was stirred at -78 °C for an additional 4 h and at 25 °C overnight, whereupon it was poured into 1 N HCl-pentane, and the organic phase was washed with K_2CO_3 solution. After drying and evaporation, Kugelrohr distillation gave 2.4 g of material, bp 90-100 °C (0.4 mmHg) which VPC (180 °C) showed contained 70% of **15**, 20% starting ester, and 10% 1-iododecane. Pure **15** was isolated by preparative VPC: ^1H NMR δ 0.03 (s, 9 H), 0.85 (m, 3 H), 1.22 (br s over t, 21 H), 1.65-1.95 (m, 2 H), 4.08 (q, J = 7 Hz, 3 H); IR 1719, 1251, 845 cm^{-1}. Anal. Calcd for $C_{17}H_{36}O_2Si$: C, 67.94; H, 12.08. Found: C, 68.13; H, 11.99.

2-(Trimethylsilyl)-1-dodecanol (16). A solution of 1.5 g (3.5 mmol) of **15**, 70% pure by VPC, in 2 mL of ether was slowly added to 0.87 g (15 mmol) of $LiAlH_4$ in 25 mL of ether. After an 18-h reflux, 3.0 mL of 1.5 N NaOH was added at 0 °C, and the resulting slurry filtered through Celite. After workup, Kugelrohr disillation gave 0.91 g of material, bp 80-90 °C (0.4 mmHg) which VPC (180 °C) showed contained **16** contaminated with 15% of a lower eluting impurity: ^1H NMR δ 0.00 (s, 9 H), 0.86 (m, 4 H, CH_3 + CHSi), 1.24 (br s, 18 H), 1.35 (s, 1 H, OH), 3.60-3.85 (*ABX* pattern, 2 H); IR 3350, 1250, 840 cm^{-1}. Anal. Calcd for $C_{15}H_{34}OSi$: C, 69.70; H, 13.26. Found: C, 69.76; H, 13.37.

1-Acetoxy-2-(trimethylsilyl)dodecane (17). A mixture of 0.39 g (1.5 mmol) of **16**, 0.20 g (2.5 mmol) of pyridine, 0.004 g of *p*-(*N*,*N*-dimethylamino)pyridine, and 0.25 g (2.5 mmol) of acetic anhydride in 2 mL of ether was allowed to stand at 25 °C for 18 h. After workup, evaporation gave 0.23 g of water-white **17**, which VPC (180 °C) indicated contained 15% of a lower eluting impurity: ^1H NMR δ 0.00 (s, 9 H), 0.86 (m, 3 H), 0.9 (m, 1 H), 1.24 (br s, 18 H), 2.01 (s, 3 H), 4.0-4.3 (*ABX* pattern, 2 H); IR 1740, 1237, 1250, 838 cm^{-1}. Anal. Calcd for $C_{17}H_{36}O_2Si$: C, 67.94; H, 12.08. Found: C, 67.92; H, 12.19.

An Improved Synthesis of *anti*-Benzo[*c*]phenanthrene-3,4-diol 1,2-Epoxide via 4-Methoxybenzo[*c*]phenanthrene

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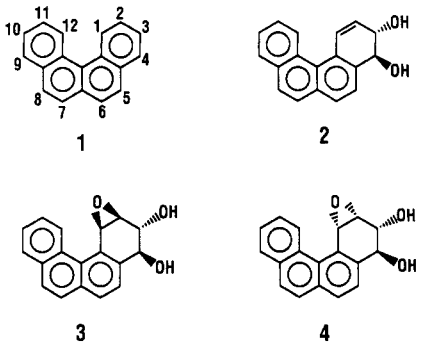
Benzo[*c*]phenanthrene (**1**) is a relatively weak carcinogen[1] widely distributed in the environment.[2] Metabolism of **1** on the benzo ring occurs almost exclusively at the 3,4-position to give *trans*-benzo[*c*]phenanthrene-3,4-diol[3] (**2**), the precursor to the bay region benzo[*c*]-phenanthrene-3,4-diol 1,2-epoxides (BcPhDE). BcPhDE exists as a pair of diastereoisomers: *syn*-BcPhDE (**3**) and *anti*-BcPhDE (**4**). Each diastereoisomer consists of a pair of enantiomers. Both diastereoisomers are highly mutagenic to bacteria and Chinese hamster V79 cells and have shown exceptionally high tumor-initiating activity on mouse skin.[4,5a] However in the newborn mouse tumor



1 **2** **3** **4**

model, **4** is more potent than **3**.[5b] The diol epoxides **3** and **4** covalently bind to calf thymus DNA at exocyclic nitrogens of guanine and adenine almost in equal proportions.[6] Similarly the enantiomeric diol epoxides (**3**, **4**) also bind to DNA in embryo cell cultures of mouse, hamster, and rat.[7] However, the major adducts obtained via metabolic activation of **1** in cell cultures were only due to **4**. During our preparation of 1-(N^2-deoxyguanosyl/N^6-deoxyadenosyl-3′-phosphate)-2,3,4-trihydroxy-1,2,3,4-tetrahydrobenzo[*c*]phenanthrene, as markers for the ^{32}P postlabeling assay,[8] we required substantial amounts of the *anti*-BcPhDE (**4**). The methods[9] available for the preparation of **4** either required multiple steps or resulted in low yields and were not suitable for our purpose. In the present study, we report the synthesis of 4-methoxybenzo[*c*]phenanthrene (**5**), the key intermediate for the preparation of **4**.

Initially, we prepared 4-hydroxybenzo[*c*]phenanthrene (**6**) using Harvey's procedure.[10] The critical step in the synthesis was the generation of the anion **7**. The formation of **7** occurs within a small range of temperatures (-42 °C to -38 °C); which is close to the boiling point of the reaction medium, liquid ammonia (-33 °C), hence care must be taken to control the reaction temperature. The cyclization of **8** to **9** was temperature dependent. At 130 °C, **8** afforded a 1:1 mixture of **9** and **10**, in contrast to exclusively **9** at 115 °C. Oxidation of **6** was carried out with Fremy's salt to afford benzo[*c*]phenanthrene-3,4-dione (**11**).[11] Subsequent steps involved $NaBH_4$ reduction of

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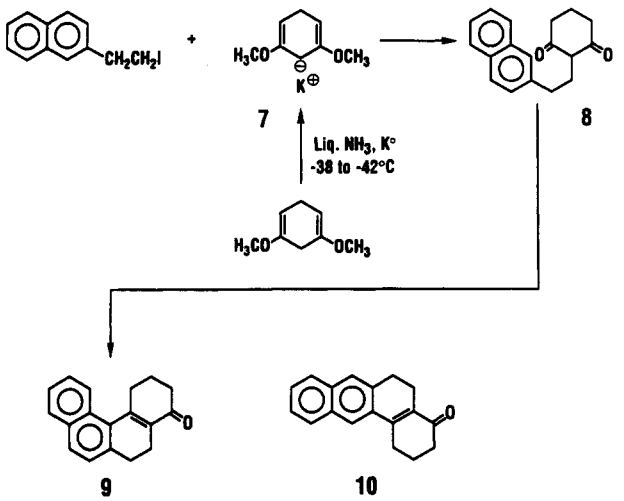
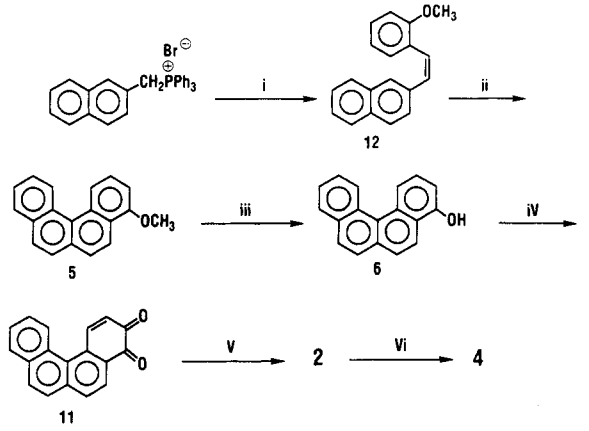
11 to 2 and mCPBA oxidation of 2 to 4. The overall yield of 4 from 2-(2-naphthyl)ethyl iodide was 5.5%.



Carruthers[12a] has reported the formation of benzo[c]-phenanthrene from β-naphth-2-ylstyrene by irradiation with UV light. We used a similar approach to prepare 4-methoxybenzo[c]phenanthrene (5), from which 6 could be obtained via demethylation. We synthesized olefin 12, the precursor to 5, from 2-(bromomethyl)naphthalene and o-anisaldehyde. Photolysis of 12 in benzene at room temperature afforded 5 in 37% yield. Interestingly, no benz[a]anthracene derivative, which could have resulted via an alternative cyclization of 12 to the 3-position of the naphthalene ring, was detected.[13] The proton assignments



(i) o-Anisaldehyde, CH_3ONa/CH_3OH; (ii) $h\nu$, C_6H_6, 25 °C; (iii) BBr_3/CH_2Cl_2; (iv) $(KSO_3)_2NO$; (v) $NaBH_4$; (vi) mCPBA

of 5 were obtained from the phase-sensitive COSY[14] spectrum (supplementary material). As would be expected, the H_1 and H_{12} fjord region protons resonate farthest downfield at 9.2 and 8.8 ppm, respectively, as a direct consequence of the deshielding experienced by the aromatic ring across the bay. The rest of the assignments are not particularly remarkable. Most interesting are the several long-range couplings that appear in the contour plot. First is a five-bond epi zig-zag coupling[15] between H_1 and H_5, the latter appearing at 8.5 ppm. The second long-range coupling is another epi zig-zag coupling between H_{12} and H_8. The most interesting however, is the six bond coupling between H_1 and H_{12}. Ittah and Jerina have reported a 14-Hz coupling between the fluorine and H_{12} in 1-fluorobenzo[c]phenanthrene.[16]

Demethylation of 5 with boron tribromide yielded 6 in 80% yield. The overall yield of 6 from 2-(bromomethyl)naphthalene was 21%. Oxidation of 6 with $(KSO_3)_2NO$ afforded benzo[c]phenanthrene-3,4-dione, which was converted to epoxide 4 in 51% overall yield from 6.

Experimental Section

Melting points are uncorrected. Chemical shifts are reported in ppm on the δ scale relative to internal standard tetramethylsilane (or appropriate solvent peaks) with coupling constants given in hertz. Chemical reagents used were procured from Aldrich Chemical Co. Elemental analysis was performed by Galbraith Laboratories, Inc.

1-(2-Naphthyl)-2-(2-methoxyphenyl)ethylene (12). (2-Naphthylmethyl)triphenylphosphonium bromide (4.82 g, 0.01 mol), 2-anisaldehyde (1.36 g, 0.01 mol) and $NaOCH_3$ (1.1 g, 0.02 mol) in CH_3OH (150 mL) were allowed to react at room temperature for 3 h. The reaction was quenched with H_2O (150 mL), the organic portion was extracted into CH_2Cl_2 (3 × 150 mL), and the residue after removal of CH_2Cl_2 was filtered through a silica gel column with elution by hexane. The filtrate upon concentration afforded a 1:1 mixture of cis and trans olefin 12 as a thick oil (1.8 g, 69%): NMR ($CDCl_3$) δ 3.8, 3.9 (s, OCH_3, 3 H) 7.8–7.1, 7.2–7.5, and 7.7–7.9 (m, aromatics, 11 H). The integration of the singlets at 3.8 and 3.9 ppm was 1:1.

4-Methoxybenzo[c]phenanthrene (5). A solution of 12 (1.3 g, 5 mmol) and iodine (5 mg) in dry benzene (1 L) was irradiated with a Pyrex filtered Havonia 450-W medium-pressure UV lamp while bubbling dry air into the solution. The cyclization of 12 was monitored by TLC. After 12 h, the solvent was removed under reduced pressure, and the yellow residue was filtered through a silica gel column with elution by hexane. The solvent was removed from the filtrate, and the residue was crystallized from EtOH to yield pure 5 (0.48 g, 37%): mp 78–79 °C; NMR ($CDCl_3$) δ 4.0 (s, 3 H, OCH_3), 7.05 (d, 1 H, H_3, $J_{2,3}$ = 7.8 Hz), 7.6–7.8 (m, 3 H, H_2, H_{10}, and H_{11}), 7.84–7.94 (m, 3 H, H_6, H_7, and H_8), 8.05 (d, 1 H, H_9, $J_{9,10}$ = 7.8 Hz), 8.48 (d, 1 H, H_5, $J_{5,6}$ = 8.8 Hz), 8.78 (d, 1 H, H_1, $J_{1,2}$ = 8.6 Hz), 9.2 (d, 1 H, H_{12}, $J_{11,12}$ = 8.4 Hz); MS *m/e* 258 (M^+, 100), 228 (3.7), 215 (65.5). Anal. Calcd for $C_{19}H_{14}O$: C, 88.37; H, 5.43. Found: C, 88.59; H, 5.43.

4-Hydroxybenzo[c]phenanthrene (6). To a stirred solution of 5 (0.52 g, 1 mmol) in CH_2Cl_2 (75 mL) was added a solution of boron tribromide (1 mL, 1 M) in CH_2Cl_2 at 0 °C under N_2 over a period of 5 min. After 12 h at room temperature, the mixture was hydrolyzed with ice-cold H_2O, the organic layer was washed several times with H_2O and dried ($MgSO_4$), and the solvent was removed to yield crude 6, which was recrystallized from CH_2Cl_2–hexane (1:1), (0.4 g, 80%), mp 108–109 °C (lit.[17] mp 110–111 °C).

Benzo[c]phenanthrene-3,4-dione (11). To a stirred mixture of 6 (0.24 g, 1 mmol), KH_2PO_4 (40 mL, 0.17 M), and Fremy's salt (0.64 g, 2.2 mmol) in 150 mL of CH_2Cl_2–benzene (16:84) was added Adogen 464 (10 drops), and the stirring was continued for 12 h at room temperature. The reaction was quenched with H_2O (200 mL), and the organic portion was extracted into benzene (2 × 100 mL). The benzene layer was washed with H_2O, dried ($MgSO_4$),

(11) Oxidation of 3-hydroxybenzo[c]phenanthrene to 11 has been reported.[9a] We expected similar results from 4-hydroxybenzo[c]-phenanthrene since steric hindrance at the fjord region would inhibit formation of a 1,4-dione.

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and concentrated under reduced pressure. The resulting residue was recrystallized from CH_2Cl_2/hexane to yield **11** as a bright red crystalline solid (0.19 g, 80%): mp 180–181 °C (lit.[9a] mp 178–180 °C); ^1H NMR ($CDCl_3$) δ 6.5 (d, 1 H, H_2, $J_{1,2}$ = 10.5 Hz), 7.6–8.1 (m, 6 H, aromatic), 8.48 (d, 1 H, H_1, $J_{1,2}$ = 10.5 Hz).

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Supplementary Material Available: The phase-sensitive COSY spectrum of 4-methoxybenzo[*c*]phenanthrene (2 pages). Ordering information is given on any current masthead page.

A General Approach to the Synthesis of Polyquinenes via the Weiss Reaction. 10. Transient Formation of *cis*-Tetracyclo[7.2.1.$0^{4,11}$.$0^{6,10}$]dodeca-3,5,7,9-tetraene and an Approach toward 10,11-Dimethyl-*cis*-tetracyclo[7.2.1.$0^{4,11}$.$0^{6,10}$]dodeca-3,5,7,9-tetraene

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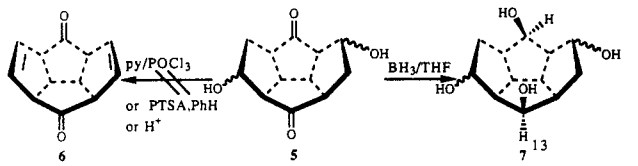
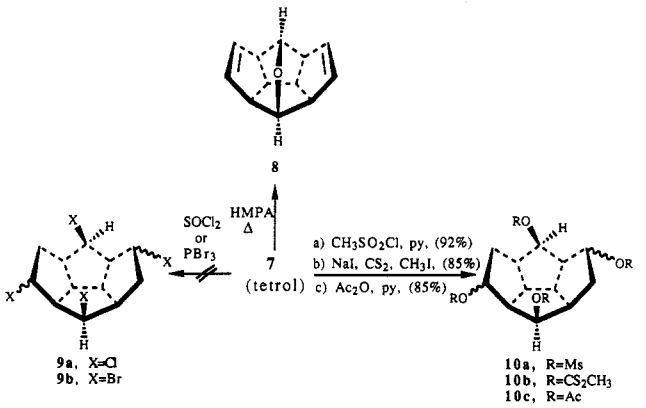
This decade has witnessed much interest in the synthesis and reactions of polyquinanes[1] and polyquinenes.[2-7] In this connection polyquinenes **1** and **2** were studied earlier from a computational point of view.[8] According to ring current criteria, Jung[9] predicted that dicyclopenta[*cd*,*gh*]pentalene **1** would be aromatic; however, Binsch[10] proposed that **1** would behave as an antiaromatic species due to its second-order double-bond fixation. Nakajima et al. employed semiempirical SCF MO theory to arrive at the same conclusion.[11] Hess[8b] and Garret[12] have also studied the stability of systems such as **1** and **2**; moreover, MNDO calculations from our laboratory[13] and MM2 computations from Paquette et al.[14] have described the increase in strain energy in going from **2** to **1** as substantial (see also ref 15). In this respect **1** should behave as a highly reactive olefin with no sign of peripheral π delocalization[15] in contrast to the earlier hypothesis of Platt.[8a]

Since the previously reported difference in the heat of formation between tetraene **3a** and isomeric olefin **3b** was

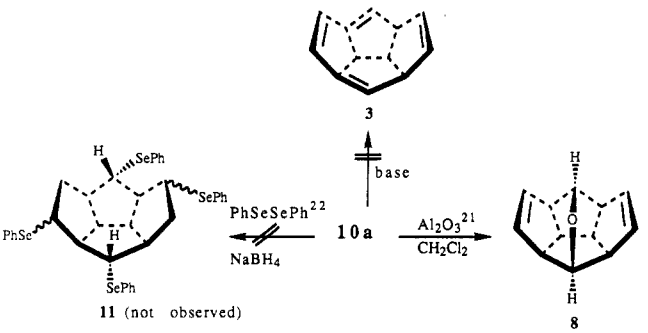
Scheme I



Scheme II



Scheme III



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