

Table 2. Positional parameters and isotropic thermal parameters *B* (Å²)

Molecule <i>A</i>	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>
O3	0.420	0.552	0.705	4.9 (1)
O11	0.1471 (9)	0.4211 (6)	0.8878 (9)	6.6 (2)
O19	0.6203 (7)	0.2119 (5)	0.6198 (7)	4.2 (1)
N4	0.5728 (8)	0.3741 (6)	0.7552 (8)	3.7 (1)
N12	0.0580 (9)	0.3196 (6)	0.6673 (9)	4.6 (2)
C1	0.3393 (9)	0.3060 (7)	0.604 (1)	3.3 (2)
C2	0.280 (1)	0.4119 (7)	0.593 (1)	4.0 (2)
C3	0.423 (1)	0.4559 (7)	0.685 (1)	4.3 (2)
C5	0.750 (1)	0.3781 (8)	0.855 (1)	5.2 (2)
C6	0.829 (1)	0.3168 (9)	1.018 (1)	5.6 (2)
C7	0.804 (1)	0.2076 (8)	0.982 (1)	4.8 (2)
C8	0.611 (1)	0.2079 (7)	0.876 (1)	3.8 (2)
C9	0.515 (1)	0.2613 (7)	0.977 (1)	4.3 (2)
C10	0.336 (1)	0.2470 (8)	0.922 (1)	4.9 (2)
C11	0.177 (1)	0.3339 (8)	0.825 (1)	5.1 (2)
C13	0.104 (1)	0.2374 (7)	0.566 (1)	3.9 (2)
C14	0.2376 (9)	0.2316 (6)	0.5341 (9)	3.2 (2)
C15	0.275 (1)	0.1502 (7)	0.433 (1)	4.3 (2)
C16	0.185 (1)	0.0767 (9)	0.374 (1)	5.8 (3)
C17	0.058 (1)	0.0840 (9)	0.411 (1)	5.6 (3)
C18	0.015 (1)	0.1617 (8)	0.506 (1)	5.4 (3)
C19	0.538 (1)	0.2705 (7)	0.712 (1)	3.4 (2)
C81	0.611 (1)	0.0931 (8)	0.835 (1)	4.5 (2)
C82	0.685 (1)	0.0196 (9)	0.984 (1)	6.7 (3)
Molecule <i>B</i>				
O203	0.7247 (8)	0.4513 (5)	0.4374 (8)	5.5 (2)
O211	0.8547 (8)	0.5765 (6)	0.1143 (8)	5.7 (2)
O219	0.4540 (7)	0.7926 (5)	0.4526 (7)	4.3 (1)
N204	0.6745 (8)	0.6286 (6)	0.4962 (8)	4.1 (2)
N212	0.5847 (9)	0.6842 (6)	-0.0384 (9)	4.4 (2)
C201	0.5002 (9)	0.6986 (6)	0.226 (1)	3.3 (2)
C202	0.544 (1)	0.5934 (7)	0.224 (1)	4.0 (2)
C203	0.654 (1)	0.5474 (7)	0.391 (1)	4.1 (2)
C205	0.775 (1)	0.6217 (9)	0.678 (1)	5.7 (3)
C206	0.907 (1)	0.6800 (9)	0.733 (1)	5.9 (3)
C207	0.819 (1)	0.7895 (8)	0.650 (1)	4.7 (2)
C208	0.720 (1)	0.7920 (7)	0.458 (1)	3.5 (2)
C209	0.852 (1)	0.7350 (7)	0.394 (1)	3.9 (2)
C210	0.796 (1)	0.7519 (7)	0.209 (1)	4.2 (2)
C211	0.747 (1)	0.6654 (7)	0.093 (1)	4.2 (2)
C213	0.440 (1)	0.7682 (7)	-0.042 (1)	4.2 (2)
C214	0.393 (1)	0.7737 (7)	0.083 (1)	3.4 (2)
C215	0.241 (1)	0.8557 (8)	0.070 (1)	4.7 (2)
C216	0.152 (1)	0.9281 (9)	-0.063 (1)	5.5 (3)
C217	0.201 (1)	0.923 (1)	-0.179 (1)	6.9 (3)
C218	0.343 (1)	0.8436 (9)	-0.173 (1)	5.6 (3)
C219	0.5855 (9)	0.7310 (6)	0.4091 (9)	3.2 (2)
C281	0.623 (1)	0.9078 (7)	0.392 (1)	4.4 (2)
C282	0.741 (1)	0.9744 (9)	0.430 (1)	5.6 (3)
Solvent (aq)				
O31	0.5070 (8)	0.3256 (5)	0.3471 (8)	5.2 (2)
O32	0.2538 (8)	0.6732 (5)	0.4098 (8)	5.1 (2)
O33	0.1966 (9)	0.4987 (6)	0.1922 (9)	6.5 (2)

Table 3. Selected distances (Å) and angles (°) with *e.s.d.*'s

O3	C3	1.27 (1)	O203	C203	1.26 (1)		
O11	C11	1.24 (1)	O211	C211	1.24 (2)		
O19	C19	1.41 (1)	O219	C219	1.43 (1)		
N4	C3	1.356 (9)	N204	C203	1.35 (1)		
N4	C5	1.44 (1)	N204	C205	1.46 (1)		
N4	C19	1.48 (1)	N204	C219	1.45 (2)		
N12	C11	1.37 (2)	N212	C211	1.354 (9)		
N12	C13	1.44 (1)	N212	C213	1.42 (1)		
C1	C2	1.34 (1)	C201	C202	1.33 (1)		
C1	C14	1.46 (1)	C201	C214	1.46 (1)		
C1	C19	1.53 (1)	C201	C219	1.54 (2)		
C2	C3	1.45 (1)	C202	C203	1.44 (1)		
C3	N4	C5	128.3 (8)	C9	C10	C11	118 (2)
C3	N4	C19	111.3 (7)	O11	C11	N12	118.5 (7)
C5	N4	C19	120.3 (6)	O11	C11	C10	120.6 (8)
C11	N12	C13	121.6 (7)	N12	C11	C10	120.9 (9)
C2	C1	C14	127.9 (7)	N12	C13	C14	120.6 (9)
C2	C1	C19	109.2 (8)	N12	C13	C18	118 (2)
C14	C1	C19	122.9 (7)	C14	C13	C18	121.0 (9)
C1	C2	C3	110.4 (7)	C1	C14	C13	121.8 (8)
O3	C3	N4	122.9 (8)	C1	C14	C15	120.4 (9)
O3	C3	C2	129.3 (6)	C13	C14	C15	117.9 (9)
N4	C3	C2	107.9 (8)	C14	C15	C16	120 (2)
N4	C5	C6	108 (2)	C15	C16	C17	120 (2)
C5	C6	C7	109.6 (9)	C16	C17	C18	122 (1)
C6	C7	C8	114.9 (7)	C13	C18	C17	119 (1)
C7	C8	C9	108.5 (8)	O19	C19	N4	110.2 (9)
C7	C8	C19	105.4 (9)	O19	C19	C1	111.2 (6)
C7	C8	C81	108.0 (6)	O19	C19	C8	109.2 (6)
C9	C8	C19	112.6 (6)	N4	C19	C1	101.2 (6)
C9	C8	C81	113 (1)	N4	C19	C8	108.6 (6)
C19	C8	C81	108.8 (8)	C1	C19	C8	116.0 (9)
C8	C9	C10	118.8 (7)	C8	C81	C82	115.4 (8)
C203	N204	C205	127.1 (8)	C209	C210	C211	118 (1)
C203	N204	C219	112.7 (7)	O211	C211	N212	119.2 (9)
C205	N204	C219	120.2 (7)	O211	C211	C210	120.9 (7)
C211	N212	C213	122.4 (7)	N212	C211	C210	119.8 (7)
C202	C201	C214	127.9 (7)	N212	C213	C214	120.2 (8)
C202	C201	C219	108.0 (7)	N212	C213	C218	119 (2)
C214	C201	C219	124.2 (7)	C214	C213	C218	120.3 (8)
C201	C202	C203	111.1 (8)	C201	C214	C213	120.6 (7)
O203	C203	N204	124.3 (7)	C201	C214	C215	120.7 (9)
O203	C203	C202	128.9 (8)	C213	C214	C215	118.6 (8)
N204	C203	C202	106.8 (7)	C214	C215	C216	118 (2)
N204	C205	C206	109 (2)	C215	C216	C217	123 (1)
C205	C206	C207	110.1 (7)	C216	C217	C218	121 (1)
C206	C207	C208	115.4 (8)	C213	C218	C217	120 (1)
C207	C208	C209	108.8 (6)	O219	C219	N204	110.6 (8)
C207	C208	C219	106.0 (9)	O219	C219	C201	110.2 (6)
C207	C208	C281	109.4 (8)	O219	C219	C208	108.7 (7)
C209	C208	C219	110.6 (8)	N204	C219	C201	101.5 (6)
C209	C208	C281	112.1 (9)	N204	C219	C208	109.4 (6)
C219	C208	C281	110.0 (6)	C201	C219	C208	116.3 (9)
C208	C209	C210	118.6 (6)	C208	C281	C282	114.7 (7)

benzene ring *ortho* disubstituted with a 4,5-dihydro-5-hydroxy-2-oxo-pyrrol-1,4,5-triyl unit and an N(CO)*R*— unit forming a twelve-membered ring.

Experimental. The alkaloid leuconolam was isolated from the plant *Rhazia stricta* (Decaisne), which has high repute in the indigenous system of medicine as a therapeutic agent in fever and chronic rheumatism. Goh, Wei & Ali (1984) previously reported the isolation of the same alkaloid from the Malaysian plant *Leuconitis griffithii*. A colorless single crystal of approximate dimensions 0.05 × 0.20 × 0.20 mm was mounted on a glass fiber. Buerger precession diagrams showed a triclinic crystal system. Structure solution was successful only in the non-centrosymmetric space group *P*1, later confirmed by the presence of twice the

same enantiomer. However, no attempt was made to determine *absolute* configuration as it seemed to be impossible (Jones, 1984). To get a better reflection/parameter ratio, all non-H atoms were assigned only individual isotropic thermal parameters in the final full-matrix least-squares refinement. More details of the intensity-data collection, structure solution and refinement are listed in Table 1. Final atomic coordinates are given in Table 2, distances and angles in Tables 3 and 4.* The two independent molecules *A* and *B* in the unit cell have the same configuration and structural features.

* Lists of structure factors, H-atom coordinates, further bond distances and angles, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51809 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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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