Anal. Calcd for $C_{16}H_{27}NO_4$: C, 64.62; H, 9.15. Found: C, 64.72; H, 9.21.

General Procedure for Selective Removal of One *tert*-Butoxycarbonyl Group. 1-[(*tert*-Butoxycarbonyl)amino]-2(*E*),4(*E*)-hexadiene (7h). Into a 25-mL round-bottom flask equipped with a magnetic stirrer were added the isomeric mixture of **5h** and **6h** (0.560 g, 1.88 mmol), methylene chloride (20 mL), and trifluoroacetic acid (0.322 g, 2.82 mmol, 1.5 equiv). The flask was fitted with a septum, and the solution was stirred at 25 °C for 20 h. The solution was then poured into ether (70 mL) and washed with 10% aqueous NaOH (1 × 10 mL) and saturated aqueous NaCl (1 × 10 mL). The colorless solution was dried ($MgSO_4$) and concentrated in vacuo to provide 0.362 g (98%) of the monoprotected amine as a mixture of isomers. When this oil was dissolved in a minimal amount of hexane, transferred to a Craig tube, and cooled to −20 °C, a white solid crystallized out of solution. Filtration and a second recrystallization provided **7h** as a white solid (in 60–80% yield): mp 51–53 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.11 (ddt, $J_{2,3}$ = 14.4 Hz, $J_{3,4}$ = 10.4 Hz, $J_{1,3}$ = 1.2 Hz, 1 H, C*H*=CHCH$_2$N), 6.00 (ddq, $J_{4,5}$ = 14.0 Hz, $J_{3,4}$ = 10.3 Hz, $J_{4,6}$ = 1.5 Hz, 1 H, CH$_3$CH=C*H*CH), 5.67 (dq, $J_{4,5}$ = 14.0 Hz, $J_{5,6}$ = 6.57 Hz, 1 H, CH$_3$C*H*=CH), 5.54 (dt, $J_{2,3}$ = 14.5 Hz, $J_{1,2}$ = 6.0 Hz, 1 H, CH=C*H*CH$_2$N), 4.65 (bs, 1 H, N*H*COO(CH$_3$)$_3$), 3.66 (bt, $J_{1,2}$ = 5.4 Hz, CH=CHC*H*$_2$N), 1.77 (d, J = 6.5 Hz, 3 H, CH$_3$CH=CH), 1.44 (s, 9 H, NHCOOC(C*H*$_3$)$_3$); ^{13}C NMR (50 MHz, $CDCl_3$) δ 155.65 (N*C*OOC(CH$_3$)$_3$), 131.88, 130.62, 129.39, and 126.84 (CH$_3$*C*H=*C*H*C*H=*C*H), 79.21 (*C*H$_2$N), 42.35 (COO*C*(CH$_3$)$_3$), 28.33 (COOC(*C*H$_3$)$_3$), 17.96 (*C*HCH$_3$); IR ($CDCl_3$) 3420, 1690 cm^{-1}; mass spectrum (CI), *m/z* (rel intensity) 197 (2, M$^+$), 141 (38, M$^+$ − C$_4$H$_8$), 96 (M$^+$ −C$_4$H$_8$ − CO$_2$), 80 (100).

Anal. Calcd for $C_{11}H_{19}NO_2$: C, 66.97; H, 9.71. Found: C, 67.18; H, 9.73.

3-[(*tert*-Butoxycarbonyl)amino]-1-hexene (7e). Following the same procedure as above, **4e** (0.237 g, 0.79 mmol) in methylene chloride (10 mL) was treated with trifluoroacetic acid (0.135 g, 1.19 mmol, 1.5 equiv) and stirred at 25 °,C for 20 h to provide 0.142 g (90%) of **7e** as a colorless oil: ^{1}H NMR (200 MHz, $CDCl_3$) δ 5.73 (ddd, $J_{1Z,2}$ = 17 Hz, $J_{1E,2}$ = 10.2 Hz, $J_{2,3}$ = 5.8 Hz, 1 H, CHC*H*=C(H$_E$)H$_Z$), 5.13 (ddd, $J_{1Z,2}$ = 17.3 Hz, $J_{1Z,1E}$ = 1.4 Hz, $J_{1Z,3}$ = 1.3 Hz, 1 H, CHCH=C(H$_E$)*H*$_Z$), 5.06 (ddd, $J_{1E,2}$ = 10.2 Hz, $J_{1Z,1E}$ = 1.4 Hz, $J_{1Z,3}$ = 1.2 Hz, 1 H, CHCH=C(*H*$_E$)H$_Z$), 4.52 (bs, N*H*COOC(CH$_3$)$_3$), 4.09 (bt, 1 H, C*H*NHCOOC(CH$_3$)$_3$), 2.10 (m, 2 H, C*H*$_2$CH$_2$CH$_3$), 1.45 (s, 9 H, NHCOOC(C*H*$_3$)$_3$), 1.30 (m, 2 H, CH$_2$C*H*$_2$CH$_3$), 0.92 (t, $J_{5,6}$ = 6.7 Hz, 3 H, CH$_2$CH$_2$C*H*$_3$); ^{13}C NMR (50 MHz, $CDCl_3$) δ 155.33 (*C*OOC(CH$_3$)$_3$), 139.14 (CH$_2$C-H(N)*C*H=CH$_2$), 114.06 (CH$_2$CH(N)C*H*=*C*H$_2$), 79.10 (CH$_2$CH-(N)CH=*C*H$_2$), 52.60 (COO*C*(CH$_3$)$_3$), 37.28 (CH$_3$CH$_2$CH$_2$CHN), 28.33 (COOC(*C*H$_3$)$_3$), 18.85 (CH$_3$*C*H$_2$CH$_2$CHN), 13.78 (*C*H$_3$C-H$_2$CH$_2$CHN); IR (neat) 3350, 1710 cm^{-1}; mass spectrum (CI), *m/z* (rel intensity) 199 (2, M$^+$), 156 (42, M + 1 − CO$_2$), 143 (38, M − C$_4$H$_8$), 100 (100, M + 1 − C$_4$H$_8$ − CO$_2$).

Anal. Calcd for $C_{11}H_{21}NO_2$: C, 66.29; H, 10.62. Found: C, 66.20; H, 10.84.

1-[(*tert*-Butoxycarbonyl)amino]-2(*E*)-hexene (7f). Following the same procedure as above, **5e** (0.626 g, 2.09 mmol), methylene chloride (15 mL), and trifluoroacetic acid (0.358 g, 3.14 mmol, 1.5 equiv) were stirred at 25 °C for 22 h. Workup as above provided 0.389 g (94%) of **7f** as a colorless oil: ^{1}H NMR (200 MHz, $CDCl_3$) δ 5.49 (dm, $J_{2,3}$ = 15.6 Hz, 2 H, CH$_2$C*H*=C*H*CH$_2$N), 4.90 (bs, 1 H, N*H*), 3.66 (bt, $J_{1,2}$ = 5.4 Hz, CH=CHC*H*$_2$NH), 1.98 (m, 2 H, CH$_2$C*H*$_2$CH=CH), 1.44 (s, 9 H, COOC(C*H*$_3$)$_3$), 1.32 (m, 2 H, CH$_3$C*H*$_2$CH$_2$), 0.89 (t, $J_{5,6}$ = 7.3 Hz, C*H*$_3$CH$_2$); ^{13}C NMR (50 MHz, $CDCl_3$) δ 155.53 (*C*OOC(CH$_3$)$_3$), 132.31 and 126.40 (*C*H=*C*H), 78.69 (CH=CH$_2$CH$_2$N), 42.32 (COO*C*(CH$_3$)$_3$), 34.00 (C-H$_3$CH$_2$CH$_2$), 28.13 (COOC(*C*H$_3$)$_3$), 22.02 (CH$_3$CH$_2$CH$_2$), 13.32 (*C*H$_3$CH$_2$CH$_2$); IR (neat) 3360, 1710 cm^{-1}; mass spectrum (CI), *m/z* (rel intensity) 199 (2, M$^+$), 143 (82, M$^+$ − C$_4$H$_8$), 100 (100, M + 1 − C$_4$H$_8$ − CO$_2$).

Anal. Calcd for $C_{11}H_{21}NO_2$: C, 66.29; H, 10.62. Found: C, 66.44; H, 10.68.

3-[(*tert*-Butoxycarbonyl)amino]-1-phenyl-1-propene (7g). Following the same procedure as above, **5g** (0.670 g, 2.01 mmol), methylene chloride (12 mL), and trifluoroacetic acid (0.329 g, 2.88 mmol) were stirred at 25 °C for 19 h. Workup as above provided 0.453 g (97%) of **7g** as a white solid: mp 83–85 °C; ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.30 (m, 5 H, Ar H), 6.48 (dt, $J_{2,3}$ = 15.8 Hz, $J_{1,3}$ = 1.5 Hz, PhC*H*=CHCH$_2$N), 6.16 (dt, $J_{2,3}$ = 15.9 Hz, $J_{1,2}$ = 6.1 Hz, PhCH=C*H*CH$_2$N), 4.71 (bs, 1 H, N*H*), 3.90 (bt, J = 5.3 Hz, PhCH=CHC*H*$_2$NH), 1.46 (s, 9 H, COOC(C*H*$_3$)$_3$); ^{13}C NMR (50 MHz, $CDCl_3$) δ 155.69 (*C*OOC(CH$_3$)$_3$), 136.61, 131.21, 128.43, 127.44, 126.30, 126.23, 79.29 (*C*H$_2$NH), 42.63 (COO*C*(CH$_3$)$_3$), 28.32 (COOC(*C*H$_3$)$_3$); IR (neat) 3190, 1690 cm^{-1}; mass spectrum (CI), *m/z* (rel intensity) 233 (2, M$^+$), 177 (64, M$^+$ − C$_4$H$_8$), 132 (38, M$^+$ − C$_4$H$_8$ − CO$_2$), 116 (100).

Anal. Calcd for $C_{14}H_{19}NO_2$: C, 72.07; H, 8.21. Found: C, 72.31; H, 7.95.

1-Amino-2(*E*),4(*E*)-hexadiene (8h). Into a 25-mL round-bottom flask equipped with a magnetic stirrer were added **7h** (0.217 g, 1.09 mmol) and ethyl ether (12 mL). Trifluoroacetic acid (0.248 g, 2.18 mmol) and concentrated HCl (5.0 μL) were added, and the solution was stirred at 23 °C for 30 h. The yellow-orange solution was poured into ether (30 mL) and extracted with saturated aqueous $NaHSO_4$ (4 × 10 mL). The aqueous portion was made basic by the addition of a cold, saturated solution of aqueous Na_2CO_3 and extracted with methylene chloride (6 × 20 mL). The organic portion was dried ($MgSO_4$) and concentrated in vacuo to provide 0.085 g (80%) of **8h** as a yellow oil with spectral properties consistent with those reported earlier.[8]

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Synthesis of 7,9-Diphenyl-8-tropyliumyl-8*H*-cyclopent[*a*]acenaphthylene Cation Having an Intramolecular Charge-Transfer Interaction and Its Transformation into the Sesquifulvalene Derivative

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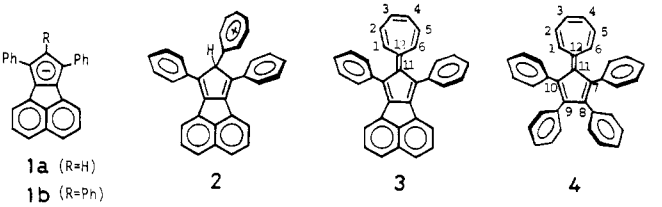
During the course of our study of phenyl-substituted cyclopent[*a*]acenaphthylenide ions **1a,b**,[2] reactivities toward various stable carbocations were investigated. This



1a (R=H) **1b** (R=Ph) **2** **3** **4**

note describes the syntheses and properties of the title cation **2**, obtained through the reaction of **1a** with tropylium ion ($C_7H_7^+$), and of the related sesquifulvalene derivative **3**. Although **3** can be looked upon as a homologue of the known sesquifulvalene **4**,[3] replacement of the two phenyl groups with a 1,8-naphthylene unit has been found

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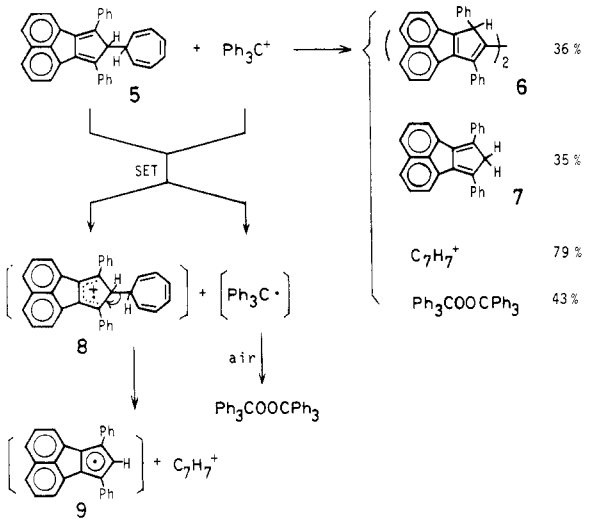
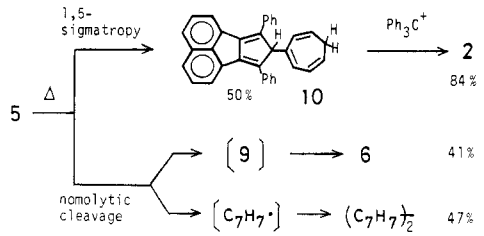
Scheme I



Scheme II



to bring about a considerable change in the properties of the cation **2** as compared with the cation formed by protonation of **4**.[3]

Reaction of **1a** with $C_7H_7^+$ in THF under vacuum afforded cycloheptatriene **5** as a single product. The attempted hydride abstraction directly from **5** using the trityl cation (Ph_3C^+) resulted in a reaction apparently involving some homolytic pathway (Scheme I). In view of the ease in oxidation of the cyclopent[*a*]acenaphthylene π-system (vide infra), this reaction is believed to be initiated by single-electron transfer (SET) from **5** to Ph_3C^+ followed by heterolytic cleavage of the cation radical **8** to $C_7H_7^+$ and **9**: radical **9** has already been shown to give dimer **6**[2] and would also abstract hydrogen from the solvent to give 7,9-diphenyl-8*H*-cyclopent[*a*]acenaphthylene (**7**).

In order to prevent this unfavorable cleavage, **5** was thermally isomerized at 230 °C for 0.5 min or at 150 °C for 2 h. The 1,5-hydrogen-shift product **10** was obtained together with comparable amounts of homolytic products (Scheme II). A similar competition of sigmatropy and homolytic dissociation has previously been observed in the thermolysis of 7-tritylcycloheptatriene.[4] Since the cation radical generated from **10** can not release $C_7H_7^+$, reaction of **10** with Ph_3C^+ smoothly afforded a salt of desired cation **2** as a black powder, which exhibits a deep blue color in polar solvents.

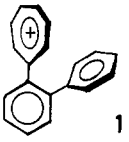
Although tropylium ions conjugated with a π-electronic system are known to absorb in the visible region,[5] it seems quite peculiar that the cation substituted with a saturated carbon such as **2** has a maximum absorption at such a long wavelength as 637 nm in CH_2Cl_2 for example. Furthermore, this absorption shows remarkable solvatochromism

Table I. Data for E_{ox} and the CT Band with $C_7H_7^+$

compd	E_{ox}[a]/V vs Ag/Ag^+	$\lambda_{CT\,max}$[b]/nm	$E_{CT\,max}$[b]/eV
7	$+0.67_5$	620	1.999
perylene	$+0.71_8$	610	2.032

[a] Anodic peak potentials (E_{pa}) by CV in CH_3CN at 0.1 V/s. [b] In CH_2Cl_2; [donor] = 2×10^{-2} M; $[C_7H_7^+] = 2 \times 10^{-3}$ M.

(see Experimental Section). In order to clarify the nature of this absorption, we examined the π-donor ability of **7** by determining the oxidation potential (E_{ox}) and charge-transfer (CT) transition energy with $C_7H_7^+$ as an acceptor. As shown by the data in Table I, **7** has an even stronger π-basicity than perylene, which exhibits a CT absorption with $C_7H_7^+$ at the longest wavelength among the polycyclic aromatic hydrocarbons so far reported.[6] Inspection of a molecular model indicates that the relative conformation of the central $C_7H_7^+$ ring and each of the two benzene rings resembles that in the case of 1-phenyl-2-tropyliumylbenzene (**11**).[7] Although each of the aromatic rings in **2**



11

and in **11** retains freedom of rotation, the presence of a through-space CT interaction has clearly been demonstrated for cation **11**.[7] Thus, all these facts taken together support assigning the longest wavelength absorption of **2** to the intramolecular CT interaction between the tropylium ring and the two benzene rings that constitute a part of the strong π-donor system.

Upon treatment with Et_3N, cation **2** was readily deprotonated to give sesquifulvalene derivative **3** as a fairly air-sensitive dark red-brown solid. In contrast to tetraphenyl derivative **4**, which has been reported to be protonated at C-7, giving the dienyl-conjugated tropylium ion,[3] protonation of **3** quantitatively regenerates cation **2**.

Comparison of the ^{13}C NMR chemical shifts for the seven-membered ring (δ 140.9, 136.3, and 133.1 for C-1 to C-6 and δ 149.6 for C-12) and C-11 (δ 132.3) in **3** with those reported for the parent sesquifulvalene[8] suggests that **3** is considerably more polarized than the latter. The electronic state of the seven-membered ring and its steric arrangement relative to the two benzene rings in **3** appear to be quite similar to those in **4**,[3] judging from the close resemblance of the ^1H NMR chemical shifts for the seven-membered-ring protons in both of these compounds.

Finally, the redox properties of **3** were examined by the use of cyclic voltammetry (CV). As shown in Figure 1, **3** exhibits two oxidation waves ($E_{1/2}$ = +0.348 V vs Ag/Ag^+ (reversible) and E_{pa} = +0.718 V (irreversible)) and one irreversible reduction wave (E_{pc} = −1.272 V). The observed difference in reversibility between the first oxidation and reduction waves is interpreted by the different stability of the produced cation radical **12** and anion radical **13** shown in Scheme III: in our previous study the fully substituted cyclopentadienyl radical, which is formed from anion **1b** and is homologous to **12**, has been shown to be persistent,[2] whereas cation **2** in the present study is irreversibly reduced at E_{pc} = −0.532 V due to the facile dimerization of the monosubstituted cycloheptatrienyl radical similar to **13**.

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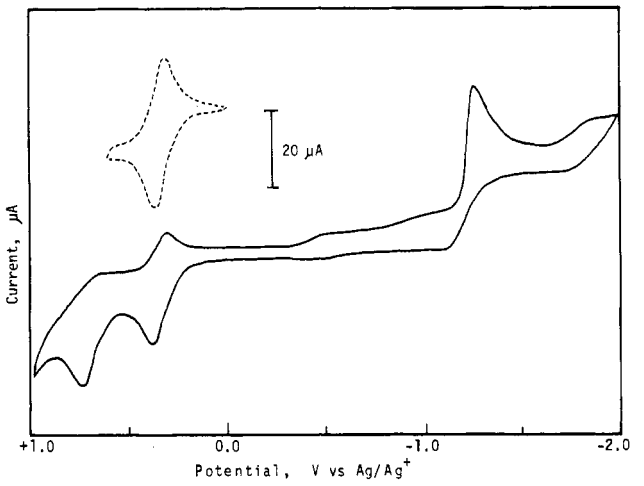
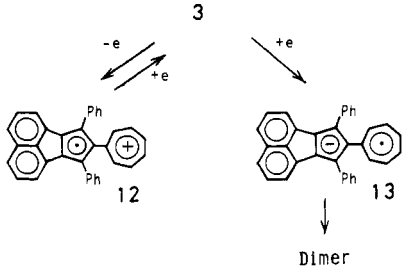


Figure 1. Cyclic voltammogram of 10^{-3} M **3** in MeCN (0.1 M $Bu_4N^+ClO_4^-$) at 0.1 V/s. The dashed inset represents the scan between 0.0 and +0.60 V.

Scheme III



Experimental Section

General. Elemental analyses were performed by the Microanalytical Center, Kyoto University, Kyoto. IR and UV–visible spectra were recorded on Hitachi 215 and Hitachi 200-10 spectrometers, respectively. ^1H NMR spectra were taken on a JEOL GX-400 (400 MHz) or a Hitachi R-24 (60 MHz) spectrometer. ^{13}C NMR spectra were taken on a JEOL FX-100 spectrometer (25 MHz). Cyclic voltammetry was performed as has previously been described.[9]

7,9-Diphenyl-8-(2,4,6-cycloheptatrienyl)-8*H*-cyclopent[*a*]acenaphthylene (5). Hydrocarbon **7**[2] (0.400 g, 1.17 mmol), *t*-BuOK (0.170 g, 1.52 mmol), and a stirring bar were placed in a flask having a side arm containing $C_7H_7^+BF_4^-$ (0.313 g, 1.76 mmol). The flask was connected to a vacuum line and evacuated. After THF (12 mL) was vacuum distilled into the flask to generate the anion **1a**, $C_7H_7^+BF_4^-$ was added from the side arm to the stirred solution of **1a** under vacuum. The mixture was stirred for 1 h and then treated with water and extracted with benzene. Recrystallization of the crude product from benzene afforded **5** (0.369 g, 73.0%) as yellow plates: mp 211–212 °C; IR (KBr) 3045, 3005, 1598, 1490, 1440, 1400, 1353, 827, 780, 740, 700 cm^{-1}; UV–vis (THF) λ_{max} (log ϵ) 250 (4.60), 280 sh (4.43), 386 nm (4.29); ^1H NMR ($CDCl_3$, 400 MHz) δ 7.82 (2 H, d, H-1,6), 7.70 (2 H, d, H-3,4), 7.67 (4 H, d, ortho H), 7.45 (6 H, dd and t, H-2,5 and meta H), 7.32 (2 H, t, para H), 6.36 (2 H, t, H-4′,5′), 5.90 (2 H, dm, H-3′,6′), 5.12 (2 H, dd, H-2′,7′), 5.11 (1 H, d, H-8), 2.05 (1 H, m, H-1′); ^{13}C NMR ($CDCl_3$) δ 147.5 (1 C, s), 144.3 (2 C, s), 140.1 (2 C, s), 136.5 (2 C, s), 132.4 (2 C, s), 131.8 (1 C, s), 130.4 (2 C, d), 128.5 (4 C, d), 128.4 (4 C, d), 127.6 (2 C, d), 127.4 (2 C, d), 125.5 (2 C, d), 124.5 (2 C, d), 123.7 (2 C, d), 118.7 (2 C, d), 62.6 (1 C, d), 40.4 (1 C, d). Anal. Calcd for $C_{34}H_{24}$: C, 94.41; H, 5.59. Found: C, 94.13; H, 5.48.

Reaction of 5 with Trityl Perchlorate. To a stirred solution of **5** (0.0480 g, 0.111 mmol) in CH_2Cl_2 (1.5 mL) and MeCN (1.5 mL) was added $Ph_3C^+ClO_4^-$ (0.0492 g, 0.143 mmol) under nitrogen. After 20 min of stirring, ether (30 mL) was added to cause the formation of a white precipitate, which was filtered and dried to give $C_7H_7^+ClO_4^-$ (0.0168 g, 79.4%). The filtrate was evaporated and separated by the use of medium-pressure liquid chromatography (MPLC) (hexane (100%) to hexane–benzene (50%:50%)/Merck SiO_2 60) to give **7** (0.0134 g, 35.1%), **6**[2] (0.0136 g, 35.9%), and $Ph_3COOCPh_3$ (0.0124 g, 43.1%).

Thermal Isomerization of 5. A solution of **5** (0.612 g, 1.41 mmol) in *p*-xylene (7 mL) was sealed in a Pyrex glass tube under vacuum and was heated in an oil bath at 150 °C for 2 h. A mixture of the approximately same composition of products was obtained when the same amount of **5** was heated at 230 °C for 0.5 min in a vacuum-sealed tube without solvent. The latter products were separated by MPLC as described above to give bitropyl (0.0599 g, 46.6%), **6** (0.198 g, 41.2%), and **10** (0.302 g, 49.6%): IR (KBr) 3045, 3005, 1598, 1490, 1440, 1390, 1355, 1025, 903, 825, 778, 740, 700 cm^{-1}; UV–vis (THF) λ_{max} (log ϵ) 252 (4.56), 283 (4.42), 396 nm (4.28); ^1H NMR ($CDCl_3$, 60 MHz) δ 8.00–7.20 (16 H, m), 6.60 (1 H, d), 5.93 (1 H, dd), 5.73 (1 H, d), 5.23 (1 H, s, H-8), 5.10 (2 H, m), 1.67 (2 H, t); ^{13}C NMR ($CDCl_3$) δ 144.4 (1 C, s), 143.9 (2 C, s), 141.6 (1 C, s), 140.6 (2 C, s), 135.7 (2 C, s), 132.5 (2 C, s), 131.9 (2 C, d), 131.2 (1 C, d), 128.2 (4 C, d), 128.1 (4 C, d), 127.6 (2 C, d), 127.2 (2 C, d), 126.1 (1 C, d), 125.5 (1 C, d), 125.4 (1 C, d), 122.2 (1 C, s), 121.1 (1 C, d), 119.0 (2 C, d), 70.8 (1 C, d), 27.3 (1 C, t).

7,9-Diphenyl-8-tropyliumyl-8*H*-cyclopent[*a*]acenaphthylene Tetrafluoroborate (2·BF_4^-). A solution of $Ph_3C^+BF_4^-$ (0.190 g, 0.576 mmol) in MeCN (1 mL) was added dropwise to a stirred solution of **10** (0.223 g, 0.516 mmol) in CH_2Cl_2 (5 mL) at 0 °C under nitrogen. After 0.5 h of stirring, ether (80 mL) was added. The resulting precipitates were filtered and washed with ether to give **2**·BF_4^- (0.225 g, 84.1%) as a black powder: mp 171 °C (dec); IR (KBr) 3050, 1600, 1520, 1485, 1442, 1410, 1260, 1080, 830, 800, 780, 760, 700 cm^{-1}; UV–vis (CH_2Cl_2) λ_{max} (log ϵ) 237 (4.65), 253 sh (4.55), 284 (4.41), 385 (4.18), 637 nm (3.83); UV–vis (MeCN) λ_{max} (log ϵ) 234 (4.65), 248 sh (4.56), 280 (4.43), 384 (4.20), 500 (3.64), 590 sh nm (3.56); ^1H NMR (CD_3CN, 400 MHz) δ 8.97 (2 H, br m, $-C_7H_6^+$), 8.74 (4 H, br m, $-C_7H_6^+$), 8.08 (2 H, d, H-1,6), 7.93 (2 H, d, H-3,4), 7.79 (4 H, d, ortho H), 7.63 (2 H, dd, H-2,5), 7.45 (4 H, t, meta H), 7.32 (2 H, t, para H), 6.37 (1 H, s, H-8); ^{13}C NMR (CD_3CN) δ 174.0 (1 C, s), 156.1 (2 C, d), 154.7 (2 C, d), 154.3 (2 C, d), 147.9 (2 C, s), 145.7 (2 C, s), 145.3 (1 C, s), 134.3 (2 C, s), 132.9 (1 C, s), 131.7 (2 C, s), 130.2 (4 C, d), 129.7 (2 C, d), 129.2 (2 C, d), 128.8 (4 C, d), 128.2 (2 C, d), 121.5 (2 C, d), 70.7 (1 C, d). Anal. Calcd for $C_{34}H_{23}BF_4$: C, 78.78; H, 4.47. Found: C, 78.29; H, 4.33.

7,9-Diphenyl-8-cycloheptatrienylidene-8*H*-cyclopent[*a*]acenaphthylene (3). To a solution of **2**·BF_4^- (0.0300 g, 0.0579 mmol) in MeCN (0.4 mL) was added Et_3N (0.012 g, 0.12 mmol) by the use of a microsyringe. The resulting precipitates were filtered and washed with MeCN (0.1 mL) and with pentane (0.2 mL × 2) to give **3** (0.0172 g, 69.0%) as a dark red-brown solid: mp 176–179 °C; IR (KBr) 3050, 1625, 1600, 1520, 1485, 1440, 1410, 1260, 1198, 830, 780, 720, 700 cm^{-1}; UV–vis (CH_2Cl_2) λ_{max} (log ϵ) 258 (4.40), 299 sh (4.07), 403 (4.30) 468 nm (4.14); UV–vis (MeCN) λ_{max} (log ϵ) 255 (4.46), 295 sh (4.15), 399 (4.35), 469 nm (4.18); ^1H NMR ($CDCl_3$, 400 MHz) δ 7.58 (4 H, d, ortho H), 7.53 (2 H, dd, H-2,5), 7.46 (4 H, t, meta H), 7.34 (2 H, t, para H), 7.29 (2 H, d, H-1,6), 7.28 (2 H, d, H-3,4), 6.66 (2 H, d, J = 11.8 Hz, H-2′,7′), 6.17 (2 H, dd, J = 5.4 and 3.6 Hz, H-4′,5′), 5.81 (2 H, ddd, J = 11.8, 5.4, and 3.6 Hz, H-3′,6′); ^{13}C NMR ($CDCl_3$) δ 149.6 (1 C, s), 143.4 (1 C, s), 140.9 (2 C, d), 139.5 (2 C, s), 136.3 (2 C, d), 134.4 (1 C, s), 133.1 (2 C, d), 132.3 (1 C, s), 129.9 (4 C, d), 129.7 (2 C, d), 129.1 (2 C, s), 128.7 (4 C, d), 127.6 (2 C, d), 126.6 (2 C, s), 125.3 (2 C, d), 118.5 (2 C, d). Anal. Calcd for $C_{34}H_{22}$: C, 94.85; H, 5.15. Found: C, 93.52; H, 5.16. Satisfactory analysis could not be obtained due to the air sensitivity of **3**.

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