Synthesis of 9-Bromo Derivatives of 4,5-, 2,7-, and 3,6-Dimethyl- and 2,4,5,7- and 3,4,5,6-Tetramethylphenanthrenes[1]

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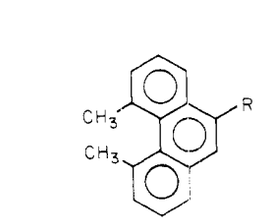
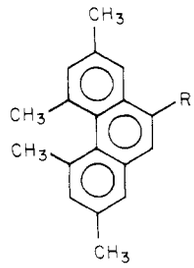
The syntheses in high yields of 4,5-dimethylphenanthrene (**1**), 2,4,5,7-tetramethylphenanthrene (**2**), 3,4,5,6-tetramethylphenanthrene (**3**), and 2,7-dimethylphenanthrene (**4**) by treatment of 2,2′-bis(chloromethyl)-6,6′-dimethylbiphenyl (**17**), 2,2′-bis(chloromethyl)-4,4′,6,6′-tetramethylbiphenyl (**18**), 2,2′-bis(chloromethyl)-5,5′,6,6′-tetramethylbiphenyl (**19**), and 2,2′-bis(chloromethyl)-4,4′-dimethylbiphenyl (**20**) with sodium amide in liquid ammonia are described. The phenanthrenes **1**, **2**, and **3** were converted with difficulty into the corresponding 9-bromo derivatives **6**, **7**, and **8** by addition of bromine to the 9,10-bond followed by elimination of hydrogen bromide. 9-Bromo-2,7-dimethylphenanthrene (**9**) and 9-bromo-3,6-dimethylphenanthrene (**10**) were readily prepared from **4** and 3,6-dimethylphenanthrene (**5**).

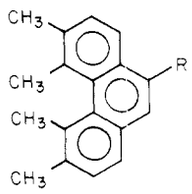
The syntheses of 4,5-dimethylphenanthrene[3] (**1**), 2,4,5,7-tetramethylphenanthrene[4] (**2**), 3,4,5,6-tetramethylphenanthrene[4] (**3**), 2,7-dimethylphenanthrene (**4**), and 3,6-dimethylphenanthrene (**5**) have been reported as



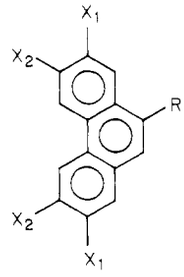
1, R = H
6, R = Br



2, R = H
7, R = Br



3, R = H
8, R = Br

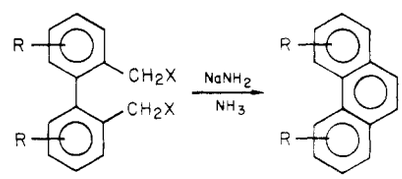


4, R = H; X_1 = CH_3; X_2 = H
9, R = Br; X_1 = CH_3; X_2 = H
5, R = H; X_1 = H; X_2 = CH_3
10, R = Br; X_1 = H; X_2 = CH_3

well as the strain energies[4,5] of **1**, **2**, and **3** as compared to nonstrained analogues by comparison of heats of combustion. For example, **1** is more strained than 2,7-dimethylphenanthrene (**4**) by 12.6 ± 1.5 kcal/mol,[5] and **3** is more strained than **2** by 7.2 ± 1.4 kcal/mol due mainly to the buttressing effect.[5]

In order to find out the difference in rates of reaction by an electrophilic substitution mechanism in strained and unstrained polycyclic aromatic hydrocarbons, we have set up a cooperative research program with Dr. Roger Taylor, University of Sussex, England. Our part was to synthesize 9-bromo derivatives of **1**–**5**, whereas Dr. Taylor was to replace the bromines by tritium and measure the rate of detritiation by protons in trifluoroacetic acid.[6]

The addition of bromine to the 9,10-bond of phenanthrene to yield 9,10-dibromo-9,10-dihydrophenanthrene followed by ready loss of hydrogen bromide to yield 9-bromophenanthrene[7a] suggested a simple way of preparing the desired 9-bromophenanthrenes, **6**–**10**. The syntheses of **1**–**4** were accomplished by the improved phenanthrene synthesis previously described,[8] the key step of which involves the cyclization of bis(chloromethyl)biphenyl-type intermediates, **11**, to the desired phenanthrenes by



11, X = Cl
12, X = Br

1–5

treatment with sodium amide in liquid ammonia. Interestingly, the cyclization occurred only when a stainless-steel stirrer was used. When a nylon-coated magnetic stirrer was used, the starting dichlorides were recovered, and the nylon stirrer was black. The syntheses of strained phenanthrenes by this method are superior to those involving phenyllithium ring closure of comparable bis(bromomethyl)biphenyls,[9] **12**. The synthesis of **5** was accomplished by cyclization of 4,4′-dimethylstilbene as described.[10a] *o*-Toluidine, 2,4-dimethylaniline, 2,3-dimethylaniline, and *p*-toluidine were converted via isatins, anthranilic acids, and diphenic acids into 2,2′-bis(hydroxymethyl)-6,6′-dimethylbiphenyl[9c] (**13**), 2,2′-bis(hydroxymethyl)-4,4′,6,6′-tetramethylbiphenyl[4] (**14**), 2,2′-bis(hydroxymethyl)-5,5′,6,6′-tetramethylbiphenyl (**15**), and 2,2′-bis(hydroxymethyl)-4,4′-dimethylbiphenyl (**16**), respectively, essentially as described[4] for the syntheses of **14** and **15**. These diols were converted via their mesylates into the corresponding dichloro compounds, **17**–**20** (all compounds of type **11**), which were cyclized in high yields to **1**–**4**.

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(2) Postdoctoral Research Associate.

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(6) Ansell, H. V.; Taylor, Roger *J. Org. Chem.*, following paper in this issue.

(7) (a) May, E. L.; Mosettig, E. *J. Org. Chem.* **1946**, *11*, 15. (b) Price, C. C. *J. Am. Chem. Soc.* **1936**, *58*, 1834 and references therein. However, the stereochemistry of dibromodihydrophenanthrene is not mentioned in any of the references cited. We are looking into this aspect.

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(9) (a) Hall, D. M.; Leslie, M. S.; Turner, E. *J. Chem. Soc.* **1950**, 711. (b) Hall, D. M.; Turner, E. *Ibid.* **1951**, 3072. (c) Wittig, G.; Zimmermann, H. *Chem. Ber.* **1953**, *86*, 629.

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The addition of bromine to the 9,10-bond of **1**, **2**, and **3** proceeded more rapidly (qualitatively) to yield the corresponding 9,10-dibromo-9,10-dihydrophenanthrenes than in the case of phenanthrene. This may be due to the noncoplanarity in these phenanthrenes forced by the 4,5-methyl groups. The fact that a competitive reaction involving the reaction of bromine with hexahelicene, a nonplanar aromatic hydrocarbon, and planar chrysene yielded mainly di- and tetrabromohexahelicene[11] provides an example of interest in this connection. Although many experiments were done with a variety (12) of reagents normally effective in eliminating HBr from a vicinal dibromide, the best experiments involved heating a solution of the dibromides in *tert*-butyl alcohol with potassium *tert*-butoxide. However, only low yields of pure 9-bromophenanthrenes, **6**, **7**, and **8**, were obtained. The chief side reaction was usually the loss of bromine to yield the starting phenanthrene. Evidently the rate of elimination of bromine from the strained dibromides is quite sensitive to reaction conditions. These results stand in marked contrast to the ready loss of HBr from phenanthrene dibromide,[7] 2,7-dimethylphenanthrene dibromide, and 3,6-dimethylphenanthrene dibromide to yield the corresponding 9-bromophenanthrenes in high yields on heating on a steam bath. The ready loss of bromine from 9,10-dibromo-9,10-dihydrophenanthrene on heating with pyridine to yield phenanthrene has been reported.[12]

Experimental Section[13]

6,6′-Dimethyl-2,2′-diphenic Acid. *o*-Toluidine was converted into 2-methylisonitrosoacetanilide in 65% yield essentially as described for similar anilines.[4] Ring closure to 7-methylisatin,[14] mp 269–271 °C, by HF in 94% yield followed by oxidation with alkaline hydrogen peroxide afforded 2-amino-3-methylbenzoic acid,[15] mp 169–171.5 °C, in 87% yield. Coupling to the diphenic acid,[16] mp 220–227 °C, was effected in 90% yield essentially as described for other aminobenzoic acids.[4]

4,4′-Dimethyl-2,2′-diphenic Acid. By a similar series of reactions *p*-toluidine was converted into the diphenic acid, mp 171–173 °C, in 40% overall yield.

Diethyl 6,6′-Dimethyl-2,2′-diphenate*. Esterification of 54 g of 6,6′-dimethyldiphenic acid, mp 220–227 °C, by refluxing a mixture with 60 mL of absolute ethanol, 350 mL of benzene, and 1 mL of H_2SO_4 into a column topped by a Dean–Stark trap for 5 days yielded 56.6 g (87%) of ester, bp 145–150 °C (0.05 torr), suitable for further work. The analytical sample melted at 53.7–54.7 °C.

Diethyl 5,5′,6,6′-Tetramethyl-2,2′-diphenate*. Esterification of 5,5′,6,6′-tetramethyl-2,2′-diphenic acid as above yielded 82% of the ester as an oil, bp 174–175 °C (0.05 torr). The analytical sample melted at 53.0–54.5 °C.

6,6′-Dimethyl-2,2′-bis(hydroxymethyl)biphenyl (13). Reduction of 56.7 g of diethyl 6,6′-dimethyl-2,2′-diphenate with 19.7 g of $LiAlH_4$ in 800 mL of ether at reflux for 16 h yielded 35.5 g (85%) of **13**,[9c] mp 121.5–122.5 °C, in two crops from benzene.

2,2′-Bis(chloromethyl)-6,6′-dimethylbiphenyl* (17). To a solution of 15.0 g of **13**[9c] in 200 mL of benzene were added 21.9 g of trimethylamine and 17.75 g of methanesulfonyl chloride with cooling. After 3.5 h at ambient temperature the triethylamine hydrochloride was filtered. The filtrate was washed with saturated KCl and was stirred overnight with a suspension of 100 g of KCl in 100 mL of saturated KCl containing 2 g of Aliquat-336.[17] After the usual workup there was obtained 17.43 g (95%) of **17** as a yellow oil, bp 148–150 °C (0.025 torr), suitable for further work. An analytical sample, mp 49.6–52.0 °C, prepared by repeated crystallization from ethanol, showed mass spectral peaks[18] at *m/e* 278, 280, and 282.

2,2′-Bis(chloromethyl)-4,4′,6,6′-tetramethylbiphenyl* (18). As described above, **14**[4] was converted into **18**, mp 116.5–118.0 °C, in 80% yield. Recrystallization from alcohol yielded the analytical sample, mp 117.9–119.1 °C.

2,2′-Bis(chloromethyl)-5,5′,6,6′-tetramethylbiphenyl* (19). As described above, **15**[4] was converted into **19**, mp 62–63 °C, in 88% yield of recrystallized product.

2,2′-Bis(chloromethyl)-4,4′-dimethylbiphenyl* (20). Conversion of **16**[16] into **20**, mp 75–75 °C, was accomplished in 86% yield essentially as described above. An analytical sample obtained by sublimination melted at 74.5–75.5 °C.

4,5-Dimethylphenanthrene[3,9c] **(1).** A suspension of sodium amide in 300 mL of liquid ammonia was prepared from 2.44 g of sodium in a 1-L three-necked flask equipped with a stainless-steel stirrer and a dry ice–isopropyl alcohol reflux condenser.[19] A solution of 11.8 g of **17** in 200 mL of toluene was added, and after most of the ammonia had evaporated overnight, 7 g of powdered NH_4Cl was added followed by 200 mL of water. Treatment in the usual way afforded 10.2 g of yellow oil. Crystallization from 20 mL of ethanol gave 5.9 g (68%) of **1**,[3] mp 76–77 °C. By chromatography of the material in the mother liquor over alumina, an additional 1.93 g (22%) of **1** (total 90%), mp 76.4–77.0 °C, was obtained.

2,7-Dimethylphenanthrene[20] **(4).** In a similar way, **4**, mp 101–102 °C, was prepared in 80% yield.

2,4,5,7-Tetramethylphenanthrene[4] **(2).** Similarly, **18** was converted into **2**, mp 110.6–111.8 °C, in 79% yield.

3,4,5,6-Tetramethylphenanthrene[4] **(3).** As above, **19** was converted into **3**, mp 69.5–70.5 °C, in 69% yield.

3,6-Dimethylphenanthrene (5). Photocyclization[10a] of 4,4′-*trans*-dimethylstilbene[10b] gave **5**, mp 142–143 °C, in 60% yield.

9-Bromo-4,5-dimethylphenanthrene* (6). A solution of 0.39 g of Br_2 in 5 mL of dry CCl_4 was added to a stirred solution at 0 °C of 0.50 g of **1** in 7 mL of CCl_4. After 1 h TLC showed no **1** present. The solvent was removed under vacuum to yield 0.87 g of almost white solid (theory, 0.89 g). Similar material was made several times. To a stirred solution under N_2 of 1.79 g of the dibromide in 40 mL of hot *tert*-butyl alcohol was added rapidly 8 mL of 4.84 mmol of potassium *tert*-butoxide in *tert*-butyl alcohol. A precipitate appeared soon, and the mixture was held at reflux overnight and poured into 40 mL of water. After the usual workup, 1.28 g of a mixture of **1** and **6** (roughly 1:2.9 by VPC) was obtained. By dry column chromatography[21] using a 2 × 26 in. flat-width nylon column containing 320 g of silica gel, activity grade III, and hexane there was obtained 650 mg (47%) of crude **6**. Recrystallization from ethanol afforded 360 mg (25%) of colorless **6**, mp 64.0–65.5 °C, the mass spectrum[18] showing peaks of about equal intensity at *m/e* 284 and 286. The NMR spectrum [($CDCl_3$, Me_4Si) δ 1.58 (perturbed s, 6, $ArCH_3$), 7.17–7.84 (m, 5, ArH), 8.00 (s, 1, $H_{pos\text{-}10}$), 8.12 (dd, $J_{H_1H_2}$ = 7 Hz, $J_{H_1H_3}$ = 2.5 Hz, 1, $H_{pos\text{-}1}$)] was consistent with that of 9-halogenated phenanthrenes.[22]

4,5-Dimethyl-9,10-phenanthrenequinone. To a solution of 0.14 g of chromic anhydride in 2 mL of acetic acid and 1 mL of water was added a warm solution of 100 mg of **6** in 4 mL of acetic acid. After 15 min the mixture was poured into water, and the

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(12) Ghiya, B. J.; Marathey, M. G. *Indian J. Chem.* **1963**, *1*, 448.
(13) All melting and boiling points are uncorrected. All new compounds marked with an asterisk gave elemental analysis (Chemalytics, Inc., Tempe, AZ) within ±0.3 units and NMR and mass spectra consistent with the formula. The term "worked up as usual" means that an ether–benzene solution of the reaction products was washed with dilute acid and/or alkali and saturated salt solution and filtered through anhydrous $MgSO_4$, and solvent was removed on a rotary evaporator.
(14) Melting point 266 °C: Giovannini, E.; Portman, P.; Johl, A.; Schnyder, K.; Knecht. B.; Zen-Ruffinen, H. P. *J. Prakt. Chem.* **1957**, *40*, 249.
(15) Melting point 172 °C: Panaotowicz, W. *J. Prakt. Chem.* **1886**, *33*, 57.
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(17) Aliquat-336, methyltricaprylylammonium chloride, supplied by the McKerson Co., Minneapolis, MN.
(18) We thank Mr. Richard Weisenberger for the mass spectral data.
(19) See: Newman, M. S. "An Advanced Organic Laboratory Course"; Macmillan: New York, 1972; pp 149–150.
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(22) Bartle, K. D.; Smith, J. A. S. *Spectrochim. Acta, Part A* **1967**, *23*, 1715

product, isolated as usual, was chromatographed over silica gel to yield 4,5-dimethyl-9,10-phenanthrenequinone, mp 161–163 °C, which was not depressed on mixing with an authentic sample,[23] mp 163–164 °C.

9-Bromo-2,4,5,7-tetramethylphenanthrene* (7). By a procedure similar to that described above **2** was converted in 38% yield into **7**: mp 110.5–112.0 °C; mass spectrum,[18] peaks at m/e 312 and 314 of about equal intensity; NMR ($CDCl_3$, Me_4Si) δ 2.55 (br s, 12, $ArCH_3$), 7.46 (m, 3 $H_{pos-3,6,8}$), 7.80 (s, 1, H_{pos-10}), 7.95 (br s, 1 H_{pos-1}). Oxidation to 2,4,5,7-tetramethyl-9,10-phenanthrenequinone was readily effected as above. The quinone was converted in high yield into the quinoxaline derivative,[23] which was identified by its melting point and mixture melting point with quinoxaline prepared from authentic **2**, mp 230.0–230.5 °C, by reaction with *o*-phenylenediamine.

9-Bromo-3,4,5,6-tetramethylphenanthrene* (8). As above, **3** was converted in 25% yield into **8**: mp 69.5–73.0 °C; mass spectral peaks at *m/e* 312 and 314 of about equal intensity;[18] NMR ($CDCl_3$, Me_4Si) δ 2.44 (m, 12, $ArCH_3$), 7.37–7.64 (m, 3, ArH), 7.77 (s, 1, H_{pos-10}), 8.07 (perturbed d, J = 8 Hz, 1, H_{pos-1}). Oxidation afforded 3,4,5,6-tetramethyl-9,10-phenanthrenequinone, identified as its quinoxaline derivative,[23] mp and mmp 178.5–180.0 °C.

9-Bromo-2,7-dimethylphenanthrene* (9) and 9-Bromo-3,6-dimethylphenanthrene* (10). These compounds were prepared by adding a slight excess of bromine to solutions of **4** and **5** in CCl_4 at 0 °C. After 2 h at ambient temperature the solvent was removed by rotary evaporation and the remainder heated on a steam bath for 30 min. After dry column chromatography on silica gel (activity grade III) using hexane, there was obtained 65% yield of **9** [mp 100–101 °C (analytical sample 102.5–103.5 °C); mass spectrum, peaks at *m/e* 284 and 286 of about equal intensity; NMR ($CDCl_3$, Me_4Si) δ 2.50 (s, 3, $ArCH_3$), 2.57 (s, 3, $ArCH_3$), 7.37–7.50 (m, 3, $H_{pos-2,6,8}$), 7.97 (s, 1, H_{pos-10}), 8.14 (br s, 1, H_{pos-1}), 8.44 and 8.57 (both br s, 2, $H_{pos-4,5}$)] and a 50% yield of **10** [mp 64–66 °C (analytical sample 66–67 °C); mass spectrum, peaks at *m/e* 284 and 286 of about equal intensity; NMR ($CDCl_3$, Me_4Si) δ 2.58 (br s, 6, $ArCH_3$), 7.27–7.74 (m, 3, $H_{pos-2,7,8}$), 7.97 (s, 1, H_{pos-10}), 8.25 (d, J = 5 Hz, 1, H_{pos-1}), 8.44 (br s, 2, $H_{pos-4,5}$)].

Registry No. **1**, 3674-69-9; **2**, 7396-38-5; **3**, 7343-06-8; **4**, 1576-69-8; **5**, 1576-67-6; **6**, 71871-02-8; **7**, 71871-03-9; **8**, 71871-04-0; **9**, 71871-05-1; **10**, 71871-06-2; **13**, 3594-91-0; **14**, 7411-15-6; **15**, 7343-08-0; **16**, 2941-81-3; **17**, 71871-07-3; **18**, 71871-08-4; **19**, 71871-09-5; **20**, 71871-10-8; 6,6′-dimethyl-2,2′-diphenic acid, 71871-11-9; *o*-toluidine, 95-53-4; 2-methylisonitrosoacetanilide, 1132-03-2; 7-methylisatin, 1127-59-9; 2-amino-3-methylbenzoic acid, 4389-45-1; 4,4′-dimethyl-2,2′-diphenic acid, 2941-79-9; *p*-toluidine, 106-49-0; diethyl 6,6′-dimethyl-2,2′-diphenate, 71871-12-0; diethyl 5,5′,6,6′-tetramethyl-2,2′-diphenate, 71871-13-1; 5,5′,6,6′-tetramethyl-2,2′-diphenic acid, 7343-07-9; sodium amide, 7782-92-5; 4,4′-*trans*-dimethylstilbene, 18869-29-9; 4,5-dimethyl-9,10-phenanthrenequinone, 17825-37-5; 2,4,5,7-tetramethyl-9,10-phenanthrenequinone, 17825-38-6; 2,4,5,7-tetramethyl-9,10-phenanthrenequinone quinoxaline derivative, 17825-39-7; *o*-phenylenediamine, 95-54-5; 3,4,5,6-tetramethyl-9,10-phenanthrenequinone, 17825-40-0; 3,4,5,6-tetramethyl-9,10-phenanthrenequinone quinoxaline derivative, 17825-41-1.

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Electrophilic Aromatic Substitution. 25.[1] Acid-Catalyzed Hydrogen Exchange of 9-Tritiated Polymethylphenanthrenes: Effect of Ring Distortion on Aromatic Reactivity and Substituent Effects[2]

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Rate coefficients have been measured for detritiation of the 9-position of some strained and unstrained polymethylphenanthrenes by anhydrous trifluoroacetic acid at 70 °C. These lead to partial rate factors (in parentheses) as follows: 2,7-dimethylphenanthrene (12 950), 3,6-dimethylphenanthrene (59 800), 3,4,5,6-tetramethylphenanthrene (356 000), 4,5-dimethylphenanthrene (38 300), 2,4,5,8-tetramethylphenanthrene (230 000). The reactivities of the former two (unstrained) compounds are in excellent agreement with those predicted from the effects of monomethyl substituents in phenanthrene. By contrast the reactivities of the latter two compounds are approximately threefold greater than predicted due to steric interaction of the 4- and 5-methyl substituents; these produce nonplanarity of the aromatic rings and hence increased reactivity through loss of ground-state resonance. The strained 3,4,5,6-tetramethylphenanthrene is by contrast *less* reactive than predicted, this being attributable to substantial reduction, through distortion, of the conjugative interaction between the 3- and 9-positions (which is normally much greater than the other substituent site interactions).

In this series of papers we have sought to determine, mainly through the use of an acid-catalyzed hydrogen-exchange reaction, quantitative reactivity data for aromatic hydrocarbons. Advantages of the reaction (among others) are freedom from steric hindrance and accuracy of the kinetic method. Recently we have determined[4] the effects of methyl substituents at each of the 1–8-positions upon the rate of exchange of tritium at the 9-position in trifluoroacetic acid at 70 °C. The data are shown in Figure 1 where the numbers indicate the activating effect of a methyl group at the appropriate position on the rate of tritium exchange at the 9-position. (Because 1,2-hydrogen shifts occur across the 9,10-bond of methylphenanthrenes during hydrogen exchange,[5] a significant error is introduced if the activating effect of a 6- and 8-methyl substituent in a symmetrically substituted polymethylphenanthrene is calculated from the raw data.[4] The corrected activating effects for these substituents are therefore given, in parentheses, in Figure 1. For the other substituents the errors are trivial so no correction need be applied.)

(1) Part 25. M. M. J. Le Guen, Y. El-din Shafig, and R. Taylor, *J. Chem. Soc., Perkin Trans. 2*, in press.

(2) This work was supported by NATO.

(3) Postdoctoral Research Associate.

(4) H. V. Ansell, P. J. Sheppard, C. F. Simpson, M. M. A. Stroud, and R. Taylor, *J. Chem. Soc., Perkin Trans. 2*, in press.

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