by the addition of water to the dimer. This ketone distilled at 55–56 °C (0.05 mm): IR 1720 cm^{-1}; NMR δ 0.10 (s, 18 H), 0.70 (t, 4 H), 2.26 (t, 4 H); mass spectrum parent peak at *m/e* 230 (theory 230).

1,4-Bis(trimethylsilyl)butane. This disilylated compound was isolated from the reaction mixture described above unless the β-trimethylsilylpropionic acid was carefully distilled. 1,4-Bis(trimethylsilyl)butane is a product from a Grignard coupling reaction in the preparation of the acid and vacuum distills at 36–38 °C (0.05 mm): NMR δ −0.10 (s, 18 H), 0.48 (m, 4 H), and 1.18 (m, 4 H).

Anal. Calcd for $C_{10}H_{26}Si_2$: C, 59.41; H, 12.87. Found: C, 59.67; H, 13.05.

General Procedure for Cycloadditions. To a solution of 0.2 mol of triethylamine and 0.2 mol of olefin in 150 ml of dry hexane at room temperature was added 0.1 mol of acid halide in 20 ml of hexane dropwise over a 0.5-h period.[10] After the addition was complete, the reaction mixture was stirred and refluxed for 2 h. The reaction was monitored by VPC analysis, and upon completion of the reaction, the salt was removed by filtration and the solvent by rotary evaporation. The residue was vacuum distilled.

***endo*-7-Trimethylsilylmethylbicyclo[3.2.0]hept-2-en-6-one (IV).** This cycloadduct of (trimethylsilylmethyl)ketene and cyclopentadiene was obtained at 65 °C (0.05 mm) (65%): IR 1800 and 1610 cm^{-1}; NMR δ 0.20 (s, 9 H), 0.84 (8 lines, 2 H), 2.64 (m, 2 H), 3.80 (m, 3 H), and 5.98 (dm, 2 H); mass spectrum parent peak at *m/e* 194 (theory 194).

Anal. Calcd for $C_{11}H_{18}OSi$: C, 68.04; H, 9.28. Found: C, 67.96; H, 9.52.

***trans*-3-Ethoxy-2-trimethylsilylmethylcyclobutanone (V).** This cycloadduct of (trimethylsilylmethyl)ketene and ethyl vinyl ether was distilled at 40–42 °C (0.05 mm) (60%): IR 1780 cm^{-1}; NMR δ 0.10 (s, 9 H), 0.88 (d, 2 H), 1.22 (t, 3 H), 2.94–3.90 (m, 5 H), and 4.27 (m, 1 H); mass spectrum parent peak at *m/e* 200 (theory 200).

Anal. Calcd for $C_{10}H_{20}O_2Si$: C, 60.00; H, 10.00. Found: C, 59.51; H, 10.82.

2-Chloro-2-methyl-3-trimethylsilylmethylcyclobutanone (VI). The cycloadduct of methylchloroketene and allyltrimethylsilane distilled at 68–70 °C (0.025 mm) (62%): IR 1780 cm^{-1}; NMR (both isomers) δ 0.24 (s, 9 H), 1.04 (8 lines, 2 H), 1.68 and 1.80 (two singlets, ratio 6:1, 3 H), 2.84 (m, 2 H), and 3.40 (m, 1 H).

Anal. Calcd for $C_9H_{17}ClOSi$: C, 52.81; H, 8.31. Found: C, 52.39; H, 8.26.

2,2-Dichloro-3-trimethylsilylmethylcyclobutanone (VII). This cycloadduct of dichloroketene and allyltrimethylsilane was vacuum distilled at 65–66 °C (0.025 mm) (54%): IR 1785 cm^{-1}; NMR δ 0.12 (s, 9 H), 1.20 (8 lines, 2 H), 2.98 (m, 2 H), and 3.40 (m, 1 H).

Anal. Calcd for $C_8H_{14}Cl_2OSi$: C, 42.67; H, 6.22. Found: C, 42.89; H, 6.11.

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Registry No.—I, 61063-48-7; II, 61063-49-8; III, 18053-95-7; IV, 61063-50-1; V, 61063-51-2; *cis*-VI, 61063-52-3; *trans*-VI, 61063-53-4; VII, 61063-54-5; β-trimethylsilylpropionyl chloride 18187-31-0; 1,4-bis(trimethylsilyl)butane, 18001-81-5; cyclopentadiene, 542-92-7; ethyl vinyl ether, 106-98-9; methylchloroketene, 13363-86-5; allyltrimethylsilane, 762-72-1; dichloroketene, 4591-28-0.

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(10) The use of a stoichiometric amount of triethylamine resulted in the same distribution of products but a lower yield.

Synthesis of 4,5-Dihydroxy-1,3,6,8-tetramethylphenanthrene[1]

Melvin S. Newman* and H. M. Dali[2]

Chemistry Department, Ohio State University, Columbus, Ohio 43210

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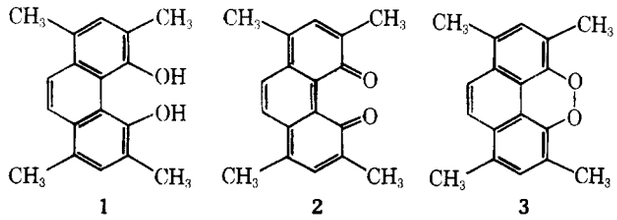
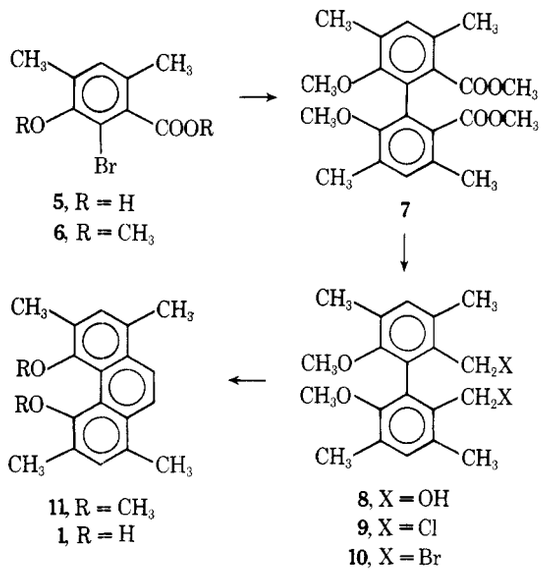
Bromination of 4,6-dimethyl-3-hydroxybenzoic acid (**4**) yielded exclusively 2-bromo-4,6-dimethyl-3-hydroxybenzoic acid (**5**), which was methylated to yield methyl 2-bromo-4,6-dimethyl-3-methoxybenzoate (**6**). Ullman coupling of **6** afforded dimethyl 6,6′-dimethoxy-3,3′,5,5′-tetramethyldiphenate (**7**). Reduction of **7** with $LiAlH_4$ yielded 2,2′-di(hydroxymethyl)-6,6′-dimethoxy-3,3′,5,5′-tetramethylbiphenyl (**8**), which was converted into 2,2′-di(chloromethyl)-6,6′-dimethoxy-3,3′,5,5′-tetramethylbiphenyl (**9**) via the dimesylate of **8**. A new phenanthrene synthesis which involved treatment of **9** with sodamide in ammonia afforded 4,5-dimethoxy-1,3,6,8-tetramethylphenanthrene (**11**) in almost quantitative yield. Demethylation of **11** by heating with pyridine hydrochloride or with anhydrous sodium sulfide yielded 4,5-dihydroxy-1,3,6,8-tetramethylphenanthrene (**1**), which could not be oxidized to a monomeric quinone.

The main objective of this work was to synthesize 4,5-dihydroxy-1,3,6,8-tetramethylphenanthrene (**1**), to see if it could be oxidized to the corresponding quinone, **2**, whose stability with respect to the tautomeric cyclic peroxide, **3**, would be of interest. It was hoped that the methyl groups would decrease the nuclear oxidation encountered in a previous attempt to synthesize 4,5-phenanthrenequinone from 4,5-dihydroxyphenanthrene.[3]



The synthesis of **1** was carried out as outlined in Scheme I. 4,6-Dimethyl-3-hydroxybenzoic acid (**4**), prepared as described[4] except that diethyl acetylenedicarboxylate, made by an improved procedure, was used in place of dimethyl acetylenedicarboxylate, was brominated cleanly to 2-bromo-4,6-dimethyl-3-hydroxybenzoic acid (**5**). This structure is supported by analysis and the fact that acid-catalyzed esterification failed. Hence a highly hindered acid, namely **5**, was at hand.

Scheme I



Methylation of the dipotassium salt of **5** with methyl iodide produced methyl 2-bromo-4,6-dimethyl-3-methoxybenzoate (**6**), which on coupling via the Ullmann reaction afforded dimethyl 6,6′-dimethoxy-3,3′,5,5′-tetramethyldiphenate (**7**). The yields in this coupling reaction were quite erratic (0–75%) as the age and type of copper used evidently affect the course of the reaction (see Experimental Section for details). One side reaction involved reduction of the bromine as in some experiments appreciable quantities of methyl 2,4-dimethyl-5-methoxybenzoate[4] were obtained. Attempts to improve the coupling reaction by utilizing the anhydride[5] in place of **7** were unpromising because of the difficulty of forming the anhydride. Reduction of **7** to **8** and conversion of **8** to **9** via the dimesylate proceeded well.

A new phenanthrene synthesis was developed to convert **9** to **11**. The use of sodamide in liquid ammonia afforded **11** in high yield, although, if a Teflon-coated magnetic stirrer was used in place of a steel stirrer, no appreciable amounts of **11** were obtained. This new phenanthrene synthesis is patterned after the intermolecular coupling of benzyl chloride to stilbene[6] and represents an improved version of the phenanthrene synthesis[7] which involves coupling of a 2,2′-di(bromomethyl)biphenyl to a 9,10-dihydrophenanthrene followed by aromatization.[8] Attempts to utilize the dibromide, **10**, in liquid ammonia as above failed to yield **11**. When the dibromo compound was treated with phenyllithium,[7] only a small yield of 9,10-dihydro-9,10-dimethyl-1,3,6,8-tetramethylphenanthrene was obtained.

The demethylation of **11** to **1** was readily effected by heating at reflux with anhydrous sodium sulfide[9] in *N*-methylpyrrolidone (NMP) or with pyridine hydrochloride[10] at 200–210 °C.

All attempts to oxidize **1** to **2** (or **3**) failed. In many cases (e.g., heating with *o*-chloranil) evidence was obtained that oxidation had occurred but all attempts at isolation of **2** or **3** resulted in the formation of polymeric materials. With most oxidants pronounced color changes occurred. The electrochemical oxidation of **1** and 2,7-dihydroxyphenanthrene were studied briefly using conventional voltammetric techniques.[11] A carbon-paste working electrode, platinum auxiliary electrode, and saturated calomel reference electrode (SCE) were used in the usual manner.[12]

A cyclic voltammogram of 2,7-dihydroxyphenanthrene in aqueous 0.1 M NaOH includes a well-defined oxidation wave with a half-peak potential of +0.10 V vs. SCE. After this initial oxidation, there appears a new, reversible redox couple centered at −0.03 V. It is most likely that the initial oxidation corresponds to formation of a quinone, which undergoes hydroxylation to form the new redox couple. The addition of nucleophiles to quinones to form electroactive products has been well characterized.[13]

A voltammogram of **1** is quite different, with the observation of a single oxidation peak, with the half-peak potential equal to +0.03 V vs. SCE. On the second voltammetric scan, the peak current for this wave is greatly reduced (by about 73%), indicating deposition of electroinactive material on the electrode surface. It is unlikely that this material is intact quinone, since in that case a desorptive reduction should have been observed on the negative-going scan.

One would conclude that although the oxidation of the 2,7-dihydroxy compound is complicated by an addition reaction, it is otherwise well behaved. In contrast, the oxidation of **1** appears to form a highly unstable product which reacts to form an insoluble, nonreducible film on the electrode. The nature of this film is unknown, but its properties are not what one would expect for **2**.

Experimental Section[14]

Diethyl Acetylenedicarboxylate.[15] To a stirred mixture of 120 g of the potassium acid salt of acetylenedicarboxylic acid, 200 ml of ethanol, and 200 ml of benzene in a 1-l. three-neck flask fitted with a dropping funnel, stirrer, and a 1.5 ft × 1 in. packed column topped with a phase-separating head, was added slowly 100 g of concentrated H_2SO_4. The mixture was refluxed for 24 h during which the lower layer in the head was removed (100 ml in all). After a conventional workup 120 g (90%) of colorless diethyl acetylenedicarboxylate, bp 140–142 °C (15 mm), was obtained.

2-Bromo-4,6-dimethyl-3-hydroxybenzoic Acid* (5). In the best of many experiments, 18 g of bromine was added at room temperature to a solution of 18.0 g of **4** in 75 ml of acetic acid. After 20 h the solvent was removed and the reaction product was taken into $(CH_2Cl)_2$ (30 ml), methanol (12 ml), and 1.25 ml of H_2SO_4. After refluxing for 20 h the reaction product was separated into acidic and neutral fractions. From the acid fraction 22.0 g (88%) of **5**, mp 155–156 °C, was obtained. Recrystallization from benzene gave the analytical sample: mp 155.5–157.0 °C; *m/e* 244, 246.[16] When the bromination was carried out at 45–50 °C the yield of **5**, mp 154–155 °C, was 85%.

Methyl 2-Bromo-4,6-dimethyl-3-methoxybenzoate* (6). In the best of many experiments, 40.0 g of **5** was dissolved in 40 ml of water containing 23 g of KOH. The water was removed on a rotary evaporator and the solid salt was stirred for 14 h with 80 ml of DMF and 55 ml of methyl iodide with slight refluxing of methyl iodide. After removal of the DMF under vacuum an ether–benzene solution of the products was extracted with a little aqueous NaOH to remove acidic material. Distillation then yielded 43.3 g (97%) of **6**, bp 169–170 °C (3 mm), as a colorless oil: *m/e* 272, 274; NMR ($CDCl_3$) δ 2.18 (s, 3, $ArCH_3$), 2.27 (s, 3, $ArCH_3$), 3.65 (s, 3, OCH_3), 3.74 (s, 3, OCH_3), 6.95 (s, 1, ArH).

Dimethyl 6,6′-Dimethoxy-3,3′,5,5′-tetramethyldiphenate* (7). Attempts to run the Ullmann coupling reaction with **6** gave results which were difficult to reproduce. In the best of many runs a stirred mixture of 3.50 g of **6**, 3.0 g of copper powder (Venus F-44, American Bronze Powder Co., from a freshly opened can), and 15 ml of freshly distilled DMF was held at reflux for 6 h. After the usual workup crystallization of the reaction mixture products from petroleum ether afforded 2.0 g (83%) of **7**: mp 122–123 °C; *m/e* 386; NMR δ 2.32 (s, 12, $ArCH_3$), 3.45 (s, 6, OCH_3), 3.55 (s, 6, OCH_3), 7. (s, 2, ArH) (Anal. Calcd for $C_{22}H_{26}O_6$: C, 68.4; H, 6.8. Found: C, 68.8; H, 7.0). However, in other similar runs with Venus F-44 copper and other copper (some activated by various treatments) yields varied from 0 to 75%. The most frequent by-product was methyl 2,4-dimethyl-5-methoxybenzoate, a reduction product.

2,2′-Di(hydroxymethyl)-6,6′-dimethoxy-3,3′,5,5′-tetramethylbiphenyl* (8). Reduction of **7** with $LiAlH_4$ in ether afforded **8**, mp 168–169 °C, *m/e* 330, in almost quantitative yields in small (0.5 g) and relatively large (10 g) runs.

2,2′-Di(chloromethyl)-6,6′-dimethoxy-3,3′,5,5′-tetramethylbiphenyl* (9). In a typical reaction a solution of 3.00 g of **8** and 4.2 ml of triethylamine in 45 ml of benzene was treated with 1.8 ml of methanesulfonyl chloride for 3 h at room temperature. After the benzene layer had been washed with water, about 0.3 g of Aliquat-

336,[17] 45 ml of saturated KCl solution, and some solid KCl were added. After stirring for 24 h at room temperature the benzene solution was passed through a short column of alumina (to remove the Aliquat-336). After the usual workup there was obtained 3.0 g (90%) of **9**:[18] mp 175–176 °C; *m/e* 366, 368; NMR δ 2.33 and 2.46 (s, 6 each, $ArCH_3$), 3.47 (s, 6, OCH_3), 4.34 (s, 4, CH_2Cl), 7.1 (s, 2, ArH).

2,2′-Di(bromomethyl)-6,6′-dimethoxy-3,3′,5,5′-tetramethylbiphenyl* (10). In reactions involving KBr similar to the above preparation of **9**, about 80% yields of **10**, mp 145–146 °C, *m/e* 454, 456, 458, after recrystallization of the distilled product from benzene–petroleum ether were obtained.

4,5-Dimethoxy-1,3,6,8-tetramethylphenanthrene[4] (11). In the best of many experiments sodium amide was prepared from 2 g of sodium in 100 ml of liquid NH_3.[19] This mixture (stirred with an iron stirrer) was cooled to about −70 to −65 °C by an external dry ice bath and then 150 ml of toluene followed (after cooling) by 5.8 g of **9** were added. After 2 h at −70 to −65 °C the cooling bath was removed and the reaction mixture was left under reflux (dry ice condenser) overnight. After the usual workup (initial treatment of the reaction mixture with 20 g of solid NH_4Cl) the crude product showed only one spot on TLC. Crystallization from ethanol afforded 4.45 g (95%) of **11**, mp 129–130 °C (lit.[4] mp 130–131 °C).

9,10-Dihydro-9,10-dimethoxy-1,3,6,8-tetramethylphenanthrene (12). A solution of 1.70 g of **10** in 10 ml of ether was added to the C_6H_5Li prepared from 0.2 g of Li and 1.75 g of C_6H_5Br in ether. After 2 h at reflux the reaction products were chromatographed over silica gel to yield a small amount of **12**, mp 101–102 °C *m/e* calcd 296.1776, found[16] 296.1782.

4,5-Dihydroxy-1,3,6,8-tetramethylphenanthrene (1). In the best of several attempts at demethylation of **11**, a solution of 1.0 g of **11** and 4.0 g of Na_2S[9] (dried at room temperature to constant weight in a desiccator over P_2O_5) in 10 ml of pure NMP[20] was held at reflux for 3.5 h. The reaction mixture was poured into water and the product isolated as usual to give a slightly yellow solid in 91% yield (in other attempts on heating with $C_5H_5N{\cdot}HCl$[10] high yields of similar product having darker colors were obtained). Various samples of this material melted in the 240–249 °C range. The analytical sample [mp 246.5–248.0 °C; *m/e* 266; NMR (Me_2SO-d_6) δ 2.40 (s, 6, $ArCH_3$, C_3, C_6), 2.59 (s, 6, $ArCH_3$, C_1, C_8), 7.43 (s, 2, ArH), 7.70 (s, 2, ArH)] was obtained by recrystallization from ethanol. The diol, **1**, was converted into the corresponding diacetate:* mp 224–225 °C; IR (KBr) 1750, 1760, cm^{-1}; NMR δ 2.23 (s, 6, $ArCH_3$), 2.30 (s, 6, $ArCH_3$), 2.65 (s, 6, CH_3CO_2), 7.26 (s, 2, ArH), 7.71 (s, 2, ArH).

Registry No.—**1**, 60935-38-8; **1** diacetate, 60935-46-8; **4**, 50790-68-6; **5**, 60935-39-9; **6**, 60935-40-2; **7**, 60935-41-3; **8**, 60935-42-4; **9**, 60935-43-5; **10**, 60935-44-6; **11**, 50790-66-4; **12**, 60935-45-7.

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(14) All melting points and boiling points are uncorrected. IR spectra were recorded using a Perkin-Elmer Infracord using NaCl disks (neat liquids) or KBr pellets. NMR spectra were recorded on a Varian A-60 instrument and are reported as δ units (Me_4Si, 0) in $CDCl_3$ unless otherwise noted. The phrase "worked up in the usual way" means that an ether–benzene solution of the products was washed with dilute acid and/or base, with saturated NaCl solution. The ether–benzene solution was then filtered through a cone of $MgSO_4$ and the solvent was removed on a rotary evaporator. All compounds marked with an asterisk gave elemental analytical data consistent (±0.3%) with the theoretical values, which were submitted for review. Analyses were by M-H-W Laboratories, Garden City, Mich.
(15) The present procedure represents an improvement over that described in "Organic Syntheses", Collect. Vol. IV, Wiley, New York, N.Y., 1963, p 329.
(16) Mass spectra were determined on an Associated Electrical Industries, Ltd., instrument by Mr. R. Weisenburger.
(17) Aliquat 336 is methyltricaprylammonium chloride, obtainable from the McKerson Corp., Minneapolis, Minn.
(18) The analytical sample was kindly prepared by Dr. R. Kannan.
(19) The technique and apparatus are described in M. S. Newman, "An Advanced Organic Laboratory Course", Macmillan, New York, N.Y., 1972, p 145.
(20) We thank the General Aniline and Film Corp. for a generous gift of NMP.

Synthesis and Properties of the Vicinal Trans Dihydrodiols of Anthracene, Phenanthrene, and Benzo[*a*]anthracene

Roland E. Lehr, Maria Schaefer-Ridder, and Donald M. Jerina*

National Institute of Arthritis, Metabolism and Digestive Diseases, National Institutes of Health, Bethesda, Maryland 20014

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The vicinal, trans, non-K-region dihydrodiols of anthracene, phenanthrene, and benzo[*a*]anthracene have been synthesized by a common approach involving bromination, dehydrobromination, and hydrolysis of the appropriate tetrahydrodiesters of the parent aromatic hydrocarbons. Utilization of tetrahydrodiacetate derivatives proved important for successful preparation of dihydrodiols in angular benzo rings, whereas tetrahydrodibenzoates served as appropriate precursors in all other cases. The NMR spectra of the dihydrodiols and dihydrodioldiesters are discussed. Of the dihydrodiols, only 3,4-dihydroxy-3,4-dihydrobenzo[*a*]anthracene (**9k**) could be metabolically activated to species highly mutagenic to bacteria, although 8,9-dihydroxy-8,9-dihydrobenzo[*a*]anthracene and 10,11-dihydroxy-10,11-dihydrobenzo[*a*]anthracene could be activated to weakly mutagenic species. The much greater biological activity of metabolically activated **9k** is in accord with the enhanced reactivity predicted by PMO calculations for the benzylic positions of many intermediate diol epoxides in which the oxirane ring occupies a bay region.

Vicinal, trans dihydrodiols, both at K-region and non-K-region (**1**, Scheme I) positions, are common metabolites of polycyclic aromatic hydrocarbons in mammals.[1] Their formation consists of initial oxidation of the hydrocarbons to arene oxides[2] which are then hydrated by the enzyme epoxide hydrase to trans dihydrodiols that are often optically active.[3] Recently, substantial interest has developed in dihydrodiols since they can be metabolically activated to diol epoxides[4] (**2**,