(III). In (I) and (III) where the pseudo bi-aryl conformation allows resonance overlap between the rings there is still inter-ring steric hindrance. In (I), the H(3)···H(8) separation is 2·19 Å [H(3)···H(8a) = 1·82 Å] and in (III) the distance is 1·97 Å, compared to the usual van der Waals H···H contact range of 2·2–2·4 Å. In (III) the formyl group on N(1) is coplanar with the pyridyl N and its adjacent atoms. It is interesting to note that in (I) the CH_3 group on N(1) is equatorial and the H atom is axial while in (II) the CH_3 group on N(1) is axial and the BH_3 moiety is equatorial. In (II), which has a density of only 1·05 Mg m^{-3}, there are no intermolecular approaches less than van der Waals radii. Molecules of (III) form hydrogen-bonded chains in the crystal with the hydroxyl H bonding to the carbonyl of the next molecule [O···O = 2·76 (1) Å, O—H···O = 158 (3)°].

Note added in proof: An independent determination of (I) at 100 K was recently published (Klein, Borne & Stevens, 1985).

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Structure of *trans*-*N*-(1-Benzyl-4-methyl-3-pyrrolidinyl)-5-chloro-2-methoxy-4-methylaminobenzamide Hydrochloride*

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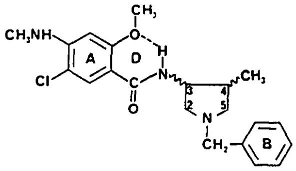
Abstract. $C_{21}H_{27}ClN_3O_2^+.Cl^-$, M_r = 424·37, monoclinic, $P2_1/c$, a = 15·863 (1), b = 12·660 (1), c = 11·264 (1) Å, β = 106·47 (1)°, V = 2169·3 (3) Å^3, D_x = 1·299 g cm^{-3}, Z = 4, room temperature, $F(000)$ = 896, $\lambda(\mathrm{Cu}\ K\alpha)$ = 1·54184 Å, μ = 28·6 cm^{-1}, R = 0·057 for 2261 observed reflections with $|F_o| > 3\sigma(|F_o|)$. An intramolecular hydrogen bond between the amide N and the methoxy O is observed. The distance between the tertiary N and the center of the benzene ring is 6·91 Å, and the deviation of the N from the plane of the benzene ring is 1·75 Å.

Introduction. The crystal structure determination of the title compound (1) was undertaken as part of serial studies for finding new potent neuroleptic drugs in the benzamide derivatives. The previous papers (Furuya, Iwanami, Takenaka & Sasada, 1982; Furuya, Iwanami, Takenaka & Sasada, 1986) have reported the crystal structures of the benzamide derivatives having a methyl group at the 2-position of the pyrrolidine ring. The present compound has a methyl group at the 4-position of the ring and it has intermediate activity.



(1) Title compound; (3*R*, 4*S*) and (3*S*, 4*R*).
cf.
(2) YM-09151-2; Methyl group at 2-position [(2*R*, 3*R*) and (2*S*, 3*S*)]
(3) YM-09151-1; Methyl group at 2-position [(2*R*, 3*S*) and (2*S*, 3*R*)]

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* New Potent Neuroleptic Drugs of Benzamide Derivatives. V.

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Experimental. Colorless plate crystals from a methanol–ethyl acetate solution; crystal size, 0·2 × 0·2 × 0·15 mm, Rigaku automated four-circle diffractometer AFC-5R, graphite-monochromated Cu *Kα* radiation. Accurate cell constants determined using 20 high-angle reflections in the range of $45 < 2\theta < 55°$. Intensities measured for $3 < 2\theta < 120°$, $-17 \leq h \leq 17$, $0 \leq k \leq 14$, $0 \leq l \leq 12$, θ–2θ scan mode, scanning rate $4°(2\theta)\,\mathrm{min}^{-1}$. Five reference reflections showed no significant intensity deterioration. 3230 independent reflections, 970 reflections with $|F_o| < 3\sigma(|F_o|)$ considered unobserved. Corrections for Lorentz and polarization factors, but not for absorption or secondary extinction. Standard deviations $\sigma^2(|F_o|) = \sigma_p^2(|F_o|) + q|F_o|^2$, with $\sigma_p(|F_o|)$ evaluated by counting statistics and q ($1·27 \times 10^{-3}$) derived from the variations of monitored reflections (McCandish & Stout, 1975).

Structure solved by direct method, *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), and block-diagonal least squares. Difference map indicated the presence of slight disordering of the benzene ring *B*. All H atoms found on a difference map, refined isotropically. $\sum w(|F_o| - |F_c|)^2$ minimized, with $w = 1/\sigma(|F_o|)$. Final $R = 0·057$ ($wR = 0·079$, $S = 1·21$) for 2261 reflections; maximum shifts of parameters $0·4\sigma$ for C, $0·2\sigma$ for N, $0·04\sigma$ for O, $0·07\sigma$ for Cl and $0·6\sigma$ for H atoms. $\Delta\rho_{\max} = 0·32$, $\Delta\rho_{\min} = -0·28$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computations performed on a Digital VAX-11/785 computer. Final atomic parameters for non-H atoms are given in Table 1.*

Discussion. The structure of the molecule and the packing diagram are shown in Figs. 1 and 2, respectively. The bond lengths and angles are listed in Table 2.

An intermolecular hydrogen bond is found between the amino nitrogen N(1) and the carbonyl oxygen O(2) in the molecule related by a *c* glide, the N(1)···O(2) distance being 3·059 (5) Å. The other intermolecular contacts are all larger than the sum of the van der Waals radii.

The lengths C(2)—C(3) [1·368 (5) Å] and C(5)—C(6) [1·377 (5) Å] are short in comparison with the other lengths in the ring [1·396 (6)–1·408 (5) Å], whereas angles C(2)—C(1)—C(6) [116·9 (3)°] and C(3)—C(4)—C(5) [116·7 (3)°] are appreciably smaller than the other angles [121·1 (3) –121·8 (3)°]. Moreover, the lengths C(1)—N(1) [1·362 (5) Å] and C(4)—

Table 1. *Fractional coordinates and equivalent isotropic temperature factors*

The *B* values accompanied by ⟨⟩ are the equivalent isotropic temperature factors calculated from the anisotropic thermal parameters using the equation $B = 8\pi^2(U_1 + U_2 + U_3)/3$, where U_1, U_2, and U_3 are the principal components of the mean-square displacement matrix U. Values in ⟨⟩ are the anisotropicity defined by $[\sum(B - 8\pi^2U_i)^2/3]^{1/2}$ and those in () are e.s.d.'s; they refer to the last decimal places.

	x	*y*	*z*	*B*(Å^2)
Cl(H)	0·52442 (7)	0·07597 (7)	0·30925 (9)	5·78 ⟨113⟩
Cl	1·07523 (7)	−0·04976 (9)	0·8684 (1)	7·07 ⟨227⟩
O(1)	0·7690 (2)	0·0386 (2)	0·4467 (2)	5·7 ⟨20⟩
O(2)	0·8056 (2)	0·2007 (3)	0·7740 (3)	9·0 ⟨52⟩
N(1)	1·0342 (2)	−0·1572 (2)	0·6242 (3)	6·0 ⟨14⟩
N(2)	0·7213 (2)	0·1845 (2)	0·5815 (3)	6·1 ⟨27⟩
N(3)	0·4979 (2)	0·2113 (2)	0·5246 (3)	5·4 ⟨17⟩
C(1)	0·9754 (2)	−0·0811 (3)	0·6324 (3)	4·9 ⟨12⟩
C(2)	0·9858 (2)	−0·0221 (3)	0·7403 (3)	5·2 ⟨13⟩
C(3)	0·9263 (2)	0·0534 (3)	0·7496 (3)	5·3 ⟨8⟩
C(4)	0·8525 (2)	0·0767 (3)	0·6507 (3)	4·9 ⟨5⟩
C(5)	0·8417 (2)	0·0173 (3)	0·5417 (3)	4·7 ⟨9⟩
C(6)	0·9018 (2)	−0·0587 (3)	0·5335 (3)	5·0 ⟨6⟩
C(7)	1·0244 (3)	−0·2204 (3)	0·5139 (4)	6·7 ⟨17⟩
C(8)	0·7514 (3)	−0·0220 (3)	0·3353 (3)	6·0 ⟨15⟩
C(9)	0·7913 (2)	0·1587 (3)	0·6733 (3)	5·4 ⟨10⟩
C(10)	0·6513 (3)	0·2523 (3)	0·6000 (3)	5·7 ⟨28⟩
C(11)	0·5773 (3)	0·1913 (3)	0·6300 (3)	6·3 ⟨18⟩
C(12)	0·6487 (3)	0·4264 (3)	0·4789 (4)	7·5 ⟨27⟩
C(13)	0·5130 (3)	0·3190 (3)	0·4804 (3)	5·8 ⟨19⟩
C(14)	0·6089 (2)	0·3175 (3)	0·4834 (3)	5·2 ⟨17⟩
C(15)	0·4154 (3)	0·1947 (4)	0·5608 (4)	7·1 ⟨13⟩
C(16)	0·3332 (3)	0·2083 (3)	0·4570 (4)	6·6 ⟨23⟩
C(17)	0·3114 (3)	0·1390 (4)	0·3575 (5)	8·2 ⟨15⟩
C(18)	0·2329 (4)	0·1499 (6)	0·2669 (5)	11·9 ⟨75⟩
C(19)	0·1804 (3)	0·2288 (6)	0·2809 (7)	16·1 ⟨158⟩
C(20)	0·1962 (5)	0·2987 (5)	0·3760 (9)	16·8 ⟨143⟩
C(21)	0·2754 (3)	0·2870 (4)	0·4607 (6)	9·7 ⟨57⟩

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

Standard deviations are given in parentheses.

Cl—C(2)	1·749 (4)	O(1)—C(5)	1·360 (4)	O(1)—C(8)	1·429 (5)
O(2)—C(9)	1·214 (5)	N(1)—C(1)	1·362 (5)	N(1)—C(7)	1·448 (5)
N(2)—C(9)	1·327 (5)	N(2)—C(10)	1·464 (5)	N(3)—C(11)	1·488 (5)
N(3)—C(13)	1·496 (5)	N(3)—C(15)	1·491 (5)	C(1)—C(2)	1·396 (5)
C(1)—C(6)	1·396 (5)	C(2)—C(3)	1·368 (5)	C(3)—C(4)	1·400 (5)
C(4)—C(5)	1·408 (5)	C(4)—C(9)	1·492 (5)	C(5)—C(6)	1·377 (5)
C(10)—C(11)	1·520 (6)	C(10)—C(14)	1·536 (5)	C(12)—C(14)	1·523 (6)
C(13)—C(14)	1·512 (5)	C(15)—C(16)	1·495 (6)	C(16)—C(17)	1·389 (7)
C(16)—C(21)	1·363 (7)	C(17)—C(18)	1·375 (8)	C(18)—C(19)	1·34 (1)
C(19)—C(20)	1·36 (1)	C(20)—C(21)	1·35 (1)		

C(5)—O(1)—C(8)	119·7 (3)	C(1)—N(1)—C(7)	122·7 (3)
C(9)—N(2)—C(10)	122·3 (3)	C(11)—N(3)—C(13)	103·9 (3)
C(11)—N(3)—C(15)	111·6 (3)	C(13)—N(3)—C(15)	117·1 (3)
N(1)—C(1)—C(2)	121·6 (3)	N(1)—C(1)—C(6)	121·5 (3)
C(2)—C(1)—C(6)	116·9 (3)	Cl—C(2)—C(1)	118·6 (3)
Cl—C(2)—C(3)	119·5 (3)	C(1)—C(2)—C(3)	121·8 (3)
C(2)—C(3)—C(4)	121·7 (3)	C(3)—C(4)—C(5)	116·7 (3)
C(3)—C(4)—C(9)	116·7 (3)	C(5)—C(4)—C(9)	126·6 (3)
O(1)—C(5)—C(4)	116·6 (3)	O(1)—C(5)—C(6)	122·3 (3)
C(4)—C(5)—C(6)	121·1 (3)	C(1)—C(6)—C(5)	121·8 (3)
O(2)—C(9)—N(2)	121·0 (4)	O(2)—C(9)—C(4)	120·4 (3)
N(2)—C(9)—C(4)	118·7 (3)	N(2)—C(10)—C(11)	113·4 (3)
N(2)—C(10)—C(14)	111·1 (3)	C(11)—C(10)—C(14)	106·6 (3)
N(3)—C(11)—C(10)	105·6 (3)	N(3)—C(13)—C(14)	103·7 (3)
C(10)—C(14)—C(12)	115·5 (3)	C(10)—C(14)—C(13)	102·7 (3)
C(12)—C(14)—C(13)	114·3 (3)	N(3)—C(15)—C(16)	114·1 (4)
C(15)—C(16)—C(17)	121·4 (4)	C(15)—C(16)—C(21)	120·4 (4)
C(17)—C(16)—C(21)	118·1 (4)	C(16)—C(17)—C(18)	120·1 (5)
C(17)—C(18)—C(19)	116·8 (6)	C(18)—C(19)—C(20)	126·7 (7)
C(19)—C(20)—C(21)	114·3 (7)	C(16)—C(21)—C(20)	123·9 (6)

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* Lists of structure factors, anisotropic thermal parameters of non-H atoms, parameters of H atoms, bond lengths and angles involving H atoms, and the least-squares plane for the benzene ring have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42949 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

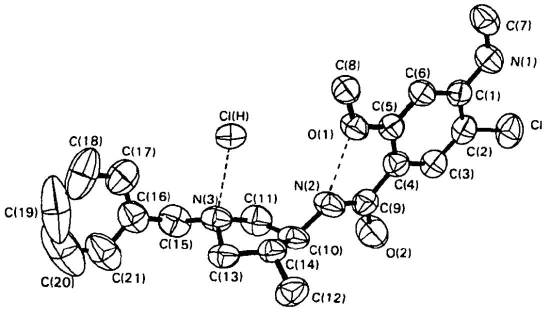

Fig. 1. Molecular structure and atom numbering.

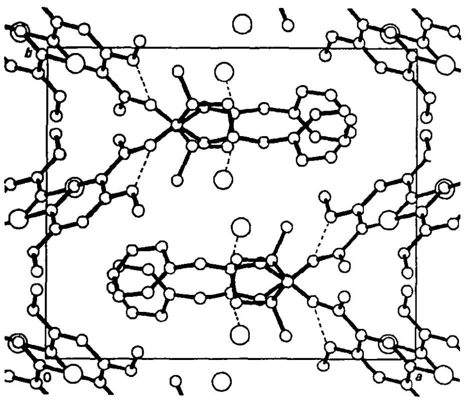

Fig. 2. Projection of the structure on the *ab* plane.

C(9) [1·492 (5) Å] are distinctly shorter than the ordinary single bond. These indicate that there is some contribution of the quinonoidal form. The benzene ring *A* [C(1)–C(6)] is almost planar with a maximum atomic deviation of 0·004 Å. The amino, methoxy and carbamoyl groups also lie on the plane.

An intramolecular hydrogen bond between the amide N(2) and methoxy O(1) results in a six-membered ring fused with the benzene ring *A*. This type of hydrogen bond is also observed in the benzamide neuroleptics we have studied and in metochlopramide (Cesario, Pascard, Moukhtari & Jung, 1981). The N(2)···O(1) length [2·635 (4) Å] is rather shorter than that of the compounds [2·662 (3)–2·716 (9) Å] in this series of studies and in metochlopramide [2·683 (6) Å]. Thus, the benzamide moiety constitutes a rigid part of the molecule by this strong hydrogen bond.

The distance between the tertiary amino N and the center of the benzene ring *A* and the deviation of the N from the mean plane of the ring *A* are used as the parameters relevant to neuroleptic activity. Comparing these values of the present compound to those of the compounds having a methyl group at the 2-position of the pyrrolidine ring, the distance, 6·91 Å, is longer than that of the free base of YM-09151-2 (6·26 Å) (Furuya *et al.*, 1982), but shorter than that of the hydrochloride of YM-09151-1 (7·25–7·5 Å) (Furuya *et al.*, 1986). On the other hand, the deviation of the present molecule, 1·75 Å, is rather greater than that of both compounds (–0·9–0·0 Å).

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Nonsteroidal Antiinflammatory Drugs. IV. Structure of (±)-(2*SR*)-2-{4-[(1*RS*,2*RS*)-2-Hydroxycyclopentylmethyl]phenyl}propionic Acid

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Abstract. $C_{15}H_{20}O_3$, $M_r = 248·3$, triclinic, $P\bar{1}$, $a = 21·511$ (1), $b = 12·275$ (1), $c = 5·5745$ (4) Å, $\alpha = 103·05$ (1), $\beta = 101·59$ (1), $\gamma = 73·73$ (1)°, $V = 1361·4$ (2) Å^3, $Z = 4$, $D_x = 1·21$ g cm^{-3}, graphite-monochromated Cu $K\alpha$, $\lambda = 1·5418$ Å, $\mu = 6·8$ cm^{-1}, $F(000) = 536$, $T = 298$ K, final $R = 0·068$ for 2042 reflections. The molecules form tetramers through four hydrogen bonds between the carboxyl and hydroxyl

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