Fig. 2. Packing diagram for 2-phenyl-1,2,3-triaziniumbetaine 1-oxide. Note the sandwich-dimer arrangement between 2-phenyl groups from different molecules.

References

BALLARD, R. E., NORRIS, E. K. & SHELDRICK, G. M. (1975). Acta Cryst. 31, 295–296.

CHATTAWAY, F. D. & ADAMSON, A. B. (1930). J. Chem. Soc. pp. 157–163, 843–850.
CHATTAWAY, F. D. & PARKES, G. D. (1935). J. Chem. Soc. pp. 1005–1008.
CHATTAWAY, F. D. & WALKER, A. J. (1927). J. Chem. Soc. pp. 323–333.
GIBSON, M. S. (1962). Tetrahedron, 18, 1377–1380.
KERBER, R. C. (1972). J. Org. Chem. 37, 1587–1592.
KOBYLECKI, R. J. (1973). Ph. D. thesis, Univ. of East Anglia.

1'-[4-(4-Fluorophenyl)-4-oxobutyl]-[1,4'-bipiperidine]-4'-carboxamide (Pipamperone)

BY J. P. DECLERCQ, G. GERMAIN AND M. H. J. KOCH

Laboratoire de Chimie Physique et de Cristallographie, Institut Lavoisier, Université de Louvain, B-1348 Louvain la Neuve, Belgium

Abstract. $C_{21}H_{30}N_3O_2F$, triclinic, $P\bar{1}$, $a=12\cdot488$ (7), $b=11\cdot177$ (7), $c=9\cdot808$ (6) Å; $\alpha=115\cdot34$ (2), $\beta=115\cdot65$ (2), $\gamma=99\cdot23$ (1)°, 25°C; M.W. 375·2; $Z=2$.

Introduction. Slow evaporation of a solution in ethanol yielded transparent crystals of this neuroleptic.

The lattice parameters were obtained from the setting angles of ten reflexions measured on a Picker card-controlled diffractometer. The experimental conditions during the measurement of the intensities are given in Table 1.

Table 1. Experimental conditions

Source Cu $K\bar{\alpha}$; $\lambda=1\cdot5418$ Å; $\omega-2\theta$ scan
$\theta_{max}=60°$; confidence level: 2·5
Total number of independent reflexions: 2998
Total observed: 2661

The structure was solved by direct methods with MULTAN (Germain, Main & Woolfson, 1971) and refined by block-diagonal least-squares calculations with the programs written by Ahmed, Hall, Pippy & Huber (1966). The positions of the hydrogen atoms were determined from a difference map. Only the positional parameters were refined for these atoms. A
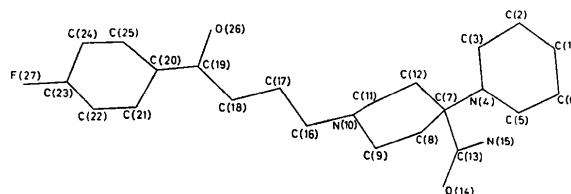

Fig. 1. Conformation of the molecule and numbering.

structure-factor calculation including all atoms resulted in $R = \sum||F_o|-|F_c||/\sum|F_o| = 0.06$ for all observed reflexions. The scattering factors used are those given in *International Tables for X-ray Crystallography* (1962). The final coordinates and standard deviations are given in Table 2.*

Discussion. The conformation of the molecule and the numbering scheme are shown in Fig. 1 and the bond distances and angles in Table 3. The conformation of the butyrophenone chain is fully extended and confirms the conformational calculations for this part of the molecule (Koch, 1974). The distance between N(10) and N(15) (4·82 Å) is within the range of distances between the basic nitrogen and a potential hydrogen bond donor found in most potent neuroleptics (Koch, 1974). The strongest intramolecular interactions occur between N(15) and O(14) [O(14): $2-x, 2-y, 4-z$: 3·02 Å] and between N(15) and O(26) [O(26): $1-x, 1-y, 3-z$: 3·09 Å].

We thank Dr Paul Janssen (Janssen Pharmaceutica, Beerse, Belgium) for providing the sample of pipam-

Table 2 (*cont.*)

Coordinates of the hydrogen atoms ($\times 10^4$) and isotropic thermal parameters

	X/a	Y/b	Z/c	B
H(11)	6715 (18)	8811 (20)	24069 (25)	5·8
H(12)	7561 (18)	7987 (21)	24684 (25)	5·8
H(21)	5490 (18)	6277 (21)	21656 (25)	5·8
H(22)	6952 (18)	6236 (20)	21667 (24)	5·8
H(31)	5397 (19)	6279 (21)	19157 (25)	5·8
H(32)	5319 (18)	7834 (20)	20606 (25)	5·8
H(51)	7291 (19)	10146 (20)	22825 (25)	5·8
H(52)	8615 (18)	10212 (21)	22731 (25)	5·8
H(61)	8941 (18)	8679 (20)	23893 (24)	5·8
H(62)	8822 (18)	10159 (20)	25272 (24)	5·8
H(81)	5334 (18)	9065 (20)	19330 (25)	5·8
H(82)	6660 (18)	10436 (20)	20273 (24)	5·8
H(91)	6306 (18)	9977 (20)	17462 (25)	5·8
H(92)	4915 (18)	9875 (20)	17435 (24)	5·8
H(111)	5147 (18)	6172 (20)	14709 (24)	5·8
H(112)	6441 (18)	7758 (21)	15970 (25)	5·8
H(121)	6667 (18)	6423 (20)	17503 (25)	5·8
H(122)	5255 (18)	6390 (20)	17229 (25)	5·8
H(151)	9772 (19)	8950 (20)	20270 (24)	5·8
H(152)	8880 (18)	7928 (20)	20300 (24)	5·8
H(161)	3551 (19)	8497 (20)	14358 (25)	5·8
H(162)	4718 (19)	8304 (21)	13914 (25)	5·8
H(171)	3621 (18)	5772 (20)	12075 (24)	5·8
H(172)	2658 (18)	5866 (20)	12833 (25)	5·8
H(181)	1811 (19)	7162 (21)	11424 (25)	5·8
H(182)	2647 (18)	6800 (20)	10465 (25)	5·8
H(211)	779 (18)	7180 (20)	8774 (25)	5·8
H(221)	−816 (18)	7060 (20)	6204 (25)	5·8
H(241)	−2512 (18)	2554 (21)	3451 (25)	5·8
H(251)	−869 (18)	2701 (20)	6087 (25)	5·8

Table 2. *Positional and thermal parameters*

Final heavy-atom positional and thermal parameters ($\times 10^4$) (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	Y/b	Z/c	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}
C(1)	7239 (2)	8242 (2)	23832 (2)	162	278	318	457	307	207
C(2)	6380 (2)	6878 (2)	21893 (2)	163	209	342	408	299	135
C(3)	5952 (1)	7287 (2)	20503 (2)	104	153	272	276	201	66
N(4)	7106 (1)	8203 (1)	20775 (1)	74	106	184	179	127	60
C(5)	7867 (1)	9566 (1)	22602 (2)	97	141	165	167	125	74
C(6)	8375 (2)	9229 (2)	24059 (2)	132	234	222	305	179	140
C(7)	6852 (1)	8461 (1)	19322 (1)	66	81	160	139	107	60
C(8)	6093 (1)	9410 (1)	19218 (2)	84	98	178	142	123	95
C(9)	5522 (1)	9398 (1)	17483 (2)	90	99	180	148	106	98
N(10)	4762 (1)	7901 (1)	15860 (1)	79	100	171	146	95	82
C(11)	5642 (1)	7180 (1)	15880 (2)	96	100	168	139	121	95
C(12)	6082 (1)	6988 (1)	17455 (2)	85	80	175	130	111	61
C(13)	8208 (1)	9187 (1)	19715 (1)	70	84	170	141	115	57
O(14)	8469 (1)	10206 (1)	19553 (2)	83	114	313	278	171	71
N(15)	9042 (1)	8613 (1)	20170 (2)	86	144	312	320	216	129
C(16)	4086 (1)	7893 (1)	14210 (2)	94	128	188	192	100	84
C(17)	3111 (1)	6369 (1)	12497 (2)	102	122	169	167	103	88
C(18)	2189 (1)	6450 (1)	10941 (2)	105	120	178	174	83	72
C(19)	1091 (1)	5003 (1)	9262 (2)	96	110	184	176	147	93
C(20)	112 (1)	4954 (1)	7662 (1)	84	102	163	144	127	75
C(21)	111 (1)	6213 (1)	7718 (2)	110	121	215	197	109	92
C(22)	−839 (2)	6127 (2)	6221 (2)	135	163	279	295	130	127
C(23)	−1761 (1)	4788 (2)	4702 (2)	105	204	217	283	100	118
C(24)	−1803 (2)	3521 (2)	4579 (2)	127	162	181	159	80	64
C(25)	−852 (1)	3611 (1)	6080 (2)	126	119	181	145	117	79
O(26)	986 (1)	3885 (1)	9224 (1)	153	112	239	216	123	98
F(27)	−2696 (1)	4687 (1)	3219 (1)	151	300	294	419	35	140

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

C(1)—C(2)	1·523 (3)	C(1)—H(11)	1·01 (3)
C(1)—C(6)	1·514 (3)	C(1)—H(12)	0·94 (2)
C(2)—C(3)	1·528 (3)	C(2)—H(21)	1·07 (3)
C(3)—N(4)	1·469 (2)	C(2)—H(22)	1·11 (3)
N(4)—C(5)	1·470 (2)	C(3)—H(31)	1·08 (2)
N(4)—C(7)	1·485 (2)	C(3)—H(32)	1·09 (3)
C(5)—C(6)	1·529 (3)	C(5)—H(51)	1·07 (3)
C(7)—C(8)	1·539 (2)	C(5)—H(52)	1·01 (3)
C(7)—C(12)	1·541 (2)	C(6)—H(61)	1·03 (3)
C(7)—C(13)	1·549 (2)	C(6)—H(62)	0·99 (2)
C(8)—C(9)	1·527 (2)	C(8)—H(81)	1·03 (3)
C(9)—N(10)	1·462 (2)	C(8)—H(82)	0·99 (2)
N(10)-C(11)	1·462 (2)	C(9)—H(91)	1·10 (3)
N(10)-C(16)	1·461 (2)	C(9)—H(92)	0·99 (3)
C(11)-C(12)	1·522 (2)	C(11)-H(111)	1·01 (2)
C(13)-O(14)	1·229 (2)	C(11)-H(112)	1·04 (3)
C(13)-N(15)	1·324 (2)	C(12)-H(121)	1·04 (3)
C(16)-C(17)	1·523 (2)	C(12)-H(122)	1·01 (3)
C(17)-C(18)	1·514 (2)	N(15)-H(151)	0·87 (3)
C(18)-C(19)	1·504 (2)	N(15)-H(152)	0·84 (2)
C(19)-C(20)	1·486 (2)	C(16)-H(161)	1·03 (3)
C(19)-O(26)	1·217 (2)	C(16)-H(162)	1·06 (3)
C(20)-C(21)	1·384 (2)	C(17)-H(171)	1·07 (2)
C(20)-C(25)	1·386 (2)	C(17)-H(172)	0·98 (3)
C(21)-C(22)	1·381 (2)	C(18)-H(181)	1·04 (3)
C(22)-C(23)	1·356 (3)	C(18)-H(182)	1·01 (2)
C(23)-C(24)	1·360 (3)	C(21)-H(211)	0·97 (2)
C(23)-F(27)	1·356 (2)	C(22)-H(221)	1·05 (3)
C(24)-C(25)	1·382 (2)	C(24)-H(241)	1·00 (2)
		C(25)-H(251)	1·02 (3)

N(10)-C(11)-C(12)	108·7 (1)	C(2)—C(1)—C(6)	109·7 (2)
C(7)—C(12)-C(11)	112·4 (1)	C(1)—C(2)—C(3)	110·8 (2)
C(7)—C(13)-O(14)	121·4 (1)	C(2)—C(3)—N(4)	110·1 (1)
C(7)—C(13)-N(15)	116·1 (1)	C(3)—N(4)—C(5)	109·2 (1)
O(14)-C(13)-N(15)	122·4 (1)	C(3)—N(4)—C(7)	116·1 (1)
N(10)-C(16)-C(17)	113·2 (1)	C(5)—N(4)—C(7)	112·7 (1)
C(16)-C(17)-C(18)	111·0 (1)	N(4)—C(5)—C(6)	110·0 (1)
C(17)-C(18)-C(19)	114·6 (1)	C(1)—C(6)—C(5)	110·2 (2)
C(18)-C(19)-C(20)	119·3 (1)	N(4)—C(7)—C(8)	113·5 (1)
C(18)-C(19)-O(26)	120·6 (1)	N(4)—C(7)—C(12)	109·9 (1)
C(20)-C(19)-O(26)	120·2 (1)	N(4)—C(7)—C(13)	106·5 (1)
C(19)-C(20)-C(21)	122·0 (1)	C(8)—C(7)—C(12)	108·9 (1)
C(19)-C(20)-C(25)	119·0 (1)	C(8)—C(7)—C(13)	111·0 (1)
C(21)-C(20)-C(25)	118·9 (1)	C(12)-C(7)—C(13)	106·7 (1)
C(20)-C(21)-C(22)	120·4 (2)	C(7)—C(8)—C(9)	115·1 (1)
C(21)-C(22)-C(23)	118·6 (2)	C(8)—C(9)—N(10)	110·9 (1)
C(22)-C(23)-C(24)	123·3 (2)	C(9)—N(10)-C(11)	108·4 (1)
C(22)-C(23)-F(27)	119·3 (2)	C(9)—N(10)-C(16)	110·6 (1)
C(24)-C(23)-F(27)	117·5 (2)	C(11)-N(10)-C(16)	113·2 (1)
C(23)-C(24)-C(25)	118·0 (2)		
C(20)-C(25)-C(24)	120·9 (2)		

Table 3 (cont.)

C(2)—C(1)—H(11)	110 (1)	N(10)—C(11)-H(112)	112 (1)
C(2)—C(1)—H(12)	110 (1)	C(12)—C(11)-H(111)	108 (1)
C(6)—C(1)—H(11)	107 (1)	C(12)—C(11)-H(112)	112 (1)
C(6)—C(1)—H(12)	110 (1)	H(111)-C(11)-H(112)	108 (2)
H(11)-C(1)—H(12)	110 (2)	C(7)——C(12)-H(121)	108 (1)
C(1)—C(2)—H(21)	110 (1)	C(7)——C(12)-H(122)	110 (1)
C(1)—C(2)—H(22)	109 (1)	C(11)—C(12)-H(121)	113 (1)
C(3)—C(2)—H(21)	105 (1)	C(11)—C(12)-H(122)	106 (1)
C(3)—C(2)—H(22)	108 (1)	H(121)-C(12)-H(122)	108 (2)
H(21)-C(2)—H(22)	114 (2)	C(13)—N(15)-H(151)	117 (2)
C(2)—C(3)—H(31)	107 (1)	C(13)—N(15)-H(152)	123 (2)
C(2)—C(3)—H(32)	110 (1)	H(151)-N(15)-H(152)	120 (2)
N(4)—C(3)—H(31)	109 (1)	N(10)—C(16)-H(161)	108 (1)
N(4)—C(3)—H(32)	111 (1)	N(10)—C(16)-H(162)	114 (1)
H(31)-C(3)—H(32)	109 (2)	C(17)—C(16)-H(161)	107 (1)
N(4)—C(5)—H(51)	112 (1)	C(17)—C(16)-H(162)	105 (1)
N(4)—C(5)—H(52)	108 (1)	H(161)-C(16)-H(162)	110 (2)
C(6)—C(5)—H(51)	107 (1)	C(16)—C(17)-H(171)	110 (1)
C(6)—C(5)—H(52)	111 (1)	C(16)—C(17)-H(172)	107 (1)
H(51)-C(5)—H(52)	108 (2)	C(18)—C(17)-H(171)	108 (1)
C(1)—C(6)—H(61)	108 (1)	C(18)—C(17)-H(172)	113 (1)
C(1)—C(6)—H(62)	106 (1)	H(171)-C(17)-H(172)	108 (2)
C(5)—C(6)—H(61)	109 (1)	C(17)—C(18)-H(181)	108 (1)
C(5)—C(6)—H(62)	108 (1)	C(17)—C(18)-H(182)	112 (1)
H(61)-C(6)—H(62)	115 (2)	C(19)—C(18)-H(181)	110 (1)
C(7)—C(8)—H(81)	109 (1)	C(19)—C(18)-H(182)	103 (1)
C(7)—C(8)—H(82)	110 (1)	H(181)-C(18)-H(182)	110 (2)
C(9)—C(8)—H(81)	108 (1)	C(20)—C(21)-H(211)	122 (1)
C(9)—C(8)—H(82)	108 (1)	C(22)—C(21)-H(211)	117 (1)
H(81)-C(8)—H(82)	106 (2)	C(21)—C(22)-H(221)	121 (1)
C(8)—C(9)—H(91)	111 (1)	C(23)—C(22)-H(221)	120 (1)
C(8)—C(9)—H(92)	109 (1)	C(23)—C(24)-H(241)	121 (1)
N(10)-C(9)—H(91)	109 (1)	C(25)—C(24)-H(241)	121 (1)
N(10)-C(9)—H(92)	107 (1)	C(20)—C(25)-H(251)	118 (1)
H(91)-C(9)—H(92)	110 (2)	C(24)—C(25)-H(251)	121 (1)
N(10)-C(11)-H(111)	109 (1)	C(19)—O(26)-H(162)	106 (1)

References

AHMED, F. R., HALL, S. R., PIPPY, M. E. & HUBER, C. P. (1966). *World list of Crystallographic Computer Programs*, 2nd ed., Appendix, p. 52. Utrecht: Oosthoek.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A27, 368–376.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

KOCH, M. H. J. (1974). *Mol. Pharmacol.* 10, 425–437.