of the particular ring. The r.m.s. deviation of all ring atoms from their respective ring planes was 0·007 Å for the set of 24 ring atoms, with a maximum deviation of 0·012 Å.

Tables 3 and 4 show interatomic distances and angles computed with the atom coordinates of Table 1. The least-squares standard deviations for the atom coordinates give standard deviations between 0·004 and 0·009 Å for all the bond lengths and 0·3 to 0·6° for all bond angles. A more realistic estimate of errors is provided by the set of 24 carbon–carbon bond lengths and angles in the four benzene rings. The 24 aromatic ring bond lengths average 1·374 Å with a standard deviation of 0·024 Å. The 24 C–C–C interior angles, Table 4, in the four benzene rings exhibit a standard deviation of 1·3° from the average of 120·0°. As may be seen from Table 3, other pairs of chemically equivalent, bonds, *e.g.* C(15)–N(4) and C(3)–N(1), or N(1)–N(2) and N(3)–N(4), agree to 0·01 Å. Rings *B* and *C* are rotated out of the plane of the conjugated system by an average of 57° about the bonds joining the rings to carbon atoms C(1) and to C(2). This permits normal van der Waals contact distances between the *ortho* hydrogen atoms on rings *B* and *C* and the nearest nitrogen atoms; an average of 2·76 Å was found for these four H···N intramolecular non-bonded distances. Within experimental error the molecular geometry is defined by a twofold molecular rotation axis perpendicular to the plane of the conjugated system and passing midway between C(1) and C(2). Each bond length shown in Fig. 1 is the average of the pair of bonds related by this twofold molecular axis. Each bond angle is likewise the average of a symmetry-related pair. The maximum deviation of any angle shown in Fig. 1 from the average is 1·0°, found for the N(1)–C(3)–C(4) angle. The azo benzene rings appear to be rotated 4° in the plane of the conjugated system; this rotation is probably real and is more likely caused by the intramolecular repulsion of the *ortho* ring hydrogen and the nearest nitrogen atom. Fig. 1 shows the rather short distance of 2·47 Å for this non-bonded atom pair. The average value of 1·25 Å found for the azo N–N bond lengths in this compound is in good agreement with the value of 1·244 Å obtained by Brown (1966*a*) for *p*-azotoluene and the average value of 1·21 Å obtained by Brown (1966*b*) for azobenzene. His C–N bond lengths of 1·433 and 1·452 Å for these two compounds are also within experimental error of the value 1·430 Å reported here for the average phenyl-*N* bond length. Brown's reported C–N–N bond angles of 113·8° (for *p*-azotoluene) and 112·4 (for azobenzene) are also within experimental error of the value of 114·4° reported here.

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1-{1-[3-(*p*-Fluorobenzoyl)propyl]-4-piperidyl}-2-benzimidazolinone (Benperidol)

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Abstract. Rhombohedral (hexagonal setting); $R\bar{3}$; $a=36\cdot6201$ (7), $c=7\cdot7053$ (1) Å; 25°C; $C_{22}H_{24}FN_3O_2$; $M=381\cdot44$; $Z=18$; $F(000)=3636$.

Introduction. Benperidol is a very potent neuroleptic. Transparent hexagonal needles were obtained by very slow evaporation from a 9:1 mixture of *N*,*N*-dimethylformamide and isopropanol.

Experimental. The lattice parameters were obtained by least-squares refinement of the setting angles of 4 reflexions measured with Cu $K\beta$ radiation.

Only reflexions for which $-h+k+l=3n$ (obverse cell) were present on Weissenberg photographs. Hence there are two possibilities for the space group: $R3$ and $R\bar{3}$. The latter was selected on the basis of the distribution of E values ($\langle E^2\rangle=0\cdot9998$; $\langle|E^2-1|\rangle=0\cdot9411$;

$\langle |E| \rangle = 0{\cdot}8185$). Intensities were collected on a Picker four circle card-controlled diffractometer. The experimental conditions are given in Table 1. The structure was solved by direct methods with *MULTAN* (Germain, Main & Woolfson, 1971).

Table 1. *Experimental data*

Source Cu $K\bar{\alpha}$; $\lambda = 1{\cdot}5418$ Å; Ni filter; ω–2θ scan; $\Delta 2\theta = \pm 1{\cdot}3°$; $\theta_{\max} = 65°$; confidence level = 2·0.
Total number of independent reflexions: 3297; total observed: 2259.

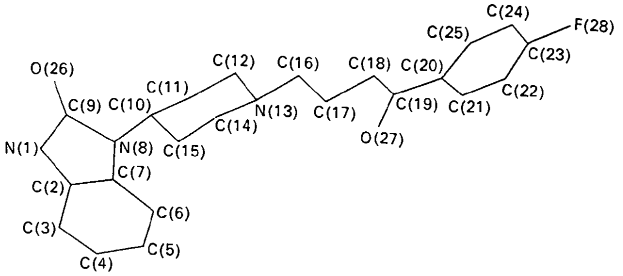

Fig. 1. Conformation and atom numbering of $C_{22}H_{24}FN_3O_2$.

Table 2. *Final positional and thermal parameters* × 10^4 (*with standard deviations in parentheses*)

$$B = \exp\left[-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{13}hl + B_{23}kl)\right]$$

	x/a	x/b	z/c	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}
N(1)	8263 (1)	8635 (1)	7341 (6)	10 (0)	11 (0)	169 (9)	6 (3)	14 (3)	12 (1)
C(2)	8700 (2)	8825 (2)	7407 (6)	10 (1)	10 (1)	118 (9)	13 (4)	3 (3)	11 (1)
C(3)	8993 (2)	9152 (2)	8420 (7)	14 (1)	11 (1)	164 (10)	−10 (4)	−17 (4)	16 (1)
C(4)	9417 (2)	9289 (2)	8129 (8)	12 (1)	14 (1)	176 (11)	−20 (5)	−21 (5)	15 (1)
C(5)	9541 (2)	9097 (2)	6913 (8)	11 (1)	13 (1)	167 (11)	−7 (4)	−12 (4)	11 (1)
C(6)	9246 (2)	8758 (2)	5915 (7)	10 (1)	12 (1)	167 (10)	−1 (4)	−4 (4)	12 (1)
C(7)	8821 (2)	8631 (2)	6158 (6)	9 (0)	10 (1)	123 (8)	5 (4)	−3 (3)	12 (1)
N(8)	8454 (1)	8327 (1)	5335 (6)	8 (0)	11 (0)	170 (9)	−10 (3)	0 (3)	9 (1)
C(9)	8112 (2)	8347 (2)	6018 (8)	8 (0)	10 (1)	210 (12)	10 (4)	11 (4)	9 (1)
C(10)	8414 (2)	8069 (2)	3812 (7)	10 (1)	9 (1)	169 (11)	−10 (4)	−7 (4)	10 (1)
C(11)	8652 (2)	8349 (2)	2248 (8)	15 (1)	11 (1)	166 (11)	−9 (4)	−10 (5)	15 (1)
C(12)	8632 (2)	8081 (2)	677 (8)	15 (1)	14 (1)	190 (12)	−23 (5)	−28 (5)	17 (1)
N(13)	8799 (1)	7801 (1)	1140 (6)	12 (1)	10 (0)	152 (8)	−21 (3)	−15 (4)	11 (1)
C(14)	8545 (2)	7514 (2)	2527 (8)	14 (1)	8 (1)	210 (12)	−14 (4)	−7 (5)	10 (1)
C(15)	8565 (2)	7758 (2)	4203 (8)	13 (1)	10 (1)	192 (12)	−4 (4)	−1 (5)	13 (1)
C(16)	8796 (3)	7578 (3)	−483 (10)	23 (1)	17 (1)	229 (15)	−30 (6)	−18 (7)	25 (2)
C(17)	8962 (3)	7292 (3)	−310 (11)	28 (2)	23 (1)	238 (18)	−25 (8)	−14 (8)	32 (3)
C(18)	8993 (3)	7127 (3)	−2129 (10)	25 (1)	22 (1)	212 (16)	−40 (7)	25 (8)	22 (2)
C(19)	9059 (3)	6763 (3)	−1979 (9)	20 (1)	17 (1)	178 (14)	12 (6)	9 (6)	11 (2)
C(20)	9117 (3)	6582 (3)	−3518 (11)	11 (1)	12 (1)	167 (19)	16 (7)	2 (7)	4 (2)
C(21)	9165 (3)	6207 (3)	−3359 (13)	17 (1)	15 (1)	302 (24)	54 (8)	−4 (8)	11 (1)
C(22)	9213 (2)	6016 (2)	−4765 (12)	16 (1)	19 (1)	401 (20)	−21 (7)	−7 (7)	17 (1)
C(23)	9222 (2)	6187 (2)	−6311 (10)	15 (1)	13 (1)	359 (14)	−21 (5)	16 (6)	12 (1)
C(24)	9192 (2)	6529 (2)	−6595 (8)	16 (1)	12 (1)	241 (11)	−16 (4)	4 (5)	11 (1)
C(25)	9137 (1)	6736 (1)	−5173 (6)	16 (0)	9 (0)	151 (11)	0 (4)	2 (3)	7 (1)
O(26)	7752 (1)	8144 (1)	5507 (6)	7 (0)	12 (0)	333 (11)	−3 (4)	5 (3)	7 (1)
O(27)	9049 (3)	5612 (3)	−560 (8)	44 (2)	31 (1)	200 (12)	20 (6)	32 (7)	33 (3)
F(28)	9259 (2)	5977 (2)	−7739 (9)	28 (1)	24 (1)	577 (19)	−90 (7)	22 (7)	30 (2)

Block-diagonal least-squares refinement with the program written by Ahmed, Hall, Pippy & Huber (1966) resulted in $R = \sum ||F_o| - |F_c|| / \sum |F_o| = 0{\cdot}12$ for all observed reflexions.

The weight of each reflexion in the refinement was

$$w = 1/(50{\cdot}0 + |F_o| + 0{\cdot}05|F_o|^2)\,.$$

A difference map did not reveal the hydrogen atoms or any other special feature. The atomic scattering factors used were those given in *International Tables for X-ray Crystallography* (1962).

Discussion. The atomic parameters and bond lengths and angles are given in Tables 2 and 3 respectively.*

The conformation of the side chain of the molecule confirms the calculations carried out for butyrophen-

Table 3. *Intramolecular bond distances and angles* (*with standard deviations in parentheses*)

N(1)—C(2)	1·388 (9) Å	N(13)–C(16)	1·489 (9) Å
N(1)—C(9)	1·369 (7)	C(14)–C(15)	1·552 (9)
C(2)—C(3)	1·383 (8)	C(16)–C(17)	1·458 (16)
C(2)—C(7)	1·394 (8)	C(17)–C(18)	1·552 (13)
C(3)—C(4)	1·390 (11)	C(18)–C(19)	1·474 (14)
C(4)—C(5)	1·378 (10)	C(19)–C(20)	1·427 (10)
C(5)—C(6)	1·399 (9)	C(19)–O(27)	1·219 (10)
C(6)—C(7)	1·398 (10)	C(20)–C(21)	1·472 (12)
C(7)—N(8)	1·396 (7)	C(20)–C(25)	1·380 (9)
N(8)—C(9)	1·393 (9)	C(21)–C(22)	1·348 (13)
N(8)—C(10)	1·468 (7)	C(22)–C(23)	1·338 (13)
C(9)—O(26)	1·212 (8)	C(23)–C(24)	1·329 (12)
C(10)–C(11)	1·539 (8)	C(23)–F(28)	1·389 (11)
C(10)–C(15)	1·524 (9)	C(24)–C(25)	1·401 (10)
C(11)–C(12)	1·537 (9)		
C(12)–N(13)	1·475 (10)		
N(13)–C(14)	1·461 (8)		

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* A list of structure factors has been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30150. Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 3 (*cont.*)

C(2)—N(1)—C(9)	109·5 (5)°	C(12)-N(13)-C(14)	109·9 (5)°
N(1)—C(2)—C(7)	107·4 (5)	C(12)-N(13)-C(16)	106·5 (5)
N(1)—C(2)—C(3)	130·9 (5)	C(14)-N(13)-C(16)	113·1 (6)
C(3)—C(2)—C(7)	121·6 (5)	N(13)-C(14)-C(15)	111·4 (5)
C(2)—C(3)—C(4)	117·8 (6)	C(10)-C(15)-C(14)	109·6 (5)
C(3)—C(4)—C(5)	121·2 (6)	N(13)-C(16)-C(17)	115·2 (8)
C(4)—C(5)—C(6)	121·5 (6)	C(16)-C(17)-C(18)	109·6 (8)
C(5)—C(6)—C(7)	117·4 (6)	C(17)-C(18)-C(19)	110·9 (8)
C(2)—C(7)—C(6)	120·5 (5)	C(18)-C(19)-O(27)	120·1 (9)
C(6)—C(7)—N(8)	132·4 (5)	C(18)-C(19)-C(20)	119·1 (8)
C(2)—C(7)—N(8)	107·1 (5)	C(20)-C(19)-O(27)	120·7 (8)
C(7)—N(8)—C(9)	108·6 (5)	C(19)-C(20)-C(21)	118·7 (7)
C(7)—N(8)—C(10)	128·4 (5)	C(21)-C(20)-C(25)	116·8 (7)
C(9)—N(8)—C(10)	122·3 (5)	C(19)-C(20)-C(25)	124·6 (7)
N(1)—C(9)—N(8)	107·0 (5)	C(20)-C(21)-C(22)	121·6 (8)
N(8)—C(9)—O(26)	125·1 (5)	C(21)-C(22)-C(23)	116·8 (9)
N(1)—C(9)—O(26)	127·9 (6)	C(22)-C(23)-C(24)	128·4 (9)
N(8)—C(10)-C(11)	110·9 (5)	C(22)-C(23)-F(28)	115·6 (8)
N(8)—C(10)-C(15)	111·5 (5)	C(24)-C(23)-F(28)	118·0 (8)
C(11)-C(10)-C(15)	110·8 (5)	C(23)-C(24)-C(25)	118·7 (8)
C(10)-C(11)-C(12)	111·2 (5)	C(20)-C(25)-C(24)	119·7 (7)
C(11)-C(12)-N(13)	110·4 (6)		

one derivatives (Koch, 1973). The torsional angles are given in Table 4.

Table 4. *Torsional angles defining the conformation of the side chain*

C(12)-N(13)-C(16)-C(17)	178·5
C(14)-N(13)-C(16)-C(17)	−60·7
N(13)-C(16)-C(17)-C(18)	−172·8
C(16)-C(17)-C(18)-C(19)	−166·2
C(17)-C(18)-C(19)-C(20)	−176·7
C(17)-C(18)-C(19)-O(27)	5·6
C(18)-C(19)-C(20)-C(21)	−177·4
C(18)-C(19)-C(20)-C(25)	3·2

The angle between the least-squares mean plane of the piperidine ring and the benzimidazole group is 86°.

The presence of an aromatic group nearly perpendicular to the mean plane of the piperidine ring seems to be a requirement for strong neuroleptic activity. In spiperone (Koch, 1973) and benperidol this conformation is rigidly fixed while it is readily accessible for the 4-hydroxy-4-phenylpiperidine derivatives (*e.g.* haloperidol).

Each molecule is hydrogen bonded to two neighbours

N(1)···N(13): 2·882 Å [N(13): y, $1+y-x$, $1-z$]
N(13)···N(1): 2·882 Å [N(1): $1+x-y$, x, $1-z$].

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