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Gram Scale Synthesis of Benzo[*ghi*]perylene and Coronene†

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Introduction

Polycyclic aromatic hydrocarbons (PAH) are formed during various kinds of combustion processes[1] and thus find their way into the environment.[2] This poses a threat for public health because of the toxic effect of some of the PAH.[3] Furthermore, PAH are used as organic conductors,[4] in solar cell research,[5] in polymer research,[6] and in many other fields of research.

Because most of the classical syntheses of PAH involve rather vigorous conditions such as pyrolysis of aromatic precursors,[7] in recent years milder methods giving higher yields and higher selectivities have been explored.[8] This usually resulted in multistep syntheses consisting of the addition of two-, three-, or four-carbon chains to the arene followed by cyclization and dehydrogenation. In this way the desired aromatic five- or six-membered rings may be formed. An example of ring formation in one step is the Diels−Alder reaction, in which the arene usually acts as the dienophile.[9] The only example known of the formation of a six-membered ring by the addition of a two-carbon unit is the Diels−Alder reaction of PAH containing a bay region with maleic anhydride or other electron-poor alkenes.[10,11] This usually gives low to moderate yields as does the subsequent decarboxylation.

In our group we focus on the synthesis of PAH via the use of anions of PAH, derived either via deprotonation or via reduction with alkali metals, because of the high selectivity of these anions. This has resulted in the synthesis of cyclopenta[*cd*]pyrene,[12] benzo[*e*]pyrene,[13] and other new PAH.[14] We now report the development of a new two-step procedure for the addition of an aromatic six-membered ring to an existing aromatic hydrocarbon. This is performed via addition and closure of a two-carbon chain over a bay region. This procedure has resulted in the very facile and high-yield, two-step synthesis of benzo[*ghi*]perylene (**4**) from perylene (**2**). Using the same procedure, **4** can be transformed into coronene (**6**) very efficiently. This reaction can easily be performed on a gram scale.

Results and Discussion

Because perylene (**2**) is not available in large quantities, we first optimized its synthesis. In most of the reported syntheses, 3,4,9,10-perylenetetracarboxylic dianhydride (**1**) is converted into perylene. Early workers have reported the synthesis of perylene from **1** in varying yields using intricate procedures such as passing superheated steam or conversion at high pressure.[15,16] We have now simplified and optimized the synthesis of perylene from **1**. This was achieved by simply mixing finely powdered barium hydroxide and **1** in a 4:1 ratio and heating this mixture in a Pyrex tube in a furnace at 80 °C for 2 h. Perylene could subsequently be harvested by sublimating it to the parts of the tube outside the oven at 380 °C, a process which lasts about 5 days. Using this very mild procedure, perylene is obtained in high purity in yields of up to 80% (Scheme 1).

Usually, the conversion of PAH into their anions with alkali metals is conducted in liquid ammonia,[17] which catalyzes the transfer of electrons. We have developed a new and facile way of generating the perylene dianion on a gram scale from neutral perylene and alkali metals. By ultrasonic vibration for 3 h of a suspension of perylene and sodium in dry degassed THF, we obtained a deep purple solution of $\mathbf{2}^{2-}$ in very high conversion (Scheme 2). When the same procedure was followed using THF-d_8 as a solvent in an NMR tube, again a deep purple solution was obtained with a ^{1}H NMR spectrum identical to that published for the perylene dianion ($\mathbf{2}^{2-}$).[18] The advantage of this method is that due to the absence of liquid ammonia, a larger scope of different reaction conditions can be applied to the anion and a greater number of electrophiles may be allowed to react with the anion.

When reduction is complete, the solution of the dianion can be brought to any desired temperature, after which it can be allowed to react with an electrophile. We found that reaction of $\mathbf{2}^{2-}$ with bromoacetaldehyde diethyl acetal at −65 °C and subsequent addition of iodine results in the formation of 1-peryleneacetaldehyde diethyl acetal (**3**) in 83% yield (Scheme 3), together with 10% of perylene and 5% of the 3-alkylated isomer. Perylene can be removed from **3** and 3-peryleneacetaldehyde diethyl acetal by means of column chromatography using toluene as the eluent. Via this method we can easily synthesize a 1-substituted perylene, contrary to Friedel−Crafts and related reactions, from which 3-substituted perylenes are obtained.[8]

Addition of sulfuric acid to a heterogeneous mixture of methanol, **3**, and 3-peryleneacetaldehyde diethyl acetal

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† Dedicated to Professor Hans-Dieter Scharf on the occasion of his 65th birthday.

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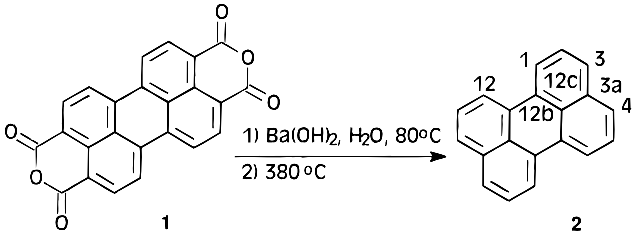
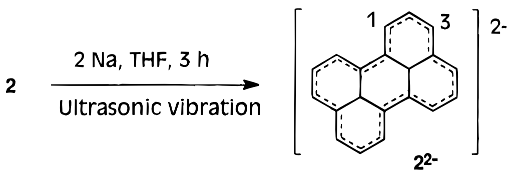
Scheme 1. Conversion of 3,4,9,10-Tetracarboxylic Dianhydride into Perylene



Scheme 2. Conversion of Perylene to its Dianion



and sonification for 1 h results in the formation of benzo[*ghi*]perylene (**4**) in 95% yield, after recrystallization from toluene, calculated from the amount of **3**. This high yield is probably related to the slight solubility of the starting compound (**3**) in the reaction mixture, causing very low concentrations of reactive intermediate. No products derived from the 3-substituted isomer were isolated. Using the same procedure with pure 3-peryleneacetaldehyde diethyl acetal results in the isolation of the starting material. Exposure of the same compound to a more acidic environment gives a black insoluble tar. With our method benzo[*ghi*]perylene can be synthesized from perylene in 79% overall yield. This exceeds the yield reported for the Diels–Alder reaction of perylene and maleic anhydride in boiling nitrobenzene (57% overall).[10] Other syntheses of **4** are usually based on photochemical cyclizations of compounds available only after multistep synthesis, which often yield **4** only on a milligram scale.[19]

Using the same procedure, compound **4** can also be converted to its dianion. After reaction at −65 °C of $\mathbf{4}^{2-}$ with bromoacetaldehyde diethyl acetal and oxidative workup with molecular iodine, we obtained 7-benzo[*ghi*]peryleneacetaldehyde diethyl acetal (**5**) in 67% yield together with 23% of benzo[*ghi*]perylene (**4**) and a small amount of 5-benzo[*ghi*]peryleneacetaldehyde diethyl acetal. Compound **5** and its 5-substituted isomer could be separated from **4** by means of column chromatography using toluene as the eluent. Via this method we can easily synthesize a 7-substituted benzo[*ghi*]perylene, contrary to Friedel–Crafts and related reactions, from which 5-substituted products are obtained.[20]

Ultrasonic vibration of a heterogeneous mixture of **5** (+ some 5-benzo[*ghi*]peryleneacetaldehyde diethyl acetal), methanol, and concentrated sulfuric acid resulted in the isolation of coronene (**6**) in 82% yield after workup and recrystallization from toluene. The yield calculated from **4** (55%) again exceeds the yield reported for the synthesis of coronene from **4** via the Diels–Alder reaction, which was reported to be 41%.[10] Other syntheses give coronene in very low yields.[21]

Reaction of $\mathbf{2}^{2-}$ with 1-bromo-2,2-dimethoxypropane and workup with molecular iodine result in a mixture of **7** and perylene. Separation of **7** and perylene by column chromatography over silica using toluene as the eluent and transformation of **7** with sulfuric acid results in the isolation of 1-methylbenzo[*ghi*]perylene (**8**) in 61% yield (Scheme 4). This is the first reported synthesis of 1-methylbenzo[*ghi*]perylene. Methyl-substituted benzo[*ghi*]perylenes have been reported to occur in the environment,[22] but the determination of the position of the methyl group has been hampered by the lack of reference material. Position 1 of **4** cannot be directly functionalized via other methods such as electrophilic substitution or conversion to its anion, which clearly proves the utility of the presently described annulation method.

Conclusion

A new method for the formation of perylene and benzo[*ghi*]perylene dianions on a gram scale has been developed. Due to the absence of liquid ammonia, the dianion solutions are very easy to handle and can be brought to a wide range of temperatures and allowed to react with various electrophiles. Using the specific reactivities of the dianions, we can easily synthesize 1-alkylated perylene **3** and 7-alkylated benzo[*ghi*]perylene **5** in good yield. Products **3** and **5** can be easily converted into benzo[*ghi*]perylene (**4**) and coronene (**6**), respectively, in high yield on a gram scale. To our knowledge these are the first multigram scale syntheses of **4** and **6**. Our preparation of 1-methylbenzo[*ghi*]perylene (**8**) is the first reported synthesis of this compound.

Experimental Section

Warning: *Many polycyclic aromatic hydrocarbons are potential mutagens and carcinogens. Guidelines for safe handling and disposal of chemical carcinogens have been published by Castegnaro and Sansone.*[23] *The use of toluene as a solvent for chromatography poses a potential safety hazard.*

3,4,9,10-Perylenetetracarboxylic dianhydride was purchased from Aldrich. Bromoacetaldehyde diethyl acetal and 1-bromo-2,2-dimethoxypropane were obtained from Janssen Chimica. $Ba(OH)_2{\cdot}8H_2O$ was obtained from Merck. THF from Acros was freshly distilled from $LiAlH_4$ directly before use. For ultrasonic vibration a Bransonic ultrasonic cleaner, Model 2200, was used. A Bruker WM 300 spectrometer was used to record 300 MHz ^1H, 75 MHz ^{13}C NMR, H–H and C–H COSY, and NOE-difference spectra. *J* values are given in Hz. Nuclear Overhauser enhancements are depicted as NOE: irradiated proton (protons which show correlation). Elemental analysis was performed by Mr. H. Eding of the Technical University of Eindhoven. Accurate masses in EI mode were measured with perfluorokerosine as the standard compound.

Acetals **3** and **5** are labile compounds, and it is therefore desirable to convert them into the stable products **4** and **6** immediately after column chromatography.

Perylene (2). A long glass tube was charged with a mixture of 48.0 g (152 mmol) of finely powdered $Ba(OH)_2{\cdot}8H_2O$, 10 mL of water, and 15.0 g of 3,4,9,10-perylenetetracarboxylic dianhydride (**1**, 38.2 mmol). Both ends of the tube were connected to bubblers to ensure a closed system. Heating this mixture in a furnace with temperature control for 2 h at 80 °C caused the color to change from red to yellow. Over a period of a few hours

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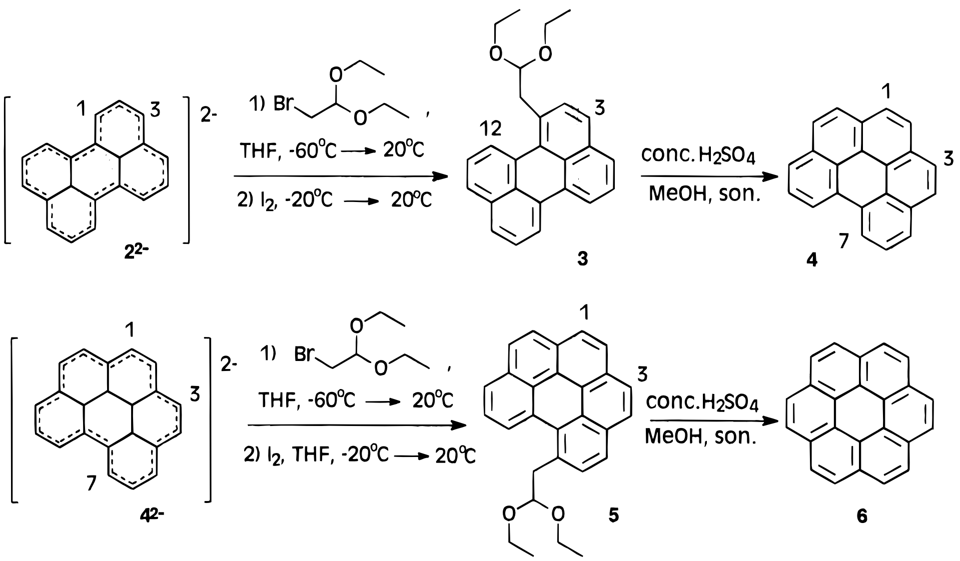
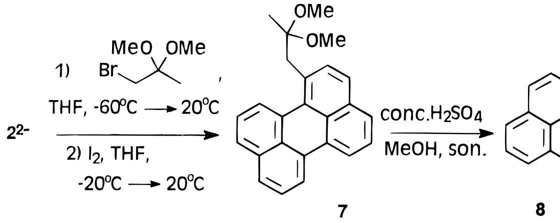
Scheme 3



Scheme 4



the water was removed at 120−200 °C under a stream of nitrogen. Pure perylene (8.10 g, 32.1 mmol, 84%) can be collected as golden plates of mp 272 °C (lit. mp 273−274 °C[24]) by sublimating it out of the oven at 380 °C, a process which lasts 5−7 days. The NMR spectrum of perylene was the same as that of Haigh et al.[25]

Generation of the Perylene Dianion and Reaction with Bromoacetaldehyde Diethyl Acetal. Into a dry 500 mL three-necked round-bottomed flask, 250 mL of THF was distilled and kept under an atmosphere of argon. Perylene (4.00 g, 15.9 mmol) was added together with 0.80 g (34.8 mmol, 2.2 equiv) of freshly cut sodium. Directly after the addition the flask was evacuated and sonificated for a period of 20−40 s. Argon was admitted, sonification was restarted, and usually the mixture turned blue. If the reaction fails to start, the evacuation−sonification procedure may have to be repeated a few times. After 3 h of sonification, during which the temperature was kept below 30 °C by the addition of ice, a deep purple solution was obtained, and sonification was stopped. The flask was then cooled to −60 °C and 2.39 mL (3.13 g, 15.9 mmol) of bromoacetaldehyde diethyl acetal was added with stirring, whereupon the color of the mixture turned to deep green. The mixture was allowed to warm up to rt and was stirred for 10 min. It was then cooled to −20 °C, 6.05 g (23.8 mmol) of iodine was added, and the temperature was again raised to rt. The color of the mixture then became brown-yellow. The reaction was quenched by adding 100 mL of a saturated aqueous sodium thiosulfate solution. Addition of diethyl ether, washing with water until neutral, washing with brine, drying over $MgSO_4$, and evaporation of the solvents in vacuo resulted in the isolation of the crude product as a highly viscous yellow oil. A first portion of 248 mg of perylene (6%) could be removed by addition of diethyl ether to the mixture and filtration over a glass funnel. Further purification of the product was achieved by column chromatography over silica gel. Starting with toluene as the eluent, 160 mg (4%) of perylene was isolated. After this, elution was continued with toluene−dichloromethane (1:1), which resulted in the isolation of 4.86 g (13.2 mmol, 83%) of 1-peryleneacetaldehyde diethyl acetal (**3**) and 286 mg (0.78 mmol, 5%) of 3-peryleneacetaldehyde diethyl acetal, both as very viscous yellow oils.

3: ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 1.23 (6H, t, J = 7.0), 3.56 (2H, d, J = 5.4), 3.54 (2H, dq, ^3J = 7.0, ^2J = 9.3), 3.70 (2H, dq, ^3J = 7.0, ^2J = 9.3), 4.98 (1H, t, J = 5.4), 7.50 (1H, t, J = 7.7), 7.52 (1H, t, J = 7.7), 7.53 (1H, t, J = 7.7), 7.58 (1H, d, J = 8.5), 7.67 (1H, d, J = 8.5), 7.70 (1H, t, J = 1.1, 7.7), 7.73 (1H d, J = 7.7), 7.75 (1H, dd, J = 1.3), 8.02 (1H, d, J = 7.7), 8.16 (2H, d, J = 7.7).

3-Peryleneacetaldehyde Diethyl Acetal: ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 1.18 (6H, t, J = 7.0), 3.33 (2H, d, J = 5.4), 3.42 (2H, dq, ^3J = 7.0, ^2J = 9.3), 3.71 (2H, dq, ^3J = 7.0, ^2J = 9.3), 4.80 (1H, t, J = 5.4), 7.73−7.56 (4H, m), 7.56 (2H, d, J = 7.7), 7.93 (1H, d, J = 8.5), 8.08−8.21 (4H, m).

Benzo[*ghi*]perylene (4). To a heterogeneous mixture of 4.29 g (11.6 mmol) of **3**, containing 5% of 3-peryleneacetaldehyde diethyl acetal, and 8 mL of methanol in a 100 mL round-bottomed flask, 4 mL of concentrated H_2SO_4 was added dropwise with stirring. After the first few drops, a yellow solid started to separate from the solution. After completion of the addition, the mixture was sonificated for half of an hour. Addition of 20 mL of water, filtration, rinsing of the filtrate with water and ether, and drying of the product resulted in 3.05 g (11.0 mmol, 95% based on **3**) of pure benzo[*ghi*]perylene (**4**). The ether fraction contained 209 mg of 3-peryleneacetaldehyde diethyl acetal. Benzo[*ghi*]perylene (**4**) was further purified by recrystallization from toluene, after which a yellow crystalline product was obtained of mp 268−272 °C (lit. mp 277−278.5 °C[26]). The NMR spectrum of **4** was the same as that of Matthews et al.[27]

Generation of the Benzo[*ghi*]perylene Dianion and Reaction with Bromoacetaldehyde Diethyl Acetal. According to the same procedure as described for perylene, 2.03 g (7.36 mmol) of benzo[*ghi*]perylene was transformed into its dianion. First a deep blue color was observed, which after 3 h had changed into a deep green color. Reaction with 1.11 mL (1.45 g, 7.36 mmol) of bromoacetaldehyde diethyl acetal resulted in a deep red color. Addition of 2.79 g (11.0 mmol) of I_2, workup, and purification according to the procedure described above resulted in 1.93 g of 7-benzo[*ghi*]peryleneacetaldehyde diethyl acetal (**5**, 4.93 mmol, 67%) and 114 mg of 5-benzo[*ghi*]peryleneacetaldehyde diethyl acetal (0.29 mmol, 4%), both as very viscous, slightly yellow oils. Furthermore, 386 mg of benzo[*ghi*]perylene (19%) was isolated.

5: ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 1.19 (6H, t, J = 7.0), 3.54 (2H, dq, ^3J = 7.0, ^2J = 9.3), 3.74 (2H, dq, ^3J = 7.0, ^2J = 9.3), 3.97 (2H, d, J = 5.2), 5.17 (1H, t, J = 5.2), 7.98 (1H, dd, J = 7.8, 8.0, H-9), 8.04−8.15 (6H, m), 8.19 (1H, dd, J = 0.9, 7.8,

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H-10), 8.31 (2H, s), 8.97 (1H, dd, $J = 0.9$, 8.0, H-8); NOE H-8 (CH_2, CH); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 15.4 (CH_3), 42.3 (O−CH_2), 62.2 (CH_2), 103.7 ($CH(OEt)_2$), 125.2 (C-9), 125.3, 125.4, 125.8, 125.9, 126.0, 126.4 (C-10), 126.6 (C-8), 126.7, 126.9, 127.1, 127.2, 128.2, 128.7, 129.0, 129.1, 129.6, 130.3, 130.5, 131.2, 131.7, 133.1. Exact mass calcd for $C_{28}H_{24}O_2$, 392.17772; found, 392.17763.

Coronene (6). According to the procedure described above for the preparation of **4**, 1.93 g of **5** (4.92 mmol) could be transformed into 1.21 g of coronene (4.03 mmol, 82%). The NMR spectrum of **6** was the same as that of Jonathan et al.[28]

1-Methylbenzo[*ghi*]perylene (8). Using the procedure described for the transformation of **2** into **3**, 1.00 g (3.96 mmol) of perylene was treated with 0.23 g (10.0 mmol) of sodium, 535 μL (725 mg, 3.96 mmol) of 1-bromo-2,2-dimethoxypropane, and 3.00 g (11.8 mmol) of iodine. In this case, after addition of the bromide the reaction mixture was stirred for 1 h at rt. Column chromatography over silica gel using toluene as the eluent yielded 250 mg (25%) of perylene, while the lower fraction was transformed into 1-methylbenzo[*ghi*]perylene (**8**) by ultrasonic vibration in 30 mL of methanol and 2 mL of H_2SO_4. Column chromatography over silica gel using toluene as the eluent yielded 701 mg (2.41 mmol, 61%) of pure **8**, which was isolated as a fluffy yellow solid with mp 209−210 °C (found: C, 95.20; H, 4.84. $C_{23}H_{14}$ requires C, 95.14; H, 4.86) after recrystallization from toluene.

8: ^1H NMR (300 MHz, $CDCl_3$) δ (ppm) 3.02 (3H, d, $J = 0.6$, CH_3), 7.94 (1H, dd, $J = 7.6$, 7.9), 7.96 (1H, dd, $J = 7.6$, 8.1), 8.00 (2H, s), 8.05 (1H, d, $J = 9.1$, H-11), 8.11 (1H, s, H-2), 8.13 (1H, dd, $J = 0.8$, 7.6), 8.14 (1H, dd, $J = 1.1$, 7.6), 8.22 (1H, d, $J = 9.1$, H-12), 8.93 (1H, dd, $J = 0.8$, 7.9), 8.94 (1H, dd, $J = 0.8$, 7.9); NOE CH_3 (H-2, H-12); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 132.2 (2-C), 131.8 (2-C), 131.2, 130.5, 130.0, 128.7, 128.2, 127.2, 127.0, 126.9, 126.4 (2-C), 126.3, 125.9, 125.7, 123.9, 123.4, 122.6, 120.5, 120.4, 20.2. Exact mass calcd for $C_{23}H_{14}$, 290.10962; found, 290.10955.

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