

different angles be calculated: 1·2 and 2·2°. In order to elucidate whether the observed ring twist is due to steric effects in the ring itself, the free cyclam amine (in the absence of water molecules) has been modelled (including all H atoms and lone electron pairs at N atoms) by the strain-energy-minimization method (program MM1, Allinger, Sprague & Yuh, 1975). It appears that the minimized strain energy for the amine molecule possessing C_{2h} symmetry may be reduced by about 5% when the symmetry of the molecule at the start of the minimization is reduced to C_i (by deleting the plane and axis restrictions). A ring twist of 3·34° is then observed as a result of ring-geometry optimization. Despite the fact that such a modelling attempt gives only a crude approximation to the truth (e.g. the internal hydrogen-bonding effect was not taken into account), it may be said that the observed ring twist in cyclam derivatives may be partly due to internal steric effects.

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Ohchinolide A, a New Limonoid from *Melia azedarach* L. var. *japonica* Makino

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Abstract. C₃₇H₄₂O₁₀, monoclinic, P2₁, *a* = 16·284 (1), *b* = 9·648 (1), *c* = 10·700 (1) Å, β = 98·08 (1)°, *Z* = 2, *D_x* = 1·29, *D_m* = 1·30 Mg m⁻³. The structure was solved by the direct method and refined by the block-diagonal least-squares technique to *R* = 0·049 for 2949 reflexions. Rings *A* and *B* are in the chair

conformation, ring *C* is in the boat conformation, and rings *D* and *E* take the envelope form.

Introduction. The molecular structures of ohchinolide A and B, isolated from an ether extract of the dry fruit of *Melia azedarach* L. var. *japonica* Makino, have been

Table 1. *Positional parameters* ($\times 10^4$) and *isotropic thermal parameters* ($\text{\AA}^2 \times 10^3$) of ohchinolide A

	x	y	z	U_{eq}
C(1)	2571 (2)	8269 (0)	2294 (3)	43 (1)
C(2)	1786 (2)	8598 (5)	1366 (3)	49 (1)
C(3)	970 (2)	8688 (5)	1933 (3)	45 (1)
C(4)	1061 (2)	9669 (5)	3060 (3)	47 (2)
C(5)	1856 (2)	9234 (5)	3925 (3)	45 (1)
C(6)	1805 (2)	9996 (5)	5128 (3)	43 (1)
C(7)	2353 (2)	9293 (5)	6197 (3)	41 (1)
C(8)	3272 (2)	9237 (4)	5933 (2)	41 (1)
C(9)	3305 (2)	8591 (5)	4581 (2)	41 (1)
C(10)	2696 (2)	9244 (5)	3464 (3)	46 (1)
C(11)	4200 (2)	8494 (7)	4226 (3)	53 (2)
C(12)	4971 (2)	8356 (5)	5176 (3)	49 (2)
C(13)	3785 (2)	8310 (5)	8186 (3)	42 (1)
C(14)	3726 (2)	8237 (4)	6922 (3)	43 (1)
C(15)	4206 (2)	7019 (5)	6532 (3)	49 (2)
C(16)	4409 (2)	6145 (5)	7702 (3)	50 (2)
C(17)	4257 (2)	7077 (5)	8830 (3)	44 (1)
C(18)	3479 (2)	9369 (5)	9035 (3)	46 (1)
C(19)	2981 (2)	10672 (5)	3019 (3)	48 (1)
C(20)	5026 (2)	7505 (5)	9661 (3)	46 (1)
C(21)	5211 (2)	7319 (6)	10919 (3)	52 (2)
C(22)	5720 (2)	8228 (6)	9295 (4)	53 (2)
C(23)	6273 (2)	8424 (6)	10357 (4)	56 (2)
O(24)	5980 (2)	7855 (5)	11365 (3)	54 (1)
O(25)	4990 (1)	7515 (4)	6164 (2)	48 (1)
O(26)	5600 (2)	8936 (5)	5017 (3)	54 (1)
O(27)	936 (1)	9971 (5)	5248 (2)	52 (1)
O(28)	2080 (1)	7875 (4)	6277 (2)	44 (1)
C(29)	1785 (2)	7453 (5)	7322 (3)	45 (1)
C(30)	464 (2)	9497 (6)	4065 (3)	53 (2)
C(31)	1010 (2)	11179 (5)	2575 (4)	53 (2)
C(32)	3663 (2)	10690 (5)	6103 (3)	50 (2)
O(33)	1617 (2)	8223 (4)	8142 (2)	52 (1)
C(34)	1724 (2)	5918 (5)	7379 (3)	46 (1)
C(35)	1415 (2)	5331 (6)	8408 (4)	48 (1)
C(36)	1356 (3)	3890 (7)	8471 (6)	56 (2)
C(37)	1598 (4)	3060 (6)	7538 (8)	60 (2)
C(38)	1907 (5)	3664 (7)	6546 (8)	60 (2)
C(39)	1974 (4)	5093 (6)	6447 (6)	53 (2)
O(40)	2508 (1)	6856 (4)	2744 (2)	44 (1)
C(41)	2974 (3)	5825 (6)	2111 (4)	54 (2)
O(42)	3067 (4)	6016 (6)	1146 (5)	64 (1)
C(43)	2670 (3)	4458 (5)	2696 (5)	57 (2)
O(44)	744 (1)	7335 (4)	2365 (2)	50 (1)
C(45)	331 (2)	6454 (6)	1523 (4)	54 (2)
O(46)	126 (2)	6764 (5)	434 (3)	61 (1)
C(47)	168 (3)	5095 (7)	2114 (6)	59 (2)

determined by spectral studies and confirmed by X-ray analysis of the former compound (Ochi, Kotsuki, Ido, Nakai, Shiro & Tokoroyama, 1979). The molecular structure of ohchinolide A, refined after recollection of the intensity data on a diffractometer available for a wider range of θ , is reported here in detail.

Crystals were obtained from an ether solution. The systematic absences are $0k0$ when k is odd. Intensities of 3148 independent reflexions with $\theta \leq 70.0^\circ$ were collected on a Rigaku diffractometer with graphite-monochromated $\text{Cu } K\alpha$ radiation and a crystal of dimensions $0.1 \times 0.1 \times 0.1$ mm. These were cor-

rected for Lorentz and polarization effects, but not for absorption. The structure was solved by use of the program *MULTAN 76* (Main, Lessinger, Woolfson, Germain & Declercq, 1976). A difference electron density map was calculated after block-diagonal least-squares refinement, from which the positions of the H atoms were revealed. After anisotropic refinement of the non-hydrogen atoms by the block-diagonal least-squares technique, the R value ($\sum |ΔF|/\sum |F_o|$) converged to 0.049 for 2949 reflexions. The atomic scattering factors were calculated by use of the analytical expression $f = \sum [a_i \exp(-b_i \lambda^{-2} \sin^2 \theta)] + c$ ($i = 1 \sim 4$) (*International Tables for X-ray Crystallography*, 1974). The weighting scheme used was $w = 1/\sigma^2(F_o)$ for $|F_c| \geq \sigma(F_o)$ and $w = 0$ for $|F_c| < \sigma(F_o)$ or $|ΔF| > 3\sigma(F_o)$. $\sigma(F_o)$ was estimated by the relation $\sigma(F_o) = [\sigma_1^2(F_o) + 0.00280|F_o|^2]^{1/2}$ where $\sigma_1(F_o)$ is the estimated standard deviation depending on

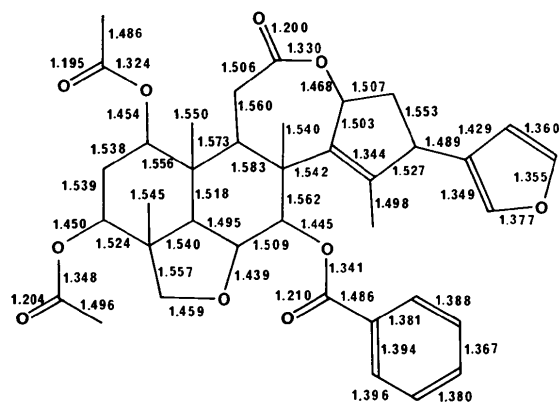


Fig. 1. Bond distances (Å). The estimated standard deviations range from 0.004 to 0.009 Å.

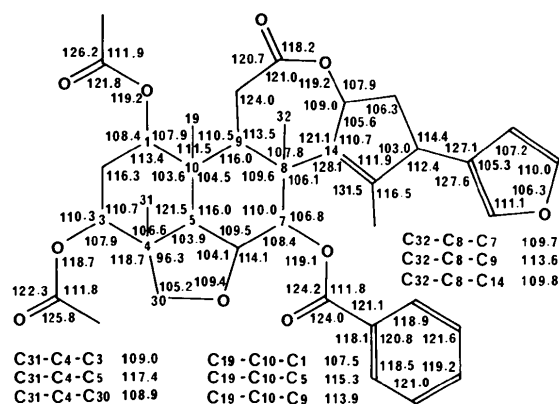


Fig. 2. Bond angles ($^\circ$). The estimated standard deviations range from 0.2 to 0.6 $^\circ$.

the counting errors (Grant, Killean & Lawrence, 1969).*

Discussion. Final positional parameters are listed in Table 1. Bond distances and angles are given in Figs. 1 and 2, respectively. The mean estimated standard deviations are 0.006 Å for bond lengths and 0.3° for angles. A perspective view of the molecule is shown in Fig. 3. Some of the bond lengths, especially those related by C(9), deviate significantly from their standard values.

The ring conformations are as follows:

	Ring	Conformation
A:	C(1), C(2), C(3), C(4), C(5), C(10)	chair
B:	C(5), C(6), C(7), C(8), C(9), C(10)	chair
C:	C(8), C(9), C(11), C(12), O(25), C(15), C(14)	boat
D:	C(13), C(14), C(15), C(16), C(17)	envelope
E:	C(4), C(5), C(6), O(27), C(30)	envelope.

Rings *A*, *B*, *C* and *E* are fused with *trans* ring junctions. The seven-membered ring may be described as consisting of the three planes C(14)–C(15)–O(25),

* Lists of structure factors, anisotropic thermal parameters and H-atom positional parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35163 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Structure of 2,4,6-Tri-*tert*-butyl-*N*-sulphonylaniline

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Abstract. C₁₈H₂₉NOS, *M_r* = 307.49, monoclinic, *P*2₁/*n*, *a* = 15.871 (2), *b* = 11.584 (2), *c* = 10.062 (2) Å, β = 90.12 (1)°, *U* = 1849.8 (5) Å³, *Z* = 4, *D_x* = 1.104 Mg m⁻³, *F*(000) = 672. The final *R* value is 0.051 for 2491 observed reflexions. The S=O and N=S lengths are 1.436 and 1.494 Å, respectively, and the OSN and SNC angles are 121.9 and 131.7°, respectively. The terminal O atom is *cis* to the C atom

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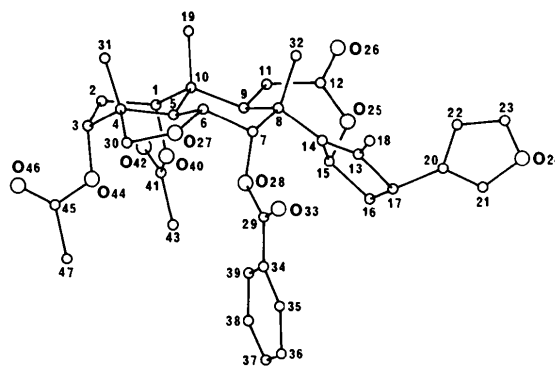


Fig. 3. Perspective view of the molecule with the atom-numbering system.

C(8)–C(12)–O(25)–C(14), and C(8)–C(9)–C(11)–C(12). The bonds C(15)–O(25) and C(17)–C(20) are *cis* in relation to each other.

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across the N=S bond with a torsion angle of 0.6°. The molecule has approximate mirror symmetry, apart from the methyl groups in the 4-*tert*-butyl group.

Introduction. Reaction of 2,4,6-tri-*tert*-butylaniline with disulphur dichloride gave a heterocyclic compound, 2,4,6-tri-*tert*-butyl-7,8,9-dithiazabicyclo[4.3.0]nona-1(9),2,4-triene (1). Treatment of (1) with Ph₃P in

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