

Fig. 2. View of two independent molecules of compound (Ib) (number of one molecule only shown).

The bond lengths and bond angles of (Ia) and (Ib) agree within experimental limits. Indeed, the space-group ambiguity for (Ib) could be uniquely resolved by comparing the coordinates of the two independent molecules. One molecule is related to the other by the approximate relationship $[-x, -y, 0.46 + z]$ which deviates significantly from the center of symmetry owing to the arbitrary value of the z coordinate. If the c axis is taken as unique, the above relation approximates to a 2_1 axis. The angle between the planar rings in the

two independent molecules (23.2°) clearly indicates the space group to be the non-centrosymmetric $P1$.

The carbon skeleton in the pseudoneolongifol moiety consists of two eight-membered rings [C(1), C(2), C(3), C(4), C(5), C(9), C(10), C(11) and C(1), C(2), C(3), C(4), C(5), C(6), C(7), C(11)] having a 'boat-chair' conformation (Bucourt, 1974). The bicyclo[2.2.1]heptane moiety takes the $S(+,+)$ conformation (Acharya, Tavale & Guru Row, 1984). The structure is stabilized by van der Waals interactions.

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Acta Cryst. (1988). **C44**, 2114–2117

Structures of 3,5-Bis(benzylidene)-4-piperidone Hydrochloride (I) and its *N*-Methyl Analog (II)

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(Received 5 May 1988; accepted 7 September 1988)

Abstract. (I): C₁₉H₁₈NO⁺.Cl⁻, $M_r = 311.80$, monoclinic, $P2_1/n$, $a = 16.787(2)$, $b = 5.7373(2)$, $c = 16.883(1)$ Å, $\beta = 99.07(1)^\circ$, $V = 1605.7$ Å³, $Z = 4$, D_m (by flotation) = 1.284, $D_x = 1.290$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 0.210$ mm⁻¹, $F(000) = 656$, $T = 287$ K, $R = 0.036$ ($wR = 0.042$) for 2824 observed reflections. (II): C₂₀H₂₀NO⁺.Cl⁻, $M_r = 325.83$, orthorhombic, $Pc2_1b$, $a = 7.065(2)$, $b = 12.302(2)$, $c = 19.354(2)$ Å, $V = 1682.3$ Å³, $Z = 4$, D_m (by flotation) = 1.290, $D_x = 1.283$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 0.196$ mm⁻¹, $F(000) = 688$, $T = 287$ K,

$R = 0.062$ ($wR = 0.060$) for 1582 observed reflections. The heterocyclic rings exhibit a boat conformation in (I) and a chair conformation in (II). The heterocyclic rings in both molecules are flattened in the moiety of C(3)–C(4)–C(5). The bond lengths and angles show good correspondence between the two molecules which, however, differ significantly with respect to torsion angles. In both molecules the steric repulsions between H atoms attached to C(2)/C(6) and those on the phenyl rings cause increases in the bond angles at the C atoms joining the rings and rotations of the phenyl groups out of the mean plane of the piperidone ring.

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Table 1. Fractional coordinates and equivalent isotropic thermal parameters for (I), with *e.s.d.*'s in parentheses
$$U_{\text{eq}} = (U_{11} + U_{22}\sin^2\beta + U_{33} + 2U_{13}\cos\beta)/3\sin^2\beta.$$

	<i>x</i>	<i>y</i>	<i>z</i>	$10^3 U_{\text{eq}} (\text{\AA}^2)$
Cl	0.27767 (3)	-0.07908 (6)	-0.17804 (2)	50
O(4)	0.18523 (8)	0.9125 (2)	0.01108 (7)	56
N(1)	0.24378 (8)	0.4150 (2)	-0.12903 (7)	40
C(2)	0.29578 (9)	0.4038 (2)	-0.04954 (9)	42
C(3)	0.27770 (9)	0.6042 (2)	0.00232 (9)	41
C(4)	0.19794 (9)	0.7242 (3)	-0.01826 (8)	43
C(5)	0.13318 (9)	0.5983 (2)	-0.07279 (8)	40
C(6)	0.15715 (9)	0.4002 (2)	-0.12246 (9)	42
C(7)	0.0567 (1)	0.6642 (3)	-0.07087 (9)	45
C(8)	0.3271 (1)	0.6797 (3)	0.06692 (9)	47
C(9)	0.4064 (1)	0.5975 (3)	0.10593 (9)	47
C(10)	0.4488 (1)	0.7420 (4)	0.1639 (1)	63
C(11)	0.5249 (1)	0.6811 (4)	0.2032 (1)	70
C(12)	0.5597 (1)	0.4769 (4)	0.1861 (1)	59
C(13)	0.5182 (1)	0.3281 (4)	0.1308 (1)	66
C(14)	0.4421 (1)	0.3866 (3)	0.0911 (1)	62
C(15)	-0.02051 (9)	0.5762 (3)	-0.11174 (9)	47
C(16)	-0.0883 (1)	0.7131 (3)	-0.1074 (1)	55
C(17)	-0.1639 (1)	0.6479 (4)	-0.1445 (1)	66
C(18)	-0.1742 (1)	0.4428 (4)	-0.1873 (1)	67
C(19)	-0.1083 (1)	0.3012 (4)	-0.1919 (1)	64
C(20)	-0.0321 (1)	0.3648 (3)	-0.1540 (1)	56

Table 2. Fractional coordinates and equivalent isotropic thermal parameters for (II), with *e.s.d.*'s in parentheses
$$U_{\text{eq}} = (U_{11} + U_{22} + U_{33})/3.$$

	<i>x</i>	<i>y</i>	<i>z</i>	$10^3 U_{\text{eq}} (\text{\AA}^2)$
Cl	0.2957 (2)	0.52090	0.03623 (7)	59
O(4)	0.8916 (7)	0.3040 (3)	0.0456 (2)	69
N(1)	0.6786 (5)	0.6048 (3)	0.0023 (2)	42
C(1)	0.6372 (9)	0.7194 (4)	-0.0165 (3)	56
C(2)	0.8042 (7)	0.5972 (4)	0.0626 (3)	44
C(3)	0.8263 (6)	0.4802 (4)	0.0853 (2)	40
C(4)	0.8418 (8)	0.3959 (4)	0.0312 (3)	48
C(5)	0.7898 (7)	0.4255 (4)	-0.0408 (2)	41
C(6)	0.7519 (7)	0.5431 (4)	-0.0583 (3)	44
C(7)	0.7712 (7)	0.3425 (4)	-0.0863 (3)	45
C(8)	0.8289 (7)	0.4478 (4)	0.1510 (3)	44
C(9)	0.8253 (6)	0.5087 (4)	0.2159 (2)	42
C(10)	0.7536 (7)	0.4593 (5)	0.2747 (3)	51
C(11)	0.7531 (7)	0.5131 (6)	0.3375 (3)	58
C(12)	0.8321 (8)	0.6158 (6)	0.3426 (3)	70
C(13)	0.9084 (8)	0.6634 (5)	0.2854 (3)	61
C(14)	0.9067 (7)	0.6126 (4)	0.2227 (3)	52
C(15)	0.7239 (7)	0.3405 (4)	-0.1594 (2)	43
C(16)	0.6424 (8)	0.2436 (5)	-0.1854 (3)	54
C(17)	0.5953 (8)	0.2345 (5)	-0.2536 (4)	64
C(18)	0.6263 (9)	0.3213 (6)	-0.2981 (3)	67
C(19)	0.7096 (8)	0.4157 (5)	-0.2746 (3)	56
C(20)	0.7550 (7)	0.4254 (4)	-0.2062 (3)	47

Introduction. α,β -Unsaturated ketones have proved to be effective as antitumour agents, which yet may be bereft of the unwanted side effects on nucleic acids due to having little or no affinity for amino groups (Baluja, Municio & Vega, 1964; Dimmock, Raghavan, Logan & Bigam, 1983). Since greater anticancer properties were found in the Mannich bases of unsaturated ketones (Dimmock & Taylor, 1975), compound (I) and some analogs were synthesized and (I) was found to have over 100 times the cytotoxicity to P388 leukemia

Table 3. Selected bond distances (\AA), angles ($^\circ$), torsion angles ($^\circ$) and some interatomic distances (\AA) for (I) and (II), with *e.s.d.*'s in parentheses

	Compound (I)	Compound (II)
O(4)–C(4)	1.221 (2)	1.216 (7)
N(1)–C(1)	–	1.485 (7)
N(1)–C(2)	1.482 (2)	1.470 (6)
N(1)–C(6)	1.478 (2)	1.489 (6)
C(2)–C(3)	1.505 (2)	1.513 (7)
C(3)–C(4)	1.497 (2)	1.478 (7)
C(3)–C(8)	1.334 (2)	1.333 (7)
C(4)–C(5)	1.495 (2)	1.485 (7)
C(5)–C(6)	1.504 (2)	1.510 (7)
C(5)–C(7)	1.343 (2)	1.354 (7)
C(7)–C(15)	1.459 (2)	1.455 (7)
C(8)–C(9)	1.467 (2)	1.463 (7)
C(9)–C(10)	1.390 (2)	1.386 (7)
C(9)–C(14)	1.391 (3)	1.408 (7)
C(15)–C(16)	1.394 (2)	1.416 (7)
C(15)–C(20)	1.405 (2)	1.400 (7)
C(1)–N(1)–C(2)	–	112.0 (4)
C(1)–N(1)–C(6)	–	111.1 (4)
C(2)–N(1)–C(6)	112.1 (1)	112.5 (4)
N(1)–C(2)–C(3)	110.4 (1)	110.7 (4)
C(2)–C(3)–C(4)	118.1 (1)	118.0 (4)
C(2)–C(3)–C(8)	124.6 (1)	124.2 (4)
C(4)–C(3)–C(8)	117.3 (1)	117.8 (4)
O(4)–C(4)–C(3)	121.3 (1)	120.7 (5)
O(4)–C(4)–C(5)	121.4 (1)	120.9 (5)
C(3)–C(4)–C(5)	117.2 (1)	118.3 (4)
C(4)–C(5)–C(6)	118.4 (1)	119.3 (3)
C(4)–C(5)–C(7)	117.0 (1)	116.7 (4)
C(6)–C(5)–C(7)	124.5 (1)	124.0 (4)
N(1)–C(6)–C(5)	110.6 (1)	111.9 (4)
C(5)–C(7)–C(15)	132.3 (1)	131.9 (6)
C(3)–C(8)–C(9)	131.8 (1)	132.0 (6)
C(8)–C(9)–C(10)	116.9 (1)	119.2 (5)
C(8)–C(9)–C(14)	125.8 (1)	122.6 (4)
C(10)–C(9)–C(14)	117.3 (2)	118.0 (5)
C(9)–C(10)–C(11)	121.1 (2)	120.8 (5)
C(9)–C(14)–C(13)	120.9 (2)	120.2 (5)
C(7)–C(15)–C(16)	116.9 (1)	117.0 (5)
C(7)–C(15)–C(20)	125.5 (1)	125.5 (5)
C(16)–C(15)–C(20)	117.7 (1)	117.5 (5)
C(15)–C(16)–C(17)	121.7 (2)	120.8 (5)
C(15)–C(20)–C(19)	120.3 (2)	121.6 (5)
C(4)–C(3)–C(2)–N(1)	20.8 (2)	39.2 (6)
N(1)–C(6)–C(5)–C(4)	-19.3 (2)	-28.7 (6)
C(2)–C(3)–C(8)–C(9)	-1.5 (3)	-4.5 (8)
C(6)–C(5)–C(7)–C(15)	-0.1 (15)	3.4 (9)
C(3)–C(8)–C(9)–C(14)	-12.9 (3)	-31.8 (8)
C(5)–C(7)–C(15)–C(20)	12.4 (3)	26.0 (9)
C(8)–C(9)–C(14)–C(13)	179.0 (2)	-176.4 (5)
C(7)–C(15)–C(20)–C(19)	-178.7 (2)	178.6 (5)
N(1)–C(7)	3.720 (2)	3.714 (10)
N(1)–C(8)	3.708 (2)	3.624 (10)
O(4)–C(7)	2.767 (2)	2.736 (10)
O(4)–C(8)	2.763 (2)	2.736 (10)
C(7)–C(8)	4.764 (2)	4.791 (11)
C(12)–C(18)	12.925 (3)	13.007 (13)

cells of 1,3-bis(2-chloroethyl)-1-nitrosourea (Warrington, Fang, Dimmock & Arora, 1987). It was of interest to determine the crystal structure of (I) which has this unusually high cytotoxicity and to compare it with the structure of its analog (II) whose bio-evaluation is underway. Preliminary data indicate an approximately 60-fold reduction in activity from (I) to (II).

Experimental. Compound synthesized by: (I) a literature procedure (Kuettel & McElvain, 1931) in a yield of 67%, m.p. 540–542 K (lit. m.p. 549–550 K), (II) a literature procedure (Krapcho & Turk, 1979) in a yield of 82%, m.p. 514–516 K (lit. m.p. 515–517 K). Crystal from: (I) 95% EtOH(hot), (II) mix. of 95% EtOH and Et_2O . Both crystals light-yellow, dimensions: (I) $0.08 \times 0.12 \times 0.45$, (II) $0.02 \times 0.28 \times 0.40$ mm. Space group (non-standard setting): (I) $P2_1/n$ (equivalent positions: x, y, z ; $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; $-x, -y, -z$; $-\frac{1}{2}+x, -\frac{1}{2}-y, -\frac{1}{2}+z$), (II) $Pc2_1b$ (equivalent positions: x, y, z ; $-x, y, \frac{1}{2}+z$; $-x, \frac{1}{2}+y, -z$; $x, \frac{1}{2}+y, \frac{1}{2}-z$). Equivalent setting: (I) $P2_1/c$, (II) $Pca2_1$. Enraf–Nonius CAD-4 diffractometer. Lattice parameters determined from 25 reflections in the θ range (I) 21.83–45.22, (II) 7.58–24.43°. Data collection: (I) 3299 unique reflections, $(\sin\theta)/\lambda_{\max} = 0.6265 \text{ \AA}^{-1}$, $-21 \leq h \leq 21, 0 \leq k \leq 7, 0 \leq l \leq 21$, (II) 1808 unique reflections, $(\sin\theta)/\lambda_{\max} = 0.6265 \text{ \AA}^{-1}$, $0 \leq h \leq 8, 0 \leq k \leq 15, 0 \leq l \leq 24$. 2824(I), 1582(II) reflections with $I > 2\sigma(I)$ used in refinement. Three intensity and orientation monitor reflections for both (I) and (II) showed no significant variation. Absorption correction applied for (II) only; transmission factors 1.480–39.482. Merging R based on intensities of 0.0083 for 374(I), 0.0075 for 106(II) replicate reflections. Structure solved by direct methods using *XTAL*, version 2.2 (Hall & Stewart, 1987), all non-H atoms found on E map, all H atoms calculated and not refined. $R(F) = 0.036$, $wR = 0.042$, $S = 2.648$ for 2824 observed reflections (I) and $R(F) = 0.062$, $wR = 0.060$, $S = 3.195$ for 1582 observed reflections (II). Parameters refined: 200(I); 209(II). $w = 1/\sigma^2(F)$. Final $(\Delta/\sigma)_{\max} = 0.27$ (I), 0.11(II), $(\Delta/\sigma)_{\text{ave}} = 0.002$ (I), 0.006(II). $\Delta\rho$ in final difference map within +0.23 and -0.21 e \AA^{-3} (I), +0.34 and -0.31 (three largest holes $-0.80, -0.56$ and -0.45 near Cl atom) e \AA^{-3} (II). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). No corrections for extinction. All calculations performed on a VAX 8650 computer at the University of Saskatchewan.

Discussion. The atomic parameters are summarized in Tables 1 and 2.* Selected bond distances, angles, torsion angles and some interatomic distances which may be of importance in understanding the bio-activities of (I) and (II) are listed in Table 3. Figs. 1 and 2 are *ORTEP* drawings (Johnson, 1976) of (I) and (II) respectively.

The bond distances and angles lie in the normal range and display a close correspondence between equivalents in the two molecules. The heterocyclic rings

* Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51273 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

exhibit a boat conformation in (I) and a chair conformation in (II). The heterocyclic rings in both molecules are flattened in the moiety of C(3)–C(4)–C(5) due to a partially conjugated dienone system. If we take C(2), C(3), C(5), C(6) as base plane (A), then the plane of C(3), C(4), O(4), C(5) is tilted 13.8°(I), 9.5°(II) from A . In both molecules the steric repulsions between H atoms attached to C(2)/C(6) and those of the phenyl rings [average contact distances of the H atoms of 2.19(I), 2.36 Å(II)] cause increases in the bond angles at the C atoms joining the rings. This is demonstrated by the angles C(5)–C(7)–C(15), C(3)–C(8)–C(9), C(8)–C(9)–C(14) and C(7)–C(15)–C(20) (Table 3), all being substantially greater than the value of 120° expected for sp^2 C atoms. Ample evidence for a similar effect has been reported (Nyberg & Faerman, 1986). To reduce the interactions further, the two phenyl rings rotate about the C(8)–C(9) and C(7)–C(15) bonds. However, (I) and (II) differ significantly with respect to

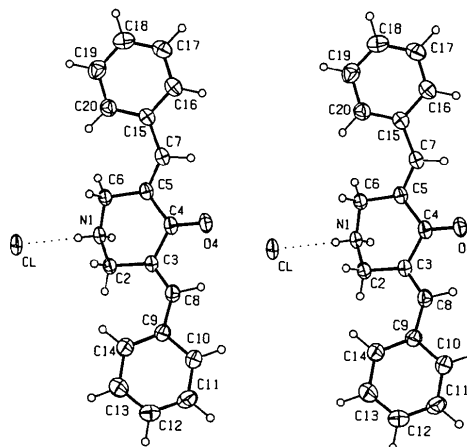


Fig. 1. Stereoscopic *ORTEP* view (Johnson, 1976) of (I) with atomic numbering.

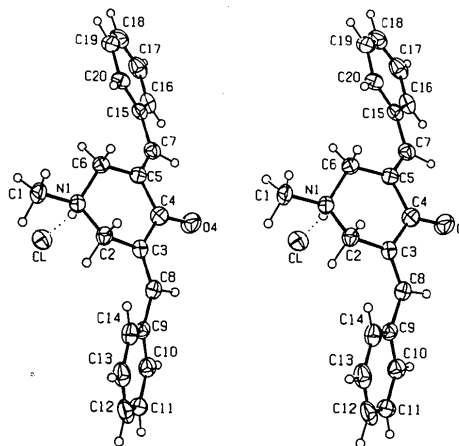


Fig. 2. Stereoscopic *ORTEP* view (Johnson, 1976) of (II) with atomic numbering.

the rotations, which are marked by torsion angle C(3)–C(8)–C(9)–C(14) and the corresponding angle C(5)–C(7)–C(15)–C(20) [12.9 (3) and 12.4 (3)° for (I), 31.5 (9) and 26.1 (10)° for (II)]. The benzylidene planes of (I) are less tilted from *A* than those of (II). In addition, (I) shows more stereosymmetry than (II) in overall geometry. The replacement of an N(1) proton of (I) by a methyl group to give (II) changes the piperidone ring from a boat to a chair conformation and changes some of the torsion angles of the aryl groups. The differences in shape between (I) and (II) may account, in part at least, for the marked disparity in cytotoxicity between the two compounds.

We thank Dr L. T. J. Delbaere for the use of facilities for collecting data and Dr L. Prasad for her valuable suggestions. ZJ thanks the Department of Chemistry, University of Saskatchewan, for financial support through a graduate scholarship.

Acta Cryst. (1988). **C44**, 2117–2120

Structures of *N,N,N',N'*-Tetramethylbenzidine (I) and 4,4'-Methylenebis(*N,N*-dimethylaniline) (II)

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(Received 29 March 1988; accepted 11 July 1988)

Abstract. (I) $C_{16}H_{20}N_2$, $M_r = 240.35$, orthorhombic, $Pna2_1$, $a = 11.445$ (2), $b = 15.598$ (3), $c = 7.588$ (1) Å, $V = 1354.6$ (4) Å³, $Z = 4$, $D_x = 1.178$ Mg m⁻³, $\lambda(Cu K\alpha) = 1.54178$ Å, $\mu(Cu K\alpha) = 0.54$ mm⁻¹, $F(000) = 520$, $T = 295$ K, $R = 0.054$ for 1170 observed reflections [$F_o > 2\sigma(F_o)$]. (II) $C_{17}H_{22}N_2$, $M_r = 254.38$, tetragonal, $P4_12_12$, $a = 6.3500$ (3), $c = 36.647$ (3) Å, $V = 1477.7$ (2) Å³, $Z = 4$, $D_x = 1.143$ Mg m⁻³, $\lambda(Cu K\alpha) = 1.54178$ Å, $\mu(Cu K\alpha) = 0.52$ mm⁻¹, $F(000) = 552$, $T = 295$ K, $R = 0.054$ for 877 observed reflections [$F_o > 3\sigma(F_o)$]. The methylene group in (II) lies on a twofold axis. One of the dimethylamino groups in (I) is planar and the other in (I) and those in (II) adopt approximately planar conformations. The dihedral angle between the two benzene rings in (I) is 13.0 (1)°.

Introduction. As part of an investigation of how substituents affect the conformation of dimethylamino groups in six-membered aromatics, the structures of (I) and (II) were determined by X-ray analysis to compare the conformations with those in 4-*N,N*-dimethylamino-pyridine *N*-oxide dihydrate (III) (Nakai, Saito &

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Yamakawa, 1988a) and *N,N*-dimethyl-1,4-phenylenediamine (IV) (Nakai, Saito & Yamakawa, 1988b). The dimethylamino group in (III) adopts a planar conformation, but that in (IV) adopts a pyramidal one in which the N atom is involved in a hydrogen bond. The two phenyl rings in (I) can conjugate with each other, but they cannot in (II) because the central methylene group interferes with the π -electron transfer between the phenyl rings.

Experimental. Rigaku AFC-5R diffractometer, graphite-monochromatized Cu $K\alpha$. Structures solved by *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978).

(I) Colorless prismatic crystals obtained from an ethyl acetate solution. Crystal of dimensions 0.4 × 0.4 × 0.5 mm, cell dimensions determined from 2θ angles for 25 reflections in the range $26 < 2\theta < 44^\circ$. Intensities measured up to $2\theta = 140^\circ$ in h 0/13, k 0/18 and l 0/8, ω - 2θ scans, ω -scan width $(1.2 + 0.2\tan\theta)^\circ$, three standard reflections monitored every 100 measurements showed no significant change. 1315 unique reflections measured, 1170 intensities observed