hydroiodide of exo isomer **3**, mp 208–212 °C) was shown by NMR to contain less than 3% of the endo isomer. Recrystallization (EtOAc) gave platelets: mp 216–220 °C; $[\alpha]^{20.0}_D$ +53 ± 0.5° (*c* 1.9, CH_3OH). Anal. Calcd for $C_{13}H_{24}NOI$: C, 46.30; H, 7.17; N, 4.15. Found: C, 46.26; H, 7.05; N, 4.24.

(+)-3-*endo*-((Dimethylamino)methyl)-*d*-camphor [(1*R*,3*R*,4*R*)-(+)-3-((Dimethylamino)methyl)camphor] (2). *d*-Camphor was treated with potassium hydride in THF followed by Eschenmoser's salt[9] by the method of Poulter and co-workers[5d] as described by Holy et al.[5c] The crude product (60% yield) was an 80:20 mixture of endo:exo isomers (free bases). Crystals were obtained from this oil at −15 °C in pentane; recrystallization gave the purified endo base (mp 34–36 °C, $[\alpha]^{20.0}_D$ +77.4 ± 1° (*c* 0.5, CH_3OH)), which retained about 3% of the exo isomer as shown by the NMR spectrum (1.95–2.10 ppm, area for $H_3 + H_4 + H_{5\beta}$ exo isomer, versus 2.58–2.68 ppm, area for H_3 endo isomer). This was converted to the hydrochloride: mp 211–212 °C dec; $[\alpha]^{20.0}_D$ +31 ± 1° (*c* 0.5, CH_3OH).

We have repeated the procedure used by Hine et al.[3] for synthesis of **2**. 3-(Hydroxymethylene)-*d*-camphor[11] (**9**) was treated with aqueous dimethylamine to give 3-((dimethylamino)-methylene)-*d*-camphor (bp 118–120 °C (3.5 Torr)) as a waxy solid (mp 50–52 °C) in 90% yield. This crude product in methanol was hydrogenated at 1–2 atm using 10% Pd–C catalyst in the presence of a slight excess of HCl added in small amounts as the reduction progressed. The product was a solid (mp 200–206 °C dec), which by NMR was an 80:20 mixture of endo:exo isomers. Several crystallizations from EtOH–EtOAc gave the purified endo hydrochloride isomer, (mp 211–212 °C dec). This hydrochloride was converted to the crystalline free base identical with that described above. The NMR spectra of the endo hydrochloride and endo free base **2** are given in Table I. The crude sample from this reaction corresponded in NMR and melting point to that of a corresponding sample supplied by Professor Hine.[12]

(1*R*,2*R*,3*S*,4*R*)-3-((Dimethylamino)methyl)isoborneol Hydrochloride (10·HCl). *exo*-3-((Dimethylamino)methyl)-*d*-camphor (**3**, 2.6 g, 12 mmol) in anhydrous ether (25 mL) was added slowly to an ether solution of $LiAlH_4$ (12 mL of 1.0 M + 75 mL of ether) at 20 °C. After 12 h, saturated NH_4Cl solution (2 mL) was added and the mixture filtered. The combined ether layer and extracts of the salts were extracted with 10% HCl. The acid extracts were made basic and extracted with ether; these extracts were dried ($MgSO_4$) and treated with dry HCl gas to give the HCl salt (**10**·HCl, 2.14 g, 72% yield), which was recrystallized from acetone: mp 259–61 °C dec; $[\alpha]_D^{27}$ +57.9° (*c* 0.88, MeOH). Anal. Calcd for $C_{13}H_{26}NOCl$: C, 63.01; H, 10.57; N, 5.65. Found: C, 62.74; H, 10.47; N, 5.49.

This salt was converted to the free base with dilute ammonia to give a clear oil. Anal. Calcd for $C_{13}H_{25}NO$: C, 73.88; H, 11.92; N, 6.63. Found: C, 73.69; H, 11.90; N, 6.51.

(1*R*,2*R*,3*S*,4*R*)-2-Benzyl-3-((dimethylamino)methyl)isoborneol (11). To an ether solution (25 mL) of Grignard reagent prepared from benzyl chloride (1.05 g, 8.3 mmol) was added *exo*-3-((dimethylamino)methyl)-*d*-camphor (**3**, 1.45 g, 6.9 mmol) in ether (75 mL). After 24 h at 20 °C the mixture was hydrolyzed (saturated NH_4Cl) and worked up, and the crude product (containing some unreacted **3**) was crystallized (EtOAc) to give 2-benzyl-3-((dimethylamino)methyl)isoborneol (**11**) hydrochloride: 0.80 g, 47% yield; mp 205–206 °C; $[\alpha]^{29}_D$ −7.55° (*c* 0.53, CH_3OH); NMR, Table I. This was converted to the free base to give a clear oil: 0.59 g, 88% yield. Anal. Calcd for $C_{20}H_{31}NO$: C, 79.68; H, 10.36; N, 4.64. Found: C, 79.76; H, 10.52; N, 4.57.

Equilibration of *endo*- and *exo*-3-((Dimethylamino)-methyl)-*d*-camphor. A purified sample of exo isomer **3** (free base) was dissolved in a 2% sodium methoxide solution in methanol at 20 °C. Aliquots were removed at intervals, the 3-((dimethylamino)methyl)-*d*-camphor was recovered as the free base, and the ratio of endo:exo isomers was determined by comparison of the integration of signals at 1.95–2.10 ppm, representing $H_3 + H_4 + H_{\beta 5}$ of the exo isomer, versus those at 2.14–2.20 ppm, representing H_4, or 2.58–2.68 ppm for H_3 of the endo isomer. Within 48 h the exo isomer was converted to a constant mixture of approximately 80:20 endo:exo. Starting with the pure endo isomer, the same 80:20 exo ratio was attained in 24 h.

Asymmetric Reductions with Lithium Aluminum Alkoxy Hydride Derived Reagent[1] from 10 and 11 (Table III). The asymmetric reductions were performed in an oven-dried 4-dram vial equipped with a serum stopper and a magnetic stirring bar. A dry argon atmosphere was maintained through a syringe needle inlet and outlet through a Drierite drying tube. A standard $LiAlH_4$ solution (about 0.2 mL, 0.2 mmol, of 1.0 M solution) was introduced via syringe. To this solution at 0 °C was introduced via syringe an ether solution of **10** or **11** as the free base. Approximately 1 mL containing a known molar equivalent of the carbinol amine **10** or **11** was introduced over a 30-s interval. In these cases a clear solution resulted. After 6.5 min an ether solution of acetophenone (0.8 molar equivalent to the $LiAlH_4$ at 0 °C) was injected, and the reaction mixture was stirred at 0 °C for 3 h. The reaction was hydrolyzed (2 mL, 1 N HCl) and extracted (ether). The ether extracts were dried ($MgSO_4$) and concentrated to give a colorless oil, which was weighed and then analyzed and preparatively purified by gas–liquid chromatography (GLC). The optical rotation of the GLC-purified methylphenylcarbinol was taken by using $[\alpha]^{20.0}_D$ 43.7° (*c* 1, CH_3OH) as the value for enantiomerically pure methylphenylcarbinol. The reliability of this determination was verified by use of the α-methoxy-α-(trifluoromethyl)phenylacetyl chloride (MTPA-Cl) reagent.[17] The results are summarized in Table III.

Acknowledgment. We gratefully acknowledge partial support of these studies by the National Institutes of Health (Grant GM 19554). We thank the National Science Foundation for an Instrument Grant (Grant CHE 1-09064) for the Varian FT400 instrument used in these studies. We also thank Dr. Lois Durham for helpful discussions on interpretation of the NMR data and Professor Jack Hine for generous cooperation.

Registry No. **1**, 464-49-3; **2**, 33162-70-8; **2**·HCl, 33162-71-9; **2**·HI, 114027-32-6; **3**, 113890-52-1; **3**·HCl, 27058-64-6; **3**·HI, 113972-49-9; **6**, 10293-06-8; **7**, 70982-26-2; **8a**, 30354-18-8; **8b**, 33797-51-2; **9**, 14681-31-3; **10**, 113890-53-2; **10**·HCl, 113972-50-2; **11**, 113812-33-2; **11**·HCl, 113812-34-3; $PhCOCH_3$, 98-86-2; (*R*)-$PhCHOHCH_3$, 1517-69-7; (*S*)-$PhCHOHCH_3$, 1445-91-6; 3-((dimethylamino)methylene)-*d*-camphor, 113890-54-3.

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Synthesis of Cyclopenta[*cd*]pyrene and Its Benzannelated Derivative Naphtho[1,2,3-*mno*]acephenanthrylene

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Received October 16, 1987

Characterization of cyclopenta[*cd*]pyrene as an important component of kerosene and gasoline soots[1-3] and its identification as a potent mutagen[4] and cell-transforming agent[5,6] have evoked considerable interest in peripherally fused cyclopenta-polycyclic aromatic hydrocarbons (cyclopenta-PAH) as potential environmental carcinogens. Cyclopenta[*cd*]pyrene is distinguished from most other

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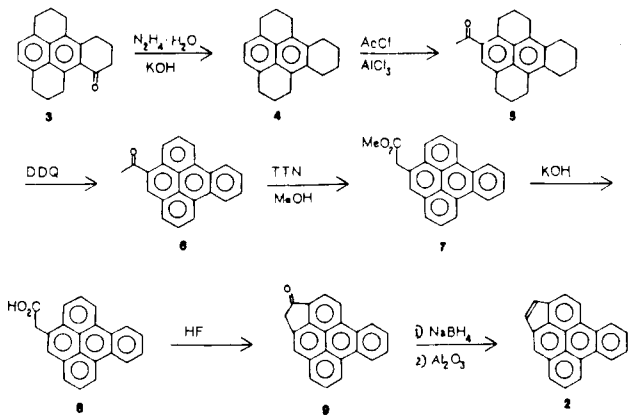
0022-3263/88/1953-2620$01.50/0

Scheme I



known PAH carcinogens by lack of the bay region feature, which has been regarded as a prerequisite for biological activity via metabolic conversion to a diol epoxide.[7] The evident need for development of a basic understanding of the relationship between molecular structure and biological activity calls for continued studies on cyclopenta[*cd*]pyrene and other peripherally fused cyclopenta systems. In pursuit of this objective, we have sought high-yield synthetic sequences that could be applied to generate a variety of peripherally fused cyclopenta systems. One sequence of general use involves formation of the cyclopenta ring by cyclodehydration of an arylacetic acid derived from the appropriate acetylarene. The methods currently available for transformation of acetylarenes to arylacetic acids are either cumbersome or variable in yield. We wish to report a simple and convenient procedure involving a thallium trinitrate (TTN) oxidative rearrangement that has been used to synthesize two cyclopenta-fused systems of interest, cyclopenta[*cd*]pyrene (**1**) and a benzannelated derivative, naphtho[1,2,3-*mno*]acephenanthrylene (**2**).

TTN is a well-established reagent[8,9] for the rapid, selective, room temperature oxidation of a variety of unsaturated organic substrates in high yield. Acetophenones have been smoothly converted into methyl phenylacetates, and in the single example of oxidation of a PAH system,[10] methyl 1-pyrenylacetate was obtained from 1-acetylpyrene in 95% yield. We applied the TTN procedure to 4-acetylpyrene[11] in a route to cyclopenta[*cd*]pyrene (starting with commerically available hexahydropyrene) via 4-pyrenylacetic acid, which is an intermediate in six of seven published syntheses.[12-18] To overcome the low solubility of PAH in the originally described methanol medium,[8] a 2:1 methanol–methylene chloride mixture was employed. The progress of the oxidation of 4-acetylpyrene[19] to the ester product could be conveniently monitored by observing the development of the sharp arylacetate bands in the UV–visible spectrum. Hydrolysis[20] to the arylacetic acid and conversion to cyclopenta[*cd*]pyrene[12] were accomplished by published methods with an overall yield of 42% from hexahydropyrene.

Naphtho[1,2,3-*mno*]acephenanthrylene (**2**), a benzannelated derivative of cyclopenta[*cd*]pyrene not described in the literature, is of interest in biological activity studies because it contains both the cyclopenta-fused ring and bay region features.[21] This compound may be conveniently synthesized from commercially available 1,2,3,6,7,8,11,12-octahydrobenzo[*e*]pyrene-9(10*H*)-one (**3**) by a scheme involving the TTN oxidation of 4-acetylbenzo[*e*]pyrene (**6**) as a key step (Scheme I). Wolff–Kishner reduction of ketone **3** afforded hydrocarbon **4**,[22] which was acetylated with acetyl chloride and $AlCl_3$ to 4-acetyl derivative **5**. Aromatization of **5** with 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ) to 4-acetylbenzo[*e*]pyrene (**6**), followed by TTN oxidation and basic hydrolysis of the resulting ester, gave 4-benzo[*e*]pyrenylacetic acid (**8**) with an overall 48% yield from **3**. A cyclodehydration–reduction–dehydration sequence[20] gave the target cyclopenta-PAH **2** in 32% overall yield from octahydrobenzo[*e*]pyrene. Composition of **2** was confirmed by elemental analysis and accurate mass measurement of the molecular ion. As in the case of cyclopenta[*cd*]pyrene[12,17] and other cyclopenta-PAH,[20] the ^1H NMR spectrum of **2** is characterized by the AX quartet of the etheno bridge protons (H_3,H_4) that appears upfield from the aromatic region. Four low-field signals from the bay region protons provide additional confirmation of the assigned structure. A doublet centered at 8.74 ppm can be unequivocally assigned to H_1 and a broad doublet at 8.95 ppm to H_8. Signals at 8.88 and 8.80 ppm, which appear as multiplets by virtue of long-range intraring coupling must arise from H_9 and H_{12}, but cannot be more specifically assigned. In accord with behavior of other cyclopenta-PAH,[12,20] **2** does not fluoresce under long-wavelength (360 nm) UV light.

Experimental Section

Physical Data. ^1H NMR spectra were obtained at 400 MHz on a Varian XL-400 spectrometer. Mass spectra were recorded on a VG 7070F Micromass mass spectrometer with an electron impact source at 70 eV. IR spectra were taken on a Beckman 4250 spectrophotometer. UV–vis spectra were recorded on a Perkin-Elmer 124 double-beam spectrophotometer. Melting points were determined on a Fisher-Johns melting point apparatus and are uncorrected. HPLC was performed with a Varian 5000 LC equipped with a Zorbax ODS 4.6 mm × 25 cm column and Perkin-Elmer LC-85B scanning UV detector. A starting eluant of 75% methanol in water was changed over to 100% methanol with a linear gradient of 1% min^{-1} and a flow rate of 0.8 mL min^{-1}. Elemental analyses were performed by Galbraith Laboratories, Knoxville, TN.

Materials. 1,2,3,6,7,8-Hexahydropyrene and 1,2,3,6,7,8,11,12-octahydrobenzo[*e*]pyrene-9(10*H*)-one (**3**) were purchased from Aldrich Chemical Co. and were used as received. 4-Acetylpyrene was synthesized from hexahydropyrene in two steps with an overall yield of 83% by a published procedure.[11]

Methyl 4-Pyrenylacetate. 4-Acetylpyrene (2.44 g, 10 mmol) was added to a solution of thallium trinitrate trihydrate (4.44 g, 10 mmol) in methanol (40 mL) and methylene chloride (20 mL)

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containing perchloric acid (70%, 10 mL). After 3 h of stirring at room temperature, the precipitated thallium(I) nitrate was removed by filtration, and the filtrate was diluted with water (100 mL). The organic phase was separated, the aqueous layer was extracted with methylene chloride (50 mL), and the combined organic phases were washed with water (100 mL), dried (Na_2SO_4), and evaporated. The resulting ester was purified by chromatography on alumina with benzene eluant; collection of the blue fluorescent fraction afforded ester: 2.20 g (80%); mp 130–131 °C (methanol) (lit.[11] mp 130.6–131.8 °C); ^{1}H NMR (400 MHz, chloroform-*d*) 3.65 (s, 3 H, ester CH_3), 4.25 (s, 2 H, benzylic CH_2), 7.90–8.35 ppm (m, 9 H, aromatic H); IR (KBr) 1740 cm^{-1} (C=O).

1,2,3,6,7,8,9,10,11,12-Decahydrobenzo[*e*]pyrene (4). A mixture of ketone **3** (1.0 g, 3.62 mmol), diethylene glycol (50 mL), hydrazine monohydrate (1.0 mL), and KOH (1.0 g) was refluxed for 6 h, cooled to room temperature, and poured into an excess of water. The product was extracted into methylene chloride (2 × 50 mL), washed with water (100 mL), and dried (Na_2SO_4), and the solvent was evaporated. The product was purified by chromatography on silica with hexane as eluant to give **4**: 770 mg (81%); mp 192 °C (lit.[22] mp 192–193 °C).

4-Acetyl-1,2,3,6,7,8,9,10,11,12-decahydrobenzo[*e*]pyrene (5). To a solution of decahydrobenzo[*e*]pyrene (**4**) (720 mg, 2.75 mmol) and acetyl chloride (0.21 mL, 2.94 mmol) in dry benzene (20 mL) was added $AlCl_3$ (463 mg, 3.45 mmol) in portions with stirring at room temperature. After stirring for 4 h, the reaction mixture was poured into ice–hydrochloride acid, the organic layer was separated, and the aqueous layer was extracted with additional benzene (100 mL). The organic phases were combined, washed with sodium bicarbonate solution (5%, 100 mL) and water (100 mL), and dried (Na_2SO_4). Following evaporation of the solvent, the product was purified by chromatography on alumina with benzene eluant to give ketone **5**: 710 mg (85%); mp 150–151 °C (hexane); ^{1}H NMR (400 MHz, CD_2Cl_2) 1.83 (m, 4 H, $H_{10,11}$), 1.96 (quintet, 2 H, J = 6.15 Hz, $H_{2\text{ or }7}$), 2.02 (quintet, 2 H, J = 6.15 Hz, $H_{7\text{ or }2}$), 2.59 (s, 3 H, $COCH_3$), 2.79 (m, 4 H, $H_{9,12}$), 2.89 (two t, 4 H, J = 6.0 Hz, $H_{1,8}$), 2.98 (t, 2 H, J = 6.15 Hz, H_6), 3.18 (t, 2 H, J = 6.15 Hz, H_3), 7.24 ppm (s, 1 H, aromatic H_5); IR (KBr) 1675 cm^{-1} (C=O); UV (CH_2Cl_2) [λ_{max}, nm ($\epsilon \times 10^{-4}$)] 360 (0.34), 319 (0.49), 309 (0.58), 299 (0.49), 269 (2.95), 265 (2.91), 233 (1.83). Anal. Calcd for $C_{22}H_{24}O$: C, 86.84; H, 7.89. Found: C, 83.96; H, 8.01.

4-Acetylbenzo[*e*]pyrene (6). A solution of **5** (684 mg, 2.25 mmol) and DDQ (2.80 g, 12.33 mmol) in dry benzene (300 mL) was refluxed under nitrogen for 12 h. Following filtration of the cooled solution, the filtrate was concentrated to 100 mL and passed through an alumina column, and the blue fluorescent fraction was collected to yield light yellow crystals of ketone **6**: 590 mg (90%); mp 214–215 °C (methanol); ^{1}H NMR (400 MHz, CD_2Cl_2) 2.90 (s, 3 H, $COCH_3$), 7.73 (m, 2 H, $H_{10,11}$), 8.05 (t, 1 H, J = 8.0 Hz, $H_{2\text{ or }7}$), 8.08 (t, 1 H, J = 8.0 Hz, $H_{7\text{ or }2}$), 8.22 (br d, 1 H, J = 7.0 Hz, H_6), 8.49 (s, 1 H, H_5), 8.82 (m, 2 H, $H_{9,12}$), 8.92–9.02 ppm (three d, 3 H, J = 8.0 Hz, $H_{1,3,8}$); IR (KBr) 1670 cm^{-1} (C=O); UV (CH_2Cl_2) [λ_{max}, nm ($\epsilon \times 10^{-4}$)] 336 (1.26), 321 (1.34), 293 (2.41), 281 (2.46), 269 (3.24), 262 (3.13). Anal. Calcd for $C_{22}H_{14}O$: C, 89.80; H, 4.76. Found: C, 89.81; H, 4.81.

Methyl 4-Benzo[*e*]pyrenylacetate (7). Ketone **6** (530 mg, 1.80 mmol) was treated by the same procedure described above for 4-acetylpyrene. The crude ester was chromatographed on alumina with benzene eluant, and collection of the intense fluorescent fraction afforded ester **7**: 479 mg (82%); mp 207–208 °C (methanol); ^{1}H NMR (400 MHz, $CDCl_3$) 3.69 (s, 3 H, ester CH_3), 4.25 (s, 2 H, CH_2CO_2), 7.72 (m, 2 H, $H_{10,11}$), 7.97 (s, 1 H, H_5), 8.00 (t, 1 H, J = 7.0 Hz, $H_{2\text{ or }7}$), 8.05 (t, 1 H, J = 7.0 Hz, $H_{7\text{ or }2}$), 8.12 (br d, 1 H, J = 8.0 Hz, $H_{3\text{ or }6}$), 8.28 (br d, 1 H, J = 8.0 Hz, $H_{6\text{ or }3}$), 8.82 (m, 2 H, $H_{9,12}$), 8.86 (br d, 1 H, J = 8.0 Hz, $H_{1\text{ or }8}$), 8.92 ppm (br d, 1 H, J = 8.0 Hz, $H_{8\text{ or }1}$); IR (KBr) 1740 cm^{-1} (C=O). Anal. Calcd for $C_{23}H_{16}O_2$: C, 85.19; H, 4.94. Found: C, 85.19; H, 4.87.

4-Benzo[*e*]pyrenylacetic Acid (8). Hydrolysis of ester **7** (450 mg, 1.39 mmol), carried out[20] in aqueous KOH (20%, 4 mL) and methanol (15 mL), gave acid **8**: 410 mg (95%); mp 231–232 °C dec (chlorobenzene); IR (KBr) 1703 cm^{-1} (C=O). Anal. Calcd for $C_{22}H_{14}O_2$: C, 85.16; H, 4.52. Found: C, 84.99; H, 4.51.

Naphtho[1,2,3-*mno*]acephenanthrylen-3(4*H*)-one (9). Acid **8** (390 mg, 1.26 mmol) was stirred in anhydrous hydrofluoric acid for 15 h at room temperature. Following standard workup,[15] the crude product was chromatographed on alumina with chloroform eluant. Collection of the yellow band with intense blue fluorescence furnished pure cyclic ketone **9**: 294 mg (80%); mp 212–213 °C (CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) 3.86 (s, 2 H, $COCH_2$), 7.79–7.84 (m, 2 H, $H_{10,11}$), 7.82 (s, 1 H, H_5), 8.04 (t, 1 H, J = 7.76, H_7), 8.16 (br d, 1 H, J = 7.16 Hz, H_6), 8.22 (d, 1 H, J = 8.09 Hz, H_2), 8.74 (m, 2 H, $H_{9,12}$), 8.81 (br d, 1 H, J = 8.34 Hz, H_8), 8.85 ppm (d, 1 H, J = 8.09 Hz, H_1); IR (KBr) 1705 cm^{-1} (C=O); UV (CH_2Cl_2) [λ_{max}, nm ($\epsilon \times 10^{-4}$)] 405 (0.79), 382 (0.65), 358 (1.56), 342 (1.29), 322 (0.94), 296 (2.91), 275 (2.35). Anal. Calcd for $C_{22}H_{12}O$: C, 90.41; H, 4.11. Found: C, 89.92; H, 4.04.

3-Hydroxy-3,4-dihydronaphtho[1,2,3-*mno*]acephenanthrylene (10). Sodium borohydride (100 mg) was added in portions to a solution of ketone **9** (263 mg, 0.90 mmol) in THF (30 mL) and methanol (15 mL) and stirred at ambient temperature for 1 h. Following addition of distilled water (25 mL), the organic solvents were removed on a rotary evaporator. The precipitated colorless alcohol **10** (256 mg, 97%) was filtered, dried in vacuo, and used directly in the next step.

Naphtho[1,2,3-*mno*]acephenanthrylene (2). After dehydration[20] of alcohol **10** (240 mg, 0.82 mmol) by 2.5 g of alumina (neutral activity I) in dry benzene (150 mL), the crude PAH was chromatographed on alumina with elution by 1:2 benzene–hexane. Collection of the yellow-orange, nonfluorescent band afforded **2**: 191 mg (85%); mp 190–191 °C; ^{1}H NMR (400 MHz, acetone-d_6) 7.26 (d, 1 H, J = 5.13 Hz, etheno H_4), 7.42 (d, 1 H, J = 5.13 Hz, etheno H_3), 7.76 (m, 2 H, $H_{10,11}$), 8.06 (t, 1 H, J = 7.80 Hz, H_7), 8.12 (d, 1 H, J = 7.76 Hz, H_2), 8.36 (s, 1 H, H_5), 8.38 (br d, 1 H, J = 7.71 Hz, H_6), 8.74 (d, 1 H, J = 7.81 Hz, H_1), 8.80 (m, 1 H, $H_{9\text{ or }12}$), 8.88 (m, 1 H, $H_{12\text{ or }9}$), 8.95 ppm (br d, 1 H, J = 7.84 Hz, H_8); IR (KBr) 3030, 1615, 1535, 1475, 1360, 1337, 1235, 1110, 915, 885, 860, 825, 750, 710 cm^{-1}; UV (heptane) [λ_{max}, nm ($\epsilon \times 10^{-4}$)] 397 (0.81), 376 (0.78), 365 (1.21), 358 (1.16), 349 (1.52), 336 (1.11), 324 (1.82), 307 (sh, 1.97), 298 (2.30), 286 (2.27), 269 (sh, 2.05), 256 (4.04), 247 (4.17), 225 (3.99); accurate mass molecular ion 276.0932, calcd for $C_{22}H_{12}$ 276.0937; mass spectrum, m/e (relative intensity) 276 (100, M^+), 274 (17, M – H_2), 138 (22, M^{2+}), 137 (9, (M – H_2)$^{2+}$); HPLC retention time 33.45 min. Anal. Calcd for $C_{22}H_{12}$: C, 95.65; H, 4.35. Found: C, 95.46; H, 4.40.

Acknowledgment. This work was supported in part by USEPA Grant CR 811817. This research has been reviewed by the Health Effects Research Laboratory of the USEPA and approved for publication. It does not necessarily reflect the views of the Agency, and no official endorsement of any kind should be inferred.

Registry No. **2**, 113779-16-1; **3**, 68151-08-6; **4**, 92387-50-3; **5**, 113779-17-2; **6**, 113779-18-3; **7**, 113779-19-4; **8**, 113779-20-7; **9**, 113779-21-8; **10**, 113779-22-9; 4-acetylpyrene, 22245-47-2; acetyl chloride, 75-36-5.

Transformation of Cycloartanyl Acetate into B-Homo Triterpenoids

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Received October 16, 1987

About 15 years ago, Lawrie et al.[1] reported that oxidation of cycloartanyl acetate (**1**) with ozone yielded 7-oxocycloartanyl acetate (**3**) as the major product. On the basis

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0022-3263/88/1953-2622$01.50/0