recrystallizations, the dimers had mp 216-8 °C (from ethyl acetate, lit.[6] mp 220 °C), 122-4 °C (from 2-propanol, lit.[4] mp 124-5 °C), and 115-7 °C (from 2-propanol, lit.[4] mp 118-9 °C), respectively.

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Registry No. 2, 118041-56-8; 3, 118101-04-5; 4, 14637-55-9; 5, 35826-08-5; tropone, 539-80-0.

Diels-Alder Reactions of Cycloalkenones. 16. Endo Diastereoselectivity of Some Cycloalkenones in Reactions with 1,3-Cyclohexadiene[1]

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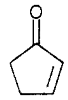
As part of a broad study of Diels-Alder reactions of 2-cycloalkenones the interaction of 2-cyclohexenones with cyclopentadiene was investigated recently.[3] Whereas this constituted the first use of a cyclic diene in the extended experimental series, the diene was a Diels-Alder substrate of high reactivity.[4] In order to complete the picture, a set of experiments involving a cyclic diene of low reactivity had to be executed. For this reason 1,3-cyclohexdiene (**1**)[4] was chosen as a Diels-Alder diene component and its cycloaddition with the following dienophiles pursued: 2-cyclopentenone (**2**), 2-cyclohexenone (**3a**), 4,4-dimethyl-2-cyclohexenone (**3b**), 5,5-dimethyl-2-cyclohexenone (**3c**), and 2-cycloheptenone (**4**).

1



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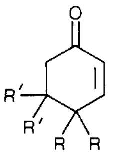
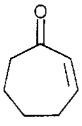


3a, R = R′ = H
b, R = Me; R′ = H
c, R = H; R′ = Me



4

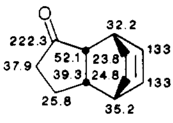
Diels-Alder Reaction Products

The cycloadditions were performed with various diene-dienophile combinations under aluminum trichloride catalysis in toluene solutions at 40 °C and led to 70-95% yields of adducts (Table I). The reactions were highly endo-diastereoselective. In view of the isolated material accounting for at least 97% of the product mixture, no more than 3% exo adducts may have been present. As in the earlier cyclopentadiene study[3] the Diels-Alder adducts were kinetically based, no exo-endo isomerization being observed throughout the course of the GC-monitored reactions nor such isomerization taking place on exposure of the pure adducts to the reaction conditions of the cycloadditions. Tricycles **5** and **6** were the products of the reactions of cyclopentenone (**2**) and the cyclohexenones **3**, respectively. The **1**-**4** reaction led to a 1.2:1 mixture of tricyles **7** and **8**, the primary product **7** having undergone acid-induced, α-keto bridgehead isomerization.

Table I. Reaction Conditions of the Diels-Alder Reactions of 1,3-Cyclohexadiene (1) and Cycloalkenones 2-4[a]

enone[b]	diene/ketone[c]	product yield,[d] %
2	12	85
3a	6	80
3b	6	70
3c	6	80
4[e]	12	95

[a] Complexation time,[11] 40 min; complexation temperature,[11] 22 °C; reaction time, 144 h; reaction temperature, 40 °C; $AlCl_3$/ketone equivalents ratio, 0.5. [b] Ketone concentration, 0.1 M. [c] Ratio of equivalents. [d] GC-based; >96% of the reaction products isolated. [e] Reaction time: 24 h.



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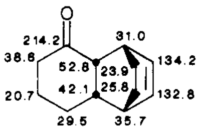
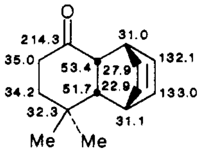


6a



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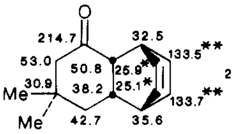
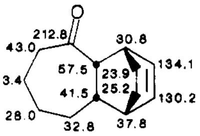


6c



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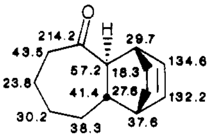

8

The structures of the cycloadducts **5**-**8** were determined by ^{1}H and ^{13}C NMR specroscopy as well as by 2-D NMR (COSY and ^{1}H-^{13}C correlation) experiments. The ^{1}H shifts and coupling characteristics are listed in the Experimental Section and the ^{13}C shifts are depicted on the formulas **5**-**8**. The stereochemistry assignment was aided greatly by NMR data on exo and endo adducts of cyclopentadiene and 2-cyclohexenones.[3]

The δ values of the hydrogens of the saturated two-carbon bridge of adducts **6** were diagnostic of the stereochemistry of these compounds. The hydrogens facing the olefinic π bond revealed a 1.29 ± 0.03 ppm shift, reminiscent of the 1.30 ± 0.06 ppm shift of the sterically identical hydrogen of the methylene bridge of the *exo*- and *endo*-3-cyclopentadiene adducts.[3] The chemical shift of the hydrogen of the one-carbon bridge of the cyclopentadiene adducts facing away from the olefinic π bond is stereochemistry-dependent, i.e., shielded (1.21 ± 0.04 ppm) in the exo compounds (presumably because of the anisotropy of the carbonyl group) and deshielded (1.45 ± 0.03 ppm) in the endo substances (because of the removal of the anisotropy of the carbon-carbon double bond). Adducts **6** show the hydrogens of the saturated two-carbon bridge facing away from the olefinic π bond to be deshielded (1.54 ± 0.07 ppm), in consonance with an endo adduct configuration. The latter conclusion is aided by the strong shift similarity of the vicinal hydrogens (the Δδ value averaging 0.05 ppm). Hydrogen shift comparison between adducts **6** and **5** and **7** indicate the latter two compounds to belong also to the endo series.

Comparison of the methylene carbon shifts (24-26 ppm) of the saturated two-carbon bridge of the Diels-Alder adducts **5**, **6a**, **6c**, and **7** with those of bicyclo[2.2.2]octene models, i.e., *exo*- and *endo*-1-acrolein cycloaddition products (endo adduct equivalent $\delta(CH_2)$ = 25 ppm),[5]

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(1) For the previous paper, see: Angell, E. C.; Fringuelli, F.; Pizzo, F.; Minuti, L.; Taticchi, A.; Wenkert, E. *J. Org. Chem.*, in press.

(2) (a) University of California. (b) Università di Perugia.

(3) Angell, E. C.; Fringuelli, F.; Guo, M.; Minuti, L.; Taticchi, A.; Wenkert, E. *J. Org. Chem.* **1988**, *53*, 4325.

(4) (a) Sauer, J.; Sustmann, R. *Angew. Chem., Int. Ed. Engl.* **1980**, *19*, 779. (b) Rucker, C.; Lang, D.; Sauer, J.; Friege, H.; Sustmann, R. *Chem. Ber.* **1980**, *113*, 1663. (c) Grieco, P. A.; Larsen, S. O. *J. Org. Chem.* **1986**, *51*, 3553. (d) Boger, D. L.; Brotherton, C. E. *Tetrahedron* **1986**, *42*, 2777.

showed the tricycles to be endo adducts. Furthermore, the strong carbon shift similarity of the two methylenes of the saturated two-carbon bridge indicates a similar environment of the two carbons, a fact explicable only on the basis of an endo configuration of the Diels-Alder products. The proximity of the *gem*-dimethyl function of tricycle **6b** to the bicyclooctene unit distorts the latter enough to make the shifts of the two-carbon bridge methylenes dissimilar.

Treatment of ketone **7** with ethanolic sodium ethoxide led to a 1.2:1 equilibrium mixture of ketones **7** and **8**, indicative of a trans juncture between the ketonic ring and the bicyclooctene unit in tricycle **8**. This configuration was substantiated by the shift dissimilarity ($\Delta\delta = 0.4$ ppm) of the hydrogens of the saturated two-carbon bridge facing the cycloheptanone unit in ketone **8** and by the dissimilarity of the carbon shifts of the methylenes of the saturated two-carbon bridge (the methylene closest to the carbonyl carbon feeling a γ-effect of 5.6 ppm from the latter).

Endo-Exo Disastereoselectivity

In previous studies of the cycloadditions of 2-cyclohexenones with (*E*)-piperylene[6] and cyclopentadiene,[3] it has been pointed out that endo-diastereoselectivity can be rationalized satisfactorily in terms of interplay of stabilizing secondary orbital interactions[7] and steric, repulsive effects between diene and dienophile in the endo transition state (on the assumption of an unsymmetrical transition state with the incipient σ bond toward the enone β-carbon more developed than that toward the α-carbon and the diene attack occurring preferentially antiparallel to the pseudoaxial enone γ-substituent[9]). The steric repulsions of methylene hydrogens of dienes from those of dienophiles in the exo transition state of reactions of the somewhat puckered cyclohexadiene (**1**) vs reactions of the planar cyclopentadiene are sufficiently great to suppress exo product formation in the cyclohexadiene (**1**) cases but permit the production of exo and endo adducts in the cyclopentadiene reactions.[3,9] Furthermore, whereas 2-methyl-2-cyclohexenones undergo reaction with cyclopentadiene (with a higher exo/endo product ratio), the 2-methylated dienophiles are totally inert toward cyclohexadiene (**1**), both thermally and under acid catalysis.

Experimental Section[10]

Melting points were taken on a Büchi melting point apparatus and are uncorrected. Infrared spectra of CCl_4 solutions were recorded on a Perkin-Elmer 257 spectrophotometer. ^1H and ^{13}C NMR spectra of $CDCl_3$ solutions were obtained on a Nicolet QE-300 spectrometer, operating in the Fourier transform mode at 300 and 75.5 MHz, respectively. The carbon shifts on formulas **5–8** are in parts per million downfield from Me_4Si; $\delta(Me_4Si) = \delta(CDCl_3) + 76.9$ ppm (starred signals being interchangeable). Mass spectra were observed on a Hewlett-Packard 5970 GC-MS instrument, calibrated with perfluorotributylamine for 70-eV operations. Analytical GC was carried out with Carlo Erba HRGC-5160 (with 30 m, 0.32 mm diameter SP-2340 fused silica capillary column and with an "on column" injection system) and Hewlett-Packard 5880A (with 30 m, 0.25 mm diameter SPB-5 capillary column and with an "on column" injection system) gas chromatographs. Absorption chromatography was executed on 0.040–0.063 mm, 230–240-mesh ASTM Merck silica gel. All (2,4-dinitrophenyl)hydrazones were purified by crystallization from ethanol.

General Procedure for the Diels–Alder Reactions. The following discussion of the **1–3a** reaction reflects the procedure used for all cycloadditions. Further details are listed in Table I.

A solution of 2.42 g (25.0 mmol) of 2-cyclohexenone (**3a**) in 100 mL of dry toluene was added to a suspension of 1.66 g (12.5 mmol) of anhydrous aluminum chloride in 100 mL of dry toluene and the mixture stirred under nitrogen at room temperature for 40 min. 1,3-Cyclohexadiene (12.1 g, 150 mmol) was poured into the mixture and enough dry toluene added to make the solution volume 250 mL. (The handling of all starting materials was performed in a drybox.) The container was closed and the contents kept at 40 °C for 144 h. The mixture then was cooled and poured into ice–water. The aqueous layer was extracted with ether and the combined extract and toluene solution washed with 10% sodium bicarbonate solution, dried (Na_2SO_4), and evaporated under vacuum. Chromatography of the residue (7.6 g) and elution with 9:1 pentane–ether gave tricyclic ketone **6a**.

Ethenohydroindanone 5: colorless liquid; IR C=CH 3050 (w), C=O 1743 (s), C=C 1650 (w) cm^{-1}; ^1H NMR δ 1.2–1.3 (m, 2, H-10a, H-11a), 1.4–1.6 (m, 3, H-4, H-10b, H-11b), 1.9–2.2 (m, 3, C-3 Hs, H-4), 2.36 (dd, 1, J = 10, 2 Hz, H-1), 2.5–2.6 (m, 1, H-5), 2.6–2.7 (m, 1, H-6), 2.9–3.0 (m, 1, H-9), 6.1–6.3 (m, 2, H-7, H-8); MS,[12] m/e (rel intensity) 162 (M^+, 17), 92 (26), 91 (25), 83 (24), 81 (8), 80 (base), 79 (39), 78 (31), 77 (20), 65 (8), 56 (8), 52 (9), 51 (13). (2,4-Dinitrophenyl)hydrazone: mp 140–141 °C.

Anal. Calcd for $C_{17}H_{18}O_4N_4$: C, 59.64; H, 5.30; N, 16.37. Found: C, 59.80; H, 5.28; N, 16.25.

Ethenodecalone 6a: colorless liquid; IR C=CH 3045 (w), C=O 1705 (s), C=C 1645 (w) cm^{-1}; ^1H NMR δ 0.9–1.0 (m, 1, H-5), 1.25–1.35 (m, 2, H-11a, H-12a), 1.47–1.55 (m, 1, H-11b), 1.53–1.62 (m, 1, H-12b), 1.7–1.9 (m, 3, C-4 Hs, H-5), 2.07 (ddd, 1, J = 18, 10, 8 Hz, H-3), 2.3–2.5 (m, 3, H-3, H-6, H-7), 2.51 (dd, 1, J = 11, 2 Hz, H-1), 3.1–3.2 (m, 1 H-10), 6.12 (t, 1, J = 7 Hz, H-8), 6.26 (t, 1, J = 7 Hz, H-9); MS, m/e (rel intensity) 176 (M^+, 19), 105 (5), 104 (11), 98 (7), 97 (41), 92 (10), 91 (19), 81 (7), 80 (base), 79 (31), 78 (9), 77 (11), 41 (6). (2,4-Dinitrophenyl)hydrazone: mp 158–159 °C.

Anal. Calcd for $C_{18}H_{20}O_4N_4$: C, 60.66; H, 5.66; N, 15.72. Found: C, 60.61; H, 5.68; N, 15.80.

Ethenodimethyldecalone 6b: colorless liquid: IR C=CH 3042 (w), C=O 1710 (s), 1650 (w) cm^{-1}; ^1H NMR δ 0.63, 1.01 (s, 3 each, methyls), 1.20–1.35 (m, 2, H-11a, H-12a), 1.45–1.55 (m, 2, H-11b, H-4), 1.50–1.60 (m, 1, H-12b), 1.73 (A of ABXY, 1, J = 14, 9, 0 Hz, H-4), 2.1–2.3 (m, 2, C-3 Hs), 2.12 (dd, 1 J = 12, 2 Hz, H-6), 2.55 (br d, 1, J = 6 Hz, H-7), 2.62 (dd, 1, J = 12, 3 Hz, H-1), 3.0–3.1 (m, 1, H-10), 6.10 (dd, 1, J = 7, 6 Hz, H-8), 6.19 (t, 1, J = 7 Hz, H-9); ^{13}C NMR δ 23.4 (Me), 30.3 (Me); MS, m/e (rel intensity) 204 (M^+, 31), 126 (8), 125 (67), 105 (8), 92 (9), 91 (13), 81 (11), 80 (base), 79 (29), 78 (11), 77 (13), 70 (7), 56 (8), 55 (11). (2,4-Dinitrophenyl)hydrazone: mp 166–167 °C.

Anal. Calcd for $C_{20}H_{24}O_4N_4$: C, 62.49; H, 6.29; N, 14.57. Found: C, 62.40; H, 6.31; N, 14.51.

Ethenodimethyldecalone 6c: colorless liquid; IR C=CH 3048 (w), C=O 1705 (s), C=C 1650 (w) cm^{-1}; ^1H NMR δ 0.97, 1.01 (s, 3 each, methyls), 1.0–1.1 (m, 1, H-5), 1.25–1.35 (m, 2, H-11a, H-12a), 1.5–1.6 (m, 3, H-5, H-11b, H-12b), 1.79 (d, 1, J = 17 Hz,

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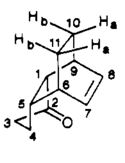
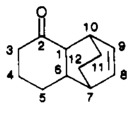
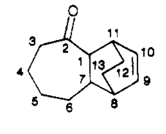
[8] Angell, E. C.; Fringuelli, F.; Pizzo, F.; Porter, B.; Taticchi, A.; Wenkert, E. *J. Org. Chem.* **1986**, *51*, 2642.

[9] The **1–3c** reaction has an even greater energy barrier for exo addition in view of the transition state requiring a parallel attack of the diene and a half-boat form of the ketone-containing ring.[8]

[10] All compounds are named on the basis of the following numbering system.







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[12] It is noteworthy that all Diels–Alder adducts (**5–7**) have a common base peak, 80 mass units, corresponding to the cyclohexadiene radical cation.

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ax H-3), 2.11 (dd, 1, $J = 17, 2$ Hz, eq H-3), 2.40 (dd, 1, $J = 10, 1$ Hz, H-1), 2.4–2.5 (m, 2 H-6, H-7), 3.01 (br s, 1, H-10), 6.1–6.3 (m, 2, H-8, H-9); ^{13}C NMR δ 27.5 (Me), 31.8 (Me); MS, m/e (rel intensity) 204 (M^+, 10), 189 (7), 125 (50), 105 (7), 92 (14), 91 (24), 81 (12), 80 (base), 79 (38), 78 (17), 77 (20), 65 (9), 56 (7), 55 (13), 53 (9), 51 (7). (2,4-Dinitrophenyl)hydrzone: mp 191–192 °C.

Anal. Calcd for $C_{20}H_{24}O_4N_4$: C, 62.49; H, 6.29; N, 14.57. Found: C, 62.60; H, 6.33; N, 14.61.

Ethenohydrobenzosuberone 7: colorless liquid; IR C=CH 3050 (w), C=O 1703 (s) cm^{-1}; ^{1}H NMR δ 0.9–1.1 (m, 1, H-6), 1.21 (dddd, 1, $J = 12, 12, 3, 3$ Hz, H-12a), 1.29 (dddd, 1, $J = 12, 12, 3, 3$ Hz, H-13a), 1.40–1.48 (m, 1, H-12b), 1.45–1.52 (m, 1, H-13b), 1.5–1.8 (m, 4, H-4, C-5 Hs, H-6), 1.8–2.0 (m, 1, H-4), 2.14 (ddd, 1, $J = 11, 2, 2$, Hz, H-7), 2.2–2.5 (m, 3, C-3 Hs, H-8), 2.7–2.8 (m, 1, H-11), 2.96 [br d, 1, $J = 11$ Hz (weak W-coupling with H-10), H-1], 6.04 (dd, 1, $J = 7, 7$ Hz, H-9), 6.37 [dd, 1, $J = 7, 7$ Hz (weak W-coupling with H-1), H-10]; MS, m/e (rel intensity) 190 (M^+, 26), 175 (8), 112 (10), 111 (52), 105 (8), 93 (11), 92 (20), 91 (28), 81 (11), 80 (base), 79 (37), 78 (13), 77 (17), 55 (13). (2,4-Dinitrophenyl)hydrazone: mp 176–177 °C.

Anal. Calcd for $C_{19}H_{22}O_4N_4$: C, 61.61; H, 5.99; N, 15.12. Found: C, 61.45; H, 6.03; N, 15.10.

Ethenohydrobenzosuberone 8: colorless liquid; IR C=CH 3040 (w), C=O 1700 (s) cm^{-1}; ^{1}H NMR δ 1.1–1.3 (m, 3, H-6, H-12a, H-13a), 1.4–1.5 (m, 1, H-13b), 1.5–1.6 (m, 2, H-5, H-7), 1.6–1.7 (m, 1, H-4), 1.7–1.9 (m, 3, H-4, H-6, H-12b), 1.9–2.1 (m, 1, H-5), 2.15 (d, 1, $J = 9$ Hz, H-1), 2.3–2.4 (m, 1, H-8), 2.4–2.6 (m, 2, C-3 Hs), 2.7–2.8 (m, 1, H-11), 6.19 (dd, 1, $J = 7, 7$ Hz, H-9), 6.32 (dd, 1, $J = 7, 7$ Hz, H-10); MS, m/e (rel intensity) 190 (M^+, 12), 112 (20), 111 (base), 105 (24), 93 (31), 92 (58), 91 (66), 81 (13), 80 (45), 79 (44), 78 (21), 77 (32), 67 (13), 66 (11), 65 (14), 55 (20), 53 (10), 51 (10). (2,4-Dinitrophenyl)hydrazone: mp 227–228 °C.

Anal. Calcd for $C_{19}H_{22}O_4N_4$: C, 61.61; H, 5.99; N, 15.12. Found: C, 61.70; H, 5.96; N, 15.15.

Epimerization of Ketone 7. A solution of 40 mg of ketone 7 in 8 mL of absolute ethanol was added to a 0.1 M ethanolic solution (6 mL) of sodium ethoxide under nitrogen and the mixture stirred at 22 °C until equilibrium was reached (monitored by GC analysis). After the usual workup the liquid product was shown by GC analysis to be a 1.2:1 **7**–**8** mixture.

Acknowledgment. F.F., L.M., F.P., and A.T. thank the Consiglio Nazionale delle Ricerche and the Ministero della Pubblica Istruzione for financial support of the work in Perugia.

Registry No. **1**, 592-57-4; **2**, 930-30-3; **3a**, 930-68-7; **3b**, 1073-13-8; **3c**, 4694-17-1; **4**, 1121-66-0; **5**, 56846-30-1; **5** ((2,4-dinitrophenyl)hydrazone), 117961-13-4; **6a**, 64989-29-3; **6a** ((2,4-dinitrophenyl)hydrazone), 117961-14-5; **6b**, 117961-10-1; **6b** ((2,4-dinitrophenyl)hydrazone), 117961-15-6; **6c**, 117961-11-2; **6c** ((2,4-dinitrophenyl)hydrazone), 117961-16-7; **7**, 117961-12-3; **7** ((2,4-dinitrophenyl)hydrazone), 117961-17-8; **8**, 118016-02-7; **8** ((2,4-dinitrophenyl)hydrazone), 118016-03-8.

Intramolecular Photochemical Rearrangement via an In-Cage Cation-Radical/Radical Pair of an Anthrylsulfonium Salt Derivative

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Since the photochemistry of triphenylsulfonium salts was reported by McEwen[1] in 1970, there has been considerable interest in the photochemical behavior of onium salts in general. This interest originates from their ability to photogenerate acid which can be used to initiate cationic polymerization as well as catalyze other chemical transformations. We have previously reported photoinduced rearrangements in a variety of sulfonium[2,3] and arsonium[4] systems. We believe the σ^* character of the lowest unoccupied molecular orbital in onium salts is responsible for the observed photochemistry[5] and have found AM1 calculations[6] helpful at predicting photochemical behavior.

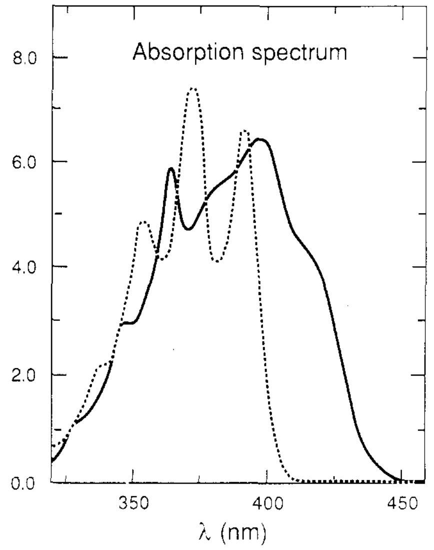

Figure 1. Absorption spectra of 9-(methylthio)anthracene (---) and 9-anthrylmethyl(p-cyanobenzyl)sulfonium hexafluorophosphate (—) in acetonitrile solvent.

We now report the photochemical behavior of 9-anthrylmethyl(p-cyanobenzyl)sulfonium hexafluorophosphate (**1**), which further supports the proposed in-cage radical pair mechanism.

The effect of a sulfonium group, i.e., dimethyl or methyl(p-cyanobenzyl), on the anthracene electronic behavior is to shift the short axis in-plane long wavelength ^1L_b electronic transition *to the red of the same transition in anthracene* (see Figure 1). There is also considerable broadening of the ^1L_b electronic transition relative to the long axis polarized ^1L_a electronic transition in **1**. We expect the lowest energy electronic transition in **1** to be $\pi \rightarrow \sigma^*$ and to be of low molecular extinction. We have assigned the ~420-nm band to this transition.

Irradiation of **1** with light of a wavelength greater than 400 nm results in a rapid bleaching of absorption in that spectral region. Five photoisomers of **1** were obtained in 56% yield (0.43 quantum yield) in addition to photosolvolysis products (44% yield, 0.34 quantum yield), which include 9-anthryl methyl sulfide and N-(p-cyanobenzyl)acetamide derived from the reaction with acetonitrile. The 10-substituted rearrangement product is predominant, being formed in 38% overall yield.

An attempt was made to rationalize the regioselectivity in terms of spin density within the 9-anthryl methyl sulfide cation-radical and the distance between the benzylic carbon in **1** and reaction sites on the anthracene ring. The intermediate singlet cation-radical/radical pair can, in theory, couple at all sites on the anthracene ring system that has spin density. However, only carbon sites pos-

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