to hydrogen-bonded dimers. This is a feature observed in all fenamate structures. The structure also contains, in common with other fenamates, an intramolecular hydrogen bond between the carbonyl group of the carboxyl acid moiety and the imino group that bridges the two six-membered rings. The parameters for both the hydrogen bonds are listed in Table 2.

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Structure of 2*H*,2′*H*-Octafluorobiphenyl*

BY J. BOWEN JONES AND D. S. BROWN

*Department of Chemistry, Loughborough University of Technology, Loughborough LE*11 3*TU, England*

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Abstract. $C_{12}H_2F_8$, orthorhombic, *Pbca*, $a = 21.710\,(5)$, $b = 7.545\,(5)$, $c = 12.79\,(1)$ Å, $Z = 8$, $\lambda(\mathrm{Cu}\,K\alpha) = 1.5418$ Å, $\mu = 2.00$ mm^{-1}. $R = 0.045$ for 1078 observed reflexions with $I > 3\sigma(I)$. The dihedral angle between the rings is 54·7 (4)° and the inter-ring bond length is 1·491 (5) Å (uncorrected for thermal libration).

Introduction. The inter-ring bond length and dihedral angle in perfluorobiphenyl (Gleason & Britton, 1976) are virtually unaffected by 2-substitution with hydrogen (Hamor & Hamor, 1978*a*). This analysis investigates the effect on these two parameters of 2,2′-disubstitution with hydrogen and is the second in a series of studies on the correlation between inter-ring bond length and dihedral angle in substituted perfluoropolyphenyls.

The compound was prepared by the method of Cohen & Massey (1966). Colourless crystals were obtained from ethanol and the selected crystal (1·1 × 0·3 × 0·15 mm) was mounted in a sealed Lindemann-glass capillary.

The intensities and cell dimensions were obtained from a Stoe Stadi-2 two-circle diffractometer by an ω scan with graphite-monochromated Cu $K\alpha$ radiation. Of 1539 reflexions, measured out to a maximum 2θ of 135°, 1078 had $I > 3\sigma(I)$ and were classed as observed. Corrections were applied for Lp but not for absorption or extinction.

The structure was solved by direct methods with *MULTAN* (Germain, Main & Woolfson, 1971). It was refined by full-matrix least squares using the XRAY system (1972) to a conventional R of 0·045. Anisotropic temperature factors were used for C and F, but the H atoms were placed in calculated positions and not refined. Calculated shifts were $<0.1\sigma$ in the final cycle of refinement with 1252 contributing reflexions. The weighting scheme used was $w = 1$ for $F_o \leq 30$ and $w = (30/F_o)^2$ for $F_o > 30$. Scattering factors were those of Cromer & Mann (1968) for C and F and of Stewart, Davidson & Simpson (1965) for H.†

* Structures of Substituted Perfluoropolyphenyls. II.

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36224 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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All calculations were carried out with *MULTAN* and the XRAY system (1972) implemented at the University of Manchester Regional Computer Centre.

Discussion. Final atomic coordinates are listed in Table 1, bond lengths and angles in Table 2, and displacements of atoms from phenyl-ring planes in Table 3. The molecular structure and atom numbering are given in Fig. 1, and the projection of the cell contents down **c** is shown in Fig. 2.

In contrast to 2*H*-nonafluorobiphenyl there is no evidence for positional disorder of *ortho* F and H atoms. The phenyl rings are planar to within ±0·005 (4) Å with substituents displaced by no more than

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

B_{eq} is given by $\frac{1}{3}(B_{11} + B_{22} + B_{33})$.

	x	y	z	B_{eq} (Å^2)
C(1)	−982 (1)	1448 (5)	3920 (3)	3·4 (3)
C(2)	−724 (2)	2415 (5)	4745 (3)	3·9 (4)
C(3)	−92 (2)	2516 (5)	4838 (3)	4·1 (4)
C(4)	289 (2)	1683 (5)	4135 (4)	4·5 (4)
C(5)	44 (2)	717 (5)	3333 (3)	4·3 (4)
C(6)	−589 (2)	612 (5)	3231 (3)	4·0 (3)
C(1′)	−1665 (2)	1314 (5)	3812 (3)	3·3 (3)
C(2′)	−2023 (2)	741 (5)	4647 (3)	3·7 (4)
C(3′)	−2654 (2)	661 (5)	4542 (3)	3·9 (4)
C(4′)	−2938 (2)	1156 (5)	3633 (4)	4·2 (4)
C(5′)	−2596 (2)	1719 (6)	2799 (3)	4·4 (4)
C(6′)	−1958 (2)	1799 (5)	2898 (3)	3·8 (4)
H(2)	−1011	3060	5281	
H(2′)	−1810	388	5345	
F(3)	159 (1)	3423 (3)	5643 (2)	6·1 (3)
F(4)	908 (1)	1794 (4)	4248 (2)	6·3 (3)
F(5)	413 (1)	−161 (4)	2670 (2)	6·5 (3)
F(6)	−814 (1)	−373 (3)	2437 (2)	5·6 (3)
F(3′)	−3007 (1)	94 (4)	5345 (2)	5·6 (3)
F(4′)	−3556 (1)	1067 (4)	3545 (2)	6·4 (3)
F(5′)	−2864 (1)	2209 (4)	1909 (2)	6·6 (3)
F(6′)	−1637 (1)	2441 (4)	2081 (2)	5·4 (3)

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)–C(2)	1·400 (5)	C(1′)–C(2′)	1·390 (5)
C(1)–C(6)	1·380 (5)	C(1′)–C(6′)	1·381 (5)
C(1)–C(1′)	1·491 (5)	C(2′)–C(3′)	1·380 (5)
C(2)–C(3)	1·378 (5)	C(3′)–C(4′)	1·368 (6)
C(3)–C(4)	1·374 (6)	C(3′)–F(3′)	1·350 (5)
C(3)–F(3)	1·351 (5)	C(4′)–C(5′)	1·368 (6)
C(4)–C(5)	1·367 (6)	C(4′)–F(4′)	1·348 (4)
C(4)–F(4)	1·353 (4)	C(5′)–C(6′)	1·392 (5)
C(5)–C(6)	1·382 (5)	C(5′)–F(5′)	1·330 (5)
C(5)–F(5)	1·342 (5)	C(6′)–F(6′)	1·346 (4)
C(6)–F(6)	1·350 (5)		

C(2)–C(1)–C(6)	118·1 (3)	C(1)–C(1′)–C(2′)	120·3 (3)
C(2)–C(1)–C(1′)	120·2 (3)	C(1)–C(1′)–C(6′)	121·3 (3)
C(6)–C(1)–C(1′)	121·6 (3)	C(2′)–C(1′)–C(6′)	118·4 (3)
C(1)–C(2)–C(3)	119·5 (3)	C(1′)–C(2′)–C(3′)	119·6 (3)
C(2)–C(3)–C(4)	121·2 (4)	C(2′)–C(3′)–C(4′)	121·2 (4)
C(2)–C(3)–F(3)	119·7 (3)	C(2′)–C(3′)–F(3′)	120·2 (3)
C(4)–C(3)–F(3)	119·1 (3)	C(4′)–C(3′)–F(3′)	118·6 (3)
C(3)–C(4)–C(5)	120·0 (3)	C(3′)–C(4′)–C(5′)	120·2 (3)
C(3)–C(4)–F(4)	120·1 (4)	C(3′)–C(4′)–F(4′)	120·4 (4)
C(5)–C(4)–F(4)	119·9 (4)	C(5′)–C(4′)–F(4′)	119·4 (4)
C(4)–C(5)–C(6)	119·2 (4)	C(4′)–C(5′)–C(6′)	118·9 (4)
C(4)–C(5)–F(5)	120·4 (3)	C(4′)–C(5′)–F(5′)	121·0 (3)
C(6)–C(5)–F(5)	120·4 (4)	C(6′)–C(5′)–F(5′)	120·1 (4)
C(5)–C(6)–C(1)	121·9 (4)	C(5′)–C(6′)–C(1′)	121·6 (3)
C(5)–C(6)–F(6)	117·6 (3)	C(5′)–C(6′)–F(6′)	117·4 (3)
C(1)–C(6)–F(6)	120·5 (3)	C(1′)–C(6′)–F(6′)	120·9 (3)

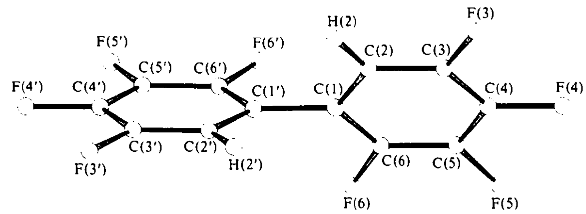

Fig. 1. *ORTEP* plot (Johnson, 1965) of the title compound, with atom labelling.

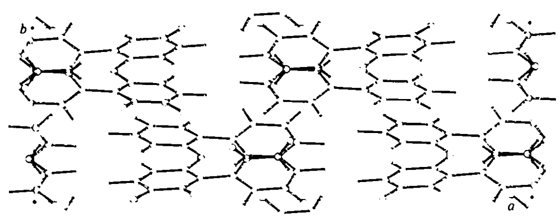

Fig. 2. Projection of the cell contents down **c**.

Table 3. *Displacements* (Å) *of atoms from least-squares planes*

Ring (I) C(1)–C(6)		Ring (II) C(1′)–C(6′)	
*C(1)	−0·004 (4)	C(1)	0·039 (5)
*C(2)	0·003 (4)	C(4)	0·106 (5)
*C(3)	0·002 (4)	*C(1′)	−0·000 (3)
*C(4)	−0·005 (4)	*C(2′)	0·002 (3)
*C(5)	0·004 (4)	*C(3′)	−0·004 (3)
*C(6)	0·001 (4)	*C(4′)	0·004 (3)
C(1′)	0·007 (5)	*C(5′)	−0·002 (3)
C(4′)	−0·012 (6)	*C(6′)	0·000 (3)
F(3)	0·031 (5)	F(3′)	−0·013 (4)
F(4)	0·002 (5)	F(4′)	−0·003 (4)
F(5)	0·054 (5)	F(5′)	0·009 (4)
F(6)	0·027 (5)	F(6′)	0·061 (4)

Equations of the planes (x, y and z are fractional coordinates)

(I) $-0{\cdot}116x - 6{\cdot}172y + 7{\cdot}356z = 2{\cdot}005$

(II) $-1{\cdot}506x + 7{\cdot}098y + 4{\cdot}246z = 2{\cdot}801$

Angle between planes (I) and (II) = 54·7 (4)°

Parametric equation of line (I), C(1)–C(1′):
$x = -0{\cdot}132 + 0{\cdot}046t$; $y = 0{\cdot}138 + 0{\cdot}009t$; $z = 0{\cdot}387 + 0{\cdot}007t$

Parametric equation of line (II), C(4)–C(1):
$x = -0{\cdot}035 + 0{\cdot}046t$; $y = 0{\cdot}157 + 0{\cdot}008t$; $z = 0{\cdot}403 + 0{\cdot}008t$

Parametric equation of line (III), C(1′)–C(4′):
$x = -0{\cdot}230 + 0{\cdot}046t$; $y = 0{\cdot}123 + 0{\cdot}006t$; $z = 0{\cdot}372 + 0{\cdot}006t$

Angle between lines (I) and (II) = 0·4 (3)°

Angle between lines (I) and (III) = 1·6 (3)°

*Atoms defining the plane.

0·061 (4) Å. The inter-ring bond length and dihedral angle are 1·491 (5) Å and 54·7 (4)° respectively, compared with 1·486 Å and 59·5° in 2*H*-nonafluorobiphenyl. Atoms C(4), C(1), C(1') and C(4') are coplanar, none being displaced by more than 0·005 (4) Å from their least-squares plane. However, they are not collinear, the ring axes C(1)—C(4) and C(1')—C(4') being inclined to the C(1)—C(1') bridge axis at 0·4 (3) and 1·6 (3)° respectively, with C(4) and C(4') *cis*. No such distortion has been reported for 2*H*-nonafluorobiphenyl.

The H atoms are *cis*, precluding intramolecular H···F interactions. In contrast, the Br atoms in 2,2'-dibromooctafluorobiphenyl are *trans* (Hamor & Hamor, 1980). The F(6)···F(6') intramolecular contact is 2·812 (4) Å and repulsion between the *cis ortho* F atoms is indicated by the fact that C(1)—C(6)—F(6) is 2·9° greater than C(5)—C(6)—F(6) and C(1')—C(6')—F(6') is 3·5° greater than C(5')—C(6')—F(6').

Principal intermolecular contacts are given in Table 4. The C(2')···F(4^{v}) and C(2)···F(3'^{vi}) intermolecular contacts of 3·393 (4) and 3·503 (4) Å would allow the possibility of very weak intermolecular hydrogen bonding. However, there are two shorter C···F intermolecular contacts, *viz* 3·129 (5) and 3·206 (5) Å, where hydrogen bonding is precluded. The shortest F···F intermolecular contact is 2·870 (4) Å, which is similar to that in 2,2'-dibromooctafluorobiphenyl (2·85 Å).

Intra-ring C—C lengths lie within the range 1·367 to 1·400 Å with a mean of 1·380 Å. C—F lengths range from 1·330 to 1·353 Å with a mean of 1·346 Å. These ranges and mean values are similar to those for C—F and intra-ring C—C lengths in perfluorobiphenyl and its derivatives (Gleason & Britton, 1976; Hamor & Hamor, 1978*a*,*b*, 1980).

Inter-ring bond lengths and dihedral angles in perfluorobiphenyl and a few of its *ortho*-substitution products are given, for comparison, in Table 5, a more comprehensive version of which has been published by Goodhand, Hamor & Hamor (1978). Replacement of F by H in the 2-position in perfluorobiphenyl has virtually no effect on the dihedral angle, whereas replacement in the 2- and 2'-positions reduces the angle by about 5°. 2-substitution with NO_2 and 2,2'-disubstitution with Br produce a marked increase in the dihedral angle (Hamor & Hamor, 1978*b*, 1980). In contrast, the inter-ring bond length is little affected by the nature of the *ortho* substituent and lies within the narrow range 1·486 (5) to 1·492 (4) Å.

No data are available for 2-bromononafluorobiphenyl. However, 1-bromo-3,4,5-trifluoro-2,6-bis-(pentafluorophenyl)benzene (Bowen Jones & Brown, 1980) may be regarded as 2-bromo-4-(pentafluorophenyl)octafluorobiphenyl. The dihedral angle in the biphenyl system of this compound is 78·3 (7)°, about 2·5° greater than in 2,2'-dibromooctafluorobiphenyl and the inter-ring bond length is 1·47 (1) Å, only slightly less than in perfluorobiphenyl and its derivatives listed in Table 5.

Clearly, for these compounds, there is no direct or inverse relationship between inter-ring bond length and dihedral angle, a conclusion reached by Goodhand *et al.* (1978) after their examination of a number of polyfluorobiphenyls.

Table 4. *Principal intermolecular contacts* (Å)

F(3')···F(5'^{i})	2·870 (4)	F(3)···C(3^{iii})	3·129 (5)
F(5)···F(4'^{ii})	2·878 (4)	C(4)···F(6^{iv})	3·206 (5)
F(3)···F(5^{i})	2·958 (4)	C(2')···F(4^{v})	3·393 (4)
F(3)···F(3^{iii})	2·974 (4)	C(2)···F(3'^{vi})	3·503 (4)

Symmetry code: (i) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (ii) $\frac{1}{2}+x, y, \frac{1}{2}-z$; (iii) $-x, 1-y, 1-z$; (iv) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (v) $-x, -y, 1-z$; (vi) $-\frac{1}{2}-x, \frac{1}{2}+y, z$.

Table 5. *Inter-ring bond lengths and dihedral angles*

	Inter-ring bond length (Å)	Dihedral angle (°)
Perfluorobiphenyl	1·486 (5)	59·6
2*H*-Nonafluorobiphenyl	1·486 (4)	59·5
2-Nitrononafluorobiphenyl	1·492 (4)	85·5
2*H*, 2'*H*-Octafluorobiphenyl	1·491 (5)	54·7 (4)
2,2'-Dibromooctafluorobiphenyl	1·489 (7)	75·9 (5)

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