2-(2,4-Cyclopentadien-1-ylidene)adamantane*

By J. Gabriel Garcia, Mark L. McLaughlin† and Frank R. Fronczek

Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803-1804, USA

Abstract. $C_{15}H_{18}$, $M_r = 198.3$, monoclinic, $P2_1/n$, $a = 6.942$ (2), $b = 15.824$ (4), $c = 10.251$ (2) Å, $\beta = 90.89$ (2)°, $V = 1126.0$ (8) Å^3, $Z = 4$, $D_x = 1.170$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.61$ cm^{-1}, $F(000) = 432$, $T = 296$ K, $R = 0.053$ for 1388 observations (of 1977 unique data). The bond lengths of the 1,3-cyclopentadienylidene and adamantane rings are normal. The bond angle *exo* from the cyclopentadienylidene ring is 111.5 (2)°. This bond angle closely mimics the analogous bond angle of a difulvene that exhibits an enhanced absorbance [McLaughlin, Cronan & Fronczek (1988). *Acta Cryst.* C44, 1747–1749]. The cyclopentadienylidene ring is planar, with maximum deviation of 0.002 (3) Å.

Experimental. The title compound was prepared as described earlier (Olah, Surya & Liang, 1977), m.p. 361–363 K, crystal size 0.30 × 0.40 × 0.45 mm. Space group from diffraction symmetry 2/*m* and systematic absences *h0l* with *h+l* odd and 0*k*0 with *k* odd. Cell dimensions from setting angles of 25 reflections having $13 > \theta > 11°$. Data collected on Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, graphite monochromator, $\omega-2\theta$ scans designed for $I > 25\sigma(I)$, subject to max. scan time = 120 s, scan rates varied 0.91–4.0° min^{-1}. Data having $1 \le \theta \le 25°$, $0 \le h \le 8$, $0 \le k \le 18$, $-12 \le l \le 12$ measured and corrected for background, Lorentz, polarization, and decay. Three standard reflections (110, 020, 002) decreased in intensity by 12.3% during data collection, apparently due to sublimation. A linear correction was applied. No absorption corrections were made. Redundant 0*kl* and 0*kl̄* data were averaged, $R_{int} = 0.021$. Structure solved by direct methods, refined by full-matrix least squares based upon F, using 1388 data for which $I > 1\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$ using Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous-dispersion coefficients of Cromer (1974), 589 unobserved data. Non-H atoms refined anisotropically; H atoms located by ΔF syntheses and refined isotropically. Final $R = 0.053$ (0.093 for all

1977 data), $wR = 0.046$, $S = 1.726$ for 209 variables. Maximum shift 0.01σ in the final cycle, max. residual density 0.16, min. -0.16 e Å^{-3}, extinction coefficient $g = 8.1$ (11) × 10^{-7} where the factor $(1 + gI_c)^{-1}$ was applied to F_c. The fractional coordinates of the title

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$$B_{eq} = \tfrac{8}{3}\pi^2(U_{11} + U_{22} + U_{33}).$$

|	x	y	z	$B_{eq}(\text{Å}^2)$
C1	0.7851 (3)	0.0818 (1)	0.1935 (2)	3.16 (4)
C2	0.8868 (3)	0.0688 (2)	0.3175 (2)	4.25 (5)
C3	0.7770 (4)	0.0231 (2)	0.3954 (2)	5.27 (6)
C4	0.5983 (4)	0.0034 (2)	0.3267 (2)	5.01 (6)
C5	0.6017 (3)	0.0380 (1)	0.2078 (2)	3.88 (5)
C6	0.8436 (3)	0.1265 (1)	0.0902 (2)	3.00 (4)
C7	0.7257 (3)	0.1391 (1)	−0.0328 (2)	3.48 (5)
C8	0.8385 (3)	0.1074 (2)	−0.1501 (2)	4.22 (6)
C9	1.0292 (3)	0.1556 (2)	−0.1584 (2)	4.24 (5)
C10	1.1452 (3)	0.1409 (2)	−0.0328 (2)	4.40 (6)
C11	1.0338 (3)	0.1725 (1)	0.0842 (2)	3.69 (5)
C12	0.6850 (3)	0.2337 (2)	−0.0487 (2)	4.02 (5)
C13	0.8755 (3)	0.2819 (1)	−0.0581 (2)	4.19 (5)
C14	0.9922 (3)	0.2675 (1)	0.0673 (2)	4.30 (5)
C15	0.9876 (3)	0.2499 (2)	−0.1743 (2)	4.56 (6)
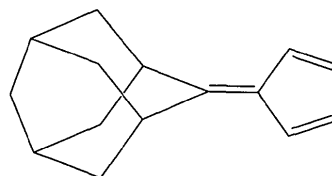
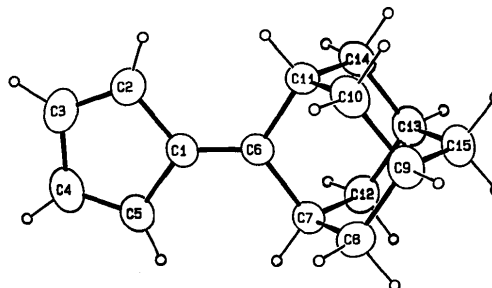Fig. 1. 2-(2,4-Cyclopentadien-1-ylidene)adamantane.

Fig. 2. *ORTEP* drawing (Johnson, 1965) of the molecule, representing heavy atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radius.

Table 2. *Bond distances* (Å), *angles* (°), *and selected torsion angles* (°)

C1 C2 1·459 (2)	C1 C5 1·459 (2)	C1 C6 1·342 (2)
C2 C3 1·327 (3)	C3 C4 1·451 (3)	C4 C5 1·337 (3)
C6 C7 1·506 (2)	C6 C11 1·510 (2)	C7 C8 1·529 (2)
C7 C12 1·533 (3)	C8 C9 1·532 (3)	C9 C10 1·526 (3)
C9 C15 1·528 (3)	C10 C11 1·522 (3)	C11 C14 1·541 (3)
C12 C13 1·531 (3)	C13 C14 1·526 (3)	C13 C15 1·519 (3)

C2 C1 C5 104·8 (2)	C2 C1 C6 127·9 (2)	C5 C1 C6 127·2 (2)
C1 C2 C3 109·1 (2)	C2 C3 C4 108·6 (2)	C3 C4 C5 109·1 (2)
C1 C5 C4 108·3 (2)	C1 C6 C7 124·3 (2)	C1 C6 C11 124·2 (2)
C7 C6 C11 111·5 (2)	C6 C7 C8 109·7 (1)	C6 C7 C12 108·3 (2)
C8 C7 C12 109·4 (2)	C7 C8 C9 109·5 (2)	C8 C9 C10 108·8 (2)
C8 C9 C15 109·2 (2)	C10 C9 C15 109·6 (2)	C9 C10 C11 110·3 (2)
C6 C11 C10 109·2 (2)	C6 C11 C14 108·2 (2)	C10 C11 C14 109·2 (2)
C7 C12 C13 109·6 (2)	C12 C13 C14 108·7 (2)	C12 C13 C15 109·7 (2)
C14 C13 C15 109·8 (2)	C11 C14 C13 109·6 (2)	C9 C15 C13 109·9 (2)

C5 C1 C6 C7	−0·3 (4)	C2 C1 C6 C11	0·5 (4)
C1 C6 C7 C8	−123·0 (2)	C1 C6 C7 C12	117·7 (2)
C11 C6 C7 C8	58·6 (2)	C11 C6 C7 C12	−60·8 (2)
C1 C6 C11 C10	123·2 (2)	C1 C6 C11 C14	−118·1 (2)
C7 C6 C11 C10	−58·4 (2)	C7 C6 C11 C14	60·4 (2)

compound are given in Table 1. Fig. 1 is a line drawing and Fig. 2 is a perspective drawing showing the atom numbering. Bond distances, angles, and selected torsion angles are presented in Table 2.*

Related literature. The structures of dimethylfulvene at 248 K (Norman & Post, 1961) and the difulvene, 1,4-bis(2,4-cyclopentadien-1-ylidene)cyclohexane (McLaughlin, Cronan & Fronczek, 1988), exhibit the

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* Lists of H-atom coordinates and isotropic thermal parameters, anisotropic thermal parameters, bond distances and angles involving H, structure-factor amplitudes, torsion angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51722 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Sqaure, Chester CH1 2HU, England.

expected single–double bond alternation within the fulvene ring system like the title compound. The bond angle C7—C6—C11 = 111·5 (2)° of the title compound compares with the analogous bond angles of dimethylfulvene, 114·0 (6)°, and 1,4-bis(2,4-cyclopentadien-1-ylidene)cyclohexane, 112·71 (9)°.

The adamantane ring system has been extensively characterized including the parent hydrocarbon by Nordman & Schmitkons (1965) and refined by Donohue & Goodman (1967). The geometry about the double bond of adamantylideneadamantane (Swen-Walstra & Visser, 1971) is similar to that of the title compound.

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Structure of 6-Tosyl-1,6,11,17,18-pentaazatricyclo[12.2.1^{1,4}.1^{8,11}]octadeca-2,4(17),8(18),9-tetraene

BY F. H. CANO AND C. FOCES-FOCES

UEI de Cristalografía, Instituto Rocasolano, CSIC, Serrano 119, 28006 Madrid, Spain

Abstract. $C_{20}H_{25}N_5O_2S$, $M_r = 399·51$, triclinic, $P\bar{1}$, $a = 10·7373$ (5), $b = 9·4581$ (5), $c = 10·8159$ (4) Å, $\alpha = 74·012$ (4), $\beta = 98·504$ (3), $\gamma = 77·587$ (3)°, $U = 1005·8$ (1) Å^3, $Z = 2$, $D_x = 1·319$ Mg m^{-3}, graphite-monochromated Cu $K\alpha$ radiation, $\lambda = 1·5418$ Å, $\mu = 1·599$ mm^{-1}, $F(000) = 424$, $T = 293$ K, $R = 0·059$ for 2903 observed reflexions $[I > 3\sigma(I)]$. The pyrazole rings, which are quite similar, are inclined at different