Polyfluorobicyclo[2.2.1]heptanes. Part IX.[1] Crystal and Molecular Structure of 1,4-Dibenzoyldecafluoronorbornane

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Crystals of the title compound are triclinic, space group $P\bar{1}$, with $Z = 2$ and cell parameters $a = 7{\cdot}321 \pm 0{\cdot}005$, $b = 11{\cdot}566 \pm 0{\cdot}006$, $c = 13{\cdot}227 \pm 0{\cdot}006$ Å, $\alpha = 110{\cdot}38 \pm 0{\cdot}05$, $\beta = 67{\cdot}18 \pm 0{\cdot}05$, $\gamma = 82{\cdot}40 \pm 0{\cdot}05°$. The structure was solved by direct methods and refined by least-squares to a final R of 4·9% for 2770 X-ray counter-data. Estimated standard deviations for bond lengths, bond angles, and torsion angles average *ca.* 0·007 Å, 0·3, and 0·5°. The decafluoronorbornane system approximates closely to C_{2v} symmetry. The bridge angle is 98·2° and the flap angle 115·7°, respectively 3 and 5° greater than the means determined by gas-phase electron diffraction studies on the parent hydrocarbon.

THE structure of the bicyclo[2.2.1]heptane (norbornane) system has been extensively studied by electron diffraction in the gas phase,[2-4] by X-ray crystallography,[5-10] and also by semi-empirical calculations [9,11-14] using different force-field approximations. We have now carried out a single-crystal X-ray structure analysis of the fluorinated derivative 1,4-dibenzoyldecafluoronorbornane,[15] to determine the effect of substituting fluorine for hydrogen on the geometry of the norbornane skeleton.

EXPERIMENTAL

Crystallographic Measurements.—Approximate cell dimensions were obtained from oscillation, Weissenberg and precession photographs. Final cell dimensions and intensity data were measured with a Hilger and Watts four-circle automatic diffractometer by use of Cu-K_α radiation and a scintillation counter. Up to θ 15° balanced filters were employed and within the range 15—75°, nickel-filtered radiation. The scan conditions were, θ—2θ mode, with 50 steps at intervals of 0·02° and a step time of 1 s for reflections with $\theta < 50°$, and 2 s for reflections within the range 50—75°. Peak and background intensities were obtained by an ordinate-analysis procedure.[16] Two hemispheres of data were measured, and after averaging the intensities of equivalent reflections, a total of 3637 independent reflection intensities was obtained, of which 2770 having $I \geqslant 3\sigma(I)$, were used in the structure analysis. Intensities were corrected for absorption effects by a semi-empirical method [17] based on the variation of intensity with azimuthal angle for the 413 reflection, and converted into structure amplitudes by application of Lorentz and polarisation factors. The crystal used had dimensions $0{\cdot}1 \times 0{\cdot}4 \times 0{\cdot}6$ mm.

Crystal Data.—$C_{21}H_{10}F_{10}O_2$, $M = 484{\cdot}3$. Triclinic, $a = 7{\cdot}321 \pm 0{\cdot}005$, $b = 11{\cdot}566 \pm 0{\cdot}006$, $c = 13{\cdot}227 \pm 0{\cdot}006$ Å, $\alpha = 110{\cdot}38 \pm 0{\cdot}05$, $\beta = 67{\cdot}18 \pm 0{\cdot}05$, $\gamma = 82{\cdot}40 \pm 0{\cdot}05°$, $U = 920{\cdot}2$, $Z = 2$, $D_c = 1{\cdot}748$, $F(000) = 484$. Space

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group $P1$ or $P\bar{1}$; $P\bar{1}$ established by the analysis. Cu-K_α radiation, $\lambda = 1{\cdot}5418$ Å, μ(Cu-K_α) = 16·8 cm^{-1}.

Structure Analysis.—Examination of the statistical properties of the normalised structure amplitudes indicated the centrosymmetric space group $P\bar{1}$ ($\langle|E|\rangle = 0{\cdot}75$, $\langle|E^2 - 1|\rangle = 0{\cdot}99$). The structure was solved by direct methods [18] by use of a multiple-solution technique [19] and tangent refinement of phases with the automatic phasing program TANFIZ.[20] Phases (0 or 180°) were determined for the 246 largest E values ($>1{\cdot}8$) and the E map calculated with these phases revealed the positions of all the atoms of the molecule (apart from hydrogen atoms). At this stage R was 32·9%, four cycles of isotropic full-matrix least-squares refinement reducing it to 13·6%. A Fourier difference synthesis was computed and from this all the hydrogen atoms were located. These were included in the calculations in theoretical positions (assuming C–H 1·08 Å) but their parameters were not refined. The heavier atoms were now allowed to vibrate anisotropically and least-squares refinement of positional and thermal parameters continued until the calculated shifts in the parameters were all $<0{\cdot}05\sigma$. Owing to the limitations of computer storage, the molecule was split into three overlapping portions for the anisotropic refinement, so that the decafluoronorbornane and the two benzoyl portions were refined separately. The calculated σ values may therefore be underestimates and should probably be increased by *ca.* 50% to give more realistic values.

The weighting scheme used in the final refinement cycles was $w^{\frac{1}{2}} = 1{\cdot}0$ if $|F_o| \leqslant 17{\cdot}0$ and $w^{\frac{1}{2}} = 17{\cdot}0/|F_o|$ if $|F_o| > 17{\cdot}0$, chosen so as to give approximately constant values for the average of $\Sigma w(|F_o| - |F_c|)^2$ when taken in groups of increasing $|F_o|$ and increasing sin θ. Six large, low-order reflections which appeared to be affected by extinction, were assigned zero weight. Atomic scattering factors were taken from ref. 21.

The final R was 4·9% for 2770 observed structure amplitudes. Observed and calculated structure factors are published in Supplementary Publication No. SUP 20878 (19 pp., 1 microfiche).*

Computations were carried out on the Oxford University KDF 9 computer (data reduction), on the IBM 360/44 of the Department of Physics, Birmingham University (phase determination, and rigid-body thermal motion analysis), and on the Birmingham University KDF 9 computer (Fourier, least-squares and molecular geometry calculations.) Local versions of FORDAP, the Zalkin Fourier program, and ORFLS and ORFFE, the Busing, Martin, and Levy least-squares and function-and-error programs were employed.

RESULTS AND DISCUSSION

The stereochemistry of the molecule is illustrated in Figure 1, which also shows the atomic numbering. Fluorine atoms are assigned the number of the carbon atom to which they are linked, with x denoting *exo* and n, *endo*. Final atomic co-ordinates are in Table 1, and thermal parameters for the non-hydrogen atoms in Table 2. Molecular dimensions are in Tables 3 and 4. Estimated standard deviations as derived from the least-squares totals are 0·003—0·005 Å for lengths, *ca.* 0·2° for bond angles, and *ca.* 0·3° for torsion angles. However, because of the refinement procedure which had to be adopted (see Experimental section), more realistic values might be some 50% greater than these, 0·007 Å for lengths, 0·3° for bond angles and 0·5° for torsion angles.

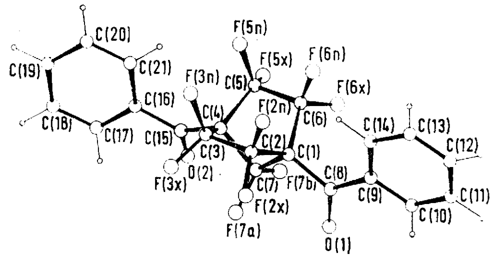

FIGURE 1 The molecule seen along the *a* axis (positive *a* is towards the viewer, *b* and *c* axes as in Figure 2)

TABLE 1

Fractional atomic co-ordinates ($\times 10^4$), with estimated standard deviations in parentheses

Atom	x	y	z
C(1)	5611(4)	3403(3)	−3032(2)
C(2)	7291(5)	2498(3)	−2988(3)
C(3)	6205(5)	1560(3)	−2554(3)
C(4)	3970(4)	1995(2)	−2387(2)
C(5)	2743(5)	3343(3)	−1302(2)
C(6)	3840(5)	4286(3)	−1728(2)
C(7)	4487(4)	2415(3)	−3431(2)
C(8)	6397(5)	4051(3)	−3901(3)
C(9)	7137(5)	5192(3)	−3591(3)
C(10)	7248(5)	5886(3)	−4280(3)
C(11)	7910(6)	6968(4)	−4047(3)
C(12)	8523(6)	7354(4)	−3145(3)
C(13)	8455(6)	6666(4)	−2472(3)
C(14)	7759(5)	5591(3)	−2680(3)
C(15)	2794(5)	966(3)	−2476(3)
C(16)	2805(4)	104(3)	−1869(3)
C(17)	2609(5)	−1113(3)	−2424(3)
C(18)	2563(5)	−1953(3)	−1892(3)
C(19)	2634(5)	−1592(4)	−805(3)
C(20)	2786(5)	−375(4)	−233(3)
C(21)	2890(5)	465(3)	−776(3)
F(2x)	8941(3)	1841(2)	−4102(2)
F(2n)	8007(3)	3112(2)	−2223(2)
F(3x)	7191(3)	322(2)	−3396(2)
F(3n)	6289(3)	1627(2)	−1540(2)
F(5x)	732(3)	3655(2)	−1058(2)
F(5n)	2730(3)	3444(2)	−253(1)
F(6x)	2493(3)	5174(2)	−1759(2)
F(6n)	4441(3)	4949(2)	−928(1)
F(7a)	5697(3)	1417(2)	−4525(1)
F(7b)	2797(3)	2984(2)	−3463(1)
O(1)	6331(4)	3579(2)	−4856(2)
O(2)	1937(4)	869(2)	−3108(2)
H[C(10)]	6812	5574	−5000
H[C(11)]	7951	7514	−4568
H[C(12)]	9054	8196	−2969
H[C(13)]	8949	6967	−1777
H[C(14)]	7695	5063	−2142
H[C(17)]	2494	−1394	−3210
H[C(18)]	2469	−2907	−2335
H[C(19)]	2572	−2256	−395
H[C(20)]	2823	−85	627
H[C(21)]	3039	1405	−345

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* See Notice to Authors No. 7, in *J.C.S. Dalton*, 1972, Index issue.

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The thermal parameters of the carbon atoms of the norbornane skeleton were analysed in terms of rigid-body motion.[22] The root-mean-square deviation between the observed U_{ij} values and those calculated on the basis of the rigid-body model is reasonably low, 0·0022 Å^2. The major axis of libration is approximately parallel to the C(1) ··· C(4) direction, the root-mean-square amplitude of libration being 4·2°. Bond lengths corrected for thermal motion using the calculated librations, are listed in the Tables with the superscript c. Carbon–fluorine bond lengths corrected on the assumption of 'riding' motion[23] are listed with the superscript r.

TABLE 2

Anisotropic thermal parameters (× 10^4) for the heavier atoms

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	513	497	403	−168	−225	207
C(2)	537	624	586	−195	−268	324
C(3)	622	534	598	−158	−290	312
C(4)	522	433	390	−137	−217	167
C(5)	620	496	388	−183	−233	196
C(6)	639	457	419	−194	−274	171
C(7)	541	466	363	−131	−201	151
C(8)	541	548	423	−171	−226	239
C(9)	476	515	442	−168	−200	208
C(10)	531	587	527	−183	−223	304
C(11)	627	627	668	−248	−247	368
C(12)	646	663	634	−311	−229	282
C(13)	774	718	585	−429	−334	277
C(14)	645	668	515	−292	−295	300
C(15)	566	461	437	−152	−215	180
C(16)	483	487	448	−162	−160	202
C(17)	561	481	557	−220	−217	205
C(18)	631	518	734	−226	−218	282
C(19)	618	692	800	−237	−275	481
C(20)	660	723	624	−263	−286	406
C(21)	638	561	511	−211	−247	249
F(2x)	541	796	804	−69	−113	429
F(2n)	822	890	1020	−446	−665	609
F(3x)	672	525	968	−42	−230	326
F(3n)	839	1026	902	−437	−563	705
F(5x)	520	558	638	−134	−149	182
F(5n)	1017	634	394	−344	−315	241
F(6x)	666	486	649	−76	−219	265
F(6n)	960	690	454	−451	−358	212
F(7a)	827	537	366	−208	−183	113
F(7b)	664	651	630	−262	−418	348
O(1)	905	728	494	−382	−406	326
O(2)	950	660	772	−387	−590	366

Temperature factors are in the form: $T = \exp[-2\pi^2(U_{11}h^2a^{*2} + \dots + 2U_{12}hka^*b^* + \dots)]$.

Bond lengths in the phenyl rings range from 1·375—1·402 Å, mean 1·387 Å, somewhat shorter than the standard aromatic length of 1·394 Å.[24] Nevertheless, none of the lengths differs significantly from the aromatic value. The C(sp^2)–C(sp^2) single bonds, C(8)–C(9) and C(15)–C(16) are 1·479 and 1·483 Å, and the C–O double bonds C(8)–O(1) and C(15)–O(2), 1·213 and 1·211 Å, in excellent agreement with the accepted[24,25] values for the lengths of these bonds (1·48—1·50, and 1·215 Å, respectively). The C(1)–C(8) and C(4)–C(15) bonds (1·555 and 1·556 Å) are, however, significantly (*ca.* 6σ) longer than a normal C(sp^3)–C(sp^2) bond[24] and this may

TABLE 3

Molecular dimensions

(*a*) Bonded distances (Å) with estimated standard deviations (× 10^3) in parentheses; superscripts are defined in the text

C(1)–C(2)	1·539(4), 1·544 ^{c}	C(16)–C(17)	1·399(4)
C(1)–C(6)	1·545(4), 1·551 ^{c}	C(16)–C(21)	1·388(4)
C(1)–C(7)	1·558(4), 1·563 ^{c}	C(17)–C(18)	1·381(5)
C(1)–C(8)	1·555(4)	C(18)–C(19)	1·375(5)
C(2)–C(3)	1·564(4), 1·568 ^{c}	C(19)–C(20)	1·390(5)
C(3)–C(4)	1·551(4), 1·556 ^{c}	C(20)–C(21)	1·389(4)
C(4)–C(5)	1·539(4), 1·545 ^{c}	C(8)–O(1)	1·213(4)
C(4)–C(7)	1·547(4), 1·552 ^{c}	C(15)–O(2)	1·211(4)
C(4)–C(15)	1·556(4)	C(2)–F(2x)	1·343(4), 1·367 ^{r}
C(5)–C(6)	1·569(4), 1·572 ^{c}	C(2)–F(2n)	1·347(3), 1·368 ^{r}
C(8)–C(9)	1·479(4)	C(3)–F(3x)	1·343(4), 1·367 ^{r}
C(9)–C(10)	1·397(4)	C(3)–F(3n)	1·342(3), 1·364 ^{r}
C(9)–C(14)	1·402(4)	C(5)–F(5x)	1·346(4), 1·363 ^{r}
C(10)–C(11)	1·379(5)	C(5)–F(5n)	1·347(3), 1·365 ^{r}
C(11)–C(12)	1·386(5)	C(6)–F(6x)	1·350(3), 1·367 ^{r}
C(12)–C(13)	1·377(5)	C(6)–F(6n)	1·336(3), 1·356 ^{r}
C(13)–C(14)	1·385(5)	C(7)–F(7a)	1·336(3), 1·354 ^{r}
C(15)–C(16)	1·483(4)	C(7)–F(7b)	1·345(3), 1·358 ^{r}

(*b*) Selected non-bonded distances (Å)

C(1) ··· C(3)	2·458	F(2x) ··· F(3x)	2·531
C(1) ··· C(4)	2·348	F(2n) ··· F(3n)	2·474
C(1) ··· C(5)	2·464	F(2n) ··· F(6n)	2·606
C(2) ··· C(4)	2·466	F(3n) ··· F(5n)	2·593
C(2) ··· C(6)	2·528	F(5x) ··· F(6x)	2·533
C(2) ··· C(7)	2·348	F(5n) ··· F(6n)	2·488
C(3) ··· C(5)	2·530		
C(3) ··· C(7)	2·347		
C(4) ··· C(6)	2·460	C(14) ··· F(2n)	3·126
C(5) ··· C(7)	2·336	C(14) ··· F(6n)	3·022
C(6) ··· C(7)	2·342	C(21) ··· F(3n)	2·984
C(2) ··· F(7a)	2·741	C(21) ··· F(5n)	3·245
C(3) ··· F(7a)	2·726	O(1) ··· F(7a)	2·752
C(5) ··· F(7b)	2·725	O(1) ··· F(7b)	2·867
C(6) ··· F(7b)	2·715	O(2) ··· F(7a)	2·962
C(8) ··· F(2x)	2·821	O(2) ··· F(7b)	2·752
C(8) ··· F(2n)	3·297	H[C(14)] ··· F(2n)	2·20
C(8) ··· F(6x)	2·839	H[C(14)] ··· F(6n)	2·35
C(8) ··· F(6n)	3·315	H[C(21)] ··· F(3n)	2·40
C(15) ··· F(3x)	2·845	H[C(21)] ··· F(5n)	2·29
C(15) ··· F(3n)	3·314		
C(15) ··· F(5x)	2·822		
C(15) ··· F(5n)	3·294		

(*c*) Bond angles (deg.); standard deviations 0·2—0·3°

C(2)–C(1)–C(6)	110·1	C(1)–C(6)–F(6n)	115·5
C(2)–C(1)–C(7)	98·6	C(5)–C(6)–F(6x)	110·4
C(2)–C(1)–C(8)	116·6	C(5)–C(6)–F(6n)	110·1
C(6)–C(1)–C(7)	98·0	F(6x)–C(6)–F(6n)	106·4
C(6)–C(1)–C(8)	117·1	C(1)–C(7)–C(4)	98·2
C(7)–C(1)–C(8)	113·4	C(1)–C(7)–F(7a)	113·7
C(1)–C(2)–C(3)	104·8	C(1)–C(7)–F(7b)	111·7
C(1)–C(2)–F(2x)	110·0	C(4)–C(7)–F(7a)	112·0
C(1)–C(2)–F(2n)	114·2	C(4)–C(7)–F(7b)	113·9
C(3)–C(2)–F(2x)	111·1	F(7a)–C(7)–F(7b)	107·3
C(3)–C(2)–F(2n)	109·7	C(1)–C(8)–C(9)	123·0
F(2x)–C(2)–F(2n)	107·0	C(1)–C(8)–O(1)	115·8
C(2)–C(3)–C(4)	104·6	C(9)–C(8)–O(1)	121·2
C(2)–C(3)–F(3x)	111·1	C(8)–C(9)–C(10)	116·6
C(2)–C(3)–F(3n)	109·8	C(8)–C(9)–C(14)	124·3
C(4)–C(3)–F(3x)	110·1	C(10)–C(9)–C(14)	119·0
C(4)–C(3)–F(3n)	114·6	C(9)–C(10)–C(11)	120·5
F(3x)–C(3)–F(3n)	106·7	C(10)–C(11)–C(12)	120·0
C(3)–C(4)–C(5)	109·9	C(11)–C(12)–C(13)	120·1
C(3)–C(4)–C(7)	98·5	C(12)–C(13)–C(14)	120·6
C(3)–C(4)–C(15)	117·0	C(13)–C(14)–C(9)	119·7
C(5)–C(4)–C(7)	98·4	C(4)–C(15)–C(16)	122·0
C(5)–C(4)–C(15)	116·4	C(4)–C(15)–O(2)	116·4
C(7)–C(4)–C(15)	113·6	C(16)–C(15)–O(2)	121·5
C(4)–C(5)–C(6)	104·6	C(15)–C(16)–C(17)	116·4
C(4)–C(5)–F(5x)	110·2	C(15)–C(16)–C(21)	123·9
C(4)–C(5)–F(5n)	114·7	C(17)–C(16)–C(21)	119·7
C(6)–C(5)–F(5x)	111·5	C(16)–C(17)–C(18)	119·4
C(6)–C(5)–F(5n)	110·0	C(17)–C(18)–C(19)	120·9
F(5x)–C(5)–F(5n)	106·0	C(18)–C(19)–C(20)	120·2
C(1)–C(6)–C(5)	104·6	C(19)–C(20)–C(21)	119·4
C(1)–C(6)–F(6x)	109·8	C(20)–C(21)–C(16)	120·4

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TABLE 3 (*Continued*)

(*d*) Torsion angles (deg.); standard deviations *ca.* 0·3°

C(6)–C(1)–C(2)–C(3)	67·8
C(6)–C(1)–C(2)–F(2x)	−172·7
C(6)–C(1)–C(2)–F(2n)	−52·3
C(7)–C(1)–C(2)–C(3)	−34·1
C(7)–C(1)–C(2)–F(2x)	85·4
C(7)–C(1)–C(2)–F(2n)	−154·2
C(8)–C(1)–C(2)–C(3)	−155·7
C(8)–C(1)–C(2)–F(2x)	−36·2
C(8)–C(1)–C(2)–F(2n)	84·2
C(1)–C(2)–C(3)–C(4)	0·3
C(1)–C(2)–C(3)–F(3x)	119·1
C(1)–C(2)–C(3)–F(3n)	−123·1
F(2x)–C(2)–C(3)–C(4)	−118·4
F(2x)–C(2)–C(3)–F(3x)	0·3
F(2x)–C(2)–C(3)–F(3n)	118·2
F(2n)–C(2)–C(3)–C(4)	123·4
F(2n)–C(2)–C(3)–F(3x)	−117·8
F(2n)–C(2)–C(3)–F(3n)	0·0
C(2)–C(3)–C(4)–C(5)	−68·4
C(2)–C(3)–C(4)–C(7)	33·8
C(2)–C(3)–C(4)–C(15)	155·9
F(3x)–C(3)–C(4)–C(5)	172·2
F(3x)–C(3)–C(4)–C(7)	−85·6
F(3x)–C(3)–C(4)–C(15)	36·4
F(3n)–C(3)–C(4)–C(5)	51·9
F(3n)–C(3)–C(4)–C(7)	154·1
F(3n)–C(3)–C(4)–C(15)	−83·8
C(3)–C(4)–C(5)–C(6)	67·7
C(3)–C(4)–C(5)–F(5x)	−172·4
C(3)–C(4)–C(5)–F(5n)	−52·9
C(7)–C(4)–C(5)–C(6)	−34·5
C(7)–C(4)–C(5)–F(5x)	85·4
C(7)–C(4)–C(5)–F(5n)	−155·1
C(15)–C(4)–C(5)–C(6)	−156·2
C(15)–C(4)–C(5)–F(5x)	−36·3
C(15)–C(4)–C(5)–F(5n)	83·2
C(4)–C(5)–C(6)–C(1)	0·2
C(4)–C(5)–C(6)–F(6x)	118·3
C(4)–C(5)–C(6)–F(6n)	−124·5
F(5x)–C(5)–C(6)–C(1)	−118·8
F(5x)–C(5)–C(6)–F(6x)	−0·7
F(5x)–C(5)–C(6)–F(6n)	116·4
F(5n)–C(5)–C(6)–C(1)	123·9
F(5n)–C(5)–C(6)–F(6x)	−118·0
F(5n)–C(5)–C(6)–F(6n)	−0·8
C(5)–C(6)–C(1)–C(2)	−68·3
C(5)–C(6)–C(1)–C(7)	33·9
C(5)–C(6)–C(1)–C(8)	155·4
F(6x)–C(6)–C(1)–C(2)	173·1
F(6x)–C(6)–C(1)–C(7)	−84·6
F(6x)–C(6)–C(1)–C(8)	36·9
F(6n)–C(6)–C(1)–C(2)	52·8
F(6n)–C(6)–C(1)–C(7)	155·1
F(6n)–C(6)–C(1)–C(8)	−83·4
C(2)–C(1)–C(7)–C(4)	55·8
C(2)–C(1)–C(7)–F(7a)	−62·7
C(2)–C(1)–C(7)–F(7b)	175·7
C(6)–C(1)–C(7)–C(4)	−56·1
C(6)–C(1)–C(7)–F(7a)	−174·6
C(6)–C(1)–C(7)–F(7b)	63·8
C(8)–C(1)–C(7)–C(4)	179·8
C(8)–C(1)–C(7)–F(7a)	61·3
C(8)–C(1)–C(7)–F(7b)	−60·4
C(3)–C(4)–C(7)–C(1)	−55·4
C(3)–C(4)–C(7)–F(7a)	64·4
C(3)–C(4)–C(7)–F(7b)	−173·6
C(5)–C(4)–C(7)–C(1)	56·4
C(5)–C(4)–C(7)–F(7a)	176·2
C(5)–C(4)–C(7)–F(7b)	−61·8
C(15)–C(4)–C(7)–C(1)	−179·9
C(15)–C(4)–C(7)–F(7a)	−60·1
C(15)–C(4)–C(7)–F(7b)	61·9
C(2)–C(1)–C(8)–C(9)	−78·7
C(2)–C(1)–C(8)–O(1)	102·7
C(6)–C(1)–C(8)–C(9)	54·8
C(6)–C(1)–C(8)–O(1)	−123·8
C(7)–C(1)–C(8)–C(9)	167·9
C(7)–C(1)–C(8)–O(1)	−10·7
C(1)–C(8)–C(9)–C(10)	−162·9
C(1)–C(8)–C(9)–C(14)	18·4
O(1)–C(8)–C(9)–C(10)	15·7
O(1)–C(8)–C(9)–C(14)	−163·1
C(3)–C(4)–C(15)–C(16)	44·1
C(3)–C(4)–C(15)–O(2)	−133·0
C(5)–C(4)–C(15)–C(16)	−88·8
C(5)–C(4)–C(15)–O(2)	94·1
C(7)–C(4)–C(15)–C(16)	158·0
C(7)–C(4)–C(15)–O(2)	−19·1
C(4)–C(15)–C(16)–C(17)	−150·8
C(4)–C(15)–C(16)–C(21)	32·5
O(2)–C(15)–C(16)–C(17)	26·1
O(2)–C(15)–C(16)–C(21)	−150·6

TABLE 4

Mean plane calculations

(i) Deviations (Å) of atoms from least squares planes. In the equations of the planes *x*, *y*, and *z* are fractional co-ordinates relative to the cell axes

Plane (*a*):

C(2), C(3), C(5), C(6) $-4{\cdot}529x - 0{\cdot}270y - 12{\cdot}078z = 0{\cdot}236$

[C(2) −0·004, C(3) 0·004, C(5) −0·004, C(6) 0·004, C(1) −0·793, C(4) −0·796, C(7) −1·810, C(8) −1·470, C(15) −1·463, F(2x) −0·619, F(3x) −0·601, F(5x) −0·611, F(6x) −0·620, F(2n) 1·262, F(3n) 1·268, F(5n) 1·260, F(6n) 1·260, F(7a) −2·611, F(7b) −2·599]

Plane (*b*):

C(1), C(4), C(7) $4{\cdot}622x - 5{\cdot}320y + 0{\cdot}148z = 0{\cdot}738$

[C(2) −1·259, C(3) −1·262, C(5) 1·268, C(6) 1·269, C(8) −0·005, C(15) −0·002, F(2x) −2·354, F(3x) −2·364, F(5x) 2·360, F(6x) 2·365, F(2n) −1·274, F(3n) −1·280, F(5n) 1·312, F(6n) 1·332, F(7a) −1·074, F(7b) 1·084]

Plane (*c*):

C(7), mid-points of C(2)–C(3) and C(5)–C(6) $3{\cdot}343x + 10{\cdot}292y - 5{\cdot}433z = 5{\cdot}849$

[C(7) 0·000, C(1) −1·176, C(4) 1·172, C(2) −0·782, C(3) 0·782, C(5) 0·785, C(6) −0·785, C(8) −2·578, C(15) 2·575, F(2x) −1·263, F(3x) 1·268, F(5x) 1·268, F(6x) −1·265, F(2n) −1·238, F(3n) 1·235, F(5n) 1·255, F(6n) −1·233, F(7a) 0·028, F(7b) −0·038]

Plane (*d*):

C(1)—(4) $-1{\cdot}364x - 3{\cdot}055y - 10{\cdot}109z = 1{\cdot}261$

[C(1) 0·001, C(2) −0·002, C(3) 0·002, C(4) −0·001, C(7) −0·857, F(2x) −1·104, F(3x) −1·093, F(2n) 1·057, F(3n) 1·059]

Plane (*e*):

C(1), C(4)—(6) $-6{\cdot}304x + 2{\cdot}622y - 10{\cdot}358z = 0{\cdot}494$

[C(1) −0·001, C(4) 0·001, C(5) −0·001, C(6) 0·001, C(7) −0·864, F(5x) −1·098, F(6x) −1·113, F(5n) 1·050, F(6n) 1·035]

Plane (*f*):

C(9)—(14) $4{\cdot}913x - 2{\cdot}690y - 2{\cdot}139z = 2{\cdot}884$

[C(9) 0·005, C(10) −0·010, C(11) 0·006, C(12) 0·002, C(13) −0·006, C(14) 0·002, C(8) −0·004]

Plane (*g*):

C(1), C(8), C(9), O(1) $5{\cdot}647x - 4{\cdot}251y + 1{\cdot}822z = 1{\cdot}172$

[C(1) 0·002, C(8) −0·007, C(9) 0·002, O(1) 0·003]

Plane (*h*):

C(16)—(21) $6{\cdot}190x - 0{\cdot}746y - 0{\cdot}234z = 1{\cdot}767$

[C(16) −0·005, C(17) 0·012, C(18) −0·009, C(19) −0·001, C(20) 0·009, C(21) −0·006, C(15) 0·052]

Plane (*i*):

C(4), C(15), C(16), O(2) $3{\cdot}459x - 3{\cdot}370y - 4{\cdot}508z = 1{\cdot}772$

[C(4) −0·005, C(15) 0·016, C(16) −0·005, O(2) −0·006]

(ii) Dihedral angles (deg.)

(*a*)–(*b*)	89·6	(*b*)–(*h*)	24·8
(*a*)–(*c*)	89·8	(*b*)–(*i*)	20·6
(*b*)–(*c*)	90·3	(*d*)–(*e*)	115·7
(*b*)–(*d*)	122·5	(*f*)–(*g*)	17·3
(*b*)–(*e*)	121·8	(*f*)–(*h*)	16·1
(*b*)–(*f*)	14·7	(*h*)–(*i*)	29·7
(*b*)–(*g*)	11·5		

be indicative of repulsive interactions between the benzoyl groups and the fluoronorbornane system. In camphane derivatives[5,6,8] and in 1,4-dichloronorbornane[3] the corresponding bonds do not show this marked lengthening over standard values.

The dimensions of the norbornane system approximate very closely to C_{2v} symmetry and in Table 5 are listed mean bond lengths, bond angles, and torsion angles with the maximum deviations from the mean values in parentheses. Apart from certain angles involving F(7a) and F(7b), the fluorine atoms linked to the bridging carbon atom C(7), no individual value differs significantly from the mean. For the carbon skeleton of the norbornane system, deviations from the mean values are all $<1{\cdot}5\sigma$.

The symmetry of the norbornane skeleton is also evident from the results of the mean plane calculations (Table 4). In the idealised skeleton with C_{2v} symmetry, the three groups of atoms used in the calculation of planes (*a*), (*d*), and (*e*) are each co-planar and the planes (*a*), (*b*), and (*c*) are mutually perpendicular. The actual deviations of atoms from their respective mean planes average 0·002 Å with maximum deviations of 0·004 Å [plane (*a*)]. The relevant dihedral angles deviate from 90° by a maximum of only 0·4°. The ' twist ' angles C(2)–C(1) ··· C(4)–C(3) and C(6)–C(1) ··· C(4)–C(5) which have been used[14] as a measure of the distortion of the system are 0·2 and 0·1°.

The norbornane carbon–carbon bonds fall into three categories, typified by C(1)–C(2), C(1)–C(7), and C(2)–C(3) (Table 5). Both the corrected and the uncorrected values show that the C(2)–C(3) bond length is significantly greater than C(1)–C(2) and probably also greater than C(1)–C(7). Theoretical calculations[9,12,13] on the parent hydrocarbon also yield a slightly greater length for the C(2)–C(3) bond but the results of the electron diffraction studies are conflicting. Carbon–fluorine bond lengths corrected for the effect of thermal motion range from 1·354—1·368, mean 1·363 Å, whereas the uncorrected lengths are in the range 1·336—1·350, mean 1·343 Å. From a consideration of corrected values (Table 5) it appears that the C(7)–F(7) length may be genuinely shorter than the C–F(x) length, with the C–F(n) bonds intermediate and not differing significantly from either. The actual differences in C–F bond lengths are, however, quite small, maximum 0·014 Å.

Hughes and Small[26] have noted that there is a variation in C–F bond length depending on the number of fluorine atoms linked to the carbon atom. For the case of two fluorine atoms, in the difluoroacetamide structure, the C–F bond distances are 1·361 and 1·364 Å (after libration correction), in very close agreement with our corrected values.

The mean F(2n) ··· F(6n) distance (2·600 ± 0·007 Å) is significantly greater than C(2) ··· C(6) (2·529 ± 0·001 Å). Electron diffraction results[3] for the hydrocarbon indicate that H(2n) ··· H(6n) at 2·25 Å is shorter by 0·19 Å than C(2) ··· C(6). The comparatively very low value for angle C(2)–C(1)–C(6) of 103·5° (*cf.* Table 6) observed in the electron diffraction study is consistent with this short H ··· H distance. In the fluoronorbornane, (Table 5) angle C(1)–C(2)–F(n) of 114·8° (mean) is significantly greater than C(1)–C(2)–F(x) (110·0°), and torsion angle C(1)–C(2)–C(3)–F(n) (123·7) significantly greater than C(1)–C(2)–C(3)–F(x) (118·7)°. These large values for the angles involving the *endo*-fluorine atoms have the effect of increasing the F(2n) ··· F(6n) distance and may be due to F ··· F repulsions.

TABLE 5

Mean dimensions for the norbornane system assuming C_{2v} symmetry (values in parentheses are the maximum deviations from the mean, superscripts are defined in the text)

(*a*) Bonded distances (Å)

C(1)–C(2)	1·544(7), 1·549(7) ^{c}	C–F(x)	1·346(4), 1·366(3) ^{r}
C(1)–C(7)	1·553(6), 1·557(6) ^{c}	C–F(n)	1·343(7), 1·363(7) ^{r}
C(2)–C(3)	1·567(3), 1·570(2) ^{c}	C(7)–F(7)	1·341(5), 1·356(2) ^{r}
C(1)–C(8)	1·556(1)		

(*b*) Non-bonded distances (Å)

C(1) ··· C(3)	2·462(4)	C(8) ··· F(2x)	2·832(13)
C(1) ··· C(4)	2·348	C(8) ··· F(2n)	3·305(11)
C(2) ··· C(6)	2·529(1)	F(2x) ··· F(3x)	2·532(1)
C(2) ··· C(7)	2·343(7)	F(2n) ··· F(3n)	2·481(7)
C(2) ··· F(7a)	2·727(14)	F(2n) ··· F(6n)	2·600(7)

(*c*) Bond angles (deg.)

C(2)–C(1)–C(6)	110·0(0·1)	C(1)–C(2)–F(n)	114·8(0·7)
C(2)–C(1)–C(7)	98·4(0·4)	C(3)–C(2)–F(x)	110·0(0·6)
C(1)–C(2)–C(3)	104·7(0·1)	C(3)–C(2)–F(n)	109·9(0·2)
C(1)–C(7)–C(4)	98·2	F(x)–C–F(n)	106·5(0·5)
C(2)–C(1)–C(8)	116·8(0·4)	C(1)–C(7)–F(7)	112·8(1·1)
C(7)–C(1)–C(8)	113·5(0·1)	F(7a)–C(7)–F(7b)	107·3
C(1)–C(2)–F(x)	110·0(0·2)		

(*d*) Torsion angles (mean of absolute values)

C(6)–C(1)–C(2)–C(3)	68·1(0·4)
C(1)–C(2)–C(3)–C(4)	0·3(0·1)
C(7)–C(1)–C(2)–C(3)	34·1(0·4)
C(2)–C(1)–C(7)–C(4)	55·9(0·5)
C(8)–C(1)–C(2)–C(3)	155·8(0·4)
C(8)–C(1)–C(7)–C(4)	179·8(0·1)
C(6)–C(1)–C(2)–F(x)	172·6(0·5)
C(6)–C(1)–C(2)–F(n)	52·5(0·6)
C(1)–C(2)–C(3)–F(x)	118·7(0·4)
C(1)–C(2)–C(3)–F(n)	123·7(0·8)
C(7)–C(1)–C(2)–F(x)	85·3(0·7)
C(7)–C(1)–C(2)–F(n)	154·6(0·5)
C(8)–C(1)–C(2)–F(x)	36·5(0·4)
C(8)–C(1)–C(2)–F(n)	83·7(0·5)
F(x)–C(2)–C(3)–F(n)	117·6(1·2)
F(x)–C(2)–C(3)–F(x)	0·5(0·2)
F(n)–C(2)–C(3)–F(n)	0·4(0·4)
C(2)–C(1)–C(7)–F(7a)	63·2(1·4)
C(2)–C(1)–C(7)–F(7b)	175·0(1·4)
C(8)–C(1)–C(7)–F(7)	60·9(1·0)

In order to assess the effect of the fluorine substitution on the geometry of the carbon skeleton, we have listed in Table 6 structural results obtained for norbornanes and camphanes by gas-phase electron diffraction,[2-4] theoretical calculations[9,12-14] and *X*-ray crystallography.[5-8] The only *X*-ray studies available for comparison (see footnote *a* to Table 6) involve unsym-

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[25] D. W. J. Cruickshank, *Tetrahedron*, 1962, **17**, 155, and references therein.

[26] D. O. Hughes and R. W. H. Small, *Acta Cryst.*, 1972, **B28**, 2520.

metrically substituted derivatives and for two of these [(8) and (9) in Table 6] the quoted standard deviations are high. However, to facilitate comparison, bond lengths and angles, and torsion angles have been averaged on the basis of C_{2v} symmetry and the maximum deviations from the mean listed in parentheses. It is clear from the results of the more accurate *X*-ray determinations [(10) and (11)] that significant deviations from the mean values occur, due, as had been noted [9] previously, to the adaptation of the bicyclo[2.2.1]-heptane system to the strain induced by the substituents. Nevertheless, the mean values from the four *X*-ray studies are in remarkably good agreement with one another and with those from the electron-diffraction studies and the theoretical calculations. In the three camphane derivatives, [(8), (9), (11)], the C(1)–C(7) bond is comparatively long, while in the norbornanol (10) the longest bond is C(2)–C(3), similar to our results for the decafluoronorbornane (14).

TABLE 6

Comparison of molecular dimensions of norbornanes [a] and camphanes.[a] Bond lengths (Å) have been rounded off to two decimal places, angles (deg.) to the nearest whole number. C_{2v} Symmetry is assumed for the bicyclo[2.2.1]heptane system. *X*-Ray results have been averaged accordingly. Figures in parentheses are the maximum deviations from the mean values. Estimated standard deviations are representative of the values quoted in the original publications and do not refer to the averaged dimensions listed for the *X*-ray analyses

	(1) [b]	(2) [c]	(3) [d]	(4) [e]	(5) [f]	(6) [g,h]	(7) [c]	(8) [i]	(9) [j]	(10) [k]	(11) [l]	(12)	(13)	(14)
(i) Bond lengths														
σ	0·02	0·015	0·025				0·015	0·045	0·03	0·008	0·002			0·007
C(1)–C(2)	1·54	1·56	1·53	1·54	1·52	1·54	1·56	1·56(3)	1·54(7)	1·54(2)	1·54(2)	$1{\cdot}54_3$	$1{\cdot}54_3$	1·54(1)
C(1)–C(7)	1·57	1·56	1·535	1·53	1·51	1·54	1·54	1·58(2)	1·57(0)	1·54(1)	1·57(2)	$1{\cdot}55_0$	1·555	1·55(1)
C(2)–C(3)	1·54	1·55	1·58	1·55	1·53	1·55	1·55	1·56(1)	1·57(3)	1·56(1)	1·55(0)	$1{\cdot}55_4$	$1{\cdot}55_7$	1·57(1)
(ii) Bond angles														
σ	1·5	1	1·5				1	3	2	0·5	0·1			0·3
C(2)–C(1)–C(6)	107 [m]	103·5	107	106	110	110	104	111(3)	110(5)	110(1)	106(2)	108	106	110(0)
C(2)–C(1)–C(7)		101	102	103	101	101	104	101(4)	101(3)	102(3)	102(2)	102	101·5	98(1)
C(1)–C(2)–C(3)	105 [m]	104	103	103	103	103	102·5	104(3)	104(4)	103(2)	104(1)	103·5	104	105(0)
C(1)–C(7)–C(4)	93	96	95	94	94	94	93	93	93	93·5	94	94	95	98
(iii) Torsion angles [n]														
σ			1 [o]				1 [o]	4 [o]	4 [o]	1 [o]	0·3 [o]			0·5
6-1-2-3		71	72 [o]			71	73	70(2)	70(3)	71(7)	71(1)	71	71·5	68(0)
1-2-3-4	0	0	0	0	0	0	0	3(2)	4(2)	7(2)	1(1)	1·4	0	0(0)
7-1-2-3		33·5	35 [o]			36	35	36(5)	36(7)	36(8)	35(2)	35	34	34(1)
2-1-7-4		53	55 [o]			56	54	57(4)	56(6)	57(4)	55(2)	55	54	56(1)
Flap angle [p]	113	108					108	115	114	114	110·5	112	110·5	116

[a] Excluded are (i) molecules containing other rings fused to the norbornane skeleton and (ii) those containing sp^2-hybridised carbon atoms within the skeleton. [b] Ref. 2. [c] Ref. 3. [d] Ref. 4. [e] Ref. 12. [f] Ref. 13. [g] Ref. 9. [h] Ref. 14. [i] Ref. 5. [j] Ref. 6. [k] Ref. 7. [l] Ref. 8. [m] From ref. 3. [n] Absolute values, ignoring signs. [o] From ref. 14. [p] Angle between mean planes of C(1)—(4) and C(1), C(4)—(6). (1)—(3) Norbornane (electron diffraction); (4)—(6) norbornane (theoretical calculation); (7) 1,4-dichloronorbornane (electron diffraction); (8) (+)-10-bromo-2-chloro-2-nitrosocamphane (*X*-ray). Averages taken over the two independent molecules in the unit cell; (9) (−)-2-bromo-2-nitrocamphane (*X*-ray); (10) 3-(*N*-benzyl-*N*-methylaminomethyl)-2-nornbornanol (*X*-ray); (11) 1-biapocamphane (*X*-ray); (12) mean of results (1)—(11); (13) mean of results (1)—(3); (14) present work, uncorrected values for bond lengths.

In an attempt to obtain representative dimensions for the norbornane skeleton we have calculated the means of the values (1)—(11) and listed these under (12), and also under (13), the means of the electron-diffraction studies on norbornane [(1)—(3)]. These two sets of values are in very good agreement, lengths differing by <0·01 Å and angles by an average of 1°, with only C(2)–C(1)–C(6) differing by as much as 2°. The first, overall average, is certainly rather arbitrary and undoubtedly torsion angle C(1)–C(2)–C(3)–C(4) would be 0, and not 1·4°, in the idealised norbornane system.

Bond lengths for the fluorinated compound are in good agreement with both sets of mean values. The C(2)–C(3) bond length (1·567 Å) is rather greater, but the order C(2)–C(3) > C(1)–C(7) > C(1)–C(2) is the same. Of the bond angles, two differ markedly from both averages. C(2)–C(1)–C(7) is smaller by 3½—4°, whereas the bridge angle C(1)–C(7)–C(4) is greater by 3—4°. Angle C(2)–C(1)–C(6) differs by 4° from the mean value derived from the electron-diffraction studies on norbornane but by only 2° from the overall mean. One torsion angle, C(6)–C(1)–C(2)–C(3), is smaller by 3—3½°, the other differ by ⩽2°. The flap angle is 115·7°, 4—5° greater than the mean, so that the fluoronorbornane skeleton appears to be somewhat flatter than the parent hydrocarbon, probably another manifestation of the F(2n) · · · F(6n) repulsive interactions. A fuller assessment of these differences must, however, await a more accurate knowledge of the geometry of the parent hydrocarbon.

The two benzoyl groups are symmetrically oriented, being related to within *ca.* 14° by the norbornane C_2 axis. Thus torsion angles C(7)–C(1)–C(8)–C(9) and C(7)–C(4)–C(15)–C(16) are 167·9 and 158·0°, respectively, and C(1)–C(8)–C(9)–C(14) and C(4)–C(15)–C(16)–C(21), 18·4 and 32·5°. The planes of the keto-groups and the phenyl rings are roughly parallel to the C(1), C(7), C(4) plane. Relevant dihedral angles are given in Table 4.

Non-bonded contacts between the fluoronorbornane system and the benzoyl groups are in Table 3(*b*). There are nine distances which are smaller than the sum of the van der Waals radii[27] by 0·1—0·3 Å. These involve two C $\cdots$ F, three O $\cdots$ F, and four H $\cdots$ F contacts, the shortest within each category being 2·984, 2·752, and 2·20 Å. Hydrogen atom H[C(14)] in its calculated position is roughly equidistant from F(2n) and F(6n) at 2·20 and 2·35 Å, a geometry which may be indicative of a bifurcated C–H $\cdots$ F hydrogen bond. Similarly, H[C(21)] is roughly equidistant from F(3n) and F(5n), the distances being 2·40 and 2·29 Å. These interactions are, however, weak compared to the non-bonded interactions within the fluoronorbornane system and would probably only have a minimal effect on its geometry.

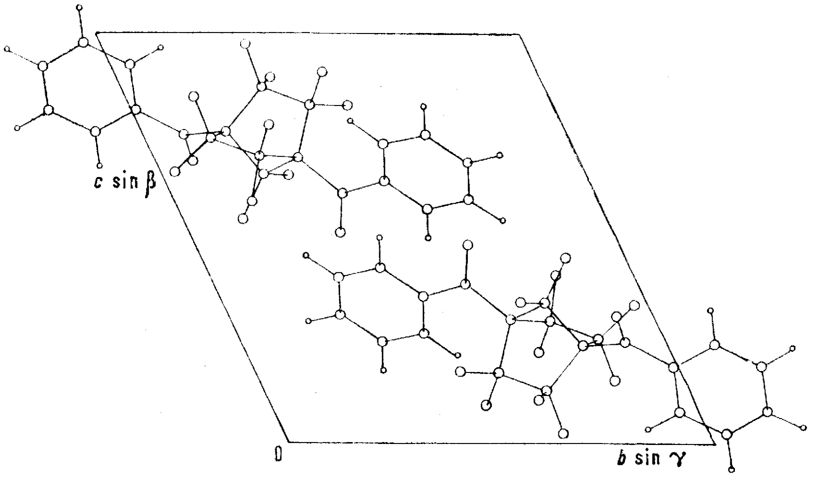

FIGURE 2 The crystal structure projected along the *a* axis

TABLE 7

Shorter intermolecular contacts (Å)

C(18) $\cdots$ C(10$^{\text{I}}$)	3·44	F(7b) $\cdots$ F(2n$^{\text{V}}$)	3·20
C(20) $\cdots$ F(3n$^{\text{II}}$)	3·38	F(5x) $\cdots$ F(5n$^{\text{IV}}$)	3·31
C(18) $\cdots$ F(6x$^{\text{I}}$)	3·39	F(5x) $\cdots$ F(6n$^{\text{IV}}$)	3·40
C(13) $\cdots$ F(5n$^{\text{III}}$)	3·42	F(5n) $\cdots$ F(6n$^{\text{III}}$)	3·42
C(19) $\cdots$ F(3n$^{\text{II}}$)	3·50	F(5n) $\cdots$ F(6x$^{\text{IV}}$)	3·43
C(14) $\cdots$ F(5n$^{\text{III}}$)	3·50	F(6n) $\cdots$ F(2n$^{\text{III}}$)	3·50
C(5) $\cdots$ F(5x$^{\text{IV}}$)	3·50	H[C(18)] $\cdots$ F(6x$^{\text{I}}$)	2·59
F(6n) $\cdots$ F(6n$^{\text{III}}$)	2·84	H[C(20)] $\cdots$ F(3n$^{\text{II}}$)	2·62
F(5x) $\cdots$ F(2n$^{\text{V}}$)	2·96	H[C(13)] $\cdots$ F(5n$^{\text{III}}$)	2·73
F(5x) $\cdots$ F(5x$^{\text{IV}}$)	3·06	H[C(18)] $\cdots$ H[C(13$^{\text{VI}}$)]	2·43
F(5x) $\cdots$ F(6x$^{\text{IV}}$)	3·18	H[C(21)] $\cdots$ H[C(13$^{\text{III}}$)]	2·44

Superscripts refer to the following equivalent positions:

I	$x, y - 1, z$	IV	$-x, 1 - y, -z$
II	$1 - x, -y, -z$	V	$x - 1, y, z$
III	$1 - x, 1 - y, -z$	VI	$x - 1, y - 1, z$

The packing of the molecules is illustrated in Figure 2. Shorter intermolecular distances are in Table 7. None of these is substantially shorter than the sum of the van der Waals radii of the atoms concerned.

We thank Dr. R. Stephens for suggesting this problem and providing suitable crystals, Professor H. M. Powell for the use of the automatic diffractometer at the Chemical Crystallography Laboratory, Oxford, and Professors M. Stacey, J. C. Robb, and J. C. Tatlow for their interest in this work.

[3/1508 *Received*, 17*th July*, 1973]

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