

Table 2. Torsion angles ($^{\circ}$) (*e.s.d.*'s $\approx 0.6^{\circ}$)

Ring A:	C(1) — C(2) — C(3) — C(4) — C(5) — C(10) — C(1)	56.1	-54.9	54.2	-52.0	48.5	-50.9
Ring B:	C(6) — C(7) — C(8) — C(9) — C(10) — C(5) — C(6)	57.8	-58.4	57.9	-55.3	52.0	-55.1
Ring C:	C(9) — C(11) — C(12) — C(13) — C(14) — C(8) — C(9)	52.5	-56.9	52.5	-48.4	49.3	-49.6
Ring D:	C(13) — C(14) — C(15) — C(16) — C(17) — C(13)	-35.6	34.6	-20.6	-1.7	23.1	
About C(17)–C(20)							
C(13)–C(17)–C(20)–C(21)	-118.1						
C(13)–C(17)–C(20)–C(22)	67.0						
C(16)–C(17)–C(20)–C(21)	118.9						
C(16)–C(17)–C(20)–C(22)	-55.9						

ring *D* has the α -envelope conformation and the lactone ring is planar.

Comparison of torsion angles in this structure (Table 2) to those in related structures (Go, Kartha & Chen, 1980; Go & Kartha, 1980) shows that the main differences between these structures are in the conformations of ring *D* and the lactone group. The conformation of methyl digitoxigenin is similar to those of digitoxigenin (Karle & Karle, 1969) and strophanthidin B (Gilardi & Flippen, 1973).

The correlation observed by Go & Kartha (1980) between the bond angles C(17)–C(20)–C(21) and C(17)–C(20)–C(22), and the torsion angle C(13)–C(17)–C(20)–C(22) is observed in methyl digitoxigenin. When this torsion angle is negative as in gitoxin (Go & Kartha, 1980) and digoxin (Go, Kartha & Chen, 1980), the above two bond angles are similar; whereas the difference between the two angles is about 14° when the torsion angle is positive as in digitoxigenin (Karle & Karle, 1969), anhydrodigitoxigenin (Gilardi & Karle, 1970) and 5β -hydroxy digitoxigenin (Przybylska & Ahmed, 1979). In the present study also, the torsion angle is positive (with the steroid

backbone having the same configuration as in the other structures) and the difference between the bond angles is 17.4° .

The packing of the molecules is similar to that of digitoxigenin (Karle & Karle, 1969). A hydrogen bond of length 2.81 (0.1) Å, between the terminal hydroxyl group O(1) and the carbonyl oxygen O(4) of the lactone group, forms infinite chains along the *b* axis.

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Structures of Polycyclic Polyamines: 1,5,9,13-Tetraazatricyclo[11.3.1.1^{5,9}]octadecane, C₁₄H₂₈N₄*

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Abstract. $M_r = 252.8$, monoclinic, $P2_1/c$, $a = 96.61$ (2) $^{\circ}$, $V = 2988.8$ Å³, $D_x = 1.122$ Mg m⁻³, $Z = 8$, $\lambda(\text{Cu K}\alpha_1) = 1.5406$ Å, $\mu = 0.491$ mm⁻¹, $T = 296$ K. Final $R = 0.056$ for 2713 observed reflections. The central 12-membered ring adopts the 'square' [3333] conformation.

* NRCC publication No. 20721.

Introduction. The structure of 1,5,9,13-tetraazatricyclo[11.3.1.1^{5,9}]octadecane has been determined as the second in a series of investigations into the solid-state stereochemistry of polycyclic polyamines (Gabe, Le Page, Prasad & Weisman, 1982). Solution conformational analysis, empirical force-field calculations and dynamic NMR studies will be reported elsewhere (Weisman, Coolidge, Petillo & Johnson, 1982).

Experimental. Prepared by condensation of aqueous formaldehyde with 1,5,9,13-tetraazacyclohexadecane (Smith, Ekstrand & Raymond, 1978) and crystallized by slow evaporation of a CH₂Cl₂/hexane solution.

Crystal 0.4 × 0.3 × 0.3 mm cut from a larger needle crystal with a solvent saw using ethanol; 5770 intensities measured ($2\theta_{\max} = 120^\circ$), 4746 unique reflections, 2713 considered significant at the $2\sigma(I)$ level, $\theta/2\theta$ scan technique, graphite-monochromatized Cu K α and profile analysis (Grant & Gabe, 1978), Picker four-circle automatic diffractometer; cell dimensions from least squares applied to 33 reflections with $2\theta \geq 62^\circ$, corrections applied for Lorentz and polarization effects but not for absorption; structure solved using *MULTAN* (Germain, Main & Woolfson, 1971), all non-H atoms appeared in the first *E* map, positional and anisotropic thermal parameters for C and N refined by block-diagonal least squares on $\sum w(F_o - F_c)^2$; H atoms included in later cycles at their calculated positions and then refined together with isotropic thermal parameters; final $R = 0.056$, $R_w = 0.039$ for observed data, $w = 1/\sigma^2(F)$ where $\sigma(F)$ is based on counting statistics; $R = 0.097$, $R_w = 0.040$ for all data; NRC system of programs for the PDP-8e (Larson & Gabe, 1978); scattering factors from *International Tables for X-ray Crystallography* (1974), $F(000) = 1120$.

Discussion. Final positional and thermal parameters listed in Table 1.*

The atomic-numbering scheme is shown in Fig. 1, together with the bond distances and angles. A stereoview of the molecules is shown in Fig. 2. Some transannular distances are given in Table 2.

The central 12-membered ring adopts the 'square' [3333] conformation, $g^\pm g^\pm a g^\pm g^\pm a g^\pm g^\pm a g^\pm g^\pm a$ (Dale, 1976), preferred by cyclododecane in the solid state (Dunitz & Shearer, 1960; Dunitz, 1968) and in solution (Anet, Cheng & Wagner, 1972). Structures of two other saturated, nitrogen-containing, 12-membered heterocycles with the [3333] conformation have been

Table 1. *Atomic coordinates and isotropic thermal parameters*

E.s.d.'s refer to the last digit printed. B_{eq} is the arithmetic mean of the principal axes of the thermal ellipsoid.

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}/B_{iso}(\text{\AA}^2)$
N(1)	0.54719 (23)	0.2291 (3)	0.56159 (7)	4.32 (14)
C(2)	0.4516 (3)	0.3236 (4)	0.53780 (10)	5.80 (21)
C(3)	0.3793 (3)	0.4175 (4)	0.56715 (10)	5.87 (22)
C(4)	0.4653 (3)	0.5244 (4)	0.59541 (10)	5.14 (20)
N(5)	0.41147 (24)	0.5659 (3)	0.63469 (8)	4.80 (15)
C(6)	0.4720 (3)	0.6994 (4)	0.65567 (11)	5.45 (21)
C(7)	0.6083 (3)	0.6745 (4)	0.67403 (11)	5.60 (21)
C(8)	0.6174 (3)	0.5401 (4)	0.70405 (10)	5.41 (21)
N(9)	0.55826 (24)	0.4115 (3)	0.68069 (7)	4.32 (14)
C(10)	0.5723 (3)	0.2783 (4)	0.70814 (10)	5.65 (21)
C(11)	0.5451 (3)	0.1362 (4)	0.68083 (10)	5.66 (22)
C(12)	0.6411 (3)	0.1155 (4)	0.64883 (10)	4.76 (19)
N(13)	0.58458 (24)	0.0236 (3)	0.61191 (8)	4.88 (16)
C(14)	0.6827 (3)	-0.0360 (4)	0.58728 (12)	5.96 (22)
C(15)	0.7386 (3)	0.0799 (4)	0.55959 (10)	5.15 (20)
C(16)	0.6362 (3)	0.1696 (4)	0.53406 (10)	4.93 (20)
C(17)	0.4917 (3)	0.1047 (4)	0.58368 (10)	5.22 (20)
C(18)	0.4258 (3)	0.4440 (4)	0.66626 (10)	4.50 (18)
H(2A)	0.3789 (24)	0.247 (3)	0.5189 (8)	6.7 (8)
H(2B)	0.511 (3)	0.396 (3)	0.5165 (9)	9.7 (10)
H(3A)	0.3255 (25)	0.346 (3)	0.5825 (8)	7.7 (9)
H(3B)	0.3130 (24)	0.477 (3)	0.5509 (8)	7.3 (9)
H(4A)	0.5590 (21)	0.489 (3)	0.6011 (7)	4.6 (7)
H(4B)	0.4701 (23)	0.619 (3)	0.5769 (7)	6.1 (8)
H(6A)	0.4173 (24)	0.713 (3)	0.6806 (8)	6.9 (8)
H(6B)	0.4754 (24)	0.777 (3)	0.6299 (8)	7.6 (9)
H(7A)	0.6430 (25)	0.766 (3)	0.6919 (9)	8.5 (9)
H(7B)	0.6645 (23)	0.667 (3)	0.6513 (8)	6.3 (8)
H(8A)	0.7131 (21)	0.520 (3)	0.7128 (7)	4.6 (7)
H(8B)	0.5637 (24)	0.556 (3)	0.7287 (8)	7.7 (9)
H(10A)	0.487 (3)	0.282 (3)	0.7272 (8)	7.8 (9)
H(10B)	0.6700 (22)	0.269 (3)	0.7199 (7)	5.4 (7)
H(11A)	0.4441 (22)	0.140 (3)	0.6662 (7)	5.5 (7)
H(11B)	0.5644 (25)	0.048 (3)	0.7021 (8)	7.7 (9)
H(12A)	0.6675 (24)	0.213 (3)	0.6384 (8)	7.0 (8)
H(12B)	0.7339 (23)	0.066 (3)	0.6617 (8)	6.7 (8)
H(14A)	0.6327 (23)	-0.116 (3)	0.5668 (7)	6.3 (8)
H(14B)	0.7482 (24)	-0.079 (3)	0.6067 (8)	6.7 (8)
H(15A)	0.7983 (23)	0.148 (3)	0.5781 (8)	6.3 (8)
H(15B)	0.798 (3)	0.032 (3)	0.5409 (9)	9.5 (10)
H(16A)	0.6808 (25)	0.266 (3)	0.5194 (8)	7.9 (9)
H(16B)	0.5824 (23)	0.098 (3)	0.5087 (8)	6.5 (8)
H(17A)	0.4115 (24)	0.156 (3)	0.6011 (8)	6.6 (8)
H(17B)	0.4461 (25)	0.026 (3)	0.5612 (8)	7.4 (8)
H(18A)	0.3847 (22)	0.347 (3)	0.6521 (7)	5.2 (7)
H(18B)	0.377 (3)	0.472 (3)	0.6928 (9)	9.5 (10)
N(1')	0.00645 (22)	0.2282 (3)	0.93875 (7)	4.17 (14)
C(2')	-0.0747 (3)	0.3201 (4)	0.96245 (10)	5.43 (21)
C(3')	-0.1678 (3)	0.4161 (4)	0.93327 (10)	5.53 (21)
C(4')	-0.0984 (3)	0.5227 (4)	0.90544 (10)	5.01 (20)
N(5')	-0.18081 (24)	0.5652 (3)	0.86591 (8)	4.63 (15)
C(6')	-0.1356 (3)	0.6986 (4)	0.84540 (11)	5.47 (20)
C(7')	-0.0102 (3)	0.6748 (4)	0.82731 (11)	5.27 (20)
C(8')	-0.0208 (3)	0.5423 (4)	0.79647 (10)	5.34 (21)
N(9')	-0.06235 (24)	0.4127 (3)	0.81961 (7)	4.26 (14)
C(10')	-0.0657 (3)	0.2777 (4)	0.79240 (10)	5.42 (20)
C(11')	-0.0742 (3)	0.1366 (4)	0.81923 (10)	5.19 (20)
C(12')	0.0404 (3)	0.1164 (4)	0.85200 (10)	4.76 (19)
N(13')	0.00958 (24)	0.0235 (3)	0.88835 (8)	4.75 (15)
C(14')	0.1241 (3)	-0.0362 (4)	0.91365 (11)	5.69 (21)
C(15')	0.2001 (3)	0.0820 (4)	0.94068 (10)	5.05 (20)
C(16')	0.1139 (3)	0.1688 (4)	0.96702 (10)	4.90 (19)
C(17')	-0.0647 (3)	0.1041 (4)	0.91670 (10)	4.94 (19)
C(18')	-0.1866 (3)	0.4437 (4)	0.83418 (10)	4.46 (18)
H(2'A)	-0.131 (3)	0.246 (3)	0.9800 (8)	7.8 (9)
H(2'B)	-0.008 (3)	0.390 (3)	0.9840 (8)	8.9 (9)
H(3'A)	-0.2313 (23)	0.343 (3)	0.9158 (8)	5.8 (7)
H(3'B)	-0.224 (3)	0.495 (3)	0.9510 (9)	9.3 (10)
H(4'A)	-0.0016 (21)	0.480 (3)	0.8993 (7)	4.8 (7)
H(4'B)	-0.0819 (24)	0.616 (3)	0.9255 (8)	7.0 (8)
H(6'A)	-0.2089 (24)	0.719 (3)	0.8224 (8)	7.4 (8)
H(6'B)	-0.1218 (25)	0.776 (3)	0.8700 (8)	7.6 (8)
H(7'A)	0.008 (3)	0.764 (4)	0.8118 (9)	9.8 (10)
H(7'B)	0.0667 (22)	0.652 (3)	0.8484 (7)	5.9 (8)
H(8'A)	0.0764 (22)	0.516 (3)	0.7867 (8)	6.4 (8)
H(8'B)	-0.0965 (25)	0.564 (3)	0.7726 (8)	7.9 (9)
H(10'A)	-0.152 (3)	0.281 (3)	0.7719 (8)	8.1 (9)
H(10'B)	0.0339 (21)	0.276 (3)	0.7813 (7)	5.3 (7)
H(11'A)	-0.1633 (21)	0.134 (3)	0.8329 (7)	4.8 (7)

* Lists of structure factors, anisotropic thermal parameters and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38167 (32 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1 (cont.)

	x	y	z	$B_{\text{iso}}(\text{\AA}^2)$
H(11'B)	-0.0818 (24)	0.041 (3)	0.7967 (8)	7.3 (8)
H(12'A)	0.0776 (22)	0.219 (3)	0.8639 (7)	6.1 (8)
H(12'B)	0.1165 (24)	0.072 (3)	0.8390 (8)	7.3 (8)
H(14'A)	0.0846 (24)	-0.123 (3)	0.9357 (8)	6.5 (8)
H(14'B)	0.168 (3)	-0.084 (3)	0.8946 (9)	9.1 (10)
H(15'A)	0.250 (3)	-0.158 (3)	0.9225 (9)	9.8 (10)
H(15'B)	0.2772 (24)	0.040 (3)	0.9603 (9)	8.0 (9)
H(16'A)	0.1750 (23)	0.265 (3)	0.9838 (8)	6.6 (8)
H(16'B)	0.0746 (25)	0.097 (3)	0.9905 (8)	7.9 (9)
H(17'A)	-0.1527 (23)	0.140 (3)	0.8993 (8)	6.6 (8)
H(17'B)	-0.089 (3)	0.023 (3)	0.9383 (9)	8.7 (9)
H(18'A)	-0.2199 (23)	0.349 (3)	0.8463 (7)	6.1 (8)
H(18'B)	-0.259 (3)	0.484 (3)	0.8094 (8)	8.2 (9)

Table 2. Transannular distances (\AA)

(E.s.d.'s are H...H: $\sim 0.040 \text{\AA}$; C...C and N...N: 0.004\AA .)	Molecule (I)	Molecule (II)
N(1)...N(9)	4.078	4.086
C(4)...C(12)	4.376	4.356
C(3)...C(11)	4.566	4.583
C(17)...C(18)	4.122	4.123
H(3A)...H(11A)	3.35	3.36
H(3A)...H(17A)	2.00	2.11
H(3A)...H(18A)	2.21	2.20
H(11A)...H(17A)	2.04	2.08
H(11A)...H(18A)	2.00	2.09
H(17A)...H(18A)	2.39	2.56
H(4A)...H(12A)	2.93	2.77

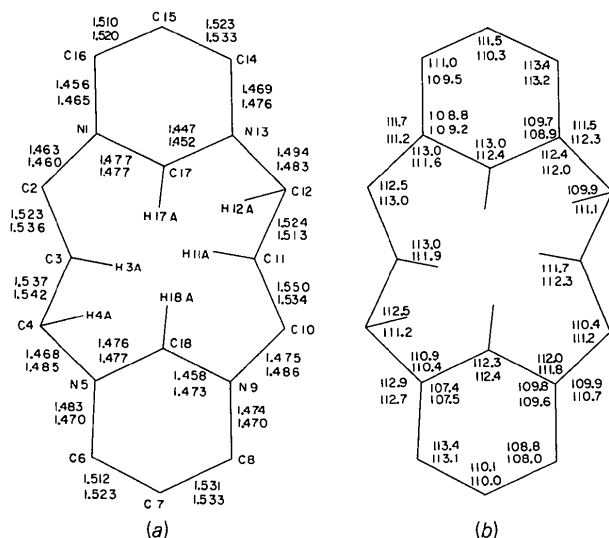


Fig. 1. (a) Atomic numbering and bond lengths (\AA) and (b) bond angles ($^\circ$) between the C and N atoms for both molecules (top line is for molecule I). E.s.d.'s of the distances are approximately 0.005\AA and of the angles 0.25° .

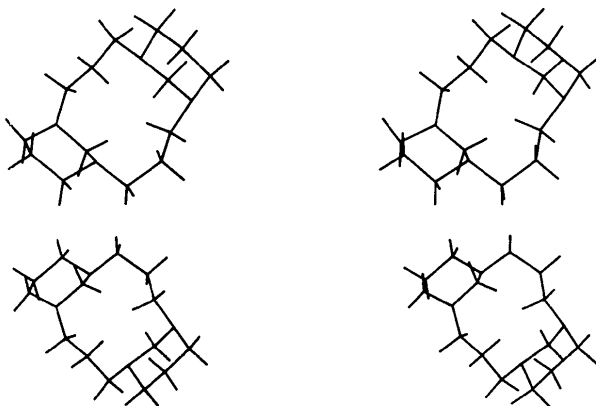


Fig. 2. A stereoview of both molecules in the asymmetric unit.

reported. The NH_2 of azacyclododecane hydrochloride is positioned at a corner of the square such that both H atoms are hydrogen-bonded to neighboring chlorides (Dunitz & Weber, 1964). The four N atoms of (2*R*,5*R*,8*R*,11*R*)-2,5,8,11-tetraethyl-1,4,7,10-tetraazacyclododecane are positioned on the four sides of the square (Sakurai, Kobayashi, Tsuboyama & Tsuboyama, 1978). 1,3,7,9-Tetraoxacyclododecane, which is formally analogous to the central ring of the title compound in terms of relative bonding order of heteroatoms, has been shown by NMR spectroscopy to prefer the [3333] conformation with 1,3-dioxa groupings across diagonal corners in solution (Borgen & Dale, 1974). On the other hand, the crystal structure of 1,4,7,10-tetraoxacyclododecane has demonstrated not the [3333] conformation but rather a centrosymmetric [66] conformation (Groth, 1978).

In the structure of the title compound, the four bridgehead N atoms occupy two diagonal corner positions [N(5), N(13)] and two opposing side positions on one face of the quadrangular 12-membered ring. This is, in fact, the only possible arrangement of N atoms that permits bridging of the two six-membered rings to extra annular positions of a [3333] conformation. Each of these hexahydropyrimidine rings is a distorted chair substituted equatorial/axial (*trans*) at the bridgehead N atoms with the propano bridges of the 12-membered ring. The two six-membered rings are *transoid/transoid* to one another with respect to the 12-membered ring. The observed hexahydropyrimidine conformation is that favored by the generalized anomeric effect (Riddell, 1980a) and that observed in the structure of 1,4,8,11-tetraazatricyclo-[9.3.1.1^{4,8}]hexadecane (Gabe *et al.*, 1982). Torsion angles of the six-membered rings are remarkably similar for the two structures.

The presence of the two opposing side-position N atoms, N(1) and N(9), relieves transannular strain relative to that in cyclododecane. In cyclododecane, each face of the ring has four intra-annular H atoms, the shortest non-bonded H...H distance lying in the range $2.0\text{--}2.1 \text{\AA}$ (Dunitz, 1968). In the title compound,

one face likewise has four intra-annular H atoms; the shortest non-bonded H...H distances (Table 2) are comparable to those observed in cyclododecane and in azacyclododecane hydrochloride (Dunitz & Weber, 1964). The other face, however, has two opposing nitrogen lone pairs. The facts that the N(1)...N(9) transannular distance is shorter than the C(17)...C(18) distance and C(4)...C(12) is shorter than C(3)...C(11) are direct reflections of the smaller van der Waals radius of nitrogen relative to that of methylene (Riddell, 1980b) (Table 2; see also the related H...H transannular distances). The decreased strain may also be part of the reason for the relatively normal C—C—N and C—N—C bond angles at the corners of the [3333] conformation. Corner bond angles in cyclododecane (Dunitz & Shearer, 1960) are considerably expanded compared to those observed in this work.

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Structure of 1-(4-Chlorobenzylidene)-5,5-dimethyl-3-pyrazolidone Betaine, C₁₂H₁₃ClN₂O*

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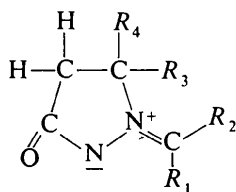
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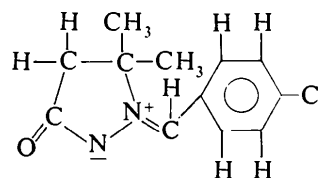
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Abstract. $M_r = 236.7$, monoclinic, $P2_1/n$, $a = 9.079$ (4), $b = 18.576$ (10), $c = 7.205$ (5) Å, $\beta = 94.26$ (2)°, $Z = 4$, $V = 1211.8$ (8) Å³, $D_x = 1.297$ (1) Mg m⁻³, $F(000) = 496$, $\mu(\text{Mo } K\alpha) = 0.302$ mm⁻¹, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $T = 296$ K. Final $R = 0.059$ for 2116 independent reflections excluding those with $|F_o| < 4\sigma(F_o)$. The azomethine imine unit of the molecule has a geometry in approximate agreement with a polymethine-like electronic structure. The whole molecule is approximately planar. Conjugation of the azomethine imine unit with the phenyl unit is indicated. There are some indications of intermolecular C—H...O hydrogen bridges.

Introduction. The compound (C₁₂H₁₃ClN₂O), abbreviated as CDPB, belongs to the class of azomethines of general formula (1) and is formally an azomethine-3 dipole (Huisgen, 1963) with the complete formula (2). The structures of the related 1-(4-nitrobenzylidene)-3-pyrazolidone betaine, NPB (Kulpe, Seidel, Geissler &



(1)



(2)

* Structures of Azomethine Imines of the 3-Pyrazolidone Type. II. Part I: Kulpe, Seidel, Geissler & Tomaschewski (1981).