The authors are grateful to a referee for pointing out a space-group discrepancy.

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Structure of a Benzamide Derivative

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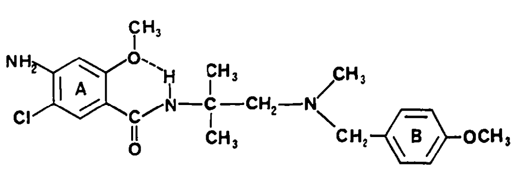
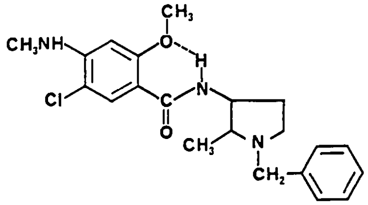
Abstract. 4-Amino-5-chloro-2-methoxy-*N*-{2-[*N*-(*p*-methoxybenzyl)methylamino]-1,1-dimethylethyl}benzamide, $C_{21}H_{28}ClN_3O_3$, $M_r = 405{\cdot}93$, monoclinic, $P2_1/c$, $a = 6{\cdot}144\ (1)$, $b = 26{\cdot}512\ (2)$, $c = 13{\cdot}741\ (2)$ Å, $\beta = 94{\cdot}76\ (1)°$, $V = 2230{\cdot}5\ (5)$ Å^3, $Z = 4$, $D_m = 1{\cdot}21$, $D_x = 1{\cdot}209$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 1{\cdot}92$ cm^{-1}, $F(000) = 864$, room temperature, $R = 0{\cdot}076$ for 3926 observed reflections. The benzamide moiety adopts a conformation and intramolecular hydrogen bond similar to those of a potent analogue, while the distance between the tertiary N atom and the center of the benzene ring is different. An intermolecular hydrogen bond of 3·019 (3) Å exists between the amino group N and the carbonyl O.

Introduction. The crystal-structure determination of the title compound (1) was undertaken as a part of continuous studies to find new potent neuroleptic drugs in benzamide derivatives in order to establish the three-dimensional structure–activity relationships. The crystal structure of a most potent compound, YM-09151-2 (2) (Furuya, Iwanami, Takenaka & Sasada, 1982), has been determined previously. Even though the present compound is not an active one contrary to expectation, the comparison of the structural features of active and inactive compounds would be important for further studies.



(1)



(2)

Experimental. Colorless plate crystals obtained from an acetone solution, density measured by flotation in a mixture of benzene and carbon tetrachloride. Crystal size 0·5 × 0·3 × 0·4 mm, Rigaku automated four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Accurate unit-cell constants determined using 40 high-angle reflections in range $20 < 2\theta < 30°$. Intensities measured for $2 < 2\theta < 50°$, $-7 \leq h \leq 7$,

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$0 \leq k \leq 31$, $0 \leq l \leq 16$, θ–2θ scan mode, scanning rate 4° (2θ) min^{-1}. Five reference reflections showed no significant intensity deterioration. 3926 independent reflections, 422 weak reflections below background considered zero reflections, observational threshold value $F_{\text{lim}} = 1{\cdot}22$. Corrections for Lorentz and polarization factors, but not for absorption or secondary extinction. Standard deviations $\sigma^2(F_o) = \sigma p^2(F_o) + qF_o^2$, with $\sigma p(F_o)$ evaluated by counting statistics and q ($5{\cdot}58 \times 10^{-5}$) derived from variations of monitored reflections (McCandlish & Stout, 1975).

Structure solved by direct methods, *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), and refined by block-diagonal least-squares calculations. All H atoms found on difference map, refined isotropically. $\sum w(|F_o| - |F_c|)^2$ minimized, with $w = 1/\sigma^2(F_o)$. In refinement procedure, zero reflections assumed to be $F_o = F_{\text{lim}}$, but those with $|F_c| < F_{\text{lim}}$ omitted. Final $R = 0{\cdot}076$ ($wR = 0{\cdot}065$); max. shifts of parameters $0{\cdot}3\sigma$ for C, $0{\cdot}05\sigma$ for N, $0{\cdot}05\sigma$ for O, $0{\cdot}07\sigma$ for Cl and $0{\cdot}4\sigma$ for H atoms. $\Delta\rho_{\text{max}} = 0{\cdot}80$, $\Delta\rho_{\text{min}} = -0{\cdot}75$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computations performed on a Hitachi HITAC M200 computer.

Discussion. Final atomic parameters for non-H atoms are given in Table 1.* The molecular structure with the atom numbering is shown in Fig. 1. The crystal structure is shown in Fig. 2.

An intermolecular hydrogen bond is found between the amino nitrogen N(1) and the carbonyl oxygen O(2) related by a c glide, the N(1)—O(2) distance being 3·019 (3) Å. The other intermolecular contacts are all larger than the sum of the van der Waals radii. The packing around the benzene ring A is especially rather loose, and it seems that the ring librates about the axis of C(1)—C(4), as seen from the thermal ellipsoids in Fig. 1.

The bond lengths and angles are listed in Table 2. The lengths C(2)—C(3) and C(5)—C(6) are short in comparison with the other lengths in the ring, whereas angles C(2)—C(1)—C(6) and C(3)—C(4)—C(5) are somewhat small. Moreover, the lengths C(1)—N(1) and C(4)—C(8) are short. These results indicate that the molecule has some contribution from the quinonoidal form. The benzene ring A is almost planar with maximum atomic deviation 0·007 Å. The amino methoxyl, and carbamoyl groups attached to this ring also lie

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

The B values accompanied by $\langle\rangle$ are the equivalent isotropic temperature factors calculated from 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 principal components of the mean-square displacement matrix **U**. Values in $\langle\rangle$ are anisotropicity defined by $[\sum(B - 8\pi^2 U_i)^2/3]^{1/2}$ and those in () are e.s.d.'s; they refer to last decimal places.

	x	y	z	B(Å^2)
Cl	0·4003 (3)	0·33175 (6)	0·91087 (9)	15·7 ⟨166⟩
O(1)	1·0995 (4)	0·20736 (9)	1·0930 (1)	7·4 ⟨62⟩
O(2)	0·8140 (3)	0·17220 (7)	0·8160 (1)	4·66 ⟨196⟩
O(3)	1·1804 (4)	0·04704 (9)	1·4813 (2)	7·6 ⟨23⟩
N(1)	0·6095 (5)	0·3461 (1)	1·1116 (2)	7·5 ⟨50⟩
N(2)	1·0943 (3)	0·15457 (8)	0·9280 (1)	3·8 ⟨10⟩
N(3)	1·3946 (3)	0·09956 (8)	1·0469 (2)	4·0 ⟨7⟩
C(1)	0·6880 (5)	0·3064 (1)	1·0599 (2)	4·9 ⟨18⟩
C(2)	0·6067 (6)	0·2944 (1)	0·9656 (2)	6·5 ⟨45⟩
C(3)	0·6872 (5)	0·2549 (1)	0·9159 (2)	5·3 ⟨30⟩
C(4)	0·8530 (4)	0·2246 (1)	0·9564 (2)	3·4 ⟨10⟩
C(5)	0·9361 (5)	0·2373 (1)	1·0516 (2)	4·2 ⟨14⟩
C(6)	0·8546 (5)	0·2766 (1)	1·1020 (2)	4·5 ⟨19⟩
C(7)	1·1870 (8)	0·2184 (2)	1·1909 (3)	12·6 ⟨119⟩
C(8)	0·9202 (4)	0·1816 (1)	0·8950 (2)	3·5 ⟨7⟩
C(9)	1·1795 (4)	0·1091 (1)	0·8828 (2)	3·8 ⟨8⟩
C(10)	1·2256 (5)	0·1193 (1)	0·7768 (2)	5·6 ⟨21⟩
C(11)	1·0199 (5)	0·0654 (1)	0·8887 (2)	5·3 ⟨10⟩
C(12)	1·4011 (5)	0·0992 (1)	0·9403 (2)	4·5 ⟨12⟩
C(13)	1·5756 (5)	0·1296 (1)	1·0941 (2)	6·6 ⟨15⟩
C(14)	1·3935 (6)	0·0490 (1)	1·0884 (2)	5·6 ⟨23⟩
C(15)	1·3316 (5)	0·0486 (1)	1·1923 (2)	4·5 ⟨9⟩
C(16)	1·4589 (5)	0·0243 (1)	1·2656 (2)	5·6 ⟨18⟩
C(17)	1·4022 (6)	0·0235 (1)	1·3595 (2)	6·3 ⟨20⟩
C(18)	1·2171 (5)	0·0479 (1)	1·3844 (2)	5·2 ⟨15⟩
C(19)	1·0845 (5)	0·0715 (1)	1·3131 (2)	5·4 ⟨7⟩
C(20)	1·1439 (5)	0·0718 (1)	1·2177 (2)	5·3 ⟨14⟩
C(21)	0·9927 (7)	0·0732 (2)	1·5106 (3)	8·3 ⟨28⟩

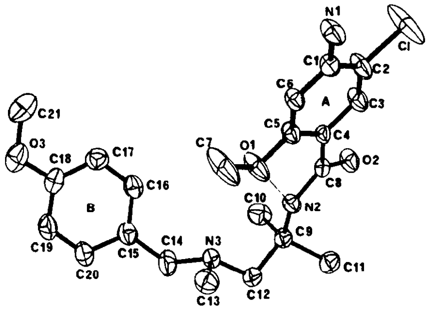

Fig. 1. Molecular structure and atom numbering.

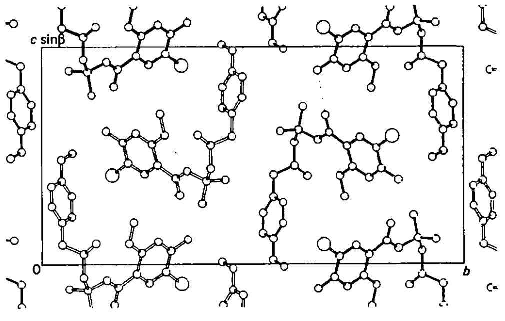

Fig. 2. Crystal structure viewed down the a axis.

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* Lists of structure factors, atomic coordinates of H atoms, anisotropic thermal parameters of non-H atoms, bond distances and angles involving H atoms and least-squares planes for the two benzene rings A and B have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42299 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

Standard deviations are given in parentheses.

Cl–C(2)	1·731 (3)	O(1)–C(5)	1·366 (3)
O(1)–C(7)	1·436 (5)	O(2)–C(8)	1·245 (3)
O(3)–C(18)	1·370 (3)	O(3)–C(21)	1·433 (4)
N(1)–C(1)	1·378 (4)	N(2)–C(8)	1·335 (3)
N(2)–C(9)	1·473 (3)	N(3)–C(12)	1·470 (3)
N(3)–C(13)	1·473 (4)	N(3)–C(14)	1·456 (3)
C(1)–C(2)	1·387 (4)	C(1)–C(6)	1·381 (4)
C(2)–C(3)	1·365 (4)	C(3)–C(4)	1·377 (3)
C(4)–C(5)	1·405 (3)	C(4)–C(8)	1·497 (3)
C(5)–C(6)	1·370 (3)	C(9)–C(10)	1·531 (4)
C(9)–C(11)	1·523 (4)	C(9)–C(12)	1·539 (3)
C(14)–C(15)	1·507 (4)	C(15)–C(16)	1·383 (4)
C(15)–C(20)	1·377 (4)	C(16)–C(17)	1·364 (4)
C(17)–C(18)	1·376 (4)	C(18)–C(19)	1·373 (4)
C(19)–C(20)	1·389 (4)		

C(5)–O(1)–C(7)	118·5 (2)	C(18)–O(3)–C(21)	117·8 (2)
C(8)–N(2)–C(9)	126·8 (2)	C(12)–N(3)–C(13)	111·1 (2)
C(12)–N(3)–C(14)	112·7 (2)	C(13)–N(3)–C(14)	110·6 (2)
N(1)–C(1)–C(2)	122·9 (2)	N(1)–C(1)–C(6)	120·0 (2)
C(2)–C(1)–C(6)	117·2 (2)	Cl–C(2)–C(1)	117·9 (2)
Cl–C(2)–C(3)	120·3 (2)	C(1)–C(2)–C(3)	121·7 (3)
C(2)–C(3)–C(4)	122·1 (2)	C(3)–C(4)–C(5)	115·9 (2)
C(3)–C(4)–C(8)	116·5 (2)	C(5)–C(4)–C(8)	127·6 (2)
O(1)–C(5)–C(4)	116·6 (2)	O(1)–C(5)–C(6)	121·2 (2)
C(4)–C(5)–C(6)	122·2 (2)	C(1)–C(6)–C(5)	120·9 (2)
O(2)–C(8)–N(2)	122·4 (2)	O(2)–C(8)–C(4)	119·6 (2)
N(2)–C(8)–C(4)	117·9 (2)	N(2)–C(9)–C(10)	110·9 (2)
N(2)–C(9)–C(11)	110·3 (2)	N(2)–C(9)–C(12)	104·7 (2)
C(10)–C(9)–C(11)	110·8 (2)	C(10)–C(9)–C(12)	107·0 (2)
C(11)–C(9)–C(12)	112·9 (2)	N(3)–C(12)–C(9)	114·5 (2)
N(3)–C(14)–C(15)	112·8 (2)	C(14)–C(15)–C(16)	121·4 (2)
C(14)–C(15)–C(20)	121·3 (2)	C(16)–C(15)–C(20)	117·2 (2)
C(15)–C(16)–C(17)	121·6 (3)	C(16)–C(17)–C(18)	120·6 (3)
O(3)–C(18)–C(17)	116·1 (2)	O(3)–C(18)–C(19)	124·5 (2)
C(17)–C(18)–C(19)	119·4 (2)	C(18)–C(19)–C(20)	119·2 (2)
C(15)–C(20)–C(19)	121·9 (2)		

in the plane. The distance between the tertiary amino nitrogen and the center of the ring, 5·83 Å, is shorter than that in YM-09151-2, 6·26 Å.

An intramolecular hydrogen bond between the amide N(2) and methoxyl O(1) forms a six-membered ring fused with the benzene ring *A*. This type of hydrogen bond is also observed in the benzamide neuroleptics (Furuya *et al.*, 1982; Cesario, Pascard, Moukhtari & Jung, 1981). The N(2)—O(1) distance 2·662 (3) Å is rather shorter than in YM-09151-2, 2·680 (3) Å, and metoclopramide, 2·683 (6) Å. Thus, the benzamide moiety constitutes a rigid part of the molecule by this strong hydrogen bond. The benzene ring *B* is almost planar with maximum atomic deviation 0·014 Å.

It is interesting that the overall structural features of this inactive compound are similar to those of the potent neuroleptic drug YM-09151-2.

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The Polymorphism of 4-Chloro-1,2-dinitrobenzene: Structures of the γ and β Forms

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Abstract. $C_6H_3ClN_2O_4$, $M_r = 202{\cdot}4$, $\lambda(\mathrm{Mo}\,K\alpha) = 0{\cdot}71069$ Å, $F(000) = 408$; γ form: orthorhombic, $P2_12_12_1$, $a = 12{\cdot}11\ (2)$, $b = 5{\cdot}57\ (1)$, $c = 11{\cdot}94\ (2)$ Å, $V = 805{\cdot}4$ Å^3, $Z = 4$, $D_m = 1{\cdot}64$, $D_x = 1{\cdot}67$ Mg m^{-3}, $\mu = 0{\cdot}395$ mm^{-1}. β form: monoclinic, $P2_1/c$, $a = 7{\cdot}64\ (1)$, $b = 7{\cdot}82\ (1)$, $c = 14{\cdot}82\ (1)$ Å, $\beta = 114{\cdot}0\ (5)°$, $V = 808{\cdot}9$ Å^3, $D_m = 1{\cdot}67$, $D_x = 1{\cdot}66$ Mg m^{-3}, $\mu = 0{\cdot}394$ mm^{-1}. For γ, $R = 0{\cdot}033$ for 1053 observed reflexions; for β, $R = 0{\cdot}065$ for 762 observed reflexions. The molecules in the two polymorphs differ only slightly in shape and dimensions; packing in γ comprises interleaved molecules, in β molecules in layers. Both structures involve close N···O and Cl···O intermolecular contacts. Because of repulsion of O atoms in adjacent rings, the nitro groups rotate relative to the aromatic rings.

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