

Table 4. Intermolecular hydrogen bonds O(aq)···O

| | | | |
|------------|------------|------------------|------------|
| O31···O19 | 2.67 (1) Å | O19···O31···O203 | 106.5 (3)° |
| O31···O203 | 2.79 (1) | | |
| O32···O3 | 2.797 (7) | O3···O32···O219 | 106.6 (2) |
| O32···O219 | 2.68 (1) | | |
| O33···O11 | 2.72 (1) | O11···O33···O211 | 97.3 (3) |
| O33···O211 | 2.72 (1) | | |

A *SCHAKAL* (Keller, 1988) plot of molecule *A* is shown in Fig. 1.

Related literature. An orthorhombic phase of leuconolam.C₂H₅OH was reported by Wei, Ali, Goh, Sinn & Butcher, 1986).

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A Tetramethylethano-Bridged Difulvene

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Abstract. 2,3-Dimethyl-2,3-bis[3-(1-methylethylidene)cyclopenta-1,4-dien-1-yl]butane, C₂₂H₃₀, *M_r* = 294.5, monoclinic, *P*2₁/*c*, *a* = 7.144 (2), *b* = 19.698 (2), *c* = 7.449 (3) Å, β = 117.51 (2)°, *V* = 929.8 (9) Å³, *Z* = 2, *D_x* = 1.052 g cm⁻³, λ(Mo *K*α) = 0.71073 Å, μ = 0.55 cm⁻¹, *F*(000) = 324, *T* = 297 K, *R* = 0.063 for 1120 data having *I* > 1σ(*I*). The cyclopentadienylidene ring exhibits the expected localized valence-bond alternation within the five-membered ring. The bond angle *exo* to the exocyclic double bond is 114.8 (2)°. Due to the centrosymmetry of the molecule, the fulvenes are *anti* and the methyl groups are all *gauche* to the fulvene rings. The cyclopentadienylidene ring is planar with maximum deviation of 0.002 (2) Å.

Experimental. The title compound is prepared by treatment of the proton-shift isomers of 2,3-bis(2,4-cyclopentadien-1-yl)-2,3-dimethylbutane with excess acetone in methanol catalyzed by pyrrolidine. Yellow crystals, dec. 417 K, suitable for single-crystal X-ray diffraction were crystallized from ethyl acetate with slow cooling from reflux temperature. All standard spectroscopic measurements can be interpreted in terms of the X-ray structure determination (Erickson, McLaughlin & Fronczek, 1989).

Intensity data were obtained from an irregular fragment of dimensions 0.15 × 0.23 × 0.33 mm mounted in a random orientation on an Enraf-Nonius CAD-4 diffractometer. Cell dimensions were determined at 297 K by a least-squares fit to setting angles of 25 reflections having 22 > 2θ > 18°. The θ values were derived from measurements at ±2θ. One quadrant of data having 2 < 2θ < 55°, 0 ≤ *h* ≤ 9, 0 ≤ *k* ≤ 25, -9 ≤ *l* ≤ 9 was measured using graphite-monochromated Mo *K*α radiation. 2128 reflections were measured. The ω-2θ scans were made at speeds ranging from 0.45 to 4.0° min⁻¹ to measure all significant data with approximately equal precision. Three standard reflections (100, 060, 002), measured every 10 000s of exposure time, exhibited only random fluctuations of less than ±2% in intensity during data collection. Data included corrections for background, Lorentz, and polarization. Absorption was negligible.

The space group was determined by systematic absences *h*0*l* with *l* odd and 0*k*0 with *k* odd. The structure was solved by direct methods and refined by full-matrix least squares based upon *F*, with weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$ using the Enraf-Nonius *SDP* (Frenz, 1985), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974), and 1120 data having *I* > 1σ(*I*). Non-H atoms were refined anisotropically; the H atoms were located

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Table 1. Atomic coordinates and equivalent isotropic thermal parameters

$$B_{eq} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

| | <i>x</i> | <i>y</i> | <i>z</i> | <i>B</i> _{eq} (Å ²) |
|-----|------------|-------------|------------|--|
| C1 | 0.4593 (3) | 0.0165 (1) | 0.3915 (3) | 3.98 (5) |
| C2 | 0.3457 (3) | 0.0828 (1) | 0.3757 (3) | 3.89 (5) |
| C3 | 0.4200 (3) | 0.1466 (1) | 0.3993 (3) | 4.35 (5) |
| C4 | 0.2525 (3) | 0.1952 (1) | 0.3633 (3) | 4.52 (5) |
| C5 | 0.0661 (3) | 0.1535 (1) | 0.3154 (3) | 5.40 (6) |
| C6 | 0.1194 (3) | 0.0882 (1) | 0.3218 (3) | 4.89 (6) |
| C7 | 0.2616 (3) | 0.2635 (1) | 0.3674 (3) | 5.62 (6) |
| C8 | 0.6445 (3) | 0.0298 (1) | 0.3450 (3) | 6.28 (6) |
| C9 | 0.3065 (3) | -0.0316 (1) | 0.2248 (3) | 6.28 (7) |
| C10 | 0.4626 (4) | 0.3004 (1) | 0.4171 (4) | 8.14 (9) |
| C11 | 0.0753 (4) | 0.3084 (1) | 0.3222 (4) | 8.28 (8) |

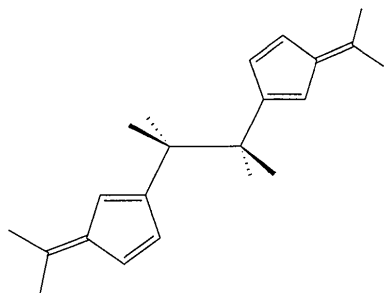


Fig. 1. 2,3-Dimethyl-2,3-bis[3-(1-methylethylidene)cyclopenta-1,4-dien-1-yl]butane.

in a ΔF map and were refined isotropically. Final $R = 0.063$ ($R = 0.169$ for all 2128 data), $wR = 0.046$, $S = 1.760$ for 160 variables. The largest shift was 0.02σ in the final cycle, maximum residual density 0.15 , minimum $-0.14 e \text{ \AA}^{-3}$. The fractional coordinates of the title compound are given in Table 1. Fig. 1 is a line drawing and Fig. 2 is a perspective drawing showing the atom numbering. Selected distances, angles, and torsion angles are presented in Table 2.*

Related literature. 6,6-Dimethylfulvene at 248 K (Norman & Post, 1961) and the difulvene 1,4-bis(2,4-cyclopentadien-1-ylidene)cyclohexane (McLaughlin, Cronan & Fronczek, 1988) exhibit a similar localized valence-bond alternation within the five-membered rings. The bond angle *exo* to the fulvene ring exocyclic double bond, $C10-C7-C11 = 114.8 (2)^\circ$, is similar to the analogous bond angles of 6,6-dimethylfulvene, $114.0 (6)^\circ$, and 1,4-bis(2,4-cyclopentadien-1-ylidene)cyclohexane, $112.71 (9)^\circ$.

* Lists of H-atom coordinates and isotropic thermal parameters, non-H-atom anisotropic thermal parameters, structure-factor amplitudes, bond distances and angles involving H, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51801 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond distances (Å), angles ($^\circ$) and selected torsion angles ($^\circ$)

| | | | | | | | | | |
|-----|-----|-----------|-----------|-----------|-----------|-----|-----------|-----|------------|
| C1 | C1' | 1.583 (3) | C3 | C4 | 1.459 (2) | | | | |
| C1 | C2 | 1.513 (2) | C4 | C5 | 1.462 (2) | | | | |
| C1 | C8 | 1.537 (2) | C4 | C7 | 1.346 (2) | | | | |
| C1 | C9 | 1.542 (3) | C5 | C6 | 1.336 (3) | | | | |
| C2 | C3 | 1.344 (2) | C7 | C10 | 1.496 (3) | | | | |
| C2 | C6 | 1.479 (2) | C7 | C11 | 1.501 (3) | | | | |
| C1' | C1 | C2 | 111.4 (2) | C2 | C3 | C4 | 110.5 (2) | | |
| C1' | C1 | C8 | 110.6 (2) | C3 | C4 | C5 | 104.6 (2) | | |
| C1' | C1 | C9 | 111.1 (2) | C3 | C4 | C7 | 128.7 (2) | | |
| C2 | C1 | C8 | 108.8 (1) | C5 | C4 | C7 | 126.6 (2) | | |
| C2 | C1 | C9 | 108.3 (2) | C4 | C5 | C6 | 108.7 (2) | | |
| C8 | C1 | C9 | 106.7 (2) | C2 | C6 | C5 | 109.8 (2) | | |
| C1 | C2 | C3 | 129.2 (2) | C4 | C7 | C10 | 121.5 (2) | | |
| C1 | C2 | C6 | 124.3 (2) | C4 | C7 | C11 | 123.7 (2) | | |
| C3 | C2 | C6 | 106.5 (2) | C10 | C7 | C11 | 114.8 (2) | | |
| C8 | C1 | C2 | C3 | -25.4 (3) | C3 | C4 | C7 | C10 | -1.2 (3) |
| C9 | C1 | C2 | C6 | 36.7 (2) | C3 | C4 | C7 | C11 | 178.4 (2) |
| C1' | C1 | C2 | C3 | 96.7 (2) | C5 | C4 | C7 | C10 | -179.4 (2) |
| C1' | C1 | C2 | C6 | -85.7 (2) | C5 | C4 | C7 | C11 | 0.2 (4) |
| C8 | C1 | C1' | C9' | 61.8 (2) | | | | | |

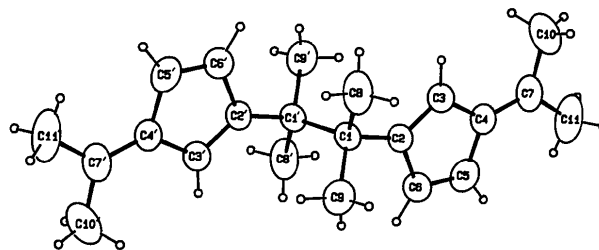


Fig. 2. ORTEP drawing (Johnson, 1965) of the molecule, representing C atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radius.

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