SUPPORTING INFORMATION FOR

**A Remarkably Efficient Synthesis of Pure *cis*-Stilbenoid Hydrocarbons
Using *trans*-Dibromoalkenes via Palladium Catalysis**

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The procedures for the preparation of various intermediates **11a**, **11b**, **12a**, and **12b** in Scheme 1 are described in detail. The spectral data for various *purified stilbenoid hydrocarbons* **1-10**, **13a**, and **13b**; and the X-ray crystal structure data for **6** and **7** are included.

Experimental Section

Various *trans*-dibromoalkenes, utilized in this study, were prepared by bromination of the corresponding disubstituted acetylenes in acetic acid using bromine, according to a reported procedure,^a and were purified by distillation/crystallization. Spectral data and yields of purified stilbenoid hydrocarbons **1-10**, **13a** and **13b**, prepared according the general procedure described in the text, are given as follows:

3,4-Bis(2,6-dimethylphenyl)hex-3-ene (1). Yield: 96 %; ¹H NMR (CDCl₃) δ 1.04 (t, 6H), 2.12 (s, 12H), 2.53 (q, 4H), 6.80 (d, 4H), 6.89 (t, 2H); ¹³C NMR (CDCl₃) δ 13.33, 21.02, 27.95, 125.79, 127.19, 135.87, 138.69, 141.14. GC-MS: m/z = 292 (M⁺) calcd. for C₂₂H₂₈. **3,4-Bis(2,4,6-trimethylphenyl)hex-3-ene (2).** Yield: 98 %; mp 161-162 °C (ethanol); ¹H NMR (CDCl₃) δ 1.05 (t, 6H), 2.10 (s, 12H), 2.18 (s, 6H), 2.50 (q, 4H), 6.64 (s, 4H); ¹³C NMR (CDCl₃) δ 14.23, 21.57, 21.67, 28.90, 127.81, 134.56, 135.37, 138.11, 138.23. GC-MS: m/z = 320 (M⁺) calcd. for C₂₄H₃₂. **3,4-Bis(2,6-dimethylanisyl)hex-3-ene (3).** Yield: 96 %; mp 121-124 °C (ethanol); ¹H NMR (CDCl₃) δ 1.05 (t, 6H), 2.11 (s, 12H), 2.49 (q, 4H), 3.71 (s, 6H), 6.39 (s, 4H); ¹³C NMR (CDCl₃) δ 14.16, 22.04, 28.90, 55.20, 112.32, 133.73, 136.91, 138.27, 156.49. GC-MS: m/z = 352 (M⁺) calcd. for C₂₄H₃₂O₂. **3,4-Bis(2,3,5,6-tetramethylphenyl)hex-3-ene (4).** Yield: 97 %; mp 133-134 °C (ethanol); ¹H NMR (CDCl₃) δ 1.08 (t, 6H), 1.95 (s, 12H), 2.06 (s, 12H), 2.56 (q, 4H), 6.69 (s, 2H); ¹³C NMR (CDCl₃) δ 13.41, 18.86, 26.71, 20.78, 29.27, 120.20, 131.86, 133.20, 139.26, 141.88. GC-MS: m/z = 348 (M⁺) calcd. for C₂₆H₃₆. **3,4-Bis(pentamethylphenyl)hex-3-ene (5).** Yield: 98 %; mp 171-174 °C (ethanol), ¹H NMR (CDCl₃) δ 1.18 (t, *J* = 7.5 Hz, 6H), 2.13 (s, 24H), 2.20 (s, 6H), 2.62 (q, *J* = 7.5 Hz, 4H); ¹³C NMR (CDCl₃) δ 13.47, 26.63, 26.71, 29.88, 29.57, 131.33, 131.82, 132.20, 139.56, 139.68.

GC-MS: $m/z = 376$ (M^+) calcd. for $C_{28}H_{40}$. **4,5-Bis(pentamethylphenyl)oct-4-ene (6)**. Yield: 94 %; mp 182-184 °C (ethanol); 1H NMR ($CDCl_3$) δ 0.97 (t, 6H), 1.55 (sym m, 4H), 2.03 (s, 12H), 2.05 (s, 12H), 2.14 (s, 6H), 2.45 (sym m, 4H); ^{13}C NMR ($CDCl_3$) δ 15.84, 17.22, 17.35, 20.44, 22.95, 39.84, 130.78, 131.29, 131.64, 138.37, 139.25. GC-MS: $m/z = 404$ (M^+) calcd. for $C_{30}H_{44}$. **1,2-Bis(pentamethylphenyl)-1-phenylhex-1-ene (7)**. Yield: 92 %; mp 191-192 °C (ethanol); 1H NMR ($CDCl_3$) δ 0.95 (t, 3H), 1.35 (sym m, 2H), 1.66 (sym m, 2H), 2.02 (s, 6H), 2.07 (s, 6H), 2.08 (s, 6H), 2.09 (s, 6H), 2.12 (s, 3H), 2.17 (s, 3H), 2.57 (sym m, 2H), 7.14-7.36 (m, 5H) ; ^{13}C NMR ($CDCl_3$) δ 14.78, 17.22, 17.41, 17.38, 20.72, 21.00, 23.98, 32.86, 37.80, 125.40, 127.14, 128.97, 130.65, 131.23, 131.32, 131.42, 131.88, 132.05, 138.47, 138.51, 138.77, 141.75, 142.78. GC-MS: $m/z = 452$ (M^+) calcd. for $C_{34}H_{44}$. **1,2-Bis(2,6-dimethylphenyl)stilbene (8)**. Yield: 96 %; mp 121-122 °C (ethanol-dichloromethane); 1H NMR ($CDCl_3$) δ 2.09 (s, 12H), 6.92-7.18 (m, 16H); ^{13}C NMR ($CDCl_3$) δ 17.22, 124.32, 127.10, 128.78, 130.70, 131.29, 133.81, 136.00, 139.53, 141.25. GC-MS: $m/z = 388$ (M^+) calcd. for $C_{30}H_{28}$. **1,2-Bis(pentamethylphenyl)stilbene (9)**. Yield: 91 %; mp 157-159 °C (ethanol-dichloromethane); 1H NMR ($CDCl_3$) δ 2.05 (s, 12H), 2.12 (s, 6H), 2.15 (s, 12H), 7.00-7.15 (m, 10H); ^{13}C NMR ($CDCl_3$) δ 16.46, 16.68, 20.07, 125.68, 127.22, 130.98, 131.79, 131.85, 132.83, 139.00, 141.59, 143.35. GC-MS: $m/z = 472$ (M^+) calcd. for $C_{36}H_{40}$. **1,2-Bis(pentamethylphenyl)-1,2-bis(4-methoxyphenyl)ethylene (10)**. Yield: 92%; mp 218-221 °C (ethanol-dichloromethane); 1H NMR ($CDCl_3$) δ 2.04 (s, 12H), 2.12 (s, 12H), 2.13 (s, 6H), 3.76 (s, 6H), 6.62 (d, 4H), 6.93 (d, 4H); ^{13}C NMR ($CDCl_3$) δ 17.20, 17.42, 20.73, 55.47, 112.52, 131.35, 131.48, 131.60, 132.23, 135.67, 138.88, 139.64, 156.75. GC-MS: $m/z = 532$ (M^+) calcd. for $C_{38}H_{44}O_2$.

1,4-Bis(1-octynyl)benzene (11a). To a stirred solution of ethylmagnesium bromide in tetrahydrofuran (45 mL, 1M) was added dropwise a solution of 1-octyne (6.6 mL, 45 mmol) in tetrahydrofuran (20 mL), during a period of 10 minutes, under an argon atmosphere at room temperature. To this mixture was added 1,4-diiodobenzene (4.95 g, 15 mmol) and Pd(PPh₃)₄ (1.0 g, ~1 mmol). The resulting yellow mixture was stirred at 50 °C for 12 h and was poured into water (100 mL) after cooling to room temperature. The organic layer was separated and the aqueous layer was extracted with dichloromethane (3 x 50 mL). Combined organic layers were dried over anhydrous magnesium sulfate and solvent was evaporated *in vacuo*. The resulting oily product was purified by flash chromatography on silica gel using hexanes-ethyl acetate (9:1) mixture as eluent. Yield: 75%; ¹H NMR (CDCl₃) δ 0.95 (t, 6H), 1.35 (sym m, 8H), 1.49 (sym m, 4H), 1.63 (sym m, 4H), 2.43 (t, 4H), 7.31 (s, 4H); ¹³C NMR (CDCl₃) δ 14.86, 20.21, 23.27, 29.26, 29.32, 31.99, 80.53, 92.01, 131.01, 131.21. GC-MS: m/z = 294 (M⁺) calcd. for C₂₂H₃₀. [Note that the preparation of the *bis*-acetylenic compounds is based on the procedure of Negishi et. al.^b, which we adapted for the preparation of **11a** and **11b**.]

4,4'-Bis(1-octynyl)biphenyl (11b). The crystalline **11b** was prepared as above. Yield: 85%; mp 74 °C (ethanol-dichloromethane); ¹H NMR (CDCl₃) δ 0.94 (t, 6H), 1.36 (sym m, 8H), 1.49 (sym m, 4H), 1.64 (sym m, 4H), 2.45 (t, 4H), 7.48 (d, *J* = 8.4 Hz, 4H), 7.51 (d, *J* = 8.4 Hz, 4H); ¹³C NMR (CDCl₃) δ 14.89, 20.26, 23.30, 29.29, 29.39, 32.03, 80.53, 91.51, 123.02, 126.40, 131.66, 139.03. GC-MS: m/z = 370 (M⁺) calcd. for C₂₈H₃₄.

4,4'-Bis(1,2-dibromo-1-octenyl)benzene (11a). The bromination of **11a** was performed in acetic acid, according to a literature procedure,^a and the resulting oily product was purified by flash chromatography on silica gel using hexanes-ethyl acetate (7:3) mixture as eluent. Yield: 68%; ¹H NMR (CDCl₃) δ 0.96 (t, 6H), 1.37 (sym m, 12H), 1.73 (sym m, 4H), 2.87 (t, 4H), 7.63 (s, 4H); ¹³C NMR (CDCl₃) δ 14.90, 23.32, 28.10, 28.94, 32.25, 41.79,

115.30, 123.87, 128.84, 140.38. $m/z = 614$ (M^+) calcd. for $C_{22}H_{30}Br_4$. **4,4'-Bis(1,2-dibromo-1-octenyl)biphenyl (12b)**. The crystalline **12b** was obtained as above from a bromination of **12b**. Yield: 88%; mp 70 °C (ethanol-dichloromethane); 1H NMR ($CDCl_3$) δ 0.96 (t, 6H), 1.41 (sym m, 12H), 1.74 (sym m, 4H), 2.88 (t, 4H), 7.44 (d, $J = 8.4$ Hz, 4H), 7.60 (d, $J = 8.4$ Hz, 4H); ^{13}C NMR ($CDCl_3$) δ 14.91, 23.31, 28.13, 28.95, 32.25, 41.78, 115.66, 123.70, 126.66, 129.42, 139.74, 140.06. $m/z = 690$ (M^+) calcd. for $C_{28}H_{34}Br_4$. **4,4'-Bis(1,2-bis(pentamethylphenyl)-1-octenyl)benzene (13a)**. The bichromophoric ligand **13a** was prepared according to the general procedure described in the text. Yield: 74%; mp 247-250 °C (ethanol-dichloromethane); 1H NMR ($CDCl_3$) δ 0.90 (t, 6H), 1.31 (sym m, 12H), 1.64 (m, 4H), 1.99 (s, 12H), 2.01 (s, 12H), 2.06 (s, 12H), 2.07 (s, 12H), 2.09 (s, 6H), 2.15 (s, 6H), 2.60 (sym m, 4H), 7.20 (s, 4H); ^{13}C NMR ($CDCl_3$) δ 14.20, 16.48, 16.90, 19.97, 20.31, 22.90, 30.01, 30.45, 31.84, 37.63, 128.45, 131.05, 131.51, 131.75, 132.16, 132.33, 138.90, 139.20, 139.36, 140.52, 142.03. $m/z = 882$ (M^+ using MALDI) calcd. for $C_{66}H_{90}$. **4,4'-Bis(1,2-bis(pentamethylphenyl)-1-octenyl)biphenyl (13b)**. The bichromophoric ligand **13b** containing biphenyl as a spacer was prepared according to the general procedure described in the text. Yield: 72%; mp 235-237 °C (ethanol-dichloromethane); 1H NMR ($CDCl_3$) δ 0.92 (t, 6H), 1.33 (sym m, 12H), 1.66 (m, 4H), 2.01 (s, 12H), 2.07 (s, 24H), 2.09 (s, 12H), 2.12 (s, 6H), 2.16 (s, 6H), 2.59 (sym m, 4H), 7.35 (d, $J = 8.7$ Hz, 4H), 7.53 (d, $J = 8.7$ Hz, 4H); ^{13}C NMR ($CDCl_3$) δ 14.97, 17.22, 17.41, 20.74, 21.01, 23.55, 30.58, 30.80, 32.38, 38.18, 125.58, 129.33, 130.65, 131.26, 131.37, 131.42, 131.87, 132.09, 137.51, 138.20, 138.56, 138.73, 141.63, 142.01. $m/z = 958$ (M^+ using MALDI) calcd. for $C_{72}H_{94}$.

X-ray Crystallography. The intensity data were collected on a Bruker CCD-1000 diffractometer with Mo K $_{\alpha}$ ($\lambda = 0.71073 \text{ \AA}$) radiation at 173(2) K. The structures were solved by direct methods^c and refined by full matrix least-squares procedure. [The details of the X-ray structures of the stilbenoid hydrocarbons **6** [CCDC Deposition No. 169899] and **7** [CCDC Deposition No. 169900] are on deposit and can be obtained from Cambridge Crystallographic Data Center, U.K.] *Crystal Data for 4,5-Bis(pentamethylphenyl)oct-4-ene (6) [C₃₀H₄₄].* A suitable crystal with approximate dimensions (0.5 x 0.3 x 0.3 mm³) was obtained from a mixture of ethanol-dichloromethane solution of stilbenoid hydrocarbon **6**. MW = 404.65, triclinic, space group P1, $a = 8.7774(9)$, $b = 10.2972(10)$, and $c = 13.6424(13) \text{ \AA}$, $\alpha = 92.513(2)^{\circ}$, $\beta = 90.577(1)^{\circ}$, $\gamma = 94.008(2)^{\circ}$, $D_c = 1.094 \text{ Mg m}^{-3}$, $V = 1228.6(2) \text{ \AA}^3$, $Z = 2$. The final least-squares refinement of 283 parameters against 4620 data resulted in residuals R (based on F^2 for $I \geq 2\sigma$) and wR (based on F^2 for all data) of 0.0483 and 0.1307, respectively. *Crystal Data 1,2-Bis(pentamethylphenyl)-1-phenylhex-1-ene (7) [C₃₄H₄₄].* An X-ray quality crystal (0.5 x 0.4 x 0.4 mm) of **7** was obtained by crystallization from ethanol/dichloromethane at 0 °C. MW = 452.69, triclinic, space group P-1, $a = 8.4910(9)$, $b = 9.8941(10)$, and $c = 16.8648(17) \text{ \AA}$, $\alpha = 98.979(2)^{\circ}$, $\beta = 100.758(2)^{\circ}$, $\gamma = 91.471(2)^{\circ}$, $D_c = 1.095 \text{ Mg m}^{-3}$, $V = 1372.7(2) \text{ \AA}^3$, $Z = 2$. The final least-squares refinement of 318 parameters against 5560 data resulted in residuals R (based on F^2 for $I \geq 2\sigma$) and wR (based on F^2 for all data) of 0.097 and 0.2721, respectively.

Tables of X-ray structure data, coordinates, bond lengths and bond angles for **6** and **7** are given below:

Table 1. Crystal data and structure refinement for **6**.

Identification code	ratha	
Empirical formula	C ₃₀ H ₄₄	
Formula weight	404.65	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P $\bar{1}$	
Unit cell dimensions	a = 8.7774(9) Å	α = 92.513(2)°.
	b = 10.2972(10) Å	β = 90.930(2)°.
	c = 13.6424(13) Å	γ = 94.008(2)°.
Volume	1228.6(2) Å ³	
Z	2	
Density (calculated)	1.094 Mg/m ³	
Absorption coefficient	0.061 mm ⁻¹	
F(000)	448	
Crystal size	0.50 x 0.30 x 0.30 mm ³	
Theta range for data collection	1.98 to 26.38°.	
Index ranges	-10 ≤ h ≤ 10, -12 ≤ k ≤ 12, 0 ≤ l ≤ 16	
Reflections collected	7175	
Independent reflections	4620 [R(int) = 0.0169]	
Completeness to theta = 26.38°	92.1 %	
Absorption correction	Empirical with SADABS	
Max. and min. transmission	0.9820 and 0.9703	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	4620 / 0 / 283	
Goodness-of-fit on F ²	1.018	
Final R indices [I > 2sigma(I)]	R1 = 0.0483, wR2 = 0.1307	
R indices (all data)	R1 = 0.0615, wR2 = 0.1393	
Largest diff. peak and hole	0.298 and -0.199 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for ratha (**6**). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	3053(2)	7009(1)	3552(1)	27(1)
C(2)	2439(2)	7964(2)	4165(1)	29(1)
C(3)	3253(2)	8463(2)	5012(1)	33(1)
C(4)	4671(2)	8009(2)	5255(1)	33(1)
C(5)	5288(2)	7058(2)	4643(1)	32(1)
C(6)	4474(2)	6546(2)	3801(1)	29(1)
C(7)	894(2)	8445(2)	3934(1)	36(1)
C(8)	2571(2)	9485(2)	5674(1)	45(1)
C(9)	5512(2)	8533(2)	6187(1)	48(1)
C(10)	6836(2)	6569(2)	4861(1)	42(1)
C(11)	5166(2)	5489(2)	3182(1)	34(1)
C(12)	2220(2)	6385(1)	2655(1)	27(1)
C(13)	1511(2)	5023(2)	2815(1)	31(1)
C(14)	31(2)	5060(2)	3386(1)	41(1)
C(15)	-711(2)	3710(2)	3561(2)	47(1)
C(16)	2123(2)	6964(1)	1792(1)	27(1)
C(17)	1592(2)	6245(2)	833(1)	30(1)
C(18)	2951(2)	5928(2)	207(1)	36(1)
C(19)	2492(2)	5305(2)	-797(1)	49(1)
C(20)	2529(2)	8396(1)	1660(1)	26(1)
C(21)	1311(2)	9195(2)	1524(1)	29(1)
C(22)	1602(2)	10519(2)	1347(1)	31(1)
C(23)	3113(2)	11048(2)	1279(1)	31(1)
C(24)	4325(2)	10251(2)	1404(1)	29(1)
C(25)	4038(2)	8929(2)	1596(1)	27(1)
C(26)	-342(2)	8659(2)	1574(1)	35(1)
C(27)	254(2)	11351(2)	1241(1)	42(1)
C(28)	3432(2)	12476(2)	1080(1)	43(1)
C(29)	5962(2)	10797(2)	1322(1)	39(1)
C(30)	5365(2)	8087(2)	1718(1)	33(1)

Table 3. Bond lengths [Å] and angles [°] for ratha (**6**).

C(1)-C(2)	1.405(2)	C(16)-C(20)	1.512(2)
C(1)-C(6)	1.407(2)	C(16)-C(17)	1.524(2)
C(1)-C(12)	1.511(2)	C(17)-C(18)	1.524(2)
C(2)-C(3)	1.408(2)	C(18)-C(19)	1.523(2)
C(2)-C(7)	1.508(2)	C(20)-C(25)	1.403(2)
C(3)-C(4)	1.400(2)	C(20)-C(21)	1.409(2)
C(3)-C(8)	1.518(2)	C(21)-C(22)	1.402(2)
C(4)-C(5)	1.402(2)	C(21)-C(26)	1.519(2)
C(4)-C(9)	1.519(2)	C(22)-C(23)	1.404(2)
C(5)-C(6)	1.406(2)	C(22)-C(27)	1.517(2)
C(5)-C(10)	1.511(2)	C(23)-C(24)	1.401(2)
C(6)-C(11)	1.515(2)	C(23)-C(28)	1.515(2)
C(12)-C(16)	1.347(2)	C(24)-C(25)	1.404(2)
C(12)-C(13)	1.520(2)	C(24)-C(29)	1.514(2)
C(13)-C(14)	1.528(2)	C(25)-C(30)	1.511(2)
C(14)-C(15)	1.524(2)		
C(2)-C(1)-C(6)	119.45(14)	C(12)-C(16)-C(20)	124.11(13)
C(2)-C(1)-C(12)	123.12(13)	C(12)-C(16)-C(17)	123.65(13)
C(6)-C(1)-C(12)	117.34(13)	C(20)-C(16)-C(17)	112.24(12)
C(1)-C(2)-C(3)	119.99(14)	C(16)-C(17)-C(18)	110.85(12)
C(1)-C(2)-C(7)	120.54(14)	C(19)-C(18)-C(17)	113.35(14)
C(3)-C(2)-C(7)	119.45(14)	C(25)-C(20)-C(21)	119.55(14)
C(4)-C(3)-C(2)	120.47(14)	C(25)-C(20)-C(16)	123.18(13)
C(4)-C(3)-C(8)	120.00(15)	C(21)-C(20)-C(16)	117.09(13)
C(2)-C(3)-C(8)	119.52(15)	C(22)-C(21)-C(20)	120.27(14)
C(3)-C(4)-C(5)	119.64(14)	C(22)-C(21)-C(26)	118.25(14)
C(3)-C(4)-C(9)	119.70(15)	C(20)-C(21)-C(26)	121.48(14)
C(5)-C(4)-C(9)	120.66(16)	C(21)-C(22)-C(23)	120.05(14)
C(4)-C(5)-C(6)	120.15(15)	C(21)-C(22)-C(27)	118.36(14)
C(4)-C(5)-C(10)	121.37(15)	C(23)-C(22)-C(27)	121.59(14)
C(6)-C(5)-C(10)	118.48(15)	C(24)-C(23)-C(22)	119.69(14)
C(5)-C(6)-C(1)	120.27(14)	C(24)-C(23)-C(28)	120.13(14)
C(5)-C(6)-C(11)	118.14(14)	C(22)-C(23)-C(28)	120.18(15)
C(1)-C(6)-C(11)	121.58(14)	C(23)-C(24)-C(25)	120.41(14)
C(16)-C(12)-C(1)	123.73(13)	C(23)-C(24)-C(29)	120.57(14)
C(16)-C(12)-C(13)	122.92(14)	C(25)-C(24)-C(29)	119.01(14)
C(1)-C(12)-C(13)	113.35(12)	C(20)-C(25)-C(24)	120.02(14)
C(12)-C(13)-C(14)	111.72(13)	C(20)-C(25)-C(30)	120.62(14)
C(15)-C(14)-C(13)	113.11(15)	C(24)-C(25)-C(30)	119.35(13)

Symmetry transformations used to generate equivalent atoms

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for ratha (6). The anisotropic Displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
C(1)	29(1)	27(1)	25(1)	4(1)	3(1)	-1(1)
C(2)	31(1)	30(1)	28(1)	4(1)	6(1)	0(1)
C(3)	38(1)	31(1)	28(1)	1(1)	7(1)	-2(1)
C(4)	38(1)	35(1)	24(1)	3(1)	1(1)	-7(1)
C(5)	30(1)	37(1)	28(1)	7(1)	1(1)	-2(1)
C(6)	30(1)	31(1)	26(1)	4(1)	3(1)	0(1)
C(7)	36(1)	36(1)	37(1)	0(1)	6(1)	7(1)
C(8)	53(1)	42(1)	39(1)	-8(1)	8(1)	1(1)
C(9)	55(1)	52(1)	34(1)	-4(1)	-6(1)	-3(1)
C(10)	36(1)	55(1)	36(1)	4(1)	-7(1)	3(1)
C(11)	32(1)	37(1)	34(1)	1(1)	-1(1)	8(1)
C(12)	23(1)	29(1)	28(1)	-1(1)	3(1)	4(1)
C(13)	34(1)	30(1)	30(1)	1(1)	-1(1)	2(1)
C(14)	39(1)	36(1)	46(1)	0(1)	9(1)	-6(1)
C(15)	44(1)	44(1)	52(1)	6(1)	7(1)	-9(1)
C(16)	19(1)	32(1)	28(1)	0(1)	1(1)	1(1)
C(17)	27(1)	32(1)	29(1)	1(1)	-3(1)	-2(1)
C(18)	32(1)	40(1)	34(1)	-5(1)	2(1)	-4(1)
C(19)	42(1)	63(1)	39(1)	-14(1)	4(1)	-6(1)
C(20)	26(1)	31(1)	21(1)	0(1)	0(1)	0(1)
C(21)	27(1)	34(1)	25(1)	1(1)	-1(1)	0(1)
C(22)	33(1)	34(1)	25(1)	0(1)	0(1)	4(1)
C(23)	38(1)	30(1)	24(1)	0(1)	3(1)	-1(1)
C(24)	30(1)	35(1)	22(1)	-2(1)	1(1)	-4(1)
C(25)	25(1)	33(1)	21(1)	-1(1)	-1(1)	0(1)
C(26)	27(1)	38(1)	41(1)	3(1)	-1(1)	2(1)
C(27)	42(1)	37(1)	49(1)	6(1)	0(1)	7(1)
C(28)	44(1)	35(1)	49(1)	5(1)	10(1)	0(1)
C(29)	35(1)	40(1)	40(1)	2(1)	2(1)	-9(1)
C(30)	25(1)	39(1)	34(1)	1(1)	0(1)	-1(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for ratha (**6**).

	x	y	z	U(eq)
H(7A)	413	7915	3384	54
H(7B)	1018	9357	3756	54
H(7C)	249	8373	4511	54
H(8A)	2100	9068	6237	67
H(8B)	1794	9912	5306	67
H(8C)	3378	10137	5907	67
H(9A)	6472	8109	6253	71
H(9B)	4875	8352	6755	71
H(9C)	5734	9476	6154	71
H(9D)	4915	9182	6521	71
H(9E)	6512	8939	6020	71
H(9F)	5653	7815	6621	71
H(10A)	7258	6996	5472	63
H(10B)	7524	6769	4321	63
H(10C)	6729	5624	4932	63
H(11A)	4519	5270	2597	51
H(11B)	5235	4712	3566	51
H(11C)	6189	5801	2982	51
H(13A)	2247	4531	3180	38
H(13B)	1296	4560	2171	38
H(14A)	251	5528	4028	49
H(14B)	-699	5557	3020	49
H(15A)	-1016	3270	2929	71
H(15B)	-1614	3793	3965	71
H(15C)	20	3199	3899	71
H(17A)	920	6792	464	36
H(17B)	993	5428	979	36
H(18A)	3595	6740	117	43
H(18B)	3575	5328	561	43
H(19A)	3401	5250	-1201	73
H(19B)	1764	5837	-1119	73
H(19C)	2014	4428	-719	73
H(19D)	1385	5093	-825	73
H(19E)	3022	4507	-907	73
H(19F)	2772	5916	-1306	73
H(26A)	-376	7734	1721	53
H(26B)	-859	8755	941	53
H(26C)	-859	9142	2090	53
H(27A)	626	12249	1124	63

H(27B)	-337	11335	1844	63
H(27C)	-397	11004	686	63
H(28A)	2474	12911	1095	64
H(28B)	3887	12556	432	64
H(28C)	4141	12885	1583	64
H(28D)	4528	12657	978	64
H(28E)	3114	13012	1641	64
H(28F)	2860	12683	490	64
H(29A)	6287	10688	641	58
H(29B)	6623	10331	1750	58
H(29C)	6037	11726	1522	58
H(30A)	5899	8329	2343	49
H(30B)	6072	8219	1176	49
H(30C)	4985	7169	1712	49

Table 6. Torsion angles [°] for ratha (6).

C(6)-C(1)-C(2)-C(3)	0.9(2)	C(20)-C(21)-C(22)-C(23)	1.6(2)
C(12)-C(1)-C(2)-C(3)	177.30(13)	C(26)-C(21)-C(22)-C(23)	-178.92(14)
C(6)-C(1)-C(2)-C(7)	-177.94(14)	C(20)-C(21)-C(22)-C(27)	-177.88(14)
C(12)-C(1)-C(2)-C(7)	-1.5(2)	C(26)-C(21)-C(22)-C(27)	1.6(2)
C(1)-C(2)-C(3)-C(4)	-0.4(2)	C(21)-C(22)-C(23)-C(24)	-0.9(2)
C(7)-C(2)-C(3)-C(4)	178.43(14)	C(27)-C(22)-C(23)-C(24)	178.53(14)
C(1)-C(2)-C(3)-C(8)	-179.42(14)	C(21)-C(22)-C(23)-C(28)	179.54(15)
C(7)-C(2)-C(3)-C(8)	-0.6(2)	C(27)-C(22)-C(23)-C(28)	-1.0(2)
C(2)-C(3)-C(4)-C(5)	0.5(2)	C(22)-C(23)-C(24)-C(25)	0.0(2)
C(8)-C(3)-C(4)-C(5)	179.57(14)	C(28)-C(23)-C(24)-C(25)	179.57(14)
C(2)-C(3)-C(4)-C(9)	-178.36(14)	C(22)-C(23)-C(24)-C(29)	179.28(14)
C(8)-C(3)-C(4)-C(9)	0.7(2)	C(28)-C(23)-C(24)-C(29)	-1.2(2)
C(3)-C(4)-C(5)-C(6)	-1.2(2)	C(21)-C(20)-C(25)-C(24)	0.5(2)
C(9)-C(4)-C(5)-C(6)	177.70(14)	C(16)-C(20)-C(25)-C(24)	175.49(13)
C(3)-C(4)-C(5)-C(10)	178.22(15)	C(21)-C(20)-C(25)-C(30)	-178.74(13)
C(9)-C(4)-C(5)-C(10)	-2.9(2)	C(16)-C(20)-C(25)-C(30)	-3.7(2)
C(4)-C(5)-C(6)-C(1)	1.7(2)	C(23)-C(24)-C(25)-C(20)	0.2(2)
C(10)-C(5)-C(6)-C(1)	-177.74(14)	C(29)-C(24)-C(25)-C(20)	-179.06(14)
C(4)-C(5)-C(6)-C(11)	-178.03(14)	C(23)-C(24)-C(25)-C(30)	179.40(14)
C(10)-C(5)-C(6)-C(11)	2.6(2)	C(29)-C(24)-C(25)-C(30)	0.2(2)
C(2)-C(1)-C(6)-C(5)	-1.5(2)	Symmetry transformations used to generate equivalent atoms:	
C(12)-C(1)-C(6)-C(5)	-178.15(13)		
C(2)-C(1)-C(6)-C(11)	178.18(14)		
C(12)-C(1)-C(6)-C(11)	1.5(2)		
C(2)-C(1)-C(12)-C(16)	78.1(2)		
C(6)-C(1)-C(12)-C(16)	-105.37(17)		
C(2)-C(1)-C(12)-C(13)	-102.52(16)		
C(6)-C(1)-C(12)-C(13)	73.98(17)		
C(16)-C(12)-C(13)-C(14)	-102.88(17)		
C(1)-C(12)-C(13)-C(14)	77.76(17)		
C(12)-C(13)-C(14)-C(15)	-179.97(14)		
C(1)-C(12)-C(16)-C(20)	-13.5(2)		
C(13)-C(12)-C(16)-C(20)	167.23(14)		
C(1)-C(12)-C(16)-C(17)	166.33(13)		
C(13)-C(12)-C(16)-C(17)	-13.0(2)		
C(12)-C(16)-C(17)-C(18)	-100.06(17)		
C(20)-C(16)-C(17)-C(18)	79.77(16)		
C(16)-C(17)-C(18)-C(19)	-175.34(14)		
C(12)-C(16)-C(20)-C(25)	78.6(2)		
C(17)-C(16)-C(20)-C(25)	-101.23(16)		
C(12)-C(16)-C(20)-C(21)	-106.25(17)		
C(17)-C(16)-C(20)-C(21)	73.91(16)		
C(25)-C(20)-C(21)-C(22)	-1.4(2)		
C(16)-C(20)-C(21)-C(22)	-176.68(13)		
C(25)-C(20)-C(21)-C(26)	179.18(14)		
C(16)-C(20)-C(21)-C(26)	3.9(2)		

Figure 1. ORTEP Diagram of **6** showing the cofacial arrangement of petamethylphenyl moieties; thermal ellipsoid shown in 30% probability.

Table 1. Crystal data and structure refinement for **7**.

Identification code	rathc	
Empirical formula	C34 H44	
Formula weight	452.69	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 8.4910(9) Å	$\alpha = 98.979(2)^\circ$.
	b = 9.8941(10) Å	$\beta = 100.758(2)^\circ$.
	c = 16.8648(17) Å	$\gamma = 91.471(2)^\circ$.
Volume	1372.7(2) Å ³	
Z	2	
Density (calculated)	1.095 Mg/m ³	
Absorption coefficient	0.061 mm ⁻¹	
F(000)	496	
Crystal size	? x ? x ? mm ³	
Theta range for data collection	2.09 to 26.39°.	
Index ranges	-10 ≤ h ≤ 10, -12 ≤ k ≤ 12, 0 ≤ l ≤ 21	
Reflections collected	5560	
Independent reflections	5560 [R(int) = 0.0000]	
Completeness to theta = 26.39°	98.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	5560 / 0 / 318	
Goodness-of-fit on F ²	0.993	
Final R indices [I > 2sigma(I)]	R1 = 0.0970, wR2 = 0.2721	
R indices (all data)	R1 = 0.1128, wR2 = 0.2879	
Largest diff. peak and hole	0.779 and -0.253 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for rathc (7). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	1680(3)	5149(3)	2166(2)	38(1)
C(2)	2283(4)	3900(3)	2080(2)	41(1)
C(3)	585(3)	5605(2)	2760(2)	35(1)
C(4)	-1011(4)	5871(2)	2447(2)	40(1)
C(5)	-2012(3)	6395(3)	2975(2)	40(1)
C(6)	-1451(3)	6598(2)	3821(2)	40(1)
C(7)	134(3)	6347(2)	4136(2)	37(1)
C(8)	1174(3)	5872(2)	3605(2)	34(1)
C(9)	-1652(4)	5631(3)	1537(2)	55(1)
C(10)	-3691(4)	6772(3)	2626(2)	55(1)
C(11)	-2568(4)	7130(4)	4385(2)	59(1)
C(12)	806(4)	6647(3)	5048(2)	49(1)
C(13)	2918(4)	5762(3)	3956(2)	44(1)
C(14)	2050(4)	6265(3)	1700(2)	42(1)
C(15)	2039(5)	6049(3)	865(2)	54(1)
C(16)	2421(6)	7122(4)	476(2)	67(1)
C(17)	2800(5)	8414(3)	921(2)	63(1)
C(18)	2766(4)	8671(3)	1731(2)	54(1)
C(19)	2398(4)	7596(3)	2119(2)	45(1)
C(20)	2033(4)	2831(2)	2604(2)	39(1)
C(21)	526(4)	2148(3)	2546(2)	41(1)
C(22)	381(4)	1134(3)	3034(2)	41(1)
C(23)	1729(4)	768(2)	3546(2)	40(1)
C(24)	3246(4)	1414(3)	3579(2)	41(1)
C(25)	3395(4)	2465(3)	3124(2)	40(1)
C(26)	-919(4)	2461(3)	1950(2)	56(1)
C(27)	-1237(4)	419(3)	3000(2)	59(1)
C(28)	1553(5)	-322(3)	4066(2)	58(1)
C(29)	4735(4)	979(3)	4108(2)	55(1)
C(30)	5044(4)	3196(3)	3235(2)	51(1)
C(31)	3278(4)	3403(3)	1444(2)	48(1)
C(32)	2295(5)	2432(3)	717(2)	56(1)
C(33)	3317(6)	1958(4)	61(2)	73(1)
C(34)	2416(9)	989(5)	-647(3)	108(2)

Table 3. Bond lengths [Å] and angles [°] for **rathc (7)**.

C(1)-C(2)	1.347(4)	C(15)-C(16)	1.393(4)
C(1)-C(14)	1.509(4)	C(16)-C(17)	1.375(5)
C(1)-C(3)	1.517(4)	C(17)-C(18)	1.357(5)
C(2)-C(20)	1.516(3)	C(18)-C(19)	1.390(4)
C(2)-C(31)	1.517(4)	C(20)-C(25)	1.407(4)
C(3)-C(8)	1.400(4)	C(20)-C(21)	1.411(4)
C(3)-C(4)	1.408(4)	C(21)-C(22)	1.409(4)
C(4)-C(5)	1.395(4)	C(21)-C(26)	1.507(4)
C(4)-C(9)	1.508(4)	C(22)-C(23)	1.392(4)
C(5)-C(6)	1.399(4)	C(22)-C(27)	1.516(4)
C(5)-C(10)	1.516(4)	C(23)-C(24)	1.410(4)
C(6)-C(7)	1.396(4)	C(23)-C(28)	1.514(4)
C(6)-C(11)	1.512(4)	C(24)-C(25)	1.402(3)
C(7)-C(8)	1.410(4)	C(24)-C(29)	1.518(4)
C(7)-C(12)	1.516(4)	C(25)-C(30)	1.525(4)
C(8)-C(13)	1.502(4)	C(31)-C(32)	1.527(5)
C(14)-C(15)	1.388(4)	C(32)-C(33)	1.553(5)
C(14)-C(19)	1.390(4)	C(33)-C(34)	1.481(6)
C(2)-C(1)-C(14)	124.0(2)	C(17)-C(16)-C(15)	119.8(3)
C(2)-C(1)-C(3)	123.6(2)	C(18)-C(17)-C(16)	120.9(3)
C(14)-C(1)-C(3)	112.4(2)	C(17)-C(18)-C(19)	119.1(3)
C(1)-C(2)-C(20)	123.8(2)	C(18)-C(19)-C(14)	122.0(3)
C(1)-C(2)-C(31)	123.8(2)	C(25)-C(20)-C(21)	120.1(2)
C(20)-C(2)-C(31)	112.5(2)	C(25)-C(20)-C(2)	117.4(2)
C(8)-C(3)-C(4)	119.9(2)	C(21)-C(20)-C(2)	122.4(3)
C(8)-C(3)-C(1)	121.1(2)	C(22)-C(21)-C(20)	119.7(3)
C(4)-C(3)-C(1)	118.8(2)	C(22)-C(21)-C(26)	119.6(3)
C(5)-C(4)-C(3)	120.3(2)	C(20)-C(21)-C(26)	120.7(3)
C(5)-C(4)-C(9)	119.0(3)	C(23)-C(22)-C(21)	120.2(3)
C(3)-C(4)-C(9)	120.7(3)	C(23)-C(22)-C(27)	119.3(3)
C(4)-C(5)-C(6)	119.9(3)	C(21)-C(22)-C(27)	120.6(3)
C(4)-C(5)-C(10)	119.7(3)	C(22)-C(23)-C(24)	120.0(2)
C(6)-C(5)-C(10)	120.4(3)	C(22)-C(23)-C(28)	119.5(3)
C(7)-C(6)-C(5)	120.1(2)	C(24)-C(23)-C(28)	120.4(3)
C(7)-C(6)-C(11)	120.7(3)	C(25)-C(24)-C(23)	120.3(3)
C(5)-C(6)-C(11)	119.2(3)	C(25)-C(24)-C(29)	119.2(3)
C(6)-C(7)-C(8)	120.3(2)	C(23)-C(24)-C(29)	120.4(2)
C(6)-C(7)-C(12)	121.2(2)	C(24)-C(25)-C(20)	119.6(3)
C(8)-C(7)-C(12)	118.4(3)	C(24)-C(25)-C(30)	117.8(3)
C(3)-C(8)-C(7)	119.4(2)	C(20)-C(25)-C(30)	122.6(2)
C(3)-C(8)-C(13)	121.4(2)	C(2)-C(31)-C(32)	111.9(3)
C(7)-C(8)-C(13)	119.1(2)	C(31)-C(32)-C(33)	111.4(3)
C(15)-C(14)-C(19)	117.3(3)	C(34)-C(33)-C(32)	113.0(4)
C(15)-C(14)-C(1)	123.8(3)		
C(19)-C(14)-C(1)	118.9(2)		
C(14)-C(15)-C(16)	120.8(3)		

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for rathe (7). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
C(1)	52(2)	34(1)	32(1)	11(1)	12(1)	7(1)
C(2)	58(2)	33(1)	35(1)	11(1)	15(1)	7(1)
C(3)	49(2)	23(1)	38(1)	11(1)	15(1)	5(1)
C(4)	53(2)	26(1)	41(1)	11(1)	9(1)	2(1)
C(5)	40(1)	27(1)	54(2)	11(1)	11(1)	-1(1)
C(6)	42(2)	27(1)	53(2)	5(1)	19(1)	-3(1)
C(7)	51(2)	23(1)	39(1)	8(1)	14(1)	-5(1)
C(8)	44(1)	22(1)	41(1)	12(1)	14(1)	0(1)
C(9)	68(2)	50(2)	46(2)	17(1)	0(2)	13(2)
C(10)	46(2)	49(2)	72(2)	13(2)	9(2)	5(1)
C(11)	55(2)	61(2)	64(2)	-1(2)	27(2)	2(2)
C(12)	63(2)	43(2)	41(2)	4(1)	14(1)	-5(1)
C(13)	46(2)	41(1)	45(2)	11(1)	9(1)	7(1)
C(14)	55(2)	39(1)	38(1)	14(1)	15(1)	12(1)
C(15)	85(2)	38(2)	38(2)	9(1)	11(2)	5(2)
C(16)	113(3)	53(2)	41(2)	18(1)	25(2)	9(2)
C(17)	97(3)	43(2)	57(2)	17(1)	28(2)	9(2)
C(18)	73(2)	37(2)	57(2)	14(1)	24(2)	5(1)
C(19)	56(2)	38(1)	45(2)	15(1)	15(1)	6(1)
C(20)	60(2)	25(1)	37(1)	9(1)	20(1)	9(1)
C(21)	61(2)	27(1)	38(1)	4(1)	11(1)	10(1)
C(22)	62(2)	24(1)	41(1)	3(1)	18(1)	2(1)
C(23)	67(2)	23(1)	35(1)	8(1)	19(1)	7(1)
C(24)	61(2)	29(1)	40(1)	12(1)	20(1)	13(1)
C(25)	54(2)	29(1)	45(2)	13(1)	22(1)	12(1)
C(26)	67(2)	35(1)	58(2)	6(1)	-4(2)	0(1)
C(27)	66(2)	41(2)	71(2)	12(2)	16(2)	-4(1)
C(28)	89(2)	34(2)	59(2)	21(1)	25(2)	2(2)
C(29)	63(2)	47(2)	63(2)	26(2)	18(2)	17(1)
C(30)	52(2)	48(2)	60(2)	22(1)	17(1)	13(1)
C(31)	69(2)	37(1)	44(2)	12(1)	21(1)	12(1)
C(32)	86(2)	43(2)	43(2)	13(1)	14(2)	16(2)
C(33)	125(4)	51(2)	50(2)	11(2)	27(2)	28(2)
C(34)	219(7)	55(2)	54(2)	4(2)	38(3)	17(3)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for rathc (7).

	x	y	z	U(eq)
H(9A)	-2708	5141	1420	82
H(9B)	-912	5083	1257	82
H(9C)	-1751	6514	1343	82
H(10A)	-3678	7153	2124	83
H(10B)	-4058	7456	3028	83
H(10C)	-4422	5952	2501	83
H(11A)	-2006	7213	4955	89
H(11B)	-3510	6491	4292	89
H(11C)	-2912	8031	4273	89
H(12A)	1206	7608	5202	73
H(12B)	1688	6052	5182	73
H(12C)	-43	6476	5348	73
H(13A)	3038	4986	4255	65
H(13B)	3332	6609	4329	65
H(13C)	3523	5618	3511	65
H(15)	1767	5159	556	64
H(16)	2419	6961	-94	80
H(17)	3089	9138	657	76
H(18)	2990	9573	2030	64
H(19)	2385	7776	2687	54
H(26A)	-1472	1602	1645	83
H(26B)	-573	3003	1567	83
H(26C)	-1652	2983	2249	83
H(27A)	-1235	-541	2745	88
H(27B)	-2080	874	2676	88
H(27C)	-1441	463	3556	88
H(28A)	943	25	4487	87
H(28B)	2618	-555	4328	87
H(28C)	982	-1142	3720	87
H(29A)	5695	1336	3947	82
H(29B)	4723	-23	4034	82
H(29C)	4748	1345	4684	82
H(30A)	5472	3492	3820	76
H(30B)	4947	3997	2957	76
H(30C)	5773	2568	3001	76
H(31A)	4201	2924	1700	57
H(31B)	3708	4202	1247	57
H(32A)	1359	2901	467	68

H(32B)	1888	1620	910	68
H(33A)	3706	2772	-137	88
H(33B)	4268	1513	318	88
H(34A)	1939	222	-451	162
H(34B)	3150	640	-1006	162
H(34C)	1565	1462	-951	162

Figure 2a. ORTEP Diagram of **7**, thermal ellipsoid shown in 50% probability.

Figure 2b. ORTEP Diagram of **7** showing the cofacial arrangement of petamethylphenyl moieties; thermal ellipsoid shown in 50% probability.

References

- a. Pincock, J.A.; Yates, K. *Can. J. Chem.* **1970**, 48, 3332.
- b. Negishi, E.; Kotori, M.; Xu, C. *J. Org. Chem.* **1997**, 62, 8957.
- c. All software and sources of the scattering factors are contained in the SHELXTL (version 5.1) program library (G. Sheldrick, Bruker Analytical X-Ray Systems, Madison, WI).