^{1}H NMR δ 4.50 (s, 4 H), 7.30 (s, 10 H). Elution with ether gave benzyl alcohol.

Physical Properties of Product Ethers. The ether **2c** was a solid: mp 148–150 °C (from methanol); MS, *m/e* 362 (M^+); ^{1}H NMR δ 4.29 (d, *J* = 4.0 Hz, 1 H), 5.37 (d, *J* = 4.0 Hz, 1 H), 6.00 (s, 1 H). Anal. Calcd for $C_{27}H_{22}O$: C, 89.50; H, 6.10. Found: C, 88.65; H, 6.08. **3c**: mp 202–203 °C (from methanol); ^{1}H NMR δ 4.14 (d, *J* = 4.0 Hz, 1 H), 5.15 (d, *J* = 4.0 Hz, 1 H), 6.34 (s, 1 H). Anal. Calcd for $C_{27}H_{22}O$: C, 89.50; H, 6.10. Found: C, 88.81; H, 6.13. **2f**: mp 87–89 °C (from methanol); ^{1}H NMR δ 2.55–3.26 (m, 2 H), 3.66–4.66 (m, 2 H), 5.67 (s, 1 H), 6.65–7.38 (m, 9 H); IR 1490, 1450, 1280 cm^{-1}. **2g**: an oil; ^{1}H NMR δ 1.24–1.88 (m, 6 H), 3.30–3.56 (m, 1 H), 3.94–4.23 (m, 2 H), 7.20 (m, 5 H). **4a**: mp 115–116 °C (from methanol); MS, *m/e* 284 (M^+); ^{1}H NMR δ 6.26 (s, 1 H), 6.48 (s, 1 H), 7.08–7.80 (m, 14 H); IR 1630, 1490, 1450 cm^{-1}. **4b**: an oil; ^{1}H NMR δ 1.92 (s, 3 H), 5.67 (s, 1 H), 6.07 (s, 1 H), 6.88–7.80 (m, 9 H).

The physical properties of **2d**, **2e**, **3d**, **3e**, and **6c** have been described previously.[1a] The ethers **2b** and **3b** were not obtained in pure states.

Registry No. **1a**, 91859-82-4; **1b**, 93645-80-8; **1c**, 89619-51-2; **1d**, 89675-06-9; **1e**, 84810-15-1; **1f**, 84847-61-0; **1g**, 84810-14-0; **1h**, 84847-60-9; **1i**, 73258-06-7; **1j**, 93645-81-9; **1k**, 23888-15-5; **1l**, 72328-17-7; **1m**, 72328-16-6; **2a**, 93645-82-0; **2b**, 93645-83-1; **2c**, 93645-84-2; **2d**, 84810-30-0; **2e**, 84810-47-9; **2f**, 2292-59-3; **2g**, 4203-44-5; **2h**, 103-50-4; **2i**, 61103-84-2; **2j**, 112-58-3; **3a**, 93645-85-3; **3b**, 93645-86-4; **3c**, 93645-87-5; **3d**, 84810-36-6; **3e**, 84810-46-8; **4a**, 93645-88-6; **4b**, 93645-89-7; **4c**, 50431-53-3; **5a**, 53067-91-7; **5b**, 93645-90-0; **5c**, 50805-40-8; **5d**, 75519-83-4; **5e**, 84810-25-3; **6a**, 93645-91-1; **6b**, 93645-92-2; **6c**, 93645-93-3; **6d**, 93645-94-4; **6e**, 22440-32-0; **6g**, 1011-61-6; **7**, 37464-86-1; **8**, 100-51-6; **9**, 111-27-3; **10a**, 16204-37-8; **10b**, 93645-95-5; **11**, 100-52-7; **12**, 108-95-2; $AlCl_2H$, 13497-97-7; $LiAlH_4$, 16853-85-3; $AlCl_3$, 7446-70-0.

Aceanthrylene

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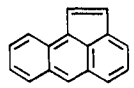
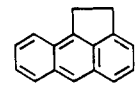
Received June 26, 1984

There is an abundance of reports dealing with the chemistry of acenaphthylene (**1**), but nothing is known about the properties of its benzo derivative aceanthrylene (**2**) which, according to *Chemical Abstracts* nomenclature,

1



2



3

is the parent compound of a hydrocarbon ring system.[1,2] As a nonalternant conjugated, peri-condensed polycyclic hydrocarbon, aceanthrylene has been of interest in the context of molecular orbital calculations,[3] but the compound itself, remarkably, has never been available or described.[4] By contrast, the synthesis of its dihydro derivative, aceanthrene (**3**), first attempted in 1911 by Liebermann and Zsuffa, was successfully carried out by Fieser and Peters in 1932 and subsequently improved by Bergmann and Ikan (1958).[5-7]

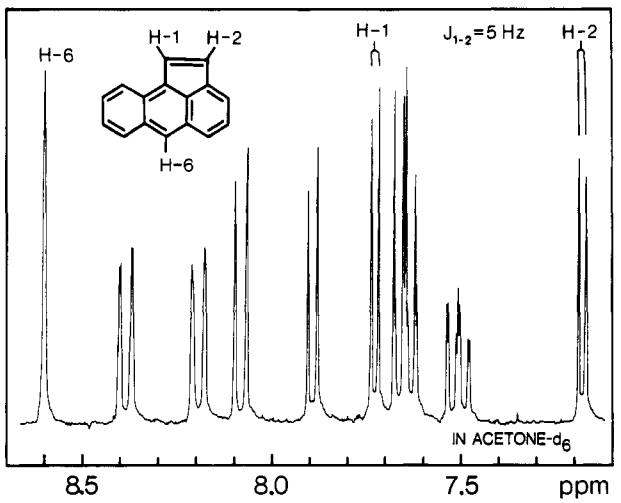
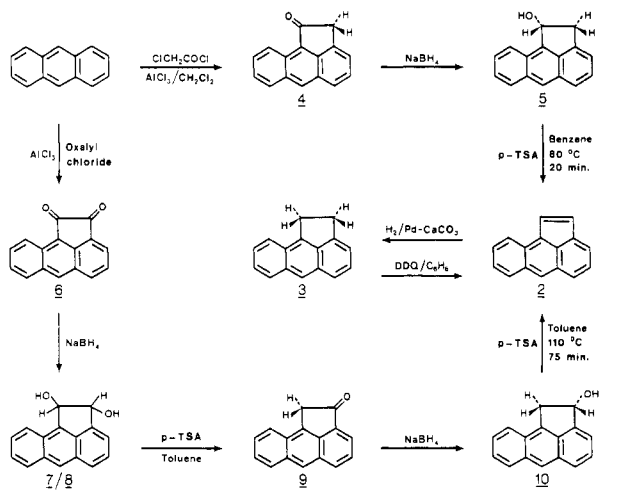


Figure 1. ^{1}H NMR spectrum (270 MHz) of aceanthrylene in acetone-d_6.

Scheme I



In conjunction with previous and current studies on the relationship between molecular geometry and photochemical properties of anthracenes,[8] we were enticed to prepare aceanthrylene. Its synthesis was accomplished by the following straightforward routes summarized in Scheme I.

Friedel–Crafts acylation of anthracene with chloroacetyl chloride in the presence of aluminum chloride (molar ratio

(1) A summary of references dealing with the chemistry of acenaphthylene can be found in *Beilstein* **1980**, *EIV 5*, 2138 and corresponding preceding volumes.

(2) Besides benzo[*a*]fluoranthene (cf. Ray, J. K.; Harvey, R. G. *J. Org. Chem.* **1982**, *47*, 3335), at least two examples of substituted aceanthrylenes are known, namely, the 1-methyl-2-chloromethyl derivative (Hauptmann, S.; Franke, L.; Dietrich, K.; Wild, G.; Schnitzker, M. *Z. Chem.* **1963**, , 147) and 1,2-dimethylaceanthrylene (Kikuchi, H.; Seki, S.; Yamamoto, G.; Mitsuhashi, T.; Nakamura, N.; Oki, M. *Bull. Chem. Soc. Jpn.* **1982**, *55*, 1514).

(3) (a) Zahradnik, R.; Michl, J.; Koutecky, J. *Collect. Czech. Chem. Commun.* **1964**, *29*, 1932. (b) DasGupta, A.; DasGupta, N. K. *Can. J. Chem.* **1976**, *54*, 3227.

(4) After this paper had been submitted, the synthesis of aceanthrylene from 2-aceanthrenol was reported: Plummer, B. F.; Al-Saigh, Z. Y.; Arfan, M. *J. Org. Chem.* **1984**, *49*, 2069. The melting point reported for 2-aceanthrenol (228–230 °C) differs significantly from that found in the present work (209–210 °C), but we have no reason to doubt the purity of our 2-aceanthrenol. The melting point of aceanthrylene reported previously is 95–96 °C, while we find a melting point of 103–104 °C. Moreover, aceanthrylene has been reported to exhibit anomalous fluorescence, i.e., second excited state emission (Plummer, B. F.; Al-Saigh, Z. Y.; Arfan, M. *Chem. Phys. Lett.* **1984**, *104*, 389). However, aceanthrylene of the present work was found to be nonfluorescent. Consequently, we believe aceanthrylene of the present work to be emission spectroscopically pure, and its electronic absorption spectrum as shown in Figure 2 to be correct.

(5) Liebermann, C.; Zsuffa, M. *Ber. Dtsch. Chem. Ges.* **1911**, *44*, 852.

(6) Fieser, L. F.; Peters, M. A. *J. Am. Chem. Soc.* **1932**, *54*, 4373.

(7) Bergmann, E. D.; Ikan, R. *J. Org. Chem.* **1958**, *23*, 907. For other references dealing with aceanthrene, see: *Beilstein* **1965**, *EIII 5*, 2235.

(8) Becker, H.-D. *Pure Appl. Chem.* **1982**, *54*, 1589. Becker, H.-D.; Andersson, K. *J. Org. Chem.* **1983**, *48*, 4542 and references cited therein.

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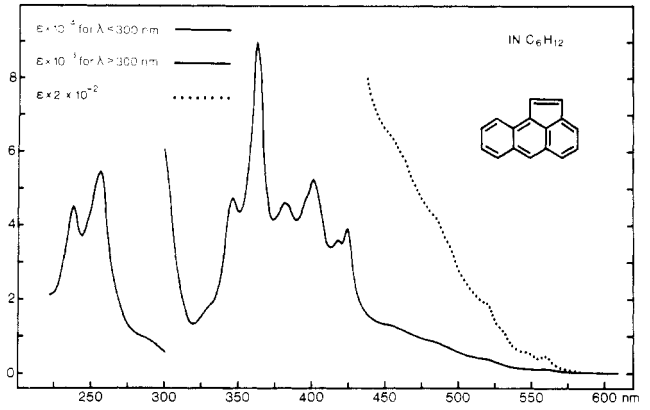

Figure 2. Electronic absorption spectrum of aceanthrylene in cyclohexane.

1:1:2) in methylene chloride gives 1-aceanthrenone[9] (**4**, 38% yield), which is converted into 1-aceanthrenol (**5**, 87% yield) by reduction with sodium borohydride. The elimination of water from 1-aceanthrenol to give aceanthrylene in 90–93% yield was accomplished by refluxing dilute solutions of **5** in benzene (220 mg/450 mL) with a catalytic amount (5 mg) of *p*-toluenesulfonic acid for 15–20 min under nitrogen.

A second, similar route to aceanthrylene involving the elimination of water from 2-aceanthrenol (**10**) was explored because the conditions for the dehydration of 1-aceanthrenol, namely, low concentration of acid catalyst and high dilution in order to avoid the formation of polymeric byproducts, were found to be of critical importance. 2-Aceanthrenol was conveniently obtained by the following reaction sequence (see Scheme I). Reduction of aceanthrene quinone[10] (**6**) with sodium borohydride gives cis and trans diols **7** and **8** which are separable by fractional crystallization. Interestingly, the dehydration of diols **7** and **8** with *p*-toluenesulfonic acid in refluxing toluene proceeds regioselectively and gives 2-aceanthrenone (**9**) in about 60% yield. Its conversion into 2-aceanthrenol is brought about by reduction with sodium borohydride. The dehydration of 2-aceanthrenol with *p*-toluenesulfonic acid in refluxing toluene is complete within 75 min and affords aceanthrylene in 82% yield.

Aceanthrylene is a scarlet crystalline substance melting between 103 and 104 °C. Its structure is supported by elemental analysis and by various spectroscopic data. In its mass spectrum, prominent peaks are found at M^+ (100%), M + 1 (46%), M − 1 (46%), M − 2 (43%), and at M^{2+} (58%). In its ^1H NMR spectrum (Figure 1), the protons of the peri-bridge are found in the "aromatic" region but are clearly distinguishable. Their chemical shifts at δ 7.18 and 7.73 suggest aceanthrylene to be an aromatic system which ^1H NMR spectroscopically is similar to acenaphthylene.[11] The electronic absorption spectrum of aceanthrylene (Figure 2) reveals that its red color is due to a broad, weakly structured absorption which is much lower in energy than is typical of a 1,9-disubstituted anthracene.

Different from its dihydro derivative **3** and most substituted anthracenes, aceanthrylene in solution at room temperature is non-fluorescent, and we have not yet found any photochemical reactivity which is typical of the anthracene chromophore.

The ground-state chemistry of aceanthrylene is that of a reactive olefin, as evidenced by its pronounced tendency to polymerize in the presence of acid. Moreover, aceanthrylene is smoothly hydrogenated in ethyl acetate over $Pd/CaCO_3$ to give aceanthrene which was isolated in 90% yield. Aceanthrylene is regenerated from aceanthrene by treatment with an equimolar amount of 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ)[12] in benzene at room temperature. Thus, if aceanthrene were an easily accessible compound, its dehydrogenation would be a most convenient way to aceanthrylene. However, by comparison with previously described routes to aceanthrene, its preparation by way of aceanthrylene as outlined in Scheme I may be regarded as a synthetically viable method.

Experimental Section

All solvents were reagent grade and were distilled before use. Benzene was dried by distillation over sodium and benzophenone. Melting points were determined on a hot-stage microscope and are uncorrected. Electronic absorption spectra were obtained on a Kontron Uvikon 810 spectrometer. ^1H NMR spectra were recorded on a Bruker 270 instrument, and chemical shifts are given in parts per million downfield from Me_4Si.

Elemental analyses were performed by NOVO Microanalytical Laboratory, Bagsvaerd, Denmark.

Aceanthrylene (2). A. From 1-Aceanthrenol. *p*-Toluenesulfonic acid monohydrate (5 mg) was added to a stirred, nitrogen-purged solution of 1-aceanthrenol (220 mg, 1 mmol) in benzene (450 mL), and the reaction mixture was refluxed under nitrogen for 20 min. The intensely red solution was then rapidly cooled to room temperature, stirred for 1 min with 3 g of basic aluminum oxide, and passed under pressure through a column (12 × 3 cm) of basic aluminum oxide in order to remove the acid catalyst and conceivable acidic byproducts (total volume of eluate about 800 mL). The solvent was then removed by vacuum evaporation, and the resulting red crystalline residue was sublimed in vacuo (0.05 mm; bath temperature 75 °C): yield 185 mg (91%) of red, flaky crystalline material; mp 103–104 °C; mass spectrum, *m/e* (relative intensity) 206 (46, M + 1), 202 (100, M^+), 201 (46, M − 1), 200 (43, M − 2), 199 (15, M − 3), 176 (23, M − 26), 175 (25, M − 27), 174 (31, M − 28), 150 (12, M − 52); M^+ found by high-resolution mass spectroscopy at *m/e* 202.0792, calcd for $C_{16}H_{10}$ *m/e* 202.0783. Anal. Calcd for $C_{16}H_{10}$: C, 95.02; H, 4.98. Found: C, 95.21; H, 4.93.

B. From 2-Aceanthrenol. *p*-Toluenesulfonic acid monohydrate (10 mg) was added to a warm solution of 2-aceanthrenol (220 mg, 1 mmol) in toluene (200 mL) under nitrogen, and the reaction mixture was refluxed for 75 min. Workup as described above gave 166 mg (82%) of aceanthrylene.

C. By Dehydrogenation of Aceanthrene. A solution of **3** (204 mg, 1 mmol) and DDQ (250 mg, 1.1 mmol) in benzene (50 mL) was stirred at room temperature for 1 h. Precipitated $DDQH_2$ was removed by filtration, and the filtrate was passed under pressure through a short column of basic aluminum oxide. Vacuum evaporation of solvent followed by vacuum sublimation as described above gave 117 mg (58%) of **2**. Its identity was established by ^1H NMR.

Aceanthrene (3). A solution of aceanthrylene (220 mg, 1 mmol) in ethyl acetate (100 mL) was hydrogenated over $Pd/CaCO_3$ (10%, 150 mg) under ambient conditions. Hydrogen uptake ceased within 5 min. Conventional workup followed by vacuum evaporation of solvent gave a yellow crystalline residue which was recrystallized from methanol by addition of water: yield 185 mg (90%) of yellow flaky crystals; mp 118–119 °C (in the literature, the melting point varies between 113 and 117 °C; cf. ref 7). The identity of **3** was verified by comparison with an authentic sample prepared according to Bergmann and Ikan.[7] ^1H NMR ($CDCl_3$): δ 8.17 (s, 1), 8.06–7.96 (m, 2), 7.71 (d, J = 8.5 Hz,

(9) Cf. German Patent D.R.P. 547644, **1930**, *Beilstein* **1968**, *EIII 7*, 2627. See also: Matsumoto, T.; Sato, M.; Hirayama, S. *Bull. Chem. Soc. Jpn.* **1974**, *47*, 358.

(10) Chang, S.-J.; Ravi Shankar, B. K.; Shechter, H. *J. Org. Chem.* **1982**, *47*, 4226.

(11) Minsky, A.; Meyer, A. Y.; Hafner, K.; Rabinovitz, M. *J. Am. Chem. Soc.* **1983**, *105*, 3975.

(12) For a review on dehydrogenations of hydroaromatic compounds by quinones, see: Becker, H.-D. In "The chemistry of quinonoid compounds"; Patai, S., Ed.; Wiley-Interscience: New York, 1974; p 335.

1), 7.48–7.40 (m, 3), 7.21 (d, J = 6.6 Hz, 1), 3.66 (center of AA'XX' system, 4 peri-bridge H).

1-Aceanthrenone (4). Aluminum chloride (8 g, 0.06 mol) was added over a period of 45 min to a solution of anthracene (5.35 g, 0.03 mol) and chloroacetyl chloride (2.4 mL, 0.03 mol) in methylene chloride at −5 to 0 °C. The dark colored reaction mixture was kept at room temperature for 24 h and then decomposed with ice–hydrochloric acid. The organic layer was subsequently washed with sodium bicarbonate and water and dried over $MgSO_4$. The dark colored solution thus obtained was treated twice with charcoal, and part of the solvent was removed by vacuum evaporation to give 300–400 mg of an insoluble yellow crystalline precipitate which consists of a mixture of disubstituted chloroacetylanthracenes (not further investigated). The filtrate was subjected to flash chromatography (SiO_2/CH_2Cl_2), and the yellow crystalline product (R_f 0.33) was first sublimed (110 °C (0.03 mm)) and then recrystallized from methylene chloride by addition of cyclohexane: yield 2.48 g (38%) of yellow crystals; mp 157–158 °C (lit.[9] mp 151–152 °C); IR (KBr) 1680 cm^{-1} (C=O); ^1H NMR ($CDCl_3$) δ 9.18 (d, J = 8.3 Hz, 1), 8.64 (s, 1), 8.14 (d, J = 7.9 Hz, 1), 7.91 (d, J = 8.6 Hz, 1), 7.77–7.71 (m, 1), 7.64–7.52 (m, 2), 7.42 (d, J = 6.7 Hz, 1), 3.90 (s, 2). Anal. Calcd for $C_{16}H_{10}O$: C, 88.05; H, 4.62. Found: C, 88.19; H, 4.56.

1-Aceanthrenol (5). Sodium borohydride (0.8 g) was added to a solution of 1-aceanthrenone (1 g, 4.6 mmol) in a mixture of methylene chloride (50 mL) and methanol (50 mL). The reaction mixture was refluxed for 15 min and then neutralized with aqueous acetic acid (50%). The methylene chloride was removed from the organic layer by vacuum evaporation, and the remaining solution was diluted with water (50 mL) to give a crystalline precipitate. It was removed by filtration and recrystallized from methylene chloride by adding hexane: yield 0.88 g (87%) of yellow, needle-shaped crystals; mp (dependent on the rate of heating) 175–185 °C (partly dec); ^1H NMR ($CDCl_3$) δ 8.36 ("d", J = 10.6 Hz, 1), 8.33 (s, 1), 8.08 ("d", J = 9.7 Hz, 1), 7.76 (d, J = 8.6 Hz, 1), 7.57–7.43 (m, 3), 7.26–7.23 (m, 1), 6.24 (dd, J = 7.8, 6.4 Hz, 1), 3.94 (dd, J = 18.0, 6.4 Hz, 1), 3.38 (d, J = 18.0 Hz, 1), 2.04 (d, J = 7.8 Hz, 1 OH). Anal. Calcd for $C_{16}H_{12}O$: C, 87.24; H, 5.49. Found: C, 87.04; H, 5.41.

Aceanthrenequinone (6) was prepared according to ref 10.

1,2-Aceanthrenediols (7/8). Sodium borohydride (2.32 g, 61 mmol) was added under nitrogen to a suspension of aceanthrenequinone (2.32 g, 10 mmol) in a mixture of methylene chloride (350 mL) and 95% ethanol (70 mL) placed in a separatory funnel. The reaction mixture was agitated by a gentle stream of nitrogen for 2 h until a light yellow solution had formed. The solution was then extracted once with 100 mL of water, and neutralization of this aqueous solution with acetic acid afforded 100 mg of virtually pure cis diol 7 (vide infra). The organic layer was washed 3 times with 100-mL portions of water whereupon part of the diol mixture precipitated from the methylene chloride solution. Partial removal of solvent by vacuum evaporation, followed by addition of hexane, gave 1.96 g of yellow crystalline cis/trans diol mixture. Separation into cis and trans isomers was accomplished by fractional crystallization from boiling ethanol in which the cis diol 7 (550 mg) is less soluble. It forms bright yellow needles, mp 239–242 °C. The yellow mother liquor was treated briefly with little sodium borohydride, and conventional workup afforded the trans diol 8 (1.10 g). It is rather soluble in acetone and can be precipitated with water. It forms almost colorless, fluffy needle-shaped crystals, mp 185–187 °C. Spectroscopic and analytical data of 7 and 8 as follow.

***cis*-1,2-Aceanthrenediol (7):** ^1H NMR ($CDCl_3$) δ 8.42 ("d", J = 8.1 Hz, 1), 8.38 (s, 1), 8.10 ("d", J = 8.1 Hz, 1), 7.91–7.88 (m, 1), 7.64–7.52 (m, 4), 6.01 (dd, J = 7.0, 6.0 Hz, 1), 5.65 (dd, J = 7.0, 6.0 Hz, 1), 2.84 (d, J = 7.0 Hz, 1 OH), 2.83 (d, J = 7.0 Hz, 1 OH). Anal. Calcd for $C_{16}H_{12}O_2$: C, 81.34; H, 5.12. Found: C, 81.16; H, 5.04.

***trans*-1,2-Aceanthrenediol (8):** ^1H NMR ($CDCl_3$) δ 8.39 (s, 1), 8.38 ("d", J = 7.3 Hz, 1), 8.10 ("d", J = 7.5 Hz, 1), 7.91 ("d", J = 8.3 Hz, 1), 7.59–7.49 (m, 4), 5.97 (d, J = 7.5 Hz, 1), 5.59 (d, J = 6.8 Hz, 1), 2.31 (d, J = 7.5 Hz, 1 OH), 2.24 (d, J = 6.8 Hz, 1 OH). Anal. Calcd for $C_{16}H_{12}O_2$: C, 81.34; H, 5.12. Found: C, 81.06; H, 5.01.

2-Aceanthrenone (9). A. From Cis Diol 7. *p*-Toluenesulfonic acid (15 mg) was added to a hot suspension of 7 (354 mg, 1.5 mmol) in toluene (15 mL), and the dark brown reaction mixture was refluxed for 1 h. Workup by extraction with sodium bicarbonate, washing, and drying, followed by flash chromatography on SiO_2/CH_2Cl_2 and vacuum sublimation (115 °C (0.04 mm)), gave 269 mg (82%) of yellow crystals, mp 167 °C. The substance may be recrystallized from methylene chloride by precipitation with hexane: ^1H NMR ($CDCl_3$, 6 mg/mL, concentration-dependent) δ 8.45 (s, 1), 8.24 (d, J = 8.4 Hz, 1), 8.12–8.09 (m, 1), 8.00–7.97 (m, 2), 7.75–7.69 (m, 1), 7.58–7.53 (m, 2), 4.16 (d, J = 0.8 Hz, 2). Irradiation of the multiplet centered at δ 7.98 changes the doublet at δ 4.16 into a singlet: IR (KBr) 1700 cm^{-1}. Anal. Calcd for $C_{16}H_{10}O$: C, 88.05; H, 4.62. Found: 87.86; H, 4.57.

B. From Trans Diol 8. The reaction was carried out as described above for diol 7 and gave 214 mg (65%) of 9.

C. From Crude Cis,Trans Diol Mixture. A suspension of crude 7/8 (2.8 g) and *p*-toluenesulfonic acid (100 mg) in toluene (100 mL) was refluxed for 1 h. Workup as described under A gave 1.5 g (59%) of 9.

2-Aceanthrenol (10). The reduction of 2-aceanthrenone was carried out in the same fashion as described for 1-aceanthrenone: yield 91% of yellow, needle-shaped crystals (from methylene chloride/hexane); mp 209–210 °C; ^1H NMR ($CDCl_3$) δ 8.26 (s, 1), 8.08–7.96 (m, 2), 7.88 (d, J = 7.8 Hz, 1), 7.58–7.47 (m, 4), 5.89 (dd, J = 7.0, 7.5 Hz, 1), 4.16 (dd, J = 7.0, 18.0 Hz, 1), 3.62 (d, J = 18.0 Hz, 1), 2.04 (d, J = 7.5 Hz, 1 OH). Anal. Calcd for $C_{16}H_{12}O$: C, 87.24; H, 5.49. Found: C, 87.18; H, 5.37.

Acknowledgment. We are indebted to Miss Carin Olsson and Mr. Gunnar Svensson for technical assistance. This work was supported by a grant from the Swedish Natural Science Research Council.

Registry No. 2, 202-03-9; 3, 641-48-5; 4, 51752-51-3; 5, 93645-78-4; 6, 6373-11-1; *cis*-7, 90047-28-2; *trans*-8, 90047-31-7; 9, 90047-29-3; 10, 90047-30-6; anthracene, 120-12-7.

Stereoselective Synthesis of (±)-11-Hydroxy-*trans*-8-dodecenoic Acid from 10-Undecenoic Acid

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In a previous paper,[1] we reported a versatile method for the stereoselective synthesis of β,γ-unsaturated ketones by reductive deconjugation of α-bromo α,β-unsaturated ketones which are readily prepared by the treatment of silyl enol ethers with dibromocarbene. This process achieves the selective introduction of a carbon–carbon double bond β,γ to ketones with one-carbon homologation. Now we describe an effective synthetic route to (±)-11-hydroxy-*trans*-8-dodecenoic acid (1), a precursor of (±)-recifeiolide (2),[2,3] from the commercially available 10-un-

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