epimerization of dialkyldihydroaromatic ring systems generally, previously accomplished with *n*-butyllithium–*N,N,N',N'*-tetramethylethylenediamine.[9c]

Experimental Section

Physical Data. Proton NMR spectra were obtained on a Varian T-60 spectrometer in $CDCl_3$ with tetramethylsilane as internal standard. The integrated NMR spectra were consistent in all cases with the structural assignments and essentially identical with the spectra of authentic standard compounds, where available. All melting points are uncorrected.

Materials. Phenanthrene-9,10-dione, chrysene-5,6-dione, and benz[*a*]anthracene-7,12-dione were commercial samples recrystallized from benzene before use. Benzo[*a*]pyrene-4,5-dione[16] and dibenz[*a,c*]anthracene-9,14-dione[17] were synthesized by the methods reported earlier. 7-Methylbenzo[*a*]pyrene-4,5-dione was prepared by adaptation of the method utilized for preparation of benzo[*a*]pyrene-4,5-dione.[16] *trans*-7,12-Dimethyl-7,12-dihydrobenz[*a*]anthracene was synthesized by the method described.[10] Benzene was purified by distillation from CaH_2 and stored over molecular sieves, type 4A. Diethyl ether was dried over sodium. The HI employed was a 57% aqueous solution (Fisher) preserved with ~1% hypophosphorous acid.

Reactions of Polycyclic Quinones with Methyllithium. A partial solution of the quinone (10 mmol) in benzene (100 mL) or an equal volume of benzene–ether (1:1) was treated with a solution of methyllithium (27 mmol, 15 mL of a 1.8 M solution) in ether under N_2 at room temperature. The resulting solution was stirred for 24 h and then worked up conventionally to provide the crude dimethyl dihydro diol (confirmed by NMR) which was reduced directly with HI in acetic acid.

Reduction of Dimethyl Dihydro Diols with HI. 1. General Procedure. A solution of the dimethyl dihydro diol (3.4 mmol) and 57% HI (2 mL, 15 mmol) in acetic acid (50 mL) was heated at reflux for 15 h and then poured into a 1% aqueous sodium bisulfite solution (100 mL). The precipitate was collected by filtration, washed with water, and dried. Purification by chromatography on silica gel with benzene–hexane (1:1) as eluant afforded the pure dimethylarenes (Table I).

2. 5,6-Dimethylchrysene (4). Reactants were heated together in refluxing acetic acid for only 5 min and then immediately poured into 1% bisulfite solution and stirred overnight. During this time the original slightly sticky precipitate became crystalline. Workup by the general procedure gave pure **4** (Table I).

3. 7,12-Dimethylbenz[*a*]anthracene (6b). Upon combination of the reactants in the proportions specified in the general procedure, **6a** precipitated out. The temperature was gradually increased until **6a** dissolved and then the reaction mixture was poured into 1% bisulfite solution and worked up in the usual manner to afford pure **6b** (Table I). The identity of **6a** was confirmed by NMR: (Me_2SO-d_6) δ 3.20 (s, 3, CH_3), 5.40 (s, 2, CH_2I), and 7.50–8.45 (m, 9, aromatic). As previously reported,[5] this compound decomposed on attempted crystallization or melting.

Reduction of 6b. These reactions were conducted on a 1-g scale according to the general procedure for the reduction of the dihydro diols. Product ratios were determined by high-resolution NMR analysis (270 MHz) in comparison with authentic samples of *cis*- and *trans*-7,12-dihydro-**6b** whose NMR spectra showed the following: trans δ 1.51 (d, 3, 7-CH_3, J = 7.31 Hz), 1.91 (d, 3, 12-CH_3, J_{12} = 6.93 Hz), 4.19 (q, 1, H_7), and 4.96 (q, 1, H_{12}); cis δ 1.60 (d, 3, 7-CH_3, J_7 = 7.30 Hz), 1.64 (d, 3, 12-CH_3, J_{12} = 7.34 Hz), 4.22 (q, 1, H_7), and 4.85 (q, 1, H_{12}). These data are in essential agreement with those reported earlier[10,12] for the less well-resolved 60-MHz spectra except that $\delta_{12\text{-}CH_3}$ of the trans isomer was found at somewhat lower field than reported (δ 1.72).[10]

Reaction for 15 min gave **6b** (25%), *trans*-7,12-dihydro-**6b** (50%), and *cis*-7,12-dihydro-**6b** (25%). After 60 min, reduction of **6b** was 85% complete, and *cis*-7,12-dihydro-**6b** was the sole dihydro isomer detectable. Recrystallization of the crude product from ethanol gave pure *cis*-7,12-dihydro-**6b**, mp 108.5–109 °C (lit.[12] mp 106–107 °C).

Epimerization of *trans*-7,12-Dihydro-6b. *trans*-7,12-Dihydro-**6b** (1 g) on treatment with HI in refluxing acetic acid by the general procedure employed for the reduction of the dihydro diols underwent essentially quantitative conversion after 1 h to the cis isomer (by NMR).

Acknowledgment. This research was supported by grants (CA 11968 and CA 09183) and a research contract (CP 033385) from the National Cancer Institute, DHEW. The HX 270 Bruker NMR spectrometer was funded through University of Chicago Cancer Research Center Grant CA 14599.

Registry No. **1**, 604-83-1; **2**, 16757-89-4; **3**, 72496-73-2; **4**, 3697-27-6; **5**, 632-53-1; **6a**, 27018-50-4; **6b**, 57-97-6; *cis*-7,12-dihydro-**6b**, 24316-23-2; *trans*-7,12-dihydro-**6b**, 23660-33-5; **6c**, 13345-62-5; phenanthrene-9,10-dione, 84-11-7; benzo[*a*]pyrene-4,5-dione, 42286-46-4; 7-methylbenzo[*a*]pyrene-4,5-dione, 72496-74-3; chrysene-5,6-dione, 2051-10-7; dibenz[*a,c*]anthracene-9,14-dione, 3228-74-8; benz[*a*]anthracene-7,12-dione, 2498-66-0.

(16) Harvey, R. G.; Goh, S. H.; Cortez, C. *J. Am. Chem. Soc.* **1975**, *97*, 3468.
(17) Harvey, R. G.; Leyba, C.; Konieczny, M.; Fu, P. P.; Sukumaran, K. B. *J. Org. Chem.* **1978**, *43*, 3423.

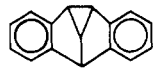
Dibenzoazasemibullvalenes

Michael J. Haire

Contribution No. 2591 from the Central Research and Development Department, E. I. du Pont de Nemours and Company, Wilmington, Delaware 19898

Received May 11, 1979

Dibenzosemibullvalene (1) has been known for many years.[1,2] However, dibenzoazasemibullvalene (2) has



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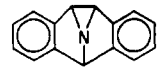
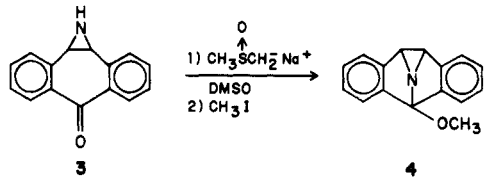


2

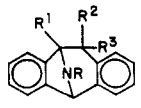
eluded all reported synthetic efforts to date.[3] The "aza" derivative of **1** is of interest because the high degree of strain associated with the semibullvalene structure should force the nitrogen and its nonbonded electrons away from the rest of the molecule, making the system more prone to form metal–ligand complexes. Also, since many 5,10-bridged dibenzocycloheptenes are known to exhibit biological activity,[4] dibenzoazasemibullvalene is of pharmaceutical interest.

I wish to report the first successful synthesis of the alkoxydibenzoazasemibullvalene **4** from the novel aziridinyl ketone **3**. The yield is high, 90–95%, and the product is



3 4

(1) E. Ciganek, *J. Am. Chem. Soc.*, **88**, 2882 (1966).
(2) G. F. Emrson, L. Watts, and R. Pettit, *J. Am. Chem. Soc.*, **87**, 131 (1965).
(3) J. J. Looker, *J. Org. Chem.*, **36**, 1045 (1971).
(4) French Patent 2 170 862 claims 5,10-iminodibenzocycloheptenes to be antidepressants, anticonvulsants, and sedatives.



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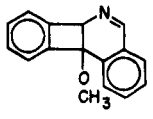
a stable, colorless, crystalline compound which can be recrystallized from 2-propanol.

The structure of **4** could be deduced from an examination of its spectral properties as well as by comparison with other dibenzocycloheptenes in this series. The proton NMR spectrum showed a single peak at δ 4.12 for the benzylic hydrogens and a singlet at δ 3.59 which was indicative of an *O*-methyl group. This eliminated the *N*-methyl derivative of **3** as a possible product. The UV spectrum of the product was consistent with two isolated, nonconjugated benzene rings. The NMR and UV evidence also appeared to rule out structures **5** and **6** as possible



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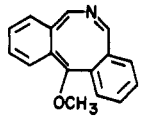
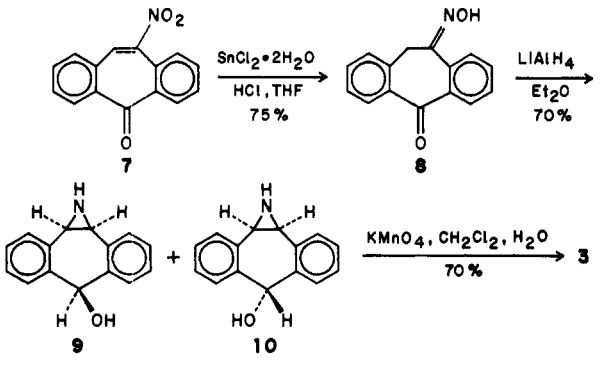


6

products. To further substantiate the assigned structure **4**, I obtained a 90-MHz ^{13}C NMR spectrum which showed nine nonequivalent carbon atoms, a fact inconsistent with **5** or **6**.[5]

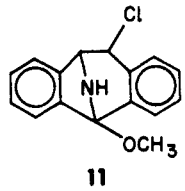
The synthesis of **4** started with the known[6] nitro olefin **7**. Reduction of **7** with stannous chloride unexpectedly[7] gave the ketoxime **8**. Further reduction of **8** with $LiAlH_4$[8]



7 **8** **9** **10**

gave a mixture of the *syn*-aziridinyl alcohol **9** and the *anti*-aziridinyl alcohol **10**. The assignment of syn and anti stereochemistry to **9** and **10**, respectively, is tentative and rests on several suggestive pieces of evidence. Compound **9** is much more soluble in $CHCl_3$ than **10**, which is consistent with intramolecular hydrogen bonding in **9** and intermolecular hydrogen bonding in **10**. In the NMR spectrum of **10** the aromatic region appears as a broad multiplet, while in **9** it appears as a slightly broadened singlet. The singlet is indicative of a single uniform environment affecting the aromatic chemical shifts. This could be attained more readily in the intramolecularly hydrogen bonded syn configuration. Thus NMR, while not diagnostic in this case, is consistent with the structure assignments **9** and **10**. Molecular models indicate the anti isomer to be more exposed and thus more readily oxidized than the syn isomer. This was indeed found to be the case. The syn isomer (**9**) could be oxidized with $KMnO_4$ in at most 10% yield, while the anti isomer (**10**) was oxidized in 70% yield under identical conditions to 10,11-dihydro-10,11-azacyclopropa-5*H*-dibenzo[*a*,*d*]cyclohepten-5-one (**3**).

Dibenzoazasemibullvalene **4** is surprisingly stable. It has been heated to 200 °C with no apparent reaction, and it does not react with strong bases. In an attempt to prepare an HCl salt of **4**, it was found that **4** is very acid sensitive. Contact with 3 N HCl in ethanol afforded 10,11-dihydro-5,10-imino-5-methoxy-11-chloro-5*H*-dibenzo[*a*,*d*]cycloheptene (**11**).



11

Studies are in progress to further elaborate the structure of **4** and investigate its chemical properties. Routes to the parent dibenzoazasemibullvalene (**2**) are also being sought.

Experimental Section

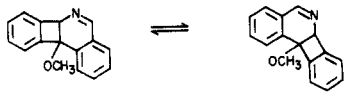
All melting points were uncorrected. NMR spectra were recorded on a Varian A-60 spectrometer using Me_4Si as an internal standard. IR spectra were recorded on a Perkin-Elmer 137 spectrometer.

10,11-Dihydro-10-(hydroxyimino)-5*H*-dibenzo[*a*,*d*]cyclohepten-5-one (8). To a solution of 40.0 g (160 mmol) of 10-nitro-5*H*-dibenzo[*a*,*d*]cyclohepten-5-one[6] in 1200 mL of tetrahydrofuran and 70 mL of concentrated hydrochloric acid at 0 °C was added slowly with stirring a solution of 111 g (585 mmol) of stannous chloride in 420 mL of tetrahydrofuran and 165 mL of concentrated hydrochloric acid. The mixture was stirred at 0 °C for 3 h followed by 2 h at room temperature. The yellow solution was then poured into 300 mL of methylene chloride and washed with water, 5% aqueous hydrochloric acid, water, saturated aqueous sodium carbonate, and water. The organic layer was dried, filtered, and concentrated in vacuo to give a yellow solid which was recrystallized from absolute ethanol to give 28.0 g (118 mmol, 74%) of the title compound as light yellow crystals: mp 186–186.5 °C; NMR ($CDCl_3$) δ 9.00–7.25 (m, 9 H, aromatic and =NOH), 5.82 (s, 2 H, benzylic); IR ($CHCl_3$) 2.82, 3.09, 3.37, 6.09, 6.29, 6.75, 6.90, 7.69, 7.84, 8.19, 8.69, 9.34, 9.64, 10.37, 10.82, 12.34, 14.08 μm.

Anal. Calcd for $C_{15}H_{11}NO_2$: C, 75.94; H, 4.67; N, 5.90. Found: C, 76.15; H, 4.77; N, 5.90; mass spectrum calcd for $C_{15}H_{11}NO_2$: *m/e* 237.0789. Found: *m/e* 237.0788.

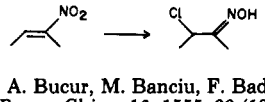
***syn*- and *anti*-10,11-Dihydro-10,11-azacyclopropa-5*H*-dibenzo[*a*,*d*]cyclohepten-5-ols (9 and 10).** The technique of Cioranescu, Bucur, Banciu, Badea, Elian, and Nenitzescu was employed.[8] To a suspension of 28.00 g (0.737 mol) of lithium aluminum hydride in 1 L of dry ether under nitrogen was added 35.00 g (0.147 mol) of 10,11-dihydro-10-(hydroxyimino)-5*H*-dibenzo[*a*,*d*]cyclohepten-5-one in 2 L of ether at room temperature over 30 min. The mixture was stirred, refluxed for 3 h, and cooled with an ice bath, and the excess lithium aluminum hydride was destroyed by carefully adding 100 mL of ice-cold water. The mixture was then extracted with ether (6×), and the ether extracts were dried, filtered, and concentrated in vacuo to give a yellow solid which was washed with hot ether (3 × 30 mL) and vacuum dried, giving 21.64 g of off-white solid. The solid was washed twice with 20 mL of chloroform, filtered, and vacuum dried to give 12.74 g (57.1 mmol, 39%) of *anti*-10,11-dihydro-10,11-azacyclopropa-5*H*-dibenzo[*a*,*d*]cyclohepten-5-ol as an off-white solid: mp 186–188 °C; NMR (Me_2SO-d_6) δ 7.80–6.90 (m, 8 H, aromatic), 5.60 (d, 1

(5) Compound **5** could exist in rapid equilibrium, too fast to observe the individual species on the NMR time scale. This seems unlikely at room temperature; however, an X-ray structure determination to firmly establish the structure of **4** is in progress.



(6) G. H. Berezin and G. A. Boswell, U.S. Patent 3 883 593 (1975).

(7) Earlier work has indicated reduction of nitro olefins with stannous chloride to give chloroximes: A. Dornov, H. D. Jordan, and A. Miller, *Chem. Ber.*, **94**, 67 (1961).



(8) E. Cioranescu, A. Bucur, M. Banciu, F. Badea, M. Elian, and C. D. Nenitzescu, *Rev. Roum. Chim.*, 16, 1555–66 (1971).

H, J = 5 Hz, CHOH), 3.68–3.10 (br m, 3 H, benzylic), 2.83–2.18 (br m, 1 H, NH); IR (Nujol) 3.07, 3.42, 6.72, 6.83, 7.27, 7.54, 7.67, 7.97, 8.17, 8.39, 8.47, 8.92, 9.57, 10.21, 10.55, 11.16, 11.67, 12.48, 12.83, 12.95, 13.27, 13.62, 14.08 μm.

Anal. Calcd for $C_{15}H_{13}NO$: C, 80.69; H, 5.87; N, 6.27. Found: C, 80.55; H, 6.07; N, 6.36; mass spectrum m/e 223 (molecular ion).

Concentration of the chloroform filtrate yielded 10.13 g (45.4 mmol, 31%) of *syn*-10,11-dihydro-10,11-azacyclopropa-5*H*-dibenzo[*a*,*d*]cycloheptene-5-ol as an off-white solid: mp 163–165 °C; NMR ($CDCl_3$) δ 7.70–7.10 (m, 8 H, aromatic), 6.28 (br d, 1 H, J = 11 Hz, CHOH), 3.75–3.45 (br s, 2 H, benzylic), 2.82–1.97 (br m, 2 H, NH and OH); IR ($CHCl_3$) 2.98, 3.30, 6.70, 6.90, 7.10, 7.17, 7.60, 7.68, 8.00, 8.45, 8.82, 9.02, 9.51, 9.90, 10.56, 11.02, 11.27, 12.20, 12.51, 14.22 μm.

Anal. Calcd for $C_{15}H_{13}NO$: C, 80.69; H, 5.87; N, 6.27. Found: C, 80.70; H, 6.08; N, 6.27. Mass spectrum calcd for $C_{15}H_{13}NO$: m/e 223.0997. Found: m/e 223.0989.

10,11-Dihydro-10,11-azacyclopropa-5*H*-dibenzo[*a*,*d*]cyclohepten-5-one (3). (a) From *anti*-10,11-Dihydro-10,11-azacyclopropa-5*H*-dibenzo[*a*,*d*]cyclohepten-5-ol (10). To 15.20 g (96.2 mmol) of finely powdered potassium permanganate was added 200 mL of saturated aqueous magnesium sulfate. After the mixture was stirred for 10 min, a suspension of 20.00 g (89.6 mmol) of *anti*-10,11-dihydro-10,11-azacyclopropa-5*H*-dibenzo[*a*,*d*]cyclohepten-5-ol (finely powdered) in 400 mL of methylene chloride was added. The mixture was stirred for 25 h, diluted with 500 mL of methylene chloride, washed with saturated aqueous sodium bisulfite and water, dried, treated with Darco, filtered, and concentrated in vacuo to give a light yellow solid which was recrystallized from ethanol to give 14.13 g (63.9 mmol, 71%) of the title compound as a white solid: mp 188–189 °C; NMR ($CDCl_3$) δ 7.71–7.10 (m, 8 H, aromatic), 3.66 (br m, 2 H, J = 9 Hz, benzylic), 3.31–2.63 (br m, 1 H, NH); IR ($CHCl_3$) 3.00, 3.30, 5.98 (s), 6.24, 6.70, 6.89, 7.19, 7.75, 8.08, 8.41, 8.68, 8.80, 9.14, 9.60, 10.50, 10.73, 11.00, 11.70, 12.20, 12.50, 14.04 μm.

Anal. Calcd for $C_{15}H_{11}NO$: C, 81.43; H, 5.01; N, 6.33. Found: C, 81.52; H, 5.09; N, 6.41; mass spectrum calcd for $C_{15}H_{11}NO$: m/e 221.0840. Found: m/e 221.0859.

(b) From *syn*-10,11-Dihydro-10,11-azacyclopropa-5*H*-dibenzo[*a*,*d*]cyclohepten-5-ol (9). To 8.10 g (51.3 mmol) of potassium permanganate (finely powdered) was added 100 mL of saturated aqueous magnesium sulfate. After the mixture was stirred for 10 min, 10.00 g (44.8 mmol) of *syn*-10,11-dihydro-10,11-azacyclopropa-5*H*-dibenzo[*a*,*d*]cyclohepten-5-ol (finely powdered) was added. The suspension was stirred for 5 min, and 200 mL of methylene chloride was added. The mixture was stirred for 16 h, diluted with 300 mL of methylene chloride, washed with saturated aqueous sodium bisulfite and water, dried, treated with Darco, filtered, and concentrated in vacuo to give a yellow solid which was recrystallized from ethanol to give 3.05 g of white solid. NMR and IR analyses showed the solid to be a 50:50 mixture of starting alcohol and 10,11-dihydro-10,11-azacyclopropa-5*H*-dibenzo[*a*,*d*]cyclohepten-5-one. Complete conversion was not obtained.

[(4b,4c-Dihydro-8b*H*-azirino[2,1,3-*cd*]dibenzo[*a*,*f*]pyrrolizin-8b-yl)oxy]methane (4). To 13.9 mmol of dimsyl sodium in 60 mL of Me_2SO at room temperature was added dropwise a solution of 3.00 g (13.6 mmol) of **3** in 25 mL of dimethyl sulfoxide. The mixture was stirred for 10 min, and 0.855 mL (1.95 g, 13.7 mmol) of methyl iodide was added dropwise. The mixture was stirred for 1 h, poured into 330 mL of brine, and extracted with methylene chloride. The organic extracts were washed with brine, dried, filtered, and concentrated in vacuo to give a yellow solid. Recrystallization from 2-propanol gave 2.91 g (12.4 mmol, 91%) of the title compound as colorless crystals: mp 118–119 °C; NMR ($CDCl_3$) δ 7.45–6.97 (m, 8 H, aromatic), 4.12 (s, 2 H, benzylic), 3.59 (s, 3 H, OCH_3); IR ($CHCl_3$) 3.32, 3.51, 6.80, 6.98, 7.37, 7.90, 8.07 (s), 8.75, 8.88, 9.18, 9.51, 9.93, 10.38, 10.61, 10.81, 11.00, 11.40, 13.98 μm; UV (EtOH) λ_{max} 202 nm (ε 30 633), 220 sh (29 920), 240 (7426), 255 (1856).

Anal. Calcd for $C_{16}H_{13}NO$: C, 81.68; H, 5.57; N, 5.95. Found: C, 82.02; H, 5.47; N, 5.65; mass spectrum calcd for $C_{16}H_{13}NO$: m/e 235.0996. Found: m/e 235.0992.

10,11-Dihydro-5,10-imino-5-methoxy-11-chloro-5*H*-dibenzo[*a*,*d*]cycloheptene (11). A solution of 235 mg (1.00 mmol) of **4** in 50 mL of 3 N hydrogen chloride in ethanol was stirred for 3 h and concentrated in vacuo to give a white solid which was taken up in 25 mL of water. The solution was added to 50 mL of saturated aqueous sodium carbonate and extracted with methylene chloride. The organic extracts were dried, filtered, and concentrated in vacuo to give 390 mg of an oily solid which was chromatographed on a 20 cm × 20 cm × 2 mm plate of silica gel. After one chloroform development the plate was divided into four bands. The second band from the top contained 190 mg (0.70 mmol, 70%) of the title compound which crystallized and was washed with 2-propanol and hexane to give a white solid: mp 132–133 °C; NMR ($CDCl_3$) δ 7.67–7.05 (m, 8 H, aromatic), 5.44 (d, 1 H, J = 5.5 Hz, PhCH–NH), 4.79 (d, 1 H, J = 5.5 Hz, PhCHCl), 3.52 (s, 3 H, OCH_3), 2.70–2.40 (br s, 1 H, NH); IR ($CHCl_3$) 3.04, 3.38, 3.54, 6.94, 7.24, 7.52, 7.94, 8.14, 8.84, 9.29, 9.75, 9.90, 10.47, 10.67, 11.04, 11.52, 11.94, 12.27, 14.28, 14.58 μm.

Anal. Mass spectrum calcd for $C_{16}H_{14}NOCl$: m/e 271.0763. Found: m/e 271.0756.

Registry No. 3, 69208-47-5; **4**, 72301-59-8; **7**, 56157-32-5; **8**, 69208-45-3; **9**, 69208-46-4; **10**, 69256-74-2; **11**, 72301-60-1.

Electrophilic Trifluoroacetylation of Dicyclopenta[*ef*,*kl*]heptalene (Azupyrene)[1,2]

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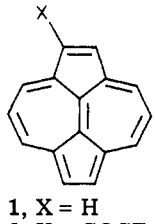
Received October 2, 1979

The spectral properties (IR, ^1H NMR, and ESR), diamagnetic susceptibility, and stability to heat and air of the nonbenzenoid, 4n π electron hydrocarbon azupyrene (**1**)



1, X = H
2, X = $COCF_3$

were indicative of aromatic character,[4] but it remained to show that electrophilic substitution rather than addition was the preferred mode of reaction and, if this were the case, to determine the most reactive position.

The symmetry of the structure simplifies the application of molecular orbital calculations to predict the primary site for bonding to an electrophile. Boekelheide et al.[5] calculated that the (equivalent) open positions on the five-membered ring would have the highest ground-state electron density. This often corresponds to the most reactive position in aromatic molecules, but the atom-localization energy is probably a more reliable indicator.[6] Accordingly, simple Hückel (HMO) and CNDO/2 calcu-

(1) Supported in part by grants from the National Science Foundation.
(2) Taken in part from the Ph.D. Thesis of G.M.M., University of Washington, 1972.
(3) NIH Predoctoral Fellow, 1968–1970.
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0022-3263/80/1945-1312$01.00/0