

Table 1. *Coordinates and equivalent isotropic thermal parameters*

The equivalent isotropic thermal parameter, for atoms refined anisotropically, is defined by the equation: $B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3$.

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}(\text{\AA}^2)$
O1	0.4356 (4)	0.2007 (3)	0.6606 (1)	5.38 (6)
O2	0.1963 (4)	-0.0296 (3)	1.01088 (9)	5.44 (6)
C1	0.3250 (6)	0.2048 (4)	0.7201 (1)	4.29 (7)
C2	0.1334 (6)	0.3012 (4)	0.7187 (2)	5.31 (9)
C3	0.0051 (7)	0.3105 (4)	0.7741 (2)	5.56 (9)
C4	0.0543 (6)	0.2246 (4)	0.8342 (2)	4.34 (8)
C5	-0.0819 (6)	0.2285 (4)	0.8919 (2)	5.57 (9)
C6	-0.0307 (6)	0.1439 (4)	0.9487 (2)	5.47 (9)
C7	0.1639 (6)	0.0506 (4)	0.9509 (2)	4.57 (8)
C8	0.2997 (5)	0.0425 (4)	0.8961 (1)	3.88 (7)
C9	0.2494 (6)	0.1310 (3)	0.8361 (1)	3.75 (7)
C10	0.3821 (6)	0.1220 (4)	0.7781 (1)	3.90 (7)
C11	0.6100 (7)	0.0888 (4)	0.6554 (2)	6.0 (1)
C12	0.3946 (7)	-0.1203 (5)	1.0187 (2)	6.4 (1)

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

O1	C1	1.353 (4)	C4	C5	1.411 (4)		
O1	C11	1.413 (4)	C4	C9	1.420 (4)		
O2	C7	1.369 (4)	C5	C6	1.354 (5)		
O2	C12	1.432 (4)	C6	C7	1.416 (5)		
C1	C2	1.414 (5)	C7	C8	1.364 (4)		
C1	C10	1.376 (4)	C8	C9	1.422 (4)		
C2	C3	1.347 (4)	C9	C10	1.405 (4)		
C3	C4	1.412 (4)					
C1	O1	C11	117.2 (3)	C4	C5	C6	121.2 (3)
C7	O2	C12	117.8 (3)	C5	C6	C7	119.9 (3)
O1	C1	C2	114.3 (3)	O2	C7	C6	114.2 (3)
O1	C1	C10	125.5 (3)	O2	C7	C8	125.0 (3)
C2	C1	C10	120.2 (3)	C6	C7	C8	120.9 (3)
C1	C2	C3	119.9 (3)	C7	C8	C9	120.1 (3)
C2	C3	C4	121.9 (3)	C4	C9	C8	118.9 (3)
C3	C4	C5	122.7 (3)	C4	C9	C10	119.4 (3)
C3	C4	C9	118.3 (3)	C8	C9	C10	121.7 (3)
C5	C4	C9	119.0 (3)	C1	C10	C9	120.3 (3)

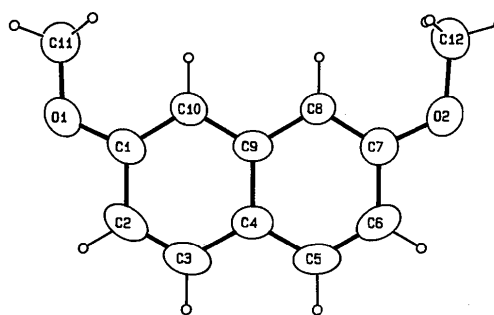


Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 40% probability level. H atoms are drawn as circles with the same arbitrary radius.

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1-Acetyl-2,7-dimethoxynaphthalene

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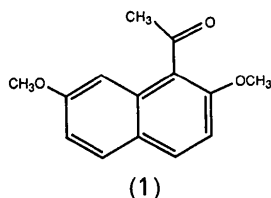
(Received 18 January 1989; accepted 6 March 1989)

Abstract. $C_{14}H_{14}O_3$, $M_r = 230.3$, monoclinic, $P2_1/c$, $a = 8.8107$ (9), $b = 18.372$ (3), $c = 7.7512$ (11) Å, $\beta = 98.49$ (1)°, $V = 1240.9$ (5) Å³, $Z = 4$, $D_x = 1.232$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.71073$ Å, $\mu = 0.80$ cm⁻¹, $F(000) = 488$, $T = 293$ K, $R = 0.047$ for 1909 observations (of 2848 unique data). The average deviation from planarity is 0.017 (2) Å with a maxi-

mum of 0.0285 (15) Å for the fused rings. The dihedral angle between the naphthalene system and the acetyl group is 117.91 (6)°. The methoxyl group *ortho* to the acetyl adopts a conformation with the methyl group *anti* to the neighboring α -carbon of the ring, with a C–C–O–C torsion angle of -178.7 (2)°. The other methoxyl group has the methyl *syn* to the neighboring α -carbon, with a C–C–O–C torsion angle of -1.3 (3)°.

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Experimental. Colorless needles of (1), m.p. 337–337.5 K, were isolated by recrystallization in hexane from the crude reaction product of 2,7-dimethoxynaphthalene and acetyl chloride in dichloroethane at room temperature (Gorelik, Reznichenko, Andronova & Luk'yanets, 1983). Crystal size 0.18 × 0.35 × 0.60 mm, space group from systematic absences $0k0$ with k odd and $h0l$ with l odd, cell dimensions from setting angles of 25 reflections having $10 < \theta < 11^\circ$. Data collection on Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, graphite monochromator, ω – 2θ scans designed for $I = 50\sigma(I)$, subject to max. scan time = 120 s, scan rates varied 0.53–4.00° min⁻¹. One quadrant of data having $2 < 2\theta < 55^\circ$, $0 \leq h \leq 11$, $0 \leq k \leq 23$, $-10 \leq l \leq 10$ measured. Data corrected for background, Lorentz and polarization effects, not for absorption. Standard reflections 300, 060, 004 varied randomly 1.6%, and no decay correction was applied. Redundant $0kl$ and $Ok\bar{l}$ data merged, $R_{\text{int}} = 0.018$, to yield 2848 unique data, 1909 observed with $I > 1\sigma(I)$. Structure solved by direct methods, using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), refined by full-matrix least squares based upon F with weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o)^2]^{-1}$ using Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Non-H atoms refined anisotropically; H atoms located by ΔF map and refined isotropically. Final $R = 0.047$, $wR = 0.046$, $S = 2.055$ for 211 variables. Max. shift 0.07 σ in the final cycle, max. residual density 0.13, min. -0.15 e \AA^{-3} , extinction coefficient $g = 6.8(11) \times 10^{-7}$ where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c . Coordinates* are given in Table 1; bond distances and angles are given in Table 2. The molecule is illustrated in Fig. 1.



Related literature. Crystal structures of 1-acetyl-2-ethoxynaphthalene: Gupta & Sahu (1972) and 1-acetyl-8-methoxynaphthalene: Schweizer, Procter, Kaftory & Dunitz (1978).

* Tables of H-atom coordinates, bond distances and angles involving H atoms, least-squares planes, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51815 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Coordinates and equivalent isotropic thermal parameters*

The equivalent isotropic thermal parameter, for atoms refined anisotropically, is defined by the equation:

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (Å ²)
O1	-0.1362 (1)	0.07604 (7)	-0.1991 (2)	6.03 (3)
O2	0.4990 (1)	0.16144 (7)	0.4738 (1)	5.69 (3)
O3	0.4323 (1)	0.11643 (7)	0.0204 (1)	5.83 (3)
C1	-0.0698 (2)	0.10363 (9)	-0.0420 (2)	4.46 (4)
C2	-0.1751 (2)	0.1342 (1)	0.0574 (2)	5.21 (4)
C3	-0.1248 (2)	0.1638 (1)	0.2135 (2)	5.04 (4)
C4	0.0332 (2)	0.16513 (8)	0.2831 (2)	3.95 (3)
C5	0.0882 (2)	0.19679 (9)	0.4458 (2)	4.83 (4)
C6	0.2389 (2)	0.1963 (1)	0.5130 (2)	4.95 (4)
C7	0.3444 (2)	0.16274 (9)	0.4185 (2)	4.21 (4)
C8	0.2970 (2)	0.13104 (8)	0.2586 (2)	3.62 (3)
C9	0.1392 (2)	0.13253 (8)	0.1849 (2)	3.55 (3)
C10	0.0829 (2)	0.10265 (8)	0.0194 (2)	3.91 (3)
C11	-0.0390 (2)	0.0449 (1)	-0.3100 (2)	5.99 (5)
C12	0.5560 (2)	0.1918 (1)	0.6396 (2)	6.42 (5)
C13	0.4125 (2)	0.09518 (9)	0.1631 (2)	4.13 (3)
C14	0.4980 (2)	0.0311 (1)	0.2455 (3)	6.24 (5)

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

O1	C1	1.368 (2)	C4	C5	1.408 (2)		
O1	C11	1.421 (2)	C4	C9	1.421 (2)		
O2	C7	1.366 (2)	C5	C6	1.353 (2)		
O2	C12	1.423 (2)	C6	C7	1.407 (2)		
O3	C13	1.210 (2)	C7	C8	1.378 (2)		
C1	C2	1.407 (2)	C8	C9	1.423 (2)		
C1	C10	1.359 (2)	C8	C13	1.496 (2)		
C2	C3	1.341 (2)	C9	C10	1.416 (2)		
C3	C4	1.417 (2)	C13	C14	1.491 (2)		
C1	O1	C11	118.1 (1)	O2	C7	C8	115.5 (1)
C7	O2	C12	118.6 (1)	C6	C7	C8	121.2 (1)
O1	C1	C2	113.9 (1)	C7	C8	C9	120.1 (1)
O1	C1	C10	125.3 (1)	C7	C8	C13	119.5 (1)
C2	C1	C10	120.8 (1)	C9	C8	C13	120.4 (1)
C1	C2	C3	120.0 (1)	C4	C9	C8	118.3 (1)
C2	C3	C4	121.7 (2)	C4	C9	C10	118.6 (1)
C3	C4	C5	122.5 (1)	C8	C9	C10	123.1 (1)
C3	C4	C9	118.4 (1)	C1	C10	C9	120.5 (1)
C5	C4	C9	119.2 (1)	O3	C13	C8	120.7 (1)
C4	C5	C6	122.0 (2)	O3	C13	C14	121.0 (2)
C5	C6	C7	119.3 (1)	C8	C13	C14	118.3 (1)
O2	C7	C6	123.3 (1)				

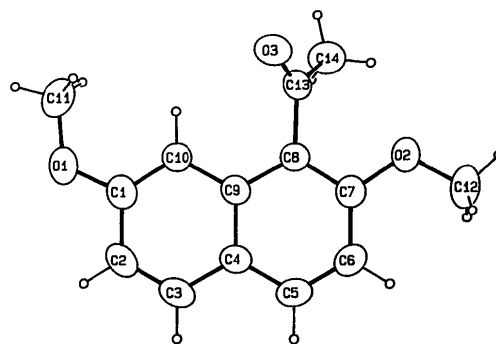


Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 40% probability level. H atoms are drawn as circles with the same arbitrary radius.

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Structure of Leuconolam Sesquihydrate

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Abstract. 8a-Ethyl-7,8,8a,10,11,12a-hexahydro-12a-hydroxyindolino[8,1-ef][1]benzazone-6,13-(5H,9H)-dione sesquihydrate, $C_{19}H_{22}N_2O_3 \cdot \frac{3}{2}H_2O$, $M_r = 353.42$, triclinic, $P1$, $a = 9.250$ (2), $b = 13.366$ (3), $c = 9.217$ (2) Å, $\alpha = 97.786$ (3), $\beta = 119.590$ (3), $\gamma = 70.726$ (3)°, $V = 934.8$ Å³, $Z = 2$, $D_x = 1.255$ g cm⁻³, $Mo K\alpha$, $\lambda = 0.71073$ Å, $\mu = 0.839$ cm⁻¹,

$F(000) = 378$, $T = 293$ 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 θ range of reflections for lattice parameters	25; 10-16°
Method used for absorption correction	No correction
Maximum value of $(\sin\theta)/\lambda$ reached in intensity measurement	0.639 Å ⁻¹
Range of h, k and l	0-11, -17-17, -11-11
Standard reflections	004, 122
Interval, standard reflections measured	2 h, no intensity variation
Total No. of reflections measured; θ range	4062; 27°
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^2 in LS refinement	F
Method of locating H atoms	H(C) calculated in idealized positions with $d(C-H) = 0.95$ Å, included in structure-factor calculation
Weighting scheme	$1/\sigma^2$
Parameters refined	203
Value of R	0.061
Value of wR	0.062
Ratio of max. LS shift to e.s.d. (d/σ)	0.0005
Max. height in final ΔF map	0.280 e Å ⁻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	<i>International Tables for X-ray Crystallography</i> (1974)
Computer used	DEC PDP 11/60
Programs used	SDP (B. A. Frenz & Associates Inc., 1985)

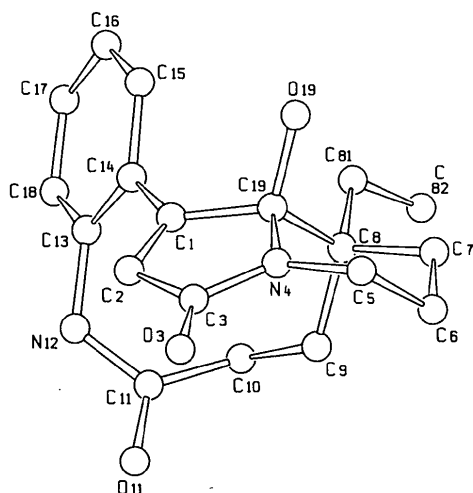


Fig. 1. View of leuconolam illustrating atom labelling and the chair conformation of the six-membered ring (N4, C5-C8, C19). The interplanar angle between the benzene ring (C13-C18) and dihydropyrrrole (C1, C2, C3, N4, C19) is 55.6 (3)° [in molecule B: 57.2 (4)°].