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N-{1-[4-Fluorophenyl-4-oxobutyl]-4-phenyl-4-piperidinylmethyl}acetamide: Aceperone

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Abstract. $C_{24}H_{29}N_2O_2F$, monoclinic, P_2_1/n ; $a = 8.071$ (5), $b = 32.365$ (5), $c = 9.021$ (5) Å, $\beta = 113.26$ (5)°; $Z = 4$. The piperidine ring is in the usual chair form; the acetamidomethyl group is equatorial and the phenyl group axial.

Introduction. Aceperone is a neuroleptic which belongs to the class of butyrophenones.

The space group and lattice parameters were determined on a Nonius CAD-4 four-circle computer-controlled diffractometer. The instrumental settings are given in Table 1. The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971). The most probable *E* map contained the whole structure. Full-matrix least-squares refinement was performed with X-RAY 72 (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). H atoms were located on a difference

Table 1. Instrumental settings for the data collection

Source Cu $K\alpha$	$\Delta 2\theta = 0.5 + 0.3 \tan \theta$
$\lambda = 1.54178$ Å	$\theta_{\min} = 2^\circ; \theta_{\max} = 72^\circ$
Scan $\omega-2\theta$	Aperture $2.5 + 0.5 \tan \theta$
Graphite monochromator	$T_{\max} = 120$ s

Confidence level = 2.5σ , where $\sigma^2 = S + B + (0.035)^2$, S being the scan and B the background count
 Total number of independent reflexions 4484
 Total observed 2885

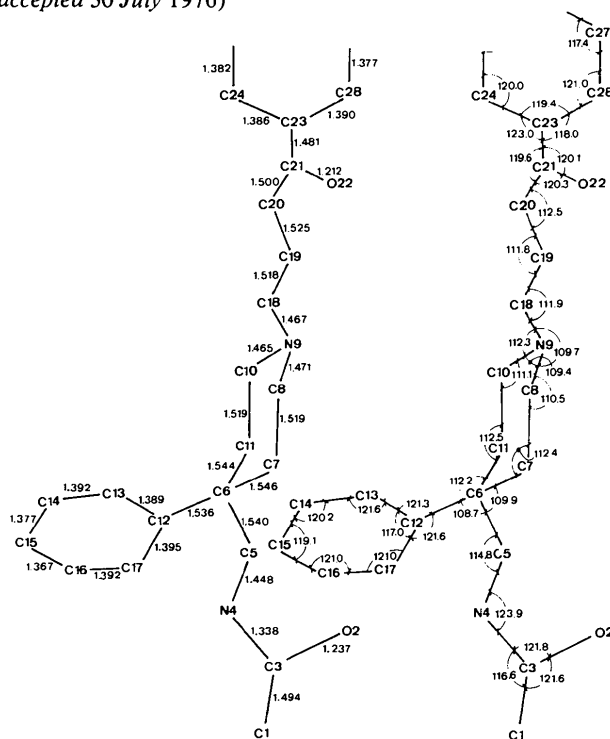


Fig. 1. Bond lengths (Å) and angles (°). The e.s.d.'s for distances and angles involving non-hydrogen atoms are in the ranges 0.003–0.006 Å and 0.3–0.6°.

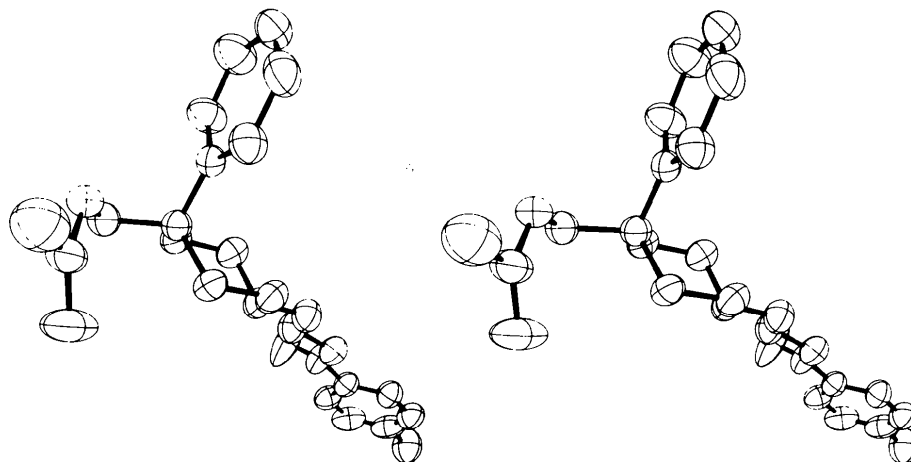


Fig. 2. Stereoscopic view of the molecule.

synthesis (CH₃ excepted). With anisotropic temperature factors for all non-H atoms and isotropic for H, the final *R* was 0.06. The scattering factors were those of Cromer & Mann (1968) except for H for which those of Stewart, Davidson & Simpson (1968) were used. The final coordinates and thermal parameters are listed in Table 2. Fig. 1 gives bond lengths and angles and the atomic numbering scheme. Table 3 gives bond lengths and angles for the H atoms.*

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32005 (46 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Discussion. Aceperone is structurally comparable to potent neuroleptics but its activity is very low. Therefore, it seemed interesting to determine the conformation as part of a structure-activity relation study. The piperidine ring is in the usual chair form but the acetamidomethyl group is equatorial and the phenyl group axial (Fig. 2), different from what is observed in related compounds. Moreover, the phenyl group is nearly perpendicular to the N(9)⋯C(6) direction (86°). The very low activity of this compound suggests that the presence of an aromatic group in equatorial position nearly perpendicular to the mean plane of the piperidine ring is a requirement for strong neuroleptic activity.

Table 2. Final coordinates ($\times 10^4$) and thermal parameters ($\times 10^4$) with standard deviations in parentheses

Thermal parameters are of the form: $T = \exp[-2\pi^2(U_{11}h^2a^{*2} + \dots + 2U_{12}hka^*b^* + \dots)]$ and $\exp[-2\pi^2U(2 \sin \theta/\lambda)^2]$.
H(11), H(12) and H(13) were not located.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(1)	6399 (7)	7977 (2)	1201 (6)	705	723	641	-82	230	-53
O(2)	7997 (5)	7442 (1)	3013 (3)	682	964	403	-161	11	1
C(3)	6862 (6)	7538 (1)	1665 (4)	436	809	375	-104	146	-27
N(4)	6040 (4)	7253 (1)	504 (4)	423	718	366	45	94	-20
C(5)	6581 (6)	6815 (1)	782 (5)	469	696	490	-72	209	20
C(6)	7768 (5)	6640 (1)	184 (5)	442	550	465	-51	177	37
C(7)	9643 (5)	6820 (1)	1208 (5)	426	621	507	-43	125	-9
C(8)	11137 (6)	6606 (1)	879 (5)	455	603	623	-49	161	59
N(9)	11194 (5)	6162 (1)	1253 (4)	477	619	647	27	217	80
C(10)	9463 (6)	5974 (1)	237 (6)	577	524	716	-27	215	66
C(11)	7917 (6)	6172 (1)	526 (5)	494	591	582	-64	174	102
C(12)	7147 (5)	6737 (1)	-1622 (5)	436	512	490	-18	206	14
C(13)	5982 (7)	6475 (2)	-2791 (5)	775	780	548	-201	204	-1
C(14)	5355 (8)	6570 (2)	-4427 (6)	844	857	515	-91	155	-99
C(15)	5865 (7)	6931 (2)	-4926 (5)	692	839	469	137	279	68
C(16)	7000 (7)	7196 (1)	-3791 (6)	755	636	678	120	414	170
C(17)	7630 (6)	7102 (1)	-2168 (5)	626	536	615	-28	286	46
C(18)	12728 (7)	5965 (2)	1049 (6)	634	734	726	58	299	87
C(19)	13007 (7)	5526 (2)	1660 (6)	613	661	785	48	309	28
C(20)	14848 (7)	5361 (2)	1871 (6)	567	665	769	24	232	26
C(21)	15155 (7)	4923 (2)	2456 (7)	558	666	1085	-11	339	42
O(22)	13898 (6)	4717 (1)	2450 (9)	795	791	2972	-31	865	332
C(23)	16986 (6)	4740 (1)	3033 (6)	578	549	734	-46	252	-39
C(24)	18362 (7)	4902 (2)	2657 (6)	629	707	786	18	306	-22
C(25)	20019 (7)	4710 (2)	3194 (7)	578	934	924	18	322	-128
C(26)	20252 (8)	4367 (2)	4137 (7)	549	968	910	222	-25	-251
C(27)	18966 (9)	4199 (2)	4572 (7)	902	663	876	40	52	-21
C(28)	17305 (8)	4387 (2)	3991 (7)	733	646	823	-88	212	-15
F(29)	21818 (5)	4186 (1)	4689 (6)	699	1207	1598	140	37	-189

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i>	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i>	
H(4)	516 (6)	735 (1)	-33 (5)	476	H(15)	545 (6)	698 (1)	-609 (5)	578
H(51)	682 (6)	674 (1)	202 (5)	547	H(16)	636 (6)	745 (1)	-413 (5)	561
H(52)	514 (6)	670 (1)	20 (5)	547	H(17)	840 (6)	728 (1)	-134 (5)	523
H(71)	996 (6)	678 (1)	241 (5)	547	H(181)	1256 (6)	597 (1)	24 (5)	600
H(72)	974 (6)	713 (1)	112 (5)	547	H(182)	1406 (6)	612 (1)	175 (5)	600
H(81)	1238 (6)	672 (1)	157 (5)	533	H(191)	1206 (6)	533 (1)	83 (5)	618
H(82)	1095 (6)	664 (1)	-35 (5)	533	H(192)	1301 (6)	550 (1)	272 (5)	618
H(101)	942 (6)	567 (1)	45 (5)	555	H(201)	1496 (6)	537 (1)	79 (5)	599
H(102)	926 (6)	599 (1)	92 (5)	555	H(202)	1593 (6)	555 (1)	265 (5)	599
H(111)	814 (6)	614 (1)	174 (5)	540	H(24)	1810 (6)	515 (1)	196 (5)	603
H(112)	671 (6)	602 (1)	11 (5)	540	H(25)	2094 (6)	484 (1)	300 (5)	659
H(13)	557 (6)	620 (1)	-241 (5)	562	H(27)	1914 (6)	394 (1)	529 (5)	728
H(14)	435 (6)	637 (1)	-527 (5)	643	H(28)	1623 (6)	428 (1)	416 (5)	644

Table 3. Bond distances (Å) and angles (°) involving H atoms

The e.s.d.'s are in the ranges 0.03–0.04 Å and 1–2°.

H(4)–N(4)	0.90	H(51)–C(5)	1.06	C(7)–C(8)–H(81)	111	N(9)–C(8)–H(82)	111
H(52)–C(5)	0.97	H(71)–C(7)	1.03	H(81)–C(8)–H(82)	109	N(9)–C(10)–H(101)	112
H(72)–C(7)	1.02	H(81)–C(8)	1.01	C(11)–C(10)–H(102)	113	H(101)–C(10)–H(102)	102
H(82)–C(8)	1.01	H(101)–C(10)	1.00	C(10)–C(11)–H(111)	109	H(111)–C(11)–H(112)	108
H(102)–C(10)	1.00	H(111)–C(11)	1.04	C(12)–C(13)–H(13)	116	C(14)–C(13)–H(13)	121
H(112)–C(11)	0.98	H(13)–C(13)	1.03	C(13)–C(14)–H(14)	121	C(15)–C(14)–H(14)	118
H(14)–C(14)	1.04	H(15)–C(15)	0.99	C(14)–C(15)–H(15)	119	C(16)–C(15)–H(15)	121
H(16)–C(16)	1.03	H(17)–C(17)	0.99	C(15)–C(16)–H(16)	121	C(17)–C(16)–H(16)	117
H(181)–C(18)	1.05	H(182)–C(18)	1.08	C(12)–C(17)–H(17)	116	C(16)–C(17)–H(17)	122
H(191)–C(19)	1.06	H(192)–C(19)	1.00	N(9)–C(18)–H(181)	110	C(19)–C(18)–H(181)	107
H(201)–C(20)	1.00	H(202)–C(20)	0.98	H(181)–C(18)–H(182)	109	C(18)–C(19)–H(191)	109
H(24)–C(24)	1.00	H(25)–C(25)	1.01	C(20)–C(19)–H(192)	103	H(191)–C(19)–H(192)	114
H(27)–C(27)	1.10	H(28)–C(28)	1.00	C(19)–C(20)–H(201)	108	C(21)–C(20)–H(202)	108
				H(201)–C(20)–H(202)	105	C(23)–C(24)–H(24)	119
C(3)–N(4)–H(4)	114	C(5)–N(4)–H(4)	121	C(25)–C(24)–H(24)	120	C(24)–C(25)–H(25)	119
N(4)–C(5)–H(51)	106	C(6)–C(6)–H(52)	111	C(26)–C(25)–H(25)	121	C(26)–C(27)–H(27)	123
H(51)–C(5)–H(52)	110	C(6)–C(7)–H(71)	110	C(28)–C(27)–H(27)	119	C(23)–C(28)–H(28)	115
C(8)–C(7)–H(72)	114	H(71)–C(7)–H(72)	101	C(27)–C(28)–H(28)	124		

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Hexadecacarbonylpentaosmium

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Abstract. Os₅(CO)₁₆, trigonal, *P*3₂1, *a* = *b* = 9.204 (2), *c* = 24.818 (4) Å, *U* = 1820.8 Å³, *Z* = 3, *D*_x = 3.83 g cm⁻³, μ(Mo *K*α) = 252.05 cm⁻¹. The structure was refined to an *R* of 0.033 for 809 unique diffractometer data. The Os(CO)₄ and four Os(CO)₃ groups form a distorted trigonal bipyramidal Os₅ cluster, with all carbonyls terminal. A crystallographic twofold axis bisects the Os(CO)₄ group, which is equatorial.

Introduction. Os₅(CO)₁₆, one of the few known pentanuclear carbonyls, was isolated from the pyrolysis products of Os₃(CO)₁₂ by Eady, Johnson & Lewis (1972, 1975). The crystal structure has been determined to confirm the predicted trigonal bipyramidal structure and locate the carbonyl groups; a preliminary communication has appeared (Eady, Johnson, Lewis, Reichert & Sheldrick, 1976).

Black crystals were grown by slow evaporation of the red–pink solution in toluene containing a little ethyl acetate. Intensities were determined with an automated Stoe STADI-2 two-circle diffractometer, graphite-monochromated Mo *K*α radiation, and a crystal in the form of a hexagonal prism 0.07 × 0.07 × 0.03 mm (layers 0–8*kl*). Empirical absorption corrections were applied, and interlayer scale factors obtained by a linear least-squares analysis of common reflexions. Equivalent reflexions were then averaged to yield 809 unique observed data. Unit-cell dimensions were obtained by a least-squares fit to diffractometer zero-layer ω angle measurements.

The Os atoms were located by multiresolution tangent refinement, and the remaining atoms by difference syntheses. The structure was refined by full-matrix least squares with anisotropic Os and isotropic light atoms, complex neutral-atom scattering factors and the