flections were used to scale the sets of intensity data from the two crystals. To compensate for the decrease in the intensities of the standard reflections during data collection, the intensities and their standard deviations were scaled by a least-squares procedure similar to that described by Ibers (1969). A trial structure, including all 29 heavy atoms, was obtained by direct methods with the computer program *MULTAN* (Germain, Main & Woolfson, 1971). The trial structure was refined by full-matrix least-squares methods; the quantity minimized was $\sum w(F_o^2-F_c^2/k^2)^2$, with k as a scale factor and the weight w equal to $1/\sigma^2(F_o^2)$. All hydrogen atoms were located in difference Fourier maps that were calculated during the later stages of refinement. In addition to the positional parameters, anisotropic temperature parameters for heavy atoms, isotropic temperature factors for hydrogen atoms, and an isotropic extinction coefficient were refined. All of the 3298 reflections were considered observable and were included in the refinement with their calculated weights. The final R_1 index $[\sum||F_o|-|F_c||/\sum|F_o|]$ for all reflections is 0·22, the R_2 index $[\sum|F_o^2-F_c^2|/\sum F_o^2]$ is 0·11, and goodness-of-fit is 1·10. If only those 1904 reflections with $I>\sigma(I)$ are considered, $R_1=0{\cdot}13$, $R_2=0{\cdot}09$ and goodness-of-fit = 1·42. A final difference Fourier map showed no peaks or troughs with magnitudes exceeding 0·8 e Å^{-3}.

Results. Table 1 gives the heavy-atom parameters and Table 2 lists the hydrogen atom parameters. A table of observed and calculated structure factors is available.* The tertiary hydroxyl group at C(2) is in the axial position, and an acetyl group is at the C(4) position. Fig. 1 depicts the molecular conformation, thermal ellipsoids and bond lengths between heavy atoms. Bond angles involving only non-hydrogen atoms are given in Table 3. The C–H bond lengths have an average value of 0·93 Å and range from 0·75 to 1·10 Å. The two O–H bond lengths are 0·85 and 0·83 Å.

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* This table has been deposited with the National Lending Library, England as Supplementary Publication No. SUP 30073 (16 pp.). Copies may be obtained from the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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4-(4-Chloro-α,α,α-trifluoro-*m*-tolyl)-1-[4, 4-bis-(*p*-fluorophenyl)butyl]-4-piperidinol (Penfluridol)

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Abstract. Orthorhombic, $P2_12_12_1$, $a=16{\cdot}141$ (9), $b=9{\cdot}559$ (5), $c=16{\cdot}794$ (10) Å, 25 °C, $C_{28}H_{27}NOF_5Cl$, $M=523{\cdot}96$, $Z=4$, $D_x=1{\cdot}337$ g cm^{-3}.

Introduction. Penfluridol is the longest-acting neuroleptic known today. Transparent needle-like crystals were obtained by slow evaporation from a (90:10) mixture of n-hexane and isopropyl alcohol.

Experimental. Lattice parameters were obtained by least-squares refinement of the setting angles of twelve reflexions. Weissenberg photographs showed absences characteristic of the space group $P2_12_12_1$.

Intensity data were collected on a Picker four-circle card-controlled diffractometer. The relevant data are given in Table 1.

Table 1. *Experimental data*

Crystal dimensions: 0·30 × 0·30 × 0·25 mm
Source Cu $K\bar{\alpha}$; $\lambda=1{\cdot}5418$ Å; Ni filter; ω–2θ scan; $\Delta 2\theta=\pm 1°$; $\theta_{max}=57{\cdot}5°$
Confidence level: 2·0
Total number of independent reflexions: 2039
Total observed: 1617

The quality of the data was rather poor as the crystal was highly mosaic and decomposed under irradiation. A gradual loss of intensity which reached 10%

after four days of exposure was observed on the standard reflexions. The data were corrected for this effect.

The structure (Fig. 1) was solved by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971). Block-diagonal least-squares refinement using the programs written by Ahmed, Hall, Pippy & Huber (1966) first with isotropic and later anisotropic thermal parameters resulted in $R=\sum||F_o|-|F_c||/\sum|F_o|=0{\cdot}12$ for all observed reflexions. The atomic scattering factors

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

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

	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}
Cl(1)	446 (1)	4583 (3)	7378 (2)	46 (1)	355 (5)	118 (1)	−26 (5)	−24 (2)	−6 (4)
O(1)	4328 (2)	2988 (4)	8121 (2)	56 (2)	100 (4)	45 (1)	43 (4)	−10 (3)	6 (5)
N(1)	5810 (3)	5630 (5)	7940 (3)	38 (2)	130 (6)	51 (2)	2 (6)	4 (3)	−6 (6)
F(1)	1120 (3)	7024 (5)	6422 (3)	95 (3)	273 (8)	102 (3)	7 (9)	−55 (5)	139 (9)
F(2)	1435 (4)	5166 (6)	5657 (3)	146 (4)	278 (8)	78 (2)	−21 (8)	−123 (5)	87 (10)
F(3)	2396 (3)	6832 (5)	5893 (3)	109 (3)	260 (7)	83 (2)	100 (8)	−24 (5)	36 (9)
F(4)	6397 (3)	6010 (9)	12299 (3)	78 (2)	648 (17)	71 (2)	17 (11)	41 (4)	79 (11)
F(5)	11623 (3)	5065 (8)	10065 (3)	61 (2)	695 (17)	93 (3)	−19 (13)	−25 (4)	130 (11)
C(1)	1517 (4)	4647 (8)	7467 (4)	49 (3)	193 (10)	59 (3)	42 (10)	−22 (5)	9 (9)
C(2)	2025 (4)	5299 (6)	6963 (4)	60 (3)	133 (7)	50 (3)	−4 (8)	−17 (5)	37 (8)
C(3)	2883 (3)	5268 (5)	7088 (4)	52 (2)	89 (6)	57 (3)	9 (8)	−27 (4)	26 (7)
C(4)	3222 (3)	4516 (6)	7677 (3)	48 (2)	112 (6)	40 (2)	−13 (7)	0 (4)	14 (7)
C(5)	2700 (4)	3899 (7)	8257 (4)	53 (3)	180 (9)	58 (3)	13 (10)	12 (5)	−58 (9)
C(6)	1841 (4)	3918 (9)	8096 (6)	64 (4)	200 (12)	89 (5)	−7 (14)	12 (8)	−39 (11)
C(7)	1737 (5)	6102 (8)	6211 (5)	93 (5)	179 (10)	93 (4)	−81 (11)	−100 (7)	112 (12)
C(8)	4160 (3)	4377 (5)	7829 (3)	44 (2)	102 (6)	38 (2)	−2 (6)	6 (4)	11 (7)
C(9)	4681 (3)	4724 (6)	7110 (3)	44 (2)	140 (7)	40 (2)	−36 (7)	−3 (4)	2 (7)
C(10)	5603 (3)	4627 (5)	7326 (3)	49 (2)	124 (6)	43 (2)	−45 (7)	0 (4)	0 (8)
C(11)	5343 (3)	5294 (7)	8683 (3)	41 (2)	163 (8)	47 (2)	−38 (8)	−6 (4)	−13 (7)
C(12)	4400 (3)	5380 (6)	8527 (3)	42 (2)	124 (6)	40 (2)	−16 (7)	1 (4)	−8 (8)
C(13)	6725 (3)	5553 (7)	8124 (4)	41 (2)	138 (8)	67 (3)	−8 (9)	−28 (5)	−2 (8)
C(14)	7018 (4)	6635 (7)	8695 (4)	46 (3)	164 (9)	73 (3)	55 (10)	−50 (5)	−29 (8)
C(15)	7944 (4)	6600 (8)	8863 (4)	63 (3)	214 (11)	55 (3)	34 (10)	−40 (5)	−35 (10)
C(16)	8191 (4)	5516 (9)	9460 (4)	54 (3)	252 (13)	55 (2)	45 (11)	1 (5)	6 (12)
C(17)	7733 (3)	5680 (7)	10268 (3)	45 (2)	177 (9)	49 (3)	20 (9)	−35 (4)	8 (9)
C(18)	7855 (4)	6824 (8)	10741 (4)	53 (3)	222 (11)	54 (3)	29 (10)	−35 (5)	5 (10)
C(19)	7402 (4)	6901 (10)	11412 (5)	50 (3)	272 (15)	93 (5)	−84 (16)	−42 (6)	−19 (12)
C(20)	6857 (4)	5909 (11)	11611 (4)	52 (3)	401 (19)	44 (3)	−46 (13)	−3 (5)	106 (14)
C(21)	6665 (5)	4791 (9)	11140 (5)	63 (4)	212 (12)	94 (5)	13 (14)	12 (7)	−13 (12)
C(22)	7141 (4)	4698 (7)	10452 (4)	69 (3)	132 (8)	79 (3)	14 (10)	−27 (6)	13 (10)
C(23)	9133 (4)	5473 (8)	9598 (3)	52 (3)	232 (11)	52 (2)	−23 (10)	10 (4)	−2 (11)
C(24)	9677 (4)	6533 (10)	9403 (4)	57 (3)	292 (15)	68 (3)	81 (13)	25 (5)	41 (12)
C(25)	10522 (5)	6467 (12)	9550 (5)	63 (4)	361 (20)	80 (4)	14 (17)	−3 (7)	−20 (17)
C(26)	10792 (4)	5244 (12)	9920 (5)	45 (3)	478 (20)	55 (4)	−23 (16)	−10 (6)	17 (15)
C(27)	10266 (5)	4177 (9)	10107 (4)	104 (5)	284 (14)	57 (3)	−80 (12)	−52 (7)	120 (15)
C(28)	9421 (4)	4243 (8)	9975 (4)	69 (3)	231 (11)	57 (3)	−32 (11)	−37 (6)	66 (11)

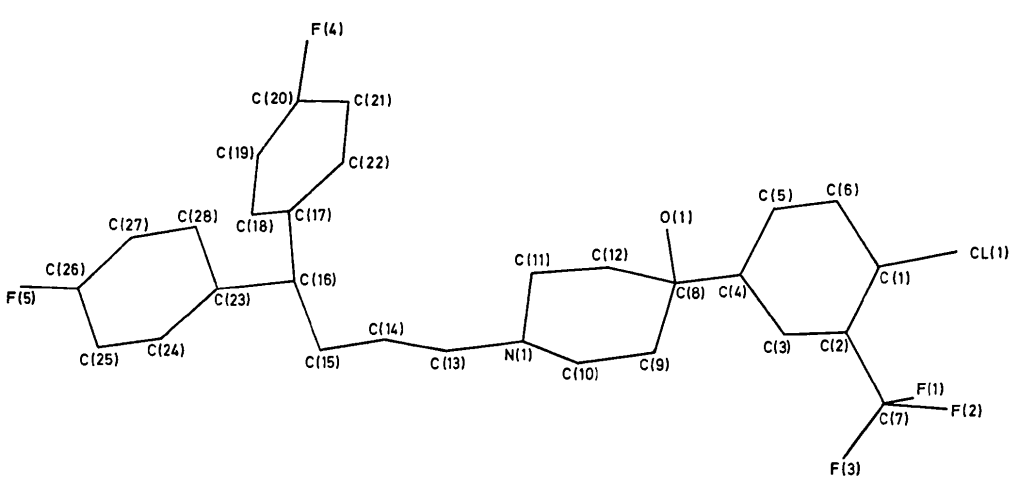

Fig. 1. Conformation and atom numbering scheme of $C_{28}H_{27}NOF_5Cl$.

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

Cl(1)–C(1)	1·74 (1) Å	C(9)–C(10)	1·53 (1) Å
O(1)–C(8)	1·44 (1)	C(11)–C(12)	1·55 (1)
N(1)–C(10)	1·45 (1)	C(13)–C(14)	1·49 (2)
N(1)–C(11)	1·49 (1)	C(14)–C(15)	1·52 (1)
N(1)–C(13)	1·51 (1)	C(15)–C(16)	1·49 (2)
F(1) –C(7)	1·38 (2)	C(16)–C(17)	1·55 (1)
F(2) –C(7)	1·38 (2)	C(16)–C(23)	1·54 (1)
F(3) –C(7)	1·38 (2)	C(17)–C(18)	1·37 (2)
F(4) –C(20)	1·38 (1)	C(17)–C(22)	1·37 (2)
F(5) –C(26)	1·37 (1)	C(18)–C(19)	1·35 (2)
C(1)–C(2)	1·33 (1)	C(19)–C(20)	1·33 (2)
C(1)–C(6)	1·37 (2)	C(20)–C(21)	1·36 (2)
C(2)–C(3)	1·40 (1)	C(21)–C(22)	1·39 (2)
C(2)–C(7)	1·55 (2)	C(23)–C(24)	1·38 (2)
C(3)–C(4)	1·34 (1)	C(23)–C(28)	1·41 (2)
C(4)–C(5)	1·42 (1)	C(24)–C(25)	1·39 (2)
C(4)–C(8)	1·54 (1)	C(25)–C(26)	1·39 (3)
C(5)–C(6)	1·41 (2)	C(26)–C(27)	1·36 (2)
C(8)–C(9)	1·51 (1)	C(27)–C(28)	1·38 (2)
C(8)–C(12)	1·56 (1)		

C(10)–N(1)–C(11)	110°	N(1)–C(11)–C(12)	110°
C(10)–N(1)–C(13)	110	C(8)–C(12)–C(11)	110
C(11)–N(1)–C(13)	108	N(1)–C(13)–C(14)	114
Cl(1)–C(1)–C(2)	125	C(13)–C(14)–C(15)	115
Cl(1)–C(1)–C(6)	115	C(14)–C(15)–C(16)	114
C(2)–C(1)–C(6)	110	C(15)–C(16)–C(17)	113
C(1)–C(2)–C(3)	120	C(15)–C(16)–C(23)	112
C(1)–C(2)–C(7)	124	C(17)–C(16)–C(23)	110
C(3)–C(2)–C(7)	115	C(16)–C(17)–C(18)	121
C(2)–C(3)–C(4)	122	C(16)–C(17)–C(22)	117
C(3)–C(4)–C(5)	119	C(18)–C(17)–C(22)	121
C(3)–C(4)–C(8)	125	C(17)–C(18)–C(19)	117
C(5)–C(4)–C(8)	116	C(18)–C(19)–C(20)	122
C(4)–C(5)–C(6)	116	F(4)–C(20)–C(19)	121
C(1)–C(6)–C(5)	122	F(4)–C(20)–C(21)	115
F(1)–C(7)–F(2)	109	C(19)–C(20)–C(21)	124
F(1)–C(7)–F(3)	109	C(20)–C(21)–C(22)	114
F(1)–C(7)–C(2)	109	C(17)–C(22)–C(21)	122
F(2)–C(7)–F(3)	110	C(16)–C(23)–C(24)	125
F(2)–C(7)–C(2)	109	C(16)–C(23)–C(28)	114
F(3)–C(7)–C(2)	109	C(24)–C(23)–C(28)	120
O(1)–C(8)–C(4)	109	C(23)–C(24)–C(25)	123
O(1)–C(8)–C(9)	112	C(24)–C(25)–C(26)	115
O(1)–C(8)–C(12)	105	F(5)–C(26)–C(25)	119
C(4)–C(8)–C(9)	113	F(5)–C(26)–C(27)	118
C(4)–C(8)–C(12)	108	C(25)–C(26)–C(27)	122
C(9)–C(8)–C(12)	109	C(26)–C(27)–C(28)	123
C(8)–C(9)–C(10)	110	C(23)–C(28)–C(27)	116
N(1)–C(10)–C(9)	111		

$\sigma = 1°$

used are those given in *International Tables for X-ray Crystallography* (1962).

Results and discussion. The final atomic parameters are listed in Table 2 and the bond distances and angles in Table 3.* The conformation of the molecule is the same as found previously in 4′-fluoro-4-{1-[4-hydroxy-4-(4′-fluoro)-phenylpiperidino]}-butyrophenone (Koch & Germain, 1972) as shown by the comparison of the corresponding torsional angles given in Table 4.

Table 4. *Torsional angles in 4′-fluoro-4-{1-[4-Hydroxy-4-(4′-fluoro)phenylpiperidino]} butyrophenone (A) and Penfluridol (B)*

	A	*B*
C(3)–C(4)–C(8)–C(9)	−28°	−20°
C(8)–C(9)–C(10)–N(1)	−62	−62
C(9)–C(10)–N(1)–C(13)	−176	−178
C(10)–N(1)–C(13)–C(14)	−179	174
N(1)–C(13)–C(14)–C(15)	−166	−178
C(13)–C(14)–C(15)–C(16)	−68	−80
C(14)–C(15)–C(16)–C(23)	−177	178

Each molecule is hydrogen-bonded to two neighbours:

$O(1)\cdots N(1)$: 2·88 Å [N(1): $1-x, -\frac{1}{2}+y, \frac{3}{2}-z$]
$N(1)\cdots O(1)$: 2·88 Å [O(1): $1-x, \frac{1}{2}+y, \frac{3}{2}-z$].

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* The structure factors have been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30087. Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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