

4'-Fluoro-4-[4-(2-pyridyl)-1-piperaziny]butyrophenone (Azaperone)

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(Received 2 February 1977; accepted 18 February 1977)

Abstract. $C_{19}H_{22}N_3OF$, $M_r = 327.39$, monoclinic, $C2/c$, $a = 24.127$ (3), $b = 5.789$ (2), $c = 25.125$ (3) Å, $\beta = 103.33^\circ$; $t = 25^\circ C$; $Z = 8$. The extended conformation of the butyrophenone chain corresponds to one of the expected energy minima [Koch, M. H. J., *Mol. Pharmacol.* (1974), **10**, 425–437] for this structural moiety.

* Chargé de Recherches du Fonds National de la Recherche Scientifique.

Table 1. *Experimental conditions*

Source Cu $K\alpha$; $\lambda = 1.5418$ Å
 ω - 2θ scan; $\theta_{max} = 55^\circ$
 Confidence level: 2.5
 Total number of independent reflexions: 2142
 Total observed: 1731

Table 2. *Final positional parameters ($\times 10^4$), with standard deviations in parentheses*

	<i>x</i>	<i>y</i>	<i>z</i>
F(1)	4702 (1)	5119 (4)	6708 (1)
C(2)	5005 (1)	5497 (6)	6324 (1)
C(3)	4906 (1)	7491 (6)	6040 (1)
C(4)	5227 (1)	7935 (5)	5646 (1)
C(5)	5619 (1)	6329 (5)	5555 (1)
C(6)	5697 (1)	4269 (5)	5853 (1)
C(7)	5392 (1)	3824 (6)	6251 (1)
C(8)	5931 (1)	6839 (5)	5129 (1)
O(9)	5909 (1)	8776 (4)	4929 (1)
C(10)	6295 (1)	5010 (5)	4956 (1)
C(11)	6570 (1)	5755 (5)	4493 (1)
C(12)	6856 (1)	3672 (5)	4294 (1)
N(13)	7133 (1)	4257 (3)	3857 (1)
C(14)	7648 (1)	5649 (5)	4037 (1)
C(15)	7898 (1)	6290 (5)	3562 (1)
N(16)	8032 (1)	4221 (3)	3282 (1)
C(17)	7526 (1)	2734 (5)	3106 (1)
C(18)	7286 (1)	2160 (5)	3600 (1)
C(19)	8400 (1)	4342 (5)	2934 (1)
C(20)	8759 (1)	6261 (5)	2933 (1)
C(21)	9141 (1)	6187 (5)	2600 (1)
C(22)	9155 (1)	4240 (6)	2273 (1)
C(23)	8770 (1)	2547 (6)	2273 (1)
N(24)	8397 (1)	2586 (4)	2605 (1)

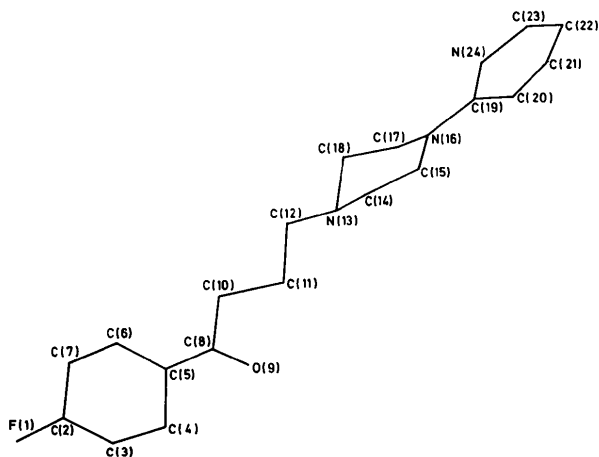
Introduction. Azaperone is a sedative neuroleptic. Transparent colourless crystals were obtained by slow evaporation of a solution in ethanol. The space group was determined from photographs. The final cell dimensions and intensities were measured on a Picker four-circle diffractometer with the experimental conditions given in Table 1.

Table 3. *Intramolecular bond distances (Å) and angles ($^\circ$), with standard deviations in parentheses*

F(1)—C(2)	1.355 (3)	F(1)—C(2)—C(3)	117.2 (3)
C(2)—C(3)	1.350 (4)	F(1)—C(2)—C(7)	118.2 (3)
C(2)—C(7)	1.387 (5)	C(3)—C(2)—C(7)	124.7 (3)
C(3)—C(4)	1.413 (4)	C(2)—C(3)—C(4)	117.9 (3)
C(4)—C(5)	1.382 (4)	C(3)—C(4)—C(5)	120.0 (3)
C(5)—C(6)	1.397 (4)	C(4)—C(5)—C(6)	119.8 (2)
C(5)—C(8)	1.473 (4)	C(4)—C(5)—C(8)	118.0 (2)
C(6)—C(7)	1.394 (4)	C(6)—C(5)—C(8)	122.2 (2)
C(8)—O(9)	1.225 (4)	C(5)—C(6)—C(7)	120.9 (3)
C(8)—C(10)	1.502 (4)	C(2)—C(7)—C(6)	116.7 (3)
C(10)—C(11)	1.529 (4)	C(5)—C(8)—O(9)	120.1 (3)
C(11)—C(12)	1.529 (4)	C(5)—C(8)—C(10)	119.9 (2)
C(12)—N(13)	1.452 (3)	O(9)—C(8)—C(10)	119.9 (3)
N(13)—C(14)	1.461 (3)	C(8)—C(10)—C(11)	114.0 (2)
N(13)—C(18)	1.461 (3)	C(10)—C(11)—C(12)	109.5 (2)
C(14)—C(15)	1.502 (4)	C(11)—C(12)—N(13)	112.7 (2)
C(15)—N(16)	1.463 (4)	C(12)—N(13)—C(14)	113.7 (2)
N(16)—C(17)	1.477 (3)	C(12)—N(13)—C(18)	110.3 (2)
N(16)—C(19)	1.383 (3)	C(14)—N(13)—C(18)	108.2 (2)
C(17)—C(18)	1.523 (4)	N(13)—C(14)—C(15)	111.0 (2)
C(19)—C(20)	1.409 (4)	C(14)—C(15)—N(16)	110.7 (2)
C(19)—N(24)	1.311 (3)	C(15)—N(16)—C(17)	111.2 (2)
C(20)—C(21)	1.382 (4)	C(15)—N(16)—C(19)	120.5 (2)
C(21)—C(22)	1.399 (5)	C(17)—N(16)—C(19)	116.6 (2)
C(22)—C(23)	1.350 (5)	N(16)—C(17)—C(18)	109.0 (2)
C(23)—N(24)	1.359 (4)	N(13)—C(18)—C(17)	111.1 (2)
		N(16)—C(19)—C(20)	121.7 (2)
		N(16)—C(19)—N(24)	116.7 (2)
		C(20)—C(19)—N(24)	121.5 (2)
		C(19)—C(20)—C(21)	118.3 (3)
		C(20)—C(21)—C(22)	119.3 (3)
		C(21)—C(22)—C(23)	118.4 (3)
		C(22)—C(23)—N(24)	122.7 (3)
		C(19)—N(24)—C(23)	119.5 (2)

Table 4. Torsion angles defining the conformation of azaperone

C(18)–N(13)–C(12)–C(11)	–167°
N(13)–C(12)–C(11)–C(10)	–179
C(12)–C(11)–C(10)–C(8)	–171
C(11)–C(10)–C(8)–C(5)	177
C(10)–C(8)–C(5)–C(4)	–171
N(24)–C(19)–N(16)–C(15)	–164

Fig. 1. Conformation and numbering scheme of $C_{19}H_{22}N_3OF$.

The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971) and refined by block-diagonal least squares (Ahmed, Hall, Pippy & Huber, 1966) to a final *R* of 0.09 for all observed reflexions.*

The scattering factors were those given in *International Tables for X-ray Crystallography* (1962).

The final coordinates 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 torsion angles defining the conformation of the molecule are given in Table 4.

Relevant structural details are given in the *Abstract*.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32507 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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.

Acta Cryst. (1977). B33, 1976–1979

Structural Studies of Precursor and Partially Oxidized Conducting Complexes. VII. A Neutron Diffraction Study of Disodium Tetracyanoplatinate(II) Trihydrate*

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(Received 7 December 1976; accepted 3 February 1977)

Abstract. $Na_2[Pt(CN)_4] \cdot 3H_2O$, triclinic $P\bar{1}(C_1)$, $a = 15.444$ (11), $b = 9.082$ (7), $c = 7.350$ (5) Å, $\alpha = 95.07$ (4), $\beta = 92.73$ (14), $\gamma = 89.04$ (2)°, $Z = 4$, $D_m =$

2.60, $D_c = 2.59$ g cm⁻³. The structure was determined with neutron diffraction data to locate all atoms. The 4067 unique reflections were refined to an agreement index of $R(F_o^2) = 0.067$. For the 3188 data with F_o^2 greater than one e.s.d., $R(F_o^2) = 0.062$. The four independent Pt–Pt spacings are 3.651 (4), 3.691 (4), 3.745 (4) and 3.754 (4) Å along the chain formed by the Pt atoms. The C–Pt–Pt–C torsion angles average 0°. This structure contains eclipsed C–N groups which

* Work performed under the auspices of the US Energy Research and Development Administration.

† Research participant sponsored by the Argonne Center for Educational Affairs, from Coe College, Cedar Rapids, Iowa.

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