(3.5).[18] The low σ_γ^* value of the norbornyl group is presumeably related to the high s character of the very strained bridgehead carbon, to which has been attributed the preference for 1-norbornyllithium in exchange experiments,[3] believed to indicate carbanion stability.

Whatever their cause, the deviations correspond to a pK_a range of less than 2 units. Two conclusions are possible: either (i) the transition state for carbanion formation does not reflect the stability of the carbanion, i.e., the transition state is early, or (ii) the tertiary alkyl carbanion stabilities are similar. If, however, the transition state were early, this would imply incomplete expression of the Δstrain differences. Consequently, the deviation of Nor would be smaller. Yet all the known features of the norbornyl group suggest that the 1-norbornyl anion *should* be the most stable of those studied here. It seems preferable, therefore, to conclude that the relative rates of alkyl group cleavage *do* express the correponding anion stabilities and that the pK_a variation amongst the different tertiary alkanes is indeed small.

Experimental Section

Alcohol Synthesis. Di-1-bicyclo[2.2.2]octyl ketone was synthesized from 1-bicyclo[2.2.2]octyl nitrile by Hartzler's method.[19] Unsymmetrical ketones were obtained by cuprous chloride catalyzed condensation of acid chlorides with organomagnesium compounds.[20] Alcohol **2** was prepared by the addition of *tert*-butyllithium to di-1-bicyclo[2.2.2]octyl ketone in ether. All other alcohols were obtained by the one-pot Barbier-type addition of alkyl halides to ketones in the presence of lithium metal.[21]

Alcohol Fragmentation Studies. In a typical experiment the alcohol (1–2 mg) was dissolved in *n*-butyllithium–hexane (1 N; 0.1 mL) at 20–25 °C. HMPT (1 mL) was then added. After a period ranging from 10 min to 24 h the reaction mixture was quenched with water, and hexane-extracted, and the ketonic products were analyzed by GLC on SE-30. All yields were checked against synthetic mixtures of the pure ketones at approximately the same relative and absolute concentrations in hexane. The results (Table I) are conveniently expressed in terms of the groups that cleaved rather than the ketones formed.

Registry No. **1**, 80514-85-8; **2**, 89849-36-5; **3**, 89849-37-6; **4**, 89849-38-7; **5**, 89849-39-8; **6**, 89849-40-1; **7**, 86458-82-4; **8**, 89849-41-2; *t*-Bu⁻, 65114-21-8; Ad⁻, 27750-87-4; Oc⁻, 89849-42-3; Nor⁻, 89849-43-4; *t*-BuH, 75-28-5; adamantane, 281-23-2; bicyclo[2.2.2]octane, 280-33-1; norbornane, 279-23-2.

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Synthesis of Aceanthrylene

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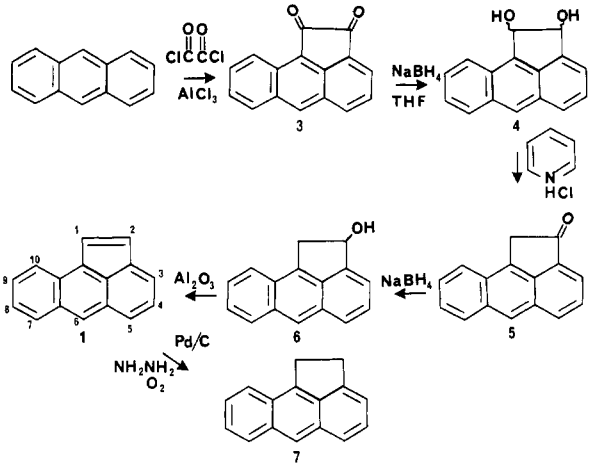
Aceanthrylene (**1**) is one of several potentially carcenogenic cyclopenta polycyclic aromatic hydrocarbons (PAH) either known or reputed to be components of diesel soot, carbon black, or of particulates from coal-fired power plants.[1-6] We view these compounds as annelated derivatives of acenaphthylene (**2**) whose derivatives show peculiar luminescent behavior under laser excitation.[7] Cyclopenta[*cd*]pyrene exhibits anomalous fluorescence and its magnetic circular dichroism is interpretable[8] from a paradigm elegantly created by Michl.[9] In accord with our expectations **1** also exhibits a characteristic anomalous fluorescence.[10] The synthesis described herein constitutes a convenient scheme for producing significant amounts of **1**.

Scheme I



Results and Discussion

One of the major problems encountered in the construction of a cyclopentano PAH is that associated with the intramolecular cyclization of appropriate substituted aryl acetic acid derivatives.[12-14] The preparation of anthracenyl-9-acetic is straightforward,[14] but our attempts to cyclize this compound to aceanthren-2-one failed.[15]

For our approach to the synthesis of **1** we chose to take advantage of the ready preparation of aceanthrenequinone (**3**)[16] (Scheme I). A slight modification of the synthetic procedure for **3** was recently reported[17] in which anthracene is treated with a large excess of oxalyl chloride and anhydrous aluminum chloride in carbon disulfide. Our experience suggests that only a slight excess of oxalyl chloride suffices and thus this constitutes a saving of the more expensive reagent. The synthesis of **3** is achieved in yields of greater than 50% on a routine basis. The quinone is not very soluble in any common organic solvent.

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We took advantage of its slight solubility in THF to reduce it with powdered sodium borohydride to the corresponding mixture of (*Z*)- and (*E*)-aceanthrene-1,2-diols (**4**).

The dehydration of mixture **4** was attempted with *p*-toluenesulfonic acid and refluxing benzene and also with P_2I_4,[18] but yields of 2-aceanthrenone (**5**) were poor. We subsequently found that a large excess of pyridine hydrochloride in benzene[19] effected the dehydration of **4** without degradation of the product. The product **5** that is formed is predominantly a single mono ketone. The structure of 2-aceanthrenone is assigned to **5** on the basis of a comparison of its NMR spectrum to that of the various substituted 1-aceanthrenones synthesized by Shechter and co-workers.[17] It is particularly noteworthy that in $CDCl_3$ the most downfield proton resonance in **5** occurs at 8.5 ppm and this is the singlet for H-6. This is to be compared to the similar singlet found for H-6 in the 1-keto isomer of **5**, which occurs at an average value of 8.6 ppm for several derivatives. In these derivatives the proton H-10 appears as a doublet of doublets resonating at an average value of 9.04 ppm, a frequency higher than any found for **5**. This increased downfield shift is expected because H-10 is geometrically very close to the diamagnetic anisotropic deshielding region of the carbonyl group of C-1. In **5** proton H-3 is far removed from the carbonyl group at C-2 and thus experiences very little downfield shift. By the use of a LIS study using europium chelate we were able to trace the origin of H-3 back to the unperturbed spectrum (see Experimental Section). Proton H-3 moved downfield at high molar Eu chelate/ketone ratios almost as rapidly as the methylene protons in **5**, thereby establishing the relative $(r^3)^{-1}$ vector distance from the oxygen contact site as being similar in magnitude for (CH_2) and H-3.[20]

When **5** is reduced in THF with sodium borohydride, a 75% yield of 2-aceanthrenol (**6**) is obtained. Treatment of **6** with *p*-TsOH in benzene produced very poor yields of **1**. Likewise when **6** was treated with pyridine hydrochloride, poor yields resulted. This is not surprising. Our PPP SCF CI calculations[10] show that there is a high degree of charge dissymmetry at the cyclopenteno bridge of **1**. The presence of a strong acid in the mixture is likely to cause protonation of **1** to form a reactive carbocation that produces dimers, oligomers, and polymers. In fact, we have isolated from the *p*-TsOH reaction mixture a dimer as determined by mass spectrometry. We hope to determine its structure shortly.

The procedure that dehydrates **6** and produces a good yield of **1** is the reaction in which anhydrous aluminum oxide and the alcohol are refluxed in benzene. As the dehydration proceeds the yellow alcohol disappears and the benzene mixture takes on the bright orange-red of **1** and produces aceanthrylene in 50% yield.

As a final verification of the structure of **1**, we transformed it by catalytic hydrogenation at ambient pressure into the dihydro derivative aceanthrene (**7**) whose physical properties corresponded to those reported by Bergmann.[11] The conditions chosen for the hydrogenation differ from the usual diimide technique used for reduction.[21] It is known that hydrazine will decompose in the presence of an active metal catalyst.[22] We mixed hydrazine, 10% Pd/C, **1**, and ethanol with vigorous magnetic stirring while exposed to the atmosphere. After a short period, the orange of **1** disappeared and upon workup the intensely fluorescent **7** was isolated. The interesting theoretical, spectroscopic, and chemical behavior of **1** is likely to pique the curiosity of future researchers. We are currently studying its photochemical and thermochemical behavior with a wide variety of reactants.

Experimental Section

Melting points were determined on a Fisher Johns hot stage and are uncorrected. ^1H NMR spectra were measured on a Varian T-60 at 60 MHz or on a JEOL FX 90Q spectrometer at 90 MHz with tetramethylsilane as an internal standard. ^{13}C NMR spectra were recorded on the FX 90Q in deuteriochloroform with $CDCl_3$ reference set at 77.00 ppm. IR spectra were recorded on a Perkin-Elmer 283 spectrophotometer. UV–visible spectra were recorded on a Cary 118C spectrophotometer. TLC runs were made with EM precoated TLC sheets, silica gel 60F-254, and aluminum oxide 60F-254 (neutral type E) with fluorescent indicator. Elemental analyses were performed by Galbraith Laboratories, Knoxville, TN.

Aceanthrenequinone (3). This compound was prepared according to the method of Liebermann and Zsuffa[16] in 50% yield.

(*Z*)- and (*E*)-Aceanthrene-1,2-diol (4). Powdered sodium borohydride (4.50 g, 119 mmol) was magnetically stirred in 100 mL of dried THF contained in a 250-mL round-bottom flask equipped with reflux condenser, and the mixture was purged through a sparge tube with dry nitrogen gas for 15 min. Aceanthrenequinone (5.5 g, 24 mmol) was added all at once and the mixture stirred at ambient temperature under positive nitrogen pressure for 4 h. The reaction was monitored by TLC during this period. The mixture was subjected to vacuum (aspirator) rotary evaporation until the bulk of the THF was removed. The yellow residue was suspended in 100 mL of water and the pH adjusted to 6 by the addition of 10% HCl. The yellow precipitate was recovered by vacuum filtration, washed with cold water, and recrystallized from acetonitrile/ethyl acetate (1:3, v/v) to produce 3.8 g, (67%), of a pale yellow mixture of diastereomeric diols as indicated by TLC.

2-Aceanthrenone (5). Freshly prepared pyridine hydrochloride[23] (2.3 g, 20 mmol) was added to 200 mL of toluene (Na dried) contained in a 500-mL round-bottom flask equipped with magnetic stirrer, condenser, Dean–Stark trap, and calcium chloride drying tube. Aceanthrene-1,2-diol (1.0 g, 4 mmol) was added and the stirred suspension was refluxed for 18 h. Upon cooling, the toluene was washed successively with 50-mL portions of 1% HCl, water, and 5% $NaHCO_3$ and then dried over anhydrous sodium sulfate. After filtration, the organic phase was vacuum rotary evaporated and the yellow residue chromatographed on aluminum oxide (neutral, grade 1) with CH_2Cl_2/*n*-hexane (1:4, v/v) to produce 0.5 g (53%) of a pale yellow solid, which after recrystallization from hexane yielded the following data: mp 165.0–166.0 °C; ^1H NMR ($CDCl_3$) δ 4.1 (s, 2 H, CH_2), 7.4–8.5 (m, 6 H), 8.1 (dd, 1 H, Ar H-3, J = 6 Hz), 8.5 (s, 1 H, Ar H-6); ^{13}C NMR ($CDCl_3$) δ 40.9, 120.8, 123.2, 124.5, 125.8, 126.2, 127.0, 127.2, 128.6, 129.7, 131.3, 131.4, 133.6, 135.3, 176.6, 202.3; IR (KBr) 3060, 1710 (s), 1430, 1400, 1260, 1225, 1015, 880, 750, 740 cm^{-1}; MS (70 eV), *m/e* (relative abundance) 81 (5), 94 (41), 10(5), 186 (8), 189 (80) 190 (50), 218 (100), 219 (20). Anal. Calcd for $C_{16}H_{10}O$: C, 88.05; H, 4.62. Found: C, 88.30; H, 4.74. The tosylhydrazone of **5** was prepared and after recrystallization from ethanol is a yellow solid, mp 215.0–216.0 °C dec.

2-Aceanthrenol (6). To a solution of 2-aceanthrenone (1.1 g, 4.6 mmol) dissolved in 100 mL of THF contained in a 250-mL round-bottom flask equipped with a magnetic stirrer was added powdered sodium borohydride (0.6 g, 15 mmol). After stirring overnight the solution was filtered, the filtrate vacuum rotary evaporated, and the residue suspended in 50 mL of water that was then acidified with 10% HCl. The pale yellow solid was collected by vacuum filtration, washed with cold water, dried, and recrystallized from benzene to yield 0.8 g (74%) of **6**: mp 228–230

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°C; ^{1}H NMR ($(CD_3)_2SO$) δ 3.5 (d, 1 H), 4.2 (m, 1 H), 5.8 (m, 2 H), 7.5–8.6 (m, 8 H); IR (KBr) 3340 (br), 3040, 2920, 1620, 1430, 1320, 1260, 1170, 1060, 900, 880, 840, 800, 770, 750, 740,cm^{-1}; MS (70 eV), *m/e* (relative abundance) 94 (5), 189 (28), 191 (38), 192 (28), 202 (80), 203 (30), 219 (18), 220 (100), 221 (19). Anal. Calcd for $C_{16}H_{12}O$: C, 87.24; H, 5.49. Found: C, 87.04; H, 5.69.

Aceanthrylene (1). Compound **6** (0.22 g, 1 mmol) was refluxed for 24 h with a magnetically stirred mixture of 100 mL of benzene and 3.0 g of Al_2O_3 (neutral activity, grade 1) in a 250-mL round-bottom flask equipped with a reflux condenser and a Dean–Stark trap. The orange solution was filtered and the benzene removed by vacuum rotary evaporation. The orange solid was recrystallized from *n*-hexane to yield 0.1 g, 50% of aceanthrylene. Sublimation at 90 °C (0.8 mm) yielded analytically pure **1**: mp 95.0–96.0 °C; UV $\lambda_{max}^{heptane}$ 560 nm (ε 10), 455 (590), 422 (1440), 399 (1970), 378 (2320), 360 (3760), 343 (2160), 250 (40 200), 235 (33 700); ^{1}H NMR ($CDCl_3$) δ 7.0 (d, 1 H, J = 5.0 Hz), 7.3–8.2 (m, 9 H); ^{13}C NMR ($CDCl_3$) δ 124.1, 124.6, 125.4, 125.6, 126.7, 127.2, 127.4, 127.6 127.9, 128.0, 129.3, 130.2, 134.3, 135.1, 137.0, 140.3; IR (KBr) 3050, 1600 (w), 1480, 1075, 880, 850, 770, 750, 735, 715 cm^{-1}; MS (70 eV, *m/e* (relative abundance) 57 (12), 88 (8), 178 (78), 200 (22), 202 (100). Anal. Calcd for $C_{16}H_{10}$: C, 95.01; H, 4.99. Found: C, 94.86; H, 5.18.

Aceanthrene (7). A mixture of aceanthrylene (54 mg, 0.2m mmol) and 1 mL of hydrazine in 10 mL of ethanol was magnetically stirred while exposed to the atmosphere and 100 mg of 10% Pd/C was added all at once. After 5 h the catalyst was removed by filtration and the fluorescent yellow solution vacuum rotary evaporated. The pale yellow residue was recrystallized from acetone to yield 49 mg (90%) of aceanthrene: mp 113–114 °C (lit.[11] mp 113–114 °C); ^{1}H NMR ($CDCl_3$) δ 3.7 (m, 4 H), 7.2–8.4 (m, 8 H).

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Registry No. 1, 202-03-9; 3, 6373-11-1; *cis*-4, 90047-28-2; *trans*-4, 90047-31-7; 5, 90047-29-3; 6, 90047-30-6; 7, 641-48-5; oxalyl chloride, 79-37-8; anthracene, 120-12-7.

The Reaction Path of Aqueous Sodium Hydroxide Induced Ring Opening of 4-Bromo-3,4-diphenyl-2-isoxazolin-2-one

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In 1979 an interesting paper on the ring-opening reactions of 3,4-disubstituted-4-bromo-2-isoxazolin-5-ones was published.[1] A noteworthy result was the formation of benzil from 4-bromo-3,4-diphenyl-2-isoxazolin-5-one (**1**).

During the course of a study on the oxidation of 3,4-disubstituted-2-isoxazolin-5-ones,[2] we showed that the mechanism proposed by the above authors for the formation of benzil was not correct. In fact, 4-hydroxy-3,4-diphenyl-2-isoxazolin-5-one (**4b**) forms benzonitrile and benzoylformic acid rather than benzil and carbon dioxide by alkaline treatment followed by acidification.

The aim of this report is to clarify the mechanism of the aqueous sodium hydroxide induced ring opening of 4-bromo-3,4-diphenyl-2-isoxazolin-5-one (**1**).

The reaction path given in Scheme I is proposed for this reaction. The initial formation of an α,β-unsaturated nitroso derivative was previously hypothesized for the ring opening of 3-methyl-4-benzyl-4-bromo-2-isoxazolin-5-one.[1]

The following experimental evidence supports this hypothesis. (i) In aqueous alkaline solution, bromoisoxazolone **1** forms a green solution, which rapidly turns colorless; the benzil precursor is a carboxylic acid salt, and benzil is formed only after acidification and decarboxylation. In fact, carbon dioxide formation is observed, and the nitrogen is present in the final solution as an ammonium salt.

(ii) Treatment of a methanol solution of the bromoisoxazolone **1** with a methanol solution of sodium methoxide affords the methyl ester of 2,3-diphenyl-2-methoxy-3-(hydroxyimino)propanoic acid (**3a**). In this case as well, addition of the methoxide solution initially leads to a green coloration. Hydrolysis of the ester **3a** with aqueous sodium hydroxide, followed by acidification, leads to benzil formation (Scheme II).

The stereochemistry of the hydroxyimine function of ester **3a** is anti with respect to the tertiary carbon atom, as shown in Scheme II. In fact, irradiation of ester **3a** in acetone (Pyrex, high-pressure Hg lamp) leads to the formation of 3,4-diphenyl-4-methoxy-2-isoxazolin-5-one (**4a**). This compound was identified on the basis of analytical and spectroscopic data (see Experimental Section).

The essential role played by the stereochemistry of the hydroxyimine function formed when a 3,4,4-trisubstituted-2-isoxazolin-2-one ring is opened under alkaline conditions is shown by the fact that 3,4-diphenyl-4-methoxy-2-isoxazolin-5-one (**4a**) dissolves in aqueous alkaline solution and is re-formed on acidification.

In the case of bromoisoxazolone **1**, alkaline treatment followed by acidification leads to the exclusive formation of benzil. The 3,4-diphenyl-4-hydroxy-2-isoxazolin-5-one (**4b**) could not be detected (TLC) in the reaction mixture. This supports the hypothesis that ring opening initially leads to the formation of an α,β-unsaturated nitroso derivative that undergoes conjguate addition of the solvent nucleophile (H_2O, MeOH). The preferential addition of the nucleophile to the unsaturated nitroso derivative in a transoid conformation to give oxime in the anti configuration has been previously reported in the literature.[3]

(iii) In the case of the methyl ester of 2,3-diphenyl-2-hydroxy-3-(hydroxyimino)propanoic acid (**3b**)[4] as well, alkaline hydrolysis followed by acidification leads to benzil formation. The stereochemistry of the hydroxyimine function of the ester **3b** is anti with respect to the tertiary carbon. Irradiation under the condition reported above of ester **3b** gave 3,4-diphenyl-4-hydroxy-2-isoxazolin-5-one (**4b**), identified by comparison with an authentic sample[2] (Scheme III).

In conclusion, the particular reactivity of the bromoisoxazolone **1** seems to be related to the following: (i) the presence of the phenyl ring in the 4-position; since a 1,5 hydrogen shift is not possible, the intermediate α,β-unsaturated nitroso derivative reacts exclusively by conjugate addition of the solvent nucleophile, and, (ii) the stereochemistry of the hydroxyimine function formed in this way, which does not allow cyclization to the isoxazolone of the intermediate hydroxyimino acid but rather allows a Neber-type reaction to give a hydroxy azirine, hydrolyzed in acid to benzil.

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