

parameters, $\Delta/\sigma < 0.20$ in final cycle; anisotropic temperature factors for nonhydrogen atoms, further Fourier difference functions permitted location of hydrogen atoms, included in refinement; model converged with $R = 3.7$ and $wR = 3.7\%$, $w = 1/\sigma^2(F)$; final difference Fourier map featureless; atomic scattering and anomalous scattering factors for all atoms from Cromer & Waber (1974).*

Discussion. Table 1 lists the atomic coordinates and equivalent isotropic thermal parameters. Table 2 contains bond distances and angles. Fig. 1 shows the single molecule and Fig. 2 is the packing diagram. Hydrogen bonds are formed from O(*e*) to O(2)[$\frac{1}{2}+x$, $\frac{1}{2}-y$, $1-z$], N(1) to O(5)[$1-x$, $\frac{1}{2}+y$, $\frac{3}{2}-z$], O(21) to O(*e*)[$1-x$, $y-\frac{1}{2}$, $\frac{3}{2}-z$] with distances of 2.788 (5), 2.855 (5) and 2.806 (5) Å respectively. O(*e*) thus acts both as a donor and as an acceptor. The structure of leuconolam shows that it is an 'opened-up' indole alkaloid artefact derived from an aspidosperma-type alkaloid. Only one other natural product, rhazinilam, C₁₉H₂₂N₂O (Abraham & Rosenstein, 1972), has previously been found to possess this structural skeleton. As shown in Fig. 1, conjugation of the benzene ring with N(1) as well as the unsaturated five-membered ring is limited by non-planarity [benzene/pyrrolidine and benzene/N(1)CO interplanar

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42572 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

angles being 125.5 (2) and 60.4 (3)° respectively]. This accounts for the weak UV absorptions and also explains other salient features in the NMR spectra. It may also be noted that the structure can allow for other possible conformational isomers owing to the conformational mobility of the nine- and six-membered rings.

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1,4-Dibromonaphthalene; a Redetermination

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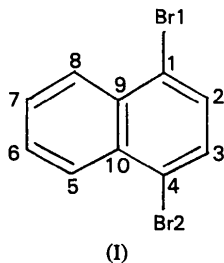
Abstract. C₁₀H₆Br₂, $M_r = 285.99$, monoclinic, $P2_1/c$, $a = 4.063$ (3), $b = 16.476$ (3), $c = 27.274$ (8) Å, $\beta = 91.84$ (3)°, $V = 1824.8$ (18) Å³, $Z = 8$ (two molecules/asymmetric unit), $D_x = 2.081$ g cm⁻³, Mo $K\alpha$, $\lambda\alpha_1 = 0.70930$ Å, $\mu = 87$ cm⁻¹, $F(000) = 1088$, $T = 293$ K, $R = 0.044$ for 1350 observed reflections. Both

molecules are planar, with bond lengths similar to those in naphthalene, but with some bond-angle differences due to the presence of the Br substituents, the endocyclic angles at the substituted 1 and 4 positions having a mean value of 123.8 (6)°.

Introduction. A two-dimensional study of the structure of 1,4-dibromonaphthalene (I) in which only y and z parameters were determined (Trotter, 1961, with a and

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c axes interchanged) has now been completed with three-dimensional data, and the results compared with those of an independent study (Bellows, Stevens & Prasad, 1978) and with those for naphthalene.



Experimental. Colourless crystals from aqueous ethanol, plates elongated along *a* with (010) developed, 0.4 × 0.02 × 0.25 mm, CAD-4 diffractometer, lattice parameters from 18 reflections ($\pm\theta$) with $\theta = 4.6$ – 18.4° . Intensities for $\theta \leq 22.5^\circ$, *hkl*: -3 to 4 , 0 to 17 , -29 to 29 , ω - 2θ scan, ω scan width $(1.0 + 0.35 \tan \theta)^\circ$ at 0.7 – $10^\circ \text{ min}^{-1}$, extended 25% on each side for background measurement, horizontal aperture $(2 + 0.5 \tan \theta)$ mm, vertical aperture 4 mm, three standard reflections showed a steady decay (crystal slightly volatile) and data scaled accordingly, *Lp* and absorption corrections (transmission factors 0.20–0.84), 3037 reflections measured, 1933 unique non-zero intensities ($R_{\text{int}} = 0.039$), 1350 with $I \geq 2\sigma(I)$, where $\sigma^2(I) = S + 4(B_1 + B_2)$, $S = \text{scan}$, B_1 and $B_2 = \text{background counts}$. Structure by Patterson (36 independent vectors) and Fourier methods, refined by full-matrix least squares on *F* with *SHELX76* (Sheldrick, 1976), H atoms in calculated positions on ring diagonals ($\text{C-H} = 1 \text{ \AA}$, $U = 0.08 \text{ \AA}^2$) not refined, $w = 1/[\sigma^2(F) + 0.01F^2]$, final $R = 0.044$, $wR = 0.067$, $S = 0.66$ for 1350 reflections with $I \geq 2\sigma(I)$, $R = 0.098$ for all 1933 reflections, $(\Delta/\sigma)_{\text{max}} = 0.056$, final difference density -0.52 to $+0.71 \text{ e \AA}^{-3}$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Final parameters are in Table 1.* The positional parameters agree well with those of Bellows *et al.* (1978), a half-normal probability plot of the differences between the 72 parameters being a straight line of zero intercept and unit slope. However, the unit-cell edges of the present study are uniformly larger (by 3–8 σ) than those of Bellows *et al.* (1978).

* Lists of structure factors, anisotropic thermal parameters, calculated H positions, bond lengths and angles, mean-planes' calculations and half-normal probability plot data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42617 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

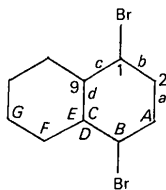
Table 1. Final positional parameters with standard deviations in parentheses (fractional, $\times 10^5$ for Br, $\times 10^4$ for C) and equivalent isotropic thermal parameters $[(U_{11} + U_{22} + U_{33})/3, \text{ \AA}^2 \times 10^3]$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Br(1)	32716 (35)	14476 (9)	10173 (5)	58
Br(2)	69694 (36)	41134 (9)	-6587 (5)	65
Br(1')	-15998 (31)	45325 (9)	14180 (4)	53
Br(2')	29772 (35)	19004 (8)	31107 (5)	56
C(1)	4449 (27)	2192 (8)	506 (4)	43
C(2)	3582 (32)	2976 (8)	588 (5)	53
C(3)	4292 (31)	3562 (8)	225 (5)	48
C(4)	5938 (29)	3301 (8)	-182 (5)	43
C(5)	8475 (30)	2240 (8)	-681 (4)	44
C(6)	9198 (31)	1437 (10)	-747 (4)	53
C(7)	8394 (30)	882 (8)	-406 (4)	48
C(8)	6843 (29)	1097 (7)	12 (4)	43
C(9)	5976 (25)	1917 (7)	93 (4)	31
C(10)	6814 (25)	2479 (7)	-270 (4)	32
C(1')	-201 (28)	3798 (8)	1924 (4)	46
C(2')	-992 (28)	2990 (8)	1861 (4)	44
C(3')	8 (31)	2430 (8)	2224 (5)	47
C(4')	1716 (26)	2691 (7)	2630 (4)	37
C(5')	4485 (28)	3793 (8)	3123 (4)	41
C(6')	5180 (31)	4607 (8)	3179 (4)	51
C(7')	4237 (32)	5156 (8)	2810 (5)	50
C(8')	2493 (29)	4916 (7)	2406 (4)	42
C(9')	1656 (26)	4093 (7)	2332 (4)	34
C(10')	2621 (27)	3513 (8)	2703 (4)	37

Both molecules are planar within experimental error. The C–C bond distances do not show any significant deviations from *mmm* symmetry for the ring system, the r.m.s. differences among equivalent bond lengths being of the same order of magnitude as the least-squares standard deviations; the mean bond lengths are quite similar to those in unsubstituted naphthalene (Table 2), with bonds of the type C(1)–C(2) being the shortest in the molecule. The C–Br distances, mean 1.917 (6) \AA , correspond to single bonds. Bond angles do show significant differences from those in naphthalene (Table 2), the endocyclic C–C–C angles at the substituted C(1) and C(4) atoms being increased to a mean of 123.8 (6) $^\circ$, as a result of the σ -electron-withdrawing power of the Br atoms (Domenicano, Vaciego & Coulson, 1975); angles of the type C(2)–C(1)–Br are decreased to a mean of 116.5 (5) $^\circ$, probably as a result of Br...H(8) (2.7 \AA) intramolecular repulsion. The molecules are stacked along the short *a* axis, with interplanar spacing 3.53 (1) \AA ; the two independent molecules are related very roughly by a pseudo-twofold axis along *a* at $y = 0.30$, $z = 0.12$. The shortest Br...Br intermolecular contacts are 3.613, 4.042, 4.151 (2) \AA , the shortest being somewhat less than the sum of the van der Waals radii, 3.9 \AA . The shortest Br...C, Br...H, C...C, C...H, and H...H contacts are 3.78 (1), 3.02, >3.5 , 2.9, and 2.5 \AA , respectively.

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Table 2. Mean bond lengths (Å) and angles (°) in naphthalene and 1,4-dibromonaphthalene (numbers in parentheses are standard deviations of the means calculated from the LS e.s.d.'s and independently from the r.m.s. deviations from the means for the dibromo compound)



	Naphthalene		Dibromonaphthalene	
	Cruickshank (1957) (no libration correction)	Pawley & Yeats (1969)	<i>mmm</i>	<i>mm</i>
<i>a</i>	1.416 (6)	1.401 (7)	1.393 (9,14)	1.410 (12,9) 1.376 (12,23)
<i>b</i>	1.357 (4)	1.372 (5)	1.368 (6,4)	1.370 (9,7) 1.366 (9,6)
<i>c</i>	1.420 (3)	1.423 (5)	1.409 (6,6)	1.407 (8,9) 1.411 (8,9)
<i>d</i>	1.405 (6)	1.412 (7)	1.422 (11,16)	1.422 (11,16)
Br—C	—	—	1.917 (6,6)	1.917 (6,6)
<i>A</i>	120.5 (2)	120.7 (1)		118.7 (6,5)
<i>B</i>	120.3 (3)	120.2 (2)		123.8 (6,5)
<i>C</i>	119.2 (2)	119.2 (1)		117.6 (6,6)
<i>D</i>	121.5 (3)	[121.6 (1)]		123.6 (6,4)
<i>E</i>	119.2 (2)	119.2 (1)		118.8 (6,9)
<i>F</i>	120.3 (3)	120.2 (2)		120.2 (6,3)
<i>G</i>	120.5 (2)	120.7 (1)		120.9 (6,5)
Br—C(1)—C(2) type	—	—		116.5 (5,7)
Br—C(1)—C(9) type	—	—		119.7 (5,3)

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Interatomic Distances and Angles in Four Planar Systems with Adjacent C—O and C—N Bonds: Structures of Pivalamide (I), Dipivalamide (II), *N*-Pivaloylpivalamidinium Pyrosulfate (III) and *N*-Pivaloylpivalamidine (IV)

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Abstract. 2,2-Dimethylpropionamide (pivalamide), C₅H₁₁NO (I): *M_r* = 101.15, monoclinic, *P*2₁/*a*, *a* = 10.303 (1), *b* = 5.896 (1), *c* = 10.404 (2) Å, β = 102.65 (1)°, *V* = 616.7 (2) Å³, *Z* = 4, *D_m* = 1.1 (1), *D_x* = 1.0894 (3) Mg m⁻³, Mo *Kα*, λ = 0.71069 Å for

graphite-monochromatized radiation, μ(Mo *Kα*) = 0.071 mm⁻¹, *T* = 140 K, *F*(000) = 224, *R* = 0.044 for 1035 unique reflections. Bis(2,2-dimethylpropionyl)-amine (dipivalamide), C₁₀H₁₉NO₂ (II): *M_r* = 185.27, orthorhombic, *Pcab*, *a* = 10.889 (8), *b* = 11.545 (15),

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