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# Structure of Leuconolam Sesquihydrate 

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#### Abstract

Ethyl-7,8,8a, 10,11,12a-hexahydro-12ahydroxyindolizino $[8,1-e f][1]$ benzazonine-6,13- ( $5 \mathrm{H}, 9 \mathrm{H}$ ) -dione sesquihydrate, $\mathrm{C}_{19} \mathrm{H}_{22} \mathrm{~N}_{2} \mathrm{O}_{3} \cdot \frac{3}{2} \mathrm{H}_{2} \mathrm{O}, M_{r}$ $=353.42$, triclinic, $P 1, a=9.250$ (2), $b=13.366$ (3), $c=9.217$ (2) $\AA, \quad \alpha=97.786$ (3),$\quad \beta=119.590$ (3), $\gamma$ $=70.726(3)^{\circ}, \quad V=934.8 \AA^{3}, \quad Z=2, \quad D_{x}=$ $1.255 \mathrm{~g} \mathrm{~cm}^{-3}$, Mo $K \alpha, \lambda=0.71073 \AA, \mu=0.839 \mathrm{~cm}^{-1}$,




Fig. 1. View of leuconolam illustrating atom labelling and the chair conformation of the six-membered ring (N4, C5-C8, C19). The interplanar angle between the benzene ring (C13-C18) and dihydropyrrole (C1, C2, C3, N4, C19) is 55.6 (3) ${ }^{\circ}$ [in molecule $B: 57 \cdot 2$ (4) ${ }^{\circ}$.
$F(000)=378, T=293 \mathrm{~K}$. The final $R$ value is 0.061 for 1646 significant $[I>3 \sigma(I)$ ] reflections. The alkaloid from the leaves of Rhazia stricta is built up by a

Table 1. Data-collection and structure-refinement parameters

| Crystal shape | Small plates |
| :---: | :---: |
| Diffractometer used | CAD-4, Enraf-Nonius |
| Method of intensity measurement | $\theta / 2 \theta$. |
| No. and $\theta$ range of reflections for lattice parameters | 25; 10-16 ${ }^{\circ}$ |
| Method used for absorption correction | No correction |
| Maximum value of $(\sin \theta) / \lambda$ reached in intensity measurement | $0.639 \AA^{-1}$ |
| Range of $h, k$ and $l$ | $0 \rightarrow 11,-17 \rightarrow 17,-11 \rightarrow 11$ |
| Standard reflections | 004, 122 |
| Interval, standard reflections measured | $\mathbf{2 h , ~ n o ~ i n t e n s i t y ~ v a r i a t i o n ~}$ |
| Total No. of reflections measured; $\theta$ range | 4062; $27^{\circ}$ |
| No. of observed reflections | 1646 with $I>3 \sigma(I)$ [ 1690 not observed, 2372 with $I>1 \sigma(I)]$ |
| Method used to solve structure | Direct methods (Sheldrick/ 1985) |
| Use of $F$ or $F^{\mathbf{2}}$ in LS refinement | $F$ |
| Method of locating H atoms | $\mathbf{H ( C )}$ calculated in idealized positions with $d(\mathrm{C}-\mathrm{H})=0.95 \AA$, included in structure-factor calculation |
| Weighting scheme | $1 / \sigma^{2}$ |
| Parameters refined | 203 |
| Value of $R$ | 0.061 |
| $V$ alue of $w R$ | 0.062 |
| Ratio of max. LS shift to e.s.d. ( $4 / \sigma$ ) | 0.0005 |
| Max. height in final $\boldsymbol{U F}$ map | $0.280 \mathrm{e} \mathrm{A}^{-3}$ |
| Error in an observation of unit weight | 0.875 |
| Secondary-extinction coefficient | 2.558 (1) $\times 10^{-7}$ (Zachariasen, 1963) |
| Source of atomic scattering factors | International Tables for X-ray Crystallography (1974) |
| Computer used | DEC PDP 11/60 |
| Programs used | SDP (B. A. Frenz \& Associates Inc., 1985) |

## Crystal shape

Method of intensity measurement
No. and $\theta$ range of reflections
Method used for absorption correction aximum value of $(\sin \theta) / \lambda$
reached in intensity measurement
Range of $h, k$ and $l$
Standard reflections
Interval, standard reflections measured
otal No. of reflections measured;

$$
\theta \text { range }
$$

No. of observed reflections

Method used to solve structure Use of $F$ or $F^{\mathbf{2}}$ in LS refinement

Weighting scheme
Parameters refined
alue of $R$
Value of $w R$
Ratio of max. LS shift to e.s.d. ( $\Delta / \sigma$ )
Error in an observation of unit weight
Secondary-extinction coefficient

Computer used
Programs used
Small plates
CAD-4, Enraf-Nonius
$\theta / 2 \theta$
$25 ; 10-16^{\circ}$
No correction
$0 \rightarrow 11,-17 \rightarrow 17,-11 \rightarrow 11$
$\mathbf{2 h}$, no intensity variation

4062; $27^{\circ}$

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Table 2. Positional parameters and isotropic thermal parameters $B\left(\AA^{2}\right)$

|  | $\boldsymbol{x}$ | $y$ | $z$ | B |
| :---: | :---: | :---: | :---: | :---: |
| Molecule A |  |  |  |  |
| 03 | 0.420 | 0.552 | 0.705 | 4.9 (1) |
| 011 | 0.1471 (9) | 0.4211 (6) | 0.8878 (9) | $6 \cdot 6$ (2) |
| 019 | 0.6203 (7) | 0.2119 (5) | 0.6198 (7) | $4 \cdot 2$ (1) |
| N4 | 0.5728 (8) | 0.3741 (6) | 0.7552 (8) | 3.7 (1) |
| N12 | 0.0580 (9) | 0.3196 (6) | 0.6673 (9) | $4 \cdot 6$ (2) |
| Cl | 0.3393 (9) | 0.3060 (7) | 0.604 (1) | $3 \cdot 3$ (2) |
| C2 | 0.280 (1) | 0.4119 (7) | 0.593 (1) | $4 \cdot 0$ (2) |
| C3 | 0.423 (1) | 0.4559 (7) | 0.685 (1) | 4.3 (2) |
| C5 | 0.750 (1) | 0.3781 (8) | 0.855 (1) | $5 \cdot 2$ (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) | $3 \cdot 8$ (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) | $5 \cdot 1$ (2) |
| C13 | $0 \cdot 104$ (1) | 0.2374 (7) | 0.566 (1) | 3.9 (2) |
| C14 | 0.2376 (9) | 0.2316 (6) | 0.5341 (9) | $3 \cdot 2$ (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 $B$ |  |  |  |  |
| O203 | 0.7247 (8) | 0.4513 (5) | 0.4374 (8) | 5.5 (2) |
| 0211 | 0.8547 (8) | 0.5765 (6) | $0 \cdot 1143$ (8) | 5.7 (2) |
| 0219 | 0.4540 (7) | 0.7926 (5) | 0.4526 (7) | 4.3 (1) |
| N204 | 0.6745 (8) | 0.6286 (6) | 0.4962 (8) | $4 \cdot 1$ (2) |
| N212 | 0.5847 (9) | $0 \cdot 6842$ (6) | -0.0384 (9) | 4.4 (2) |
| C201 | 0.5002 (9) | 0.6986 (6) | 0.226 (1) | $3 \cdot 3$ (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) | $3 \cdot 5$ (2) |
| C209 | 0.852 (1) | 0.7350 (7) | 0.394 (1) | 3.9 (2) |
| C210 | 0.796 (1) | 0.7519 (7) | 0.209 (1) | $4 \cdot 2$ (2) |
| C211 | 0.747 (1) | 0.6654 (7) | 0.093 (1) | $4 \cdot 2$ (2) |
| C213 | 0.440 (1) | 0.7682 (7) | -0.042 (1) | $4 \cdot 2$ (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) | $5 \cdot 6$ (3) |
| C219 | 0.5855 (9) | 0.7310 (6) | 0.4091 (9) | $3 \cdot 2$ (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) |  |  |  |  |
| 031 | 0.5070 (8) | 0.3256 (5) | 0.3471 (8) | $5 \cdot 2$ (2) |
| 032 | $0 \cdot 2538$ (8) | 0.6732 (5) | 0.4098 (8) | $5 \cdot 1$ (2) |
| 033 | 0.1966 (9) | 0.4987 (6) | 0.1922 (9) | $6 \cdot 5$ (2) |

benzene ring ortho disubstituted with a 4,5 -dihydro-5-hydroxy-2-oxo-pyrrol-1,4,5-triyl unit and an $\mathrm{N}(\mathrm{CO}) R$ - 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 $0.05 \times 0.20 \times 0.20 \mathrm{~mm}$ 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

Table 3. Selected distances ( $\AA$ ) and angles ( ${ }^{\circ}$ ) with e.s.d.'s

| O3 | C3 |  | 1.27 (1) | 0203 | C203 |  | 1.26 (1) |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 011 | C11 |  | 1.24 (1) | 0211 | C211 |  | 1.24 (2) |
| 019 | 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) | 011 | C11 | N12 | 118.5 (7) |
| C5 | N4 | C19 | $120 \cdot 3$ (6) | 011 | C11 | C10 | $120 \cdot 6$ (8) |
| C11 | N12 | C13 | 121.6 (7) | N12 | C11 | C10 | 120.9 (9) |
| C2 | C1 | C14 | 127.9 (7) | N12 | C13 | C14 | $120 \cdot 6$ (9) |
| C2 | CI | C19 | 109.2 (8) | N12 | C13 | C18 | 118 (2) |
| C14 | C1 | C19 | 122.9 (7) | C14 | C13 | C18 | 121.0 (9) |
| CI | 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) | 019 | C19 | N4 | 110.2 (9) |
| C7 | C8 | C19 | 105.4 (9) | 019 | C19 | C1 | 111.2 (6) |
| C7 | C8 | C81 | 108.0 (6) | 019 | C19 | C8 | 109.2 (6) |
| C9 | C8 | C19 | $112 \cdot 6$ (6) | N4 | C19 | C1 | 101.2 (6) |
| C9 | C8 | C81 | 113 (1) | N4 | C19 | C8 | $108 \cdot 6$ (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 | $120 \cdot 2$ (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 | $120 \cdot 2$ (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 | $111 \cdot 1$ (8) | C201 | C214 | C213 | $120 \cdot 6$ (7) |
| O203 | C203 | N204 | $124 \cdot 3$ (7) | C201 | C214 | C215 | 120.7 (9) |
| O203 | C203 | C202 | 128.9 (8) | C213 | C214 | C215 | 118.6 (8) |
| N204 | C203 | C202 | $106 \cdot 8$ (7) | C214 | C215 | C216 | 118 (2) |
| N204 | C205 | C206 | 109 (2) | C215 | C216 | C217 | 123 (1) |
| C205 | C206 | C207 | $110 \cdot 1$ (7) | C216 | C217 | C218 | 121 (2) |
| 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 | $106 \cdot 0$ (9) | O219 | C219 | C201 | $110 \cdot 2$ (6) |
| C207 | C208 | C281 | 109.4 (8) | O219 | C219 | C208 | 108.7 (7) |
| C209 | C208 | C219 | $110 \cdot 6$ (8) | N204 | C219 | C201 | 101.5 (6) |
| C209 | C208 | C281 | 112.1 (9) | N204 | C219 | C208 | 109.4 (6) |
| C219 | C208 | C281 | $110 \cdot 0$ (6) | C201 | C219 | C208 | 116.3 (9) |
| C208 | C209 | C210 | 118.6 (6) | C208 | C281 | C282 | 114.7 (7) |

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.

[^0]Table 4. Intermolecular hydrogen bonds $\mathrm{O}(a q) \cdots \mathrm{O}$

| 031..O19 | 2.67 (1) $\AA$ | O19...O31...O203 | $106.5(3)^{\circ}$ |
| :---: | :---: | :---: | :---: |
| O31...O203 | 2.79 (1) |  |  |
| 032...O3 | 2.797 (7) | O3..O32 $\ldots 0219$ | $106 \cdot 6$ (2) |
| O32 $\cdots$ O219 | 2.68 (1) |  |  |
| 033..O11 | 2.72 (1) | O11..033..0211 | $97 \cdot 3$ (3) |
| O33...0211 | $2 \cdot 72$ (1) |  |  |

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

Related literature. An orthorhombic phase of leuconolam. $\mathrm{C}_{2} \mathrm{H}_{5} \mathrm{OH}$ was reported by Wei, Ali, Goh, Sinn \& Butcher, 1986).

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# A Tetramethylethano-Bridged Difulvene 

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#### Abstract

Dimethyl-2,3-bis[3-(1-methylethylidene)-cyclopenta-1,4-dien-1-yl]butane, $\mathrm{C}_{22} \mathrm{H}_{30}, \quad M_{r}=294 \cdot 5$, monoclinic, $P 2_{1} / c, a=7.144$ (2), $b=19.698$ (2), $c$ $=7.449$ (3) $\AA, \quad \beta=117.51(2)^{\circ}, \quad V=929.8$ (9) $\AA^{3}, Z$ $=2, \quad D_{x}=1.052 \mathrm{~g} \mathrm{~cm}^{-3}, \quad \lambda(\mathrm{Mo} K \alpha)=0.71073 \AA, \quad \mu$ $=0.55 \mathrm{~cm}^{-1}, F(000)=324, T=297 \mathrm{~K}, R=0.063$ for 1120 data having $I>1 \sigma(I)$. 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) ${ }^{\circ}$. 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) $\AA$.


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).

[^1]0108-2701/89/081260-02803.00

Intensity data were obtained from an irregular fragment of dimensions $0.15 \times 0.23 \times 0.33 \mathrm{~mm}$ 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 $22>2 \theta>18^{\circ}$. The $\theta$ values were derived from measurements at $\pm 2 \theta$. One quadrant of data having $2<2 \theta<55^{\circ}, \quad 0 \leq h \leq 9, \quad 0 \leq k \leq 25$, $-9 \leq l \leq 9$ was measured using graphite-monochromated Mo $\mathrm{K} \mathrm{\alpha}$ radiation. 2128 reflections were measured. The $\omega-2 \theta$ scans were made at speeds ranging from 0.45 to $4.0^{\circ} \mathrm{min}^{-1}$ to measure all significant data with approximately equal precision. Three standard reflections ( $100,060,002$ ), measured every 10000 s of exposure time, exhibited only random fluctuations of less than $\pm 2 \%$ 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 $w=4 F_{o}{ }^{2}\left[\sigma^{2}(I)+\left(0.02 F_{o}{ }^{2}\right)^{2}\right]^{-1}$ using the Enraf-Nonius SDP (Frenz, 1985), scattering factors of Cromer \& Waber (1974), anomalous coefficients of Cromer (1974), and 1120 data having $I>1 \sigma(I)$. Non-H atoms were refined anisotropically; the H atoms were located © 1989 International Union of Crystallography


[^0]:    * 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.

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