

Hydrogen bonding

Bond distances in Table 3 show that, in terms of the bond-length bond-strength curve for U(VI) (Zachariasen & Plettinger, 1959), some oxygen atoms exhibit valences below the value 2 v.u. (valence units).

Observed bond distances give bond-strength sums for U(1) and U(2) of 5.91 and 6.04 v.u. respectively, indicating that bond distances have been determined with reasonable accuracy. Bond strengths for O(3) [bonded to U(1), U(2), U(2')] and O(4') [bonded to U(1), U(2)] are 2.1 v.u. and 2.05 v.u. respectively. However, bond strengths for O(1) and O(5) fall slightly below 2 v.u., while the bond-strength for O(2) is the very low value: 1.26 v.u. This is taken as an indication that a hydrogen atom is strongly associated with O(2).

The IR spectrum of $\text{H}_2\text{U}_3\text{O}_{10}$ exhibits a single O-H stretching absorption at 3460 cm^{-1} (Hoekstra *et al.*, 1972). Application of a relationship between IR stretching frequencies and the hydrogen bond distances in crystals (Bellamy & Owen, 1969) leads to a predicted O-H...O bond length of 2.83 Å. If hydrogen bonding occurs to both O(1) and O(5'), our data give two bonding distances for each hydrogen atom in $\text{H}_2\text{U}_3\text{O}_{10}$: O(2)-H...O(1)=2.88 Å, and O(2)-H...O(5')=2.91 Å. Within error estimates, these bond lengths are equal and in accord with predictions by Bellamy & Owen. Although it is reasonable, based on bond-strength considerations and IR data, to place the hydrogen atoms as indicated (and at corresponding centrosymmetric positions as shown in Fig. 1), a more precise picture of the bonding must be derived from a neutron-diffraction study.

As indicated above, the IR spectrum of $\text{H}_2\text{U}_3\text{O}_{10}$ is similar to that of $\text{CuU}_3\text{O}_{10}$ (Urbanec, 1966; Hoekstra

& Marshall, 1967). The symmetry of copper triuranate is monoclinic with $a=7.57$, $b=6.47$, $c=16.68$ Å, and $\beta=91.07^\circ$. Preliminary structural results indicate that the stacking of uranium atoms along c is similar to that found for $\text{H}_2\text{U}_3\text{O}_{10}$ along the c^* direction, with Cu atoms probably located at the hole defined by the octahedral oxygen array in the hydrate. A pseudo-repeat distance of 16.88 Å ($4d_{001}$) in $\text{H}_2\text{U}_3\text{O}_{10}$ gives a pseudo-cell of $a=6.87$, $b=7.42$, $c'=16.88$ Å, with α' , β' and γ' differing only by a few degrees from 90° , thus completing the similarity to $\text{CuU}_3\text{O}_{10}$.

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The Crystal and Molecular Structure of 4'-Fluoro-4-{1-[4-hydroxy-4-(4'-fluoro)-phenylpiperidino]}butyrophenone and its Hydrochloride

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The structures of these compounds were determined by X-ray diffraction. The base crystallizes in the space group $P2_1/c$ with dimensions $a=7.855$, $b=8.924$, $c=28.060$ Å and $\beta=105.45^\circ$ with $Z=4$. The hydrochloride crystallizes in the space group $P2_1/a$ with $a=16.611$, $b=7.056$, $c=17.458$ Å, $\beta=102.42^\circ$ and $Z=4$. Both structures were refined by block-diagonal least squares. Final R indexes of 0.10 and 0.07 respectively were obtained.

This butyrophenone derivative is related to a class of potent neuroleptics. Therefore it seemed interesting to determine the conformation of this compound in various environments as part of a structure-activity correlation study.

Experimental

Both compounds were recrystallized from hot isopropanol to form colourless prisms. Intensity data were collected on a Picker four-circle automatic diffrac-

tometer. Crystallographic and experimental data are listed in Table 1. To determine the cell parameters a

Table 1. *Crystallographic and experimental data*

Hydrochloride
C₂₁H₂₃O₂NF₂·HCl F.W. 395.9

Monoclinic *P*2₁/*a* *a* = 16.611 (2) Å
b = 7.056 (1)
c = 17.458 (2)
β = 102.42 (1)°

Density, calculated (*Z* = 4) 1.312 g.cm⁻³
measured 1.319 g.cm⁻³
V = 1998.3 Å³
F(000) = 832

Crystal dimensions 0.30 × 0.31 × 0.30 mm
Source Mo *Kα*; Zr filter; *λ* = 0.7114 Å [average of *λ*(*α*₁) and *λ*(*α*₂)]; *ω*-2*θ* scan; *Δ*2*θ* = ± 0.7°; *θ*_{max} = 22.5°

Confidence level: 2.5;
total number of independent reflexions: 1860
total observed: 1467.

Base
C₂₁H₂₃O₂NF₂ F.W. 359.4

Monoclinic *P*2₁/*c* *a* = 7.855 (2) Å
b = 8.924 (2)
c = 28.060 (3)
β = 105.45 (1)°

Density, calculated (*Z* = 4) 1.264 g.cm⁻³
measured 1.266 g.cm⁻³
V = 1895.9
F(000) = 760

Crystal dimensions: 0.30 × 0.20 × 0.25 mm
Source Mo *Kα*; Zr filter; *λ* = 0.7114 Å [average of *λ*(*α*₁) and *λ*(*α*₂)]; *ω*-2*θ* scan; *Δ*2*θ* = ± 0.7°; *θ*_{max} = 22.5°

Confidence level: 2.5;
total number of independent reflexions: 2372;
total observed: 1229.

few reflexions were centred through a narrow vertical slit at a take-off angle of 0.7° using either *Kα*₁ or *Kβ* radiation. The standard deviations which are given are thus only a measure of precision and not necessarily of accuracy.

To check the electronic and crystal stability during the data collection the intensity of a standard reflexion was measured every 35th reflexion. No significant shift in these standards was observed.

The Lorentz and polarization factors were calculated for a *θ*-value corresponding to the middle of the scan, hence the use of the simple mean of *λ*(*α*₁) and *λ*(*α*₂). The scale and overall temperature factor computed by Wilson's (1942) method were used to derive normalized structure factors. The values of the statistical averages of |*E*| and |*E*² - 1| were 0.746 and 0.925 respectively for the hydrochloride and 0.711 and 1.084 for the base.

Table 2. *Origin and symbols*

(a) Origin and symbols for the structure of the hydrochloride.

<i>h</i>	<i>k</i>	<i>l</i>	<i>s</i>	<i>E</i>	--
6	5	1	+	3.94	
$\bar{3}$	1	4	+	2.93	
13	0	2	+	3.32	
0	2	0	<i>A</i>	2.56	
13	2	2	<i>B</i>	2.88	
$\bar{3}$	0	4	<i>C</i>	2.73	
5	1	5	<i>D</i>	2.65	
$\bar{3}$	2	4	<i>E</i>	2.23	
$\bar{1}$	4	4	<i>F</i>	2.58	

(b) Origin and symbols for the structure of the base.

<i>h</i>	<i>k</i>	<i>l</i>	<i>s</i>	<i>E</i>
1	1	3	+	5.03
$\bar{3}$	5	10	+	3.54
5	4	11	+	3.83
$\bar{3}$	1	11	<i>A</i>	4.42
1	1	23	<i>B</i>	3.61

Table 3. *Figures of merit*

$$m(1) = \sum_{hh'} s(h)s(h')s(h-h')p_{hh'} \quad (p_{hh'} \text{ is the probability that the product of signs is positive})$$

$$m(2) = \sum_{hh'} s(h)s(h')s(h-h') |E_h E_{h'} E_{h-h'}|$$

$$m(3) = \text{number of positive relationships}$$

$$m(4) = \text{number of signs determined}$$

(a) Figures of merit for the sets of signs corresponding to the structure of the hydrochloride.

Group	Signs of symbols						<i>m</i> (1)	<i>m</i> (2)	<i>m</i> (3)	<i>m</i> (4)
	<i>A</i>	<i>B</i>	<i>C</i>	<i>D</i>	<i>E</i>	<i>F</i>				
1	-	-	-	-	+	-	1374	1593	1661	328
2	+	+	-	-	-	-	1174	1377	1458	324
3	-	-	+	+	-	+	1190	1393	1575	382
4	-	-	-	-	-	-	1374	1593	1661	328
5	-	-	-	+	+	-	1105	1298	1486	349
6	-	-	-	+	+	+	1037	1221	1474	367
7	-	+	-	-	+	-	1456	1673	1771	382

(b) Figures of merit for the sets of signs corresponding to the structure of the base.

Group	Signs of symbols		<i>m</i> (1)	<i>m</i> (2)	<i>m</i> (3)	<i>m</i> (4)
	<i>A</i>	<i>B</i>				
1	+	+	1633	2665	1854	435
2	-	+	1538	2542	1790	429
3	+	-	1564	2581	1805	430
4	-	-	1649	2690	1863	435

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

Structure determination

The structures were solved automatically with the symbolic addition method program for centrosymmetric crystals written by Germain, Main & Woolfson (1969).

Structure of the hydrochloride

The reflexions which correspond to the highest E -values were used in the process. The origin and symbols given in Table 2(a) were chosen by the program. Seven sets of signs were generated, each being characterized by four figures of merit which are listed in Table 3(a). An E -map computed with the signs of the most reliable set (set 7) showed the complete structure.

Table 4. *Final coordinates and thermal parameters and their standard deviations ($\times 10^4$)*

$$B = \exp(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{13}hl + B_{23}kl) \times 10^{-4}$$

(a) Structure of the hydrochloride

	x	y	z	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}
O(1)	4421 (2)	9854 (4)	7255 (1)	69 (2)	301 (9)	52 (1)	69 (6)	- 4 (2)	- 113 (6)
O(2)	2286 (2)	4090 (4)	2734 (1)	64 (1)	114 (5)	44 (1)	13 (4)	- 3 (2)	14 (5)
N	3589 (1)	2165 (4)	4433 (1)	25 (1)	166 (7)	33 (1)	11 (5)	13 (2)	6 (5)
C(1')	4563 (2)	8183 (6)	2010 (2)	33 (1)	247 (11)	38 (2)	0 (7)	17 (2)	-16 (7)
C(2')	4356 (2)	9541 (7)	1406 (2)	47 (2)	343 (13)	42 (2)	32 (8)	5 (3)	-12 (8)
C(3')	3742 (3)	9178 (8)	752 (3)	65 (3)	430 (16)	45 (2)	16 (10)	11 (4)	18 (11)
C(4')	3350 (3)	7461 (8)	718 (2)	45 (2)	527 (18)	39 (2)	-57 (9)	-10 (3)	-44 (10)
C(5')	3525 (3)	6109 (8)	1268 (3)	72 (2)	387 (15)	51 (2)	3 (10)	1 (4)	- 104 (11)
C(6')	4143 (2)	6453 (7)	1930 (2)	53 (2)	360 (14)	44 (2)	-17 (8)	- 6 (3)	-77 (9)
C(1)	4116 (2)	3022 (5)	5161 (2)	33 (2)	180 (9)	31 (1)	25 (6)	0 (2)	-12 (6)
C(2)	4034 (2)	1885 (5)	5891 (2)	46 (2)	207 (10)	30 (1)	10 (6)	7 (3)	-22 (7)
C(3)	4640 (2)	2693 (5)	6600 (2)	41 (2)	216 (10)	25 (1)	41 (6)	- 3 (2)	7 (7)
C(4)	200 (2)	6366 (5)	2720 (2)	37 (1)	199 (9)	42 (1)	10 (7)	20 (3)	36 (7)
C(2'')	2691 (2)	2661 (6)	4363 (2)	26 (2)	234 (11)	39 (2)	-25 (7)	14 (3)	26 (7)
C(3'')	2175 (2)	1587 (5)	3672 (2)	33 (2)	214 (10)	29 (1)	1 (6)	10 (3)	- 4 (7)
C(4'')	2428 (2)	2105 (5)	2884 (2)	40 (2)	138 (9)	39 (2)	- 1 (6)	12 (3)	7 (6)
C(5'')	3367 (2)	1822 (5)	2996 (2)	34 (2)	209 (10)	35 (2)	11 (7)	23 (3)	11 (7)
C(6'')	3855 (2)	2839 (6)	3701 (2)	40 (2)	223 (10)	28 (1)	18 (6)	19 (3)	-18 (7)
C(1''')	1956 (2)	933 (5)	2225 (2)	40 (2)	170 (9)	29 (1)	15 (6)	7 (3)	11 (7)
C(2''')	1387 (2)	1730 (6)	1594 (2)	59 (2)	245 (11)	45 (2)	6 (8)	2 (3)	10 (8)
C(3''')	959 (3)	642 (7)	985 (2)	65 (2)	262 (12)	48 (2)	-30 (9)	- 3 (3)	9 (9)
C(4''')	1092 (2)	8723 (6)	1013 (2)	55 (2)	266 (11)	31 (1)	-54 (7)	2 (3)	-42 (9)
C(5''')	1642 (2)	7824 (6)	1609 (2)	53 (2)	220 (11)	43 (2)	-26 (7)	9 (3)	-19 (8)
C(6''')	2065 (2)	8923 (6)	2222 (2)	52 (2)	199 (10)	41 (2)	17 (7)	14 (3)	-13 (8)
F(1)	2735 (2)	7156 (6)	74 (2)	76 (2)	754 (14)	53 (1)	-14 (7)	-19 (2)	-84 (8)
F(2)	684 (2)	7615 (4)	410 (2)	80 (1)	343 (8)	54 (1)	-66 (5)	- 6 (2)	-25 (6)
Cl	3676 (1)	7835 (1)	4400 (1)	46 (0)	136 (2)	54 (0)	21 (2)	42 (1)	25 (2)

(b) Structure of the base

	x	y	z	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}
O(1)	4290 (5)	3992 (5)	3403 (1)	198 (8)	164 (7)	14 (0)	4 (3)	28 (4)	-59 (13)
O(2)	9096 (4)	2963 (4)	1900 (1)	164 (7)	79 (5)	17 (0)	3 (3)	26 (4)	-59 (11)
F(1)	8280 (6)	5306 (6)	4693 (1)	354 (10)	456 (10)	20 (0)	19 (4)	78 (5)	104 (17)
F(2)	3078 (7)	5308 (6)	399 (2)	562 (15)	373 (12)	26 (0)	15 (5)	158 (6)	-92 (11)
N(1'')	8394 (5)	5644 (5)	2748 (1)	108 (8)	86 (6)	15 (0)	9 (3)	4 (4)	36 (11)
C(1')	3650 (7)	5053 (8)	4101 (2)	143 (11)	242 (14)	12 (0)	14 (6)	31 (5)	-26 (21)
C(2')	3756 (8)	6296 (9)	4417 (2)	207 (14)	294 (16)	17 (1)	11 (7)	22 (6)	243 (26)
C(3')	3003 (9)	6170 (9)	4824 (2)	232 (15)	246 (13)	18 (1)	11 (6)	31 (7)	192 (24)
C(4')	2318 (8)	4826 (9)	4898 (2)	211 (14)	323 (14)	16 (1)	20 (7)	62 (7)	53 (26)
C(5')	2195 (9)	3559 (10)	4602 (3)	267 (16)	316 (16)	21 (1)	23 (7)	42 (7)	-52 (27)
C(6')	2880 (8)	3751 (10)	4197 (2)	183 (14)	372 (19)	15 (1)	26 (7)	33 (6)	-43 (27)
C(1)	7485 (7)	5446 (8)	3145 (2)	159 (11)	176 (12)	17 (1)	23 (6)	50 (6)	96 (20)
C(2)	5912 (7)	6502 (7)	3085 (2)	165 (11)	140 (10)	19 (1)	16 (5)	39 (6)	61 (19)
C(3)	5257 (8)	6571 (6)	3555 (2)	212 (13)	96 (8)	20 (1)	14 (5)	59 (6)	71 (17)
C(4)	4409 (6)	5105 (6)	3664 (2)	127 (10)	112 (9)	14 (0)	- 9 (5)	-12 (5)	71 (16)
C(2'')	9884 (7)	4547 (6)	2843 (2)	150 (11)	87 (8)	18 (1)	- 2 (5)	33 (5)	79 (17)
C(3'')	10943 (6)	4774 (6)	2454 (2)	139 (10)	124 (9)	11 (1)	-11 (1)	2 (5)	-21 (17)
C(4'')	9751 (6)	4464 (6)	1929 (2)	116 (9)	57 (7)	17 (1)	12 (4)	29 (5)	- 4 (14)
C(5'')	8148 (7)	5519 (6)	1839 (2)	175 (12)	122 (9)	15 (1)	- 9 (5)	23 (5)	27 (18)
C(6'')	7184 (7)	5333 (7)	2259 (2)	155 (11)	138 (10)	13 (1)	0 (5)	3 (5)	14 (18)
C(1''')	690 (7)	4751 (7)	1528 (2)	176 (11)	114 (9)	12 (1)	2 (5)	8 (5)	47 (18)
C(2''')	2050 (8)	5783 (7)	1574 (2)	220 (14)	122 (9)	20 (1)	8 (6)	37 (6)	-68 (19)
C(3''')	2857 (9)	5972 (8)	1191 (2)	301 (17)	187 (12)	23 (1)	22 (6)	89 (8)	-75 (25)
C(4''')	2264 (9)	5155 (8)	769 (2)	333 (17)	193 (13)	19 (1)	25 (6)	77 (8)	-78 (25)
C(5''')	946 (10)	4157 (9)	710 (2)	394 (20)	194 (13)	17 (1)	0 (6)	76 (8)	-72 (27)
C(6''')	137 (9)	3905 (8)	1083 (2)	251 (15)	202 (12)	16 (1)	-25 (6)	42 (6)	-44 (23)

Only seven signs in 382 were wrong as compared with those obtained at the end of the refinement process.

Structure of the base

The origin and symbols are listed in Table 2(b). The following relationships between symbols were found:

$$\begin{aligned} AB &+ (1.0) \\ B &- (0.0) \\ A &+ (0.96) \end{aligned}$$

The numbers between brackets are the probabilities for the sign to be positive. Four sets of signs were generated [Table 3(b)]. The *E*-map computed with the signs of set 4 showed the whole molecule but shifted by approximately $(\frac{1}{2}, 0, \frac{1}{2})$. The correct solution was given by set 1. This is a good example of the advantage of a multisolution programme.

Refinement

Both structures were refined by least squares in the block-diagonal approximation (scheme 3 × 3, 6 × 6) minimizing $\sum w(F_o - F_c)^2$. The weight *w* is given by:

$$w = \frac{1}{P_1 + |F_o| + P_2|F_c|^2} \quad (\text{Cruickshank, 1961}).$$

$P_1 = 16$; $P_2 = 0.01$ for the base and $P_1 = 8$; $P_2 = 0.01$ for the hydrochloride. At the end of the isotropic refinement $R = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}$ was 0.18 for both structures. This was readily reduced to 0.076 and 0.10 for the hydrochloride and the base respectively by anisotropic refinement.

Final coordinates and thermal parameters for both compounds are listed in Table 4. A listing of observed and calculated structure factors is available upon request.

Description of the structures

Fig. 1 shows the atom numbering scheme on a perspective view of the molecule in the base crystal. Bond distances and angles for both structures are listed in Table 5. The phenyl groups are planar within experimental error.

The conformation of the molecules can be described in terms of torsional angles (Table 6). The angle $\varphi(i, i + 1)$

Table 5. Bond distances and angles

(a) Structure of the hydrochloride			
F(1)—C(4')	1.36 Å	C(6'')—C(5'')	1.50
C(4')—C(3')	1.37	C(2'')—C(3'')	1.52
C(4')—C(5')	1.34	C(3'')—C(4'')	1.56
C(5')—C(6')	1.39	C(5'')—C(4'')	1.54
C(3')—C(2')	1.38	C(4'')—O(2)	1.43
C(2')—C(1')	1.41	C(4'')—C(1''')	1.49
C(1')—C(6')	1.39	C(1''')—C(2''')	1.40
C(1')—C(4)	1.48	C(1''')—C(6''')	1.43
C(4)—O(1)	1.23	C(2''')—C(3''')	1.38
C(4)—C(3)	1.48	C(3''')—C(4''')	1.37
C(3)—C(2)	1.53	C(6''')—C(5''')	1.38
C(2)—C(1)	1.53	C(5''')—C(4''')	1.38
C(1)—N	1.50	C(4''')—F(2)	1.37
N—C(2'')	1.51	N—Cl	3.06
N—C(6'')	1.51		
$\sigma_a \approx 0.01 \text{ \AA}$			
F(1)—C(4')—C(3')	116°	N—C(2'')—C(3'')	109°
F(1)—C(4')—C(5')	119	N—C(6'')—C(5'')	109
C(4')—C(5')—C(6')	118	C(6'')—C(5'')—C(4'')	113
C(5')—C(4')—C(3')	124	C(2'')—C(3'')—C(4'')	111
C(4')—C(3')—C(2')	117	C(3'')—C(4'')—C(5'')	108
C(3')—C(2')—C(1')	120	C(3'')—C(4'')—C(1''')	110
C(5')—C(6')—C(1')	119	C(5'')—C(4'')—C(1''')	112
C(6')—C(1')—C(2')	118	C(3'')—C(4'')—O(2)	108
C(6')—C(1')—C(4)	121	C(5'')—C(4'')—O(2)	105
C(2')—C(1')—C(4)	119	C(4'')—C(1''')—C(2''')	120
C(1')—C(4)—O(1)	118	C(4'')—C(1''')—C(6''')	122
O(1)—C(4)—C(3)	121	C(6''')—C(1''')—C(2''')	117
C(1')—C(4)—C(3)	120	C(6''')—C(5''')—C(4''')	117
C(4)—C(3)—C(2)	112	C(2''')—C(3''')—C(4''')	118
C(3)—C(2)—C(1)	108	C(1''')—C(2''')—C(3''')	122
C(2)—C(1)—N	110	C(1''')—C(6''')—C(5''')	121
C(1)—N—C(2'')	111	C(5''')—C(4''')—C(3''')	123
C(1)—N—C(6'')	111	C(5''')—C(4''')—F(2)	117
C(2'')—N—C(6'')	108	C(3''')—C(4''')—F(2)	119
$\sigma_a \approx 1^\circ$			
(b) Structure of the base			
F(1)—C(4')	1.34 Å	N(1'')—C(6'')	1.47
C(4')—C(3')	1.36	C(6'')—C(5'')	1.57
C(4')—C(5')	1.39	C(2'')—C(3'')	1.55
C(5')—C(6')	1.39	C(3'')—C(4'')	1.55
C(3')—C(2')	1.42	C(5'')—C(4'')	1.54
C(2')—C(1')	1.40	C(4'')—O(2)	1.43
C(6')—C(1')	1.38	C(4'')—C(1''')	1.52
C(1')—C(4)	1.50	C(1''')—C(2''')	1.40
C(4)—O(1)	1.22	C(1''')—C(6''')	1.42
C(4)—C(3)	1.53	C(2''')—C(3''')	1.39
C(3)—C(2)	1.53	C(5''')—C(4''')	1.35
C(2)—C(1)	1.53	C(6''')—C(5''')	1.38
C(1)—N(1')	1.49	C(3''')—C(4''')	1.37
N(1'')—C(2'')	1.49	C(4''')—F(2)	1.37
$\sigma_a = 0.01 \text{ \AA}$			

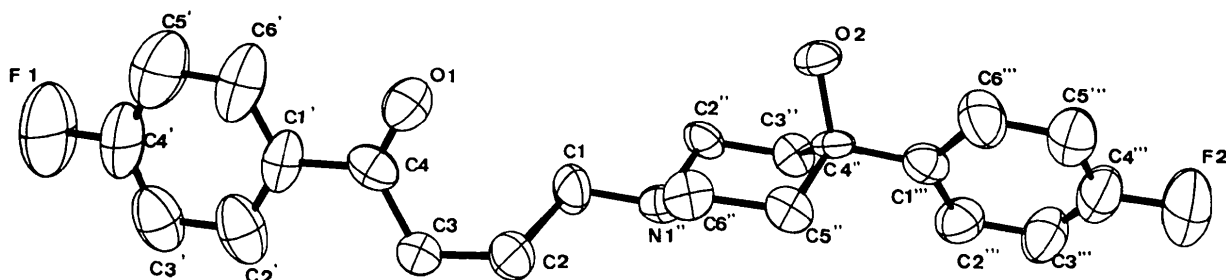


Fig. 1. Perspective view of a molecule in the base crystal.

Table 5 (cont.)

F(1)—C(4')—C(3')	116°	N(1')—C(2'')—C(3'')	109°
F(1)—C(4')—C(5')	117	N(1'')—C(6'')—C(5'')	111
C(3')—C(4')—C(5')	125	C(2'')—C(3'')—C(4'')	110
C(4')—C(3')—C(2')	117	C(6'')—C(5'')—C(4'')	111
C(4')—C(5')—C(6')	115	C(5'')—C(4'')—O(2)	107
C(3')—C(2')—C(1')	119	C(3'')—C(4'')—O(2)	109
C(5')—C(6')—C(1')	122	C(3'')—C(4'')—C(1''')	113
C(6')—C(1')—C(2')	120	C(5'')—C(4'')—C(1''')	109
C(6')—C(1')—C(4)	118	C(1''')—C(4'')—O(2)	111
C(2')—C(1')—C(4)	122	C(4'')—C(1''')—C(2''')	123
C(1')—C(4)—C(3)	118	C(4'')—C(1''')—C(6''')	118
C(1')—C(4)—O(1)	119	C(2''')—C(1''')—C(6''')	119
O(1)—C(4)—C(3)	122	C(1''')—C(2''')—C(3''')	120
C(4)—C(3)—C(2)	114	C(1''')—C(6''')—C(5''')	119
C(3)—C(2)—C(1)	112	C(6''')—C(5''')—C(4''')	120
C(2)—C(1)—N(1'')	112	C(2''')—C(3''')—C(4''')	118
C(1)—N(1'')—C(2'')	107	C(3''')—C(4''')—F(2)	119
C(1)—N(1'')—C(6'')	111	C(5''')—C(4''')—F(2)	118
C(2'')—N(1'')—C(6'')	109	C(5''')—C(4''')—C(3''')	123

$$\sigma_z = 1^\circ$$

is the angle between the planes defined by $C(i-1)$, $C(i)$, $C(i+1)$ and $C(i)$, $C(i+1)$, $C(i+2)$. The zero angle corresponds to the *trans*-conformation except in the piperidine ring where the *cis*-conformation is the reference. The conformation of the molecule in both crystals is quite different as can be seen from the values of these angles. The phenyl group is perpendicular to the piperidine ring in the hydrochloride but not in the base. The piperidine ring is in the chair conformation in both cases with the hydroxyl in axial position and the phenyl group and the lateral chain equatorial as expected from conformational energy considerations.

In the crystal of the base there is one hydrogen bond between the nitrogen atom and the hydroxyl group

$N \cdots H-O(2)$: [O(2): $2-x$, $y-0.5$, $0.5-z$; 2.85 \AA]. The molecules of the hydrochloride are held together by one hydrogen bond between the hydroxyl group and a carbonyl group $O(2)-H \cdots O(1)$: [O(1): $0.5-x$, $0.5+y$, $1-z$; 2.89 \AA].

All calculations were carried out in the Computing Center of the University using programs written by Dr F. R. Ahmed (1966). The drawing was produced by Dr C. K. Johnson's program *ORTEP* (1965).

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Table 6. Conformational angles

	C(4)—C(3)	C(3)—C(2)	C(2)—C(1)	C(1)—N	C(4'')—C(1''')
Base	3	112	13	1	74°
Hydrochloride	354	18	355	257	90°