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3-{2-[4-(4-Fluorobenzoyl)piperidino]-ethyl}-2-methyl-4H-pyrido[1,2-a]-pyrimidin-4-one (Pirenperone)†

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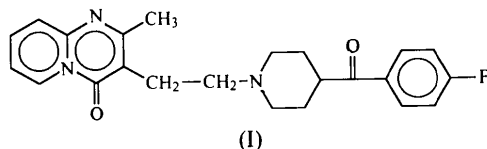
Abstract

Pirenperone, $C_{23}H_{24}FN_3O_2$, a serotonin-2 antagonist, has a nearly planar pyrido–pyrimidine moiety connected by an ethylpiperidyl bridge to an almost planar fluorobenzoyl moiety. The planar parts are perpendicular to each other [98.7 (2)°]. The packing of the molecules is governed only by van der Waals contacts.

Comment

Pirenperone, (I), which is chemically related to Ketanserin (Peeters, Blaton & De Ranter, 1982), has been reported to be a selective serotonin-2 (5-HT$_2$) antagonist and a specific D-LSD antagonist (Colpaert, Niemegeers & Janssen, 1982). The X-ray structural study of the title compound has been carried out as part of our studies on serotonin antagonists in an attempt to correlate structural features with the antagonistic activities (Blaton, Peeters & De Ranter, 1985).

(I)

The atoms in the phenyl ring are nearly coplanar: the largest deviation is 0.016 (7) Å for C7. The endocyclic angles reflect the *para*-disubstitution (Domenicano &

† Internal code of the Janssen Research Foundation: R47465.

Murray-Rust, 1979). The carbonyl group is slightly inclined with respect to the phenyl group, the torsion angle C4—C5—C8—O9 being 175.1 (7)°.

The piperidine ring has rotational disorder around the C10—N13 axis, as can be deduced from the large U_{33} values for atoms C11, C12, C13 and C15, and their displacement ellipsoids in Fig. 1. The appreciable shortening of the N—C bond lengths [1.43 (1) and 1.41 (1) Å compared to the value of 1.473 Å given by Allen *et al.* (1987)] is caused by the displacement of the atomic positions towards the rotation axis. This shift is a consequence of the approximation of a real kidney-like displacement of an ellipsoid. Because the motion is not known exactly, the effect has not been treated; this explains the relatively high R value of 0.0748.

The ethyl bridge between the piperidine ring and the pyrido–pyrimidine moiety has an antiperiplanar conformation. The geometry of the latter is in good agreement with those of similar structures (Hermecs, Vasvari-Debreczy & Simon, 1988). The pyrido–pyrimidine ring system deviates significantly from planarity with maximum deviations for C18 and C27 [0.058 (7) and 0.046 (7) Å, respectively]. The dihedral angles between the least-squares planes of the three-ring system [phenyl (A), piperidine (atoms C11, C12, C14 and C15) (B) and pyrido–pyrimidine (C)] are: $A{\wedge}B$ 59.2 (3), $A{\wedge}C$ 98.7 (2) and $B{\wedge}C$ 40.3 (2)°.

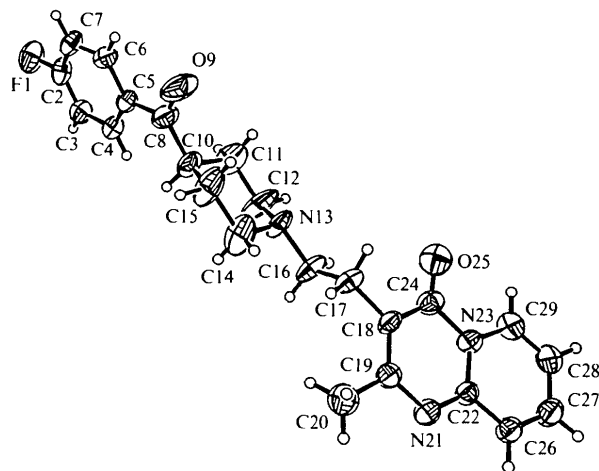The packing of the molecules is governed only by van der Waals contacts.

Fig. 1. *ORTEX* (McArdle, 1993) diagram of the molecule showing the atomic numbering scheme. Displacement ellipsoids are plotted at the 40% probability level.

Experimental

Crystal data

$C_{23}H_{24}FN_3O_2$	Cu $K\alpha$ radiation
$M_r = 393.45$	$\lambda = 1.54184$ Å

$C_{23}H_{24}FN_3O_2$

Monoclinic
$P2_1/c$
$a = 17.144 \ (1)$ Å
$b = 11.450 \ (3)$ Å
$c = 10.179 \ (2)$ Å
$\beta = 90.757 \ (8)°$
$V = 1997.9 \ (6)$ Å^3
$Z = 4$
$D_x = 1.308$ Mg m^{-3}
$D_m = 1.32$ Mg m^{-3}
D_m measured by flotation in
n-heptane/CCl$_4$

Cell parameters from 23
reflections
$\theta = 20-25°$
$\mu = 0.740$ mm^{-1}
$T = 293$ K
Prism
$0.50 \times 0.20 \times 0.10$ mm
Colourless
Crystal source: Janssen
Research Foundation,
Belgium

Data collection

Hilger & Watts four-circle
diffractometer
$\omega/2\theta$ scans (fixed speed 1.2°
min^{-1}, width 1.2°)
Absorption correction:
empirical (North, Phillips
& Mathews, 1968)
$T_{min} = 0.915$, $T_{max} =$
0.997
3491 measured reflections
3170 independent reflections

1167 observed reflections
$[F > 6.0\sigma(F)]$
$R_{int} = 0.0244$
$\theta_{max} = 70.00°$
$h = 0 \rightarrow 20$
$k = -13 \rightarrow 0$
$l = -12 \rightarrow 12$
3 standard reflections
monitored every 50
reflections
intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.075$
$wR(F^2) = 0.165$
$S = 1.44$
3170 reflections
264 parameters
H-atom parameters not
refined (calculated, riding)
$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2$
$+ 2.5198P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.026$

$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Extinction correction:
SHELXL93 (Sheldrick,
1993)
Extinction coefficient:
0.0014 (3)
Atomic scattering factors
from *International Tables
for X-ray Crsytallography*
(1974. Vol. IV, Tables
2.2B and 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
F1	0.9128 (2)	0.5792 (4)	0.5902 (4)	0.095 (2)
C2	0.8561 (4)	0.5108 (6)	0.5350 (7)	0.060 (3)
C3	0.7848 (4)	0.5081 (6)	0.5914 (7)	0.067 (3)
C4	0.7276 (4)	0.4390 (6)	0.5351 (6)	0.060 (3)
C5	0.7430 (3)	0.3752 (6)	0.4224 (6)	0.053 (2)
C6	0.8168 (3)	0.3828 (6)	0.3692 (6)	0.060 (3)
C7	0.8745 (4)	0.4487 (6)	0.4270 (7)	0.065 (3)
C8	0.6837 (4)	0.2991 (7)	0.3561 (7)	0.069 (3)
O9	0.7008 (3)	0.2527 (5)	0.2529 (6)	0.113 (3)
C10	0.6034 (4)	0.2822 (6)	0.4094 (7)	0.069 (3)
C11	0.5496 (4)	0.3727 (7)	0.3537 (9)	0.093 (4)
C12	0.4654 (4)	0.3543 (7)	0.396 (1)	0.116 (5)
N13	0.4385 (3)	0.2410 (5)	0.3587 (6)	0.069 (2)
C14	0.4848 (4)	0.1534 (7)	0.4190 (9)	0.103 (4)
C15	0.5703 (4)	0.1630 (7)	0.376 (1)	0.106 (4)
C16	0.3550 (4)	0.2253 (7)	0.3914 (8)	0.085 (3)
C17	0.3183 (3)	0.1273 (6)	0.3109 (7)	0.065 (3)
C18	0.2304 (3)	0.1227 (6)	0.3237 (6)	0.055 (2)
C19	0.1902 (4)	0.0515 (6)	0.4068 (6)	0.057 (2)
C20	0.2323 (4)	-0.0301 (6)	0.4993 (7)	0.082 (3)
N21	0.1116 (3)	0.0444 (4)	0.4124 (5)	0.056 (2)
C22	0.0711 (3)	0.1124 (5)	0.3326 (6)	0.048 (2)
N23	0.1050 (3)	0.1919 (4)	0.2487 (5)	0.050 (2)
C24	0.1885 (4)	0.2022 (6)	0.2409 (7)	0.057 (3)
O25	0.2147 (3)	0.2746 (4)	0.1654 (5)	0.079 (2)
C26	-0.0116 (4)	0.1055 (6)	0.3312 (6)	0.060 (3)
C27	-0.0550 (4)	0.1717 (6)	0.2487 (7)	0.068 (3)
C28	-0.0182 (4)	0.2521 (7)	0.1661 (7)	0.069 (3)
C29	0.0593 (4)	0.2608 (6)	0.1673 (7)	0.063 (3)

Table 2. *Selected geometric parameters* (Å, °)

F1—C2	1.362 (8)	C14—C15	1.54 (1)
C2—C3	1.36 (1)	C16—C17	1.52 (1)
C2—C7	1.35 (1)	C17—C18	1.516 (8)
C3—C4	1.379 (9)	C18—C19	1.367 (9)
C4—C5	1.388 (9)	C18—C24	1.428 (9)
C5—C6	1.385 (8)	C19—C20	1.50 (1)
C5—C8	1.494 (9)	C19—N21	1.352 (8)
C6—C7	1.371 (9)	N21—C22	1.316 (8)
C8—O9	1.22 (1)	C22—N23	1.381 (8)
C8—C10	1.50 (1)	C22—C26	1.421 (8)
C10—C11	1.49 (1)	N23—C24	1.441 (8)
C10—C15	1.51 (1)	N23—C29	1.379 (8)
C11—C12	1.53 (1)	C24—O25	1.219 (8)
C12—N13	1.43 (1)	C26—C27	1.35 (1)
N13—C14	1.41 (1)	C27—C28	1.40 (1)
N13—C16	1.486 (8)	C28—C29	1.33 (1)
F1—C2—C7	117.7 (6)	N13—C16—C17	111.2 (5)
F1—C2—C3	118.7 (6)	C16—C17—C18	112.6 (5)
C3—C2—C7	123.6 (6)	C17—C18—C24	114.8 (5)
C2—C3—C4	118.5 (6)	C17—C18—C19	125.7 (6)
C3—C4—C5	120.2 (6)	C19—C18—C24	119.4 (6)
C4—C5—C8	123.0 (6)	C18—C19—N21	124.9 (6)
C4—C5—C6	118.4 (6)	C18—C19—C20	121.1 (6)
C6—C5—C8	118.7 (6)	C20—C19—N21	114.0 (6)
C5—C6—C7	121.6 (6)	C19—N21—C22	117.2 (5)
C2—C7—C6	117.7 (6)	N21—C22—C26	119.4 (6)
C5—C8—C10	122.3 (6)	N21—C22—N23	123.3 (5)
C5—C8—O9	118.4 (6)	N23—C22—C26	117.2 (5)
O9—C8—C10	119.2 (6)	C22—N23—C29	120.6 (5)
C8—C10—C15	112.3 (6)	C22—N23—C24	120.9 (5)
C8—C10—C11	109.8 (6)	C24—N23—C29	118.4 (5)
C11—C10—C15	108.3 (6)	C18—C24—N23	114.1 (6)
C10—C11—C12	112.3 (6)	N23—C24—O25	117.7 (6)
C11—C12—N13	110.8 (6)	C18—C24—O25	128.3 (6)
C12—N13—C16	111.1 (6)	C22—C26—C27	121.2 (6)
C12—N13—C14	110.7 (6)	C26—C27—C28	119.6 (6)
C14—N13—C16	110.8 (6)	C27—C28—C29	120.0 (7)
N13—C14—C15	110.9 (7)	N23—C29—C28	121.4 (6)
C10—C15—C14	110.9 (6)		
C4—C5—C8—O9	175.1 (7)	C14—N13—C16—C17	-77.3 (7)
C5—C8—C10—C11	89.9 (8)	C16—N13—C14—C15	175.3 (6)
O9—C8—C10—C11	-87.5 (8)	N13—C16—C17—C18	-169.9 (5)
C12—N13—C16—C17	159.3 (6)	C16—C17—C18—C19	-96.9 (8)

Data collection: Hilger & Watts Y290 software. Cell refinement: *XRAY76 PARAM* (Stewart *et al.*, 1976). Data reduction: *REDU4* (Stoe & Cie, 1992). Program(s) used to solve structure: *MULTAN80* (Main *et al.*, 1980). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEX* (McArdle, 1993). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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(I)

The tetraethylammonium ion has S_4 symmetry and its shape is very regular. The symmetry, bond lengths and bond angles agree with those reported previously [for example, by Vicente, Knop, Linden, Cameron & Robertson (1988)]. Two of the O atoms in the perchlorate ion, O2 and O3, are disordered with refined site occupancies of 0.71 (1) and 0.74 (1), respectively. As a result, the tetrahedral shape of the ion is slightly distorted. The bond lengths and angles still have acceptable values. In the crystal lattice the ions are stacked in the direction of the b axis. The shortest Cl···N distance is 4.795 (3) Å and the shortest O···N distance is 4.033 (7) Å. There are no significantly short distances between the ions, hence the crystal lattice is held together by weak electrostatic forces.

Fig. 1. A view of the cation and anion showing the numbering scheme (O atom disorder and H atoms not shown). Displacement ellipsoids are plotted at the 50% probability level.

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Tetraethylammonium Perchlorate at 150 K

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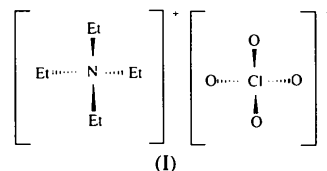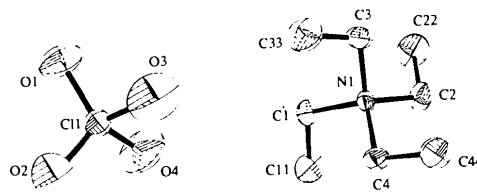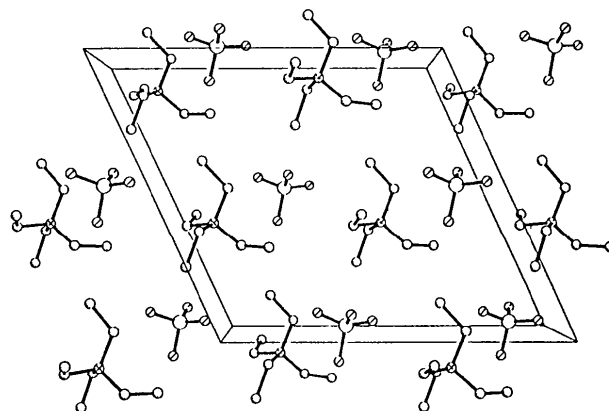
Abstract

Tetraethylammonium perchlorate, $C_8H_{20}N^+.ClO_4^-$, has been crystallized and its crystal structure is reported.

Comment

Both the tetraethylammonium cation and the perchlorate anion are widely used counterions. During the course of work aimed at understanding the structure of complexes formed between tetraalkylammonium ions and lipophilic cyclodextrins (Bates, Kataky & Parker, 1994a,b), we accidentally managed to co-crystallize tetraethylammonium with perchlorate to form the title compound, (I). Views of the ions and the unit-cell contents are shown in Figs. 1 and 2, respectively.

Fig. 2. The unit-cell contents viewed down **b**.

Experimental

Crystal data

$C_8H_{20}N^+.ClO_4^-$	Mo $K\alpha$ radiation
$M_r = 229.70$	$\lambda = 0.71073$ Å