

Table 3. Average intra- and intermolecular non-bonded distances (Å)

The quantity following each slash is the number of distances averaged for the contact.

	C_s	C_2
Intramolecular		
F	2.780/20	2.846/20
O(C—NO ₂)	2.909/64	2.890/60
O(N—NO ₂)	2.834/16	2.845/16
Intermolecular		
F	3.493/10	3.364/13
O(C—NO ₂)	3.391/65	3.338/71
O(N—NO ₂)	3.433/20	3.402/20

On the basis of previous work, AGGE had proposed that the more 'a molecule is stressed, the more loosely it is packed in the crystal'.* In support of this hypothesis (see *Introduction*), they tabulated the average intra- and intermolecular contacts for the F and O atoms in the two conformers, which showed that the conformer (C_s) with the longer intramolecular contacts is the same one that is associated with the shorter intermolecular contacts. Unfortunately, the precise boundary conditions used in these tabulations were not reported and we have been unable to repeat their calculations exactly with our atomic-coordinate data. Our calculations were carried out in the following manner: the intra- and intermolecular contacts were limited to those less than 3.8 Å, and no more than 10 of the shortest contacts to a particular atom were used to construct the appropriate non-bonded contact averages. These data, which are summarized in Table 3, show no significant trends that could be interpreted as support for the AGGE hypothesis. We find, in fact, that it is the C_s conformer, not the C_2 conformer, which is involved in the shorter intramolecular/longer intermolecular contacts. The

* This quotation is from a translation of the original Russian paper. We believe that the word 'strain' would be more in line with common chemical usage than 'stress'. The inference here is to intramolecular, non-bonded contacts.

tabulated data further show that it is primarily the F...F contacts that provide a basis for concluding that the C_s conformer is associated with the shorter intramolecular contacts.

These data, in conjunction with our calculated molecular volumes, indicate that there are no significant differences between the intra- and intermolecular environments of the two molecules. While the hypothesis of AGGE in the matter of the saturability of van der Waals contacts may be valid, it is our conclusion that the crystal structure in question cannot be used to support this idea.

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3'-Fluorobiphenyl-4-carboxylic Acid, C₁₃H₉FO₂

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Abstract. $M_r = 216.2$, monoclinic, $P2_1/c$, $a = 3.92$ (1), $b = 8.04$ (1), $c = 32.50$ (2) Å, $\beta = 101.6$ (1)°, $V = 1003.4$ Å³, $Z = 4$, $D_m = 1.42$, $D_x = 1.43$ Mg m⁻³, Cu K α , $\lambda = 1.5418$ Å, $\mu = 0.927$ mm⁻¹, $F(000) = 448$, $T = 293$ K, $R = 0.087$ for 899 observed reflexions. The

structure was solved by Patterson synthesis from densitometer and visually measured equi-inclination Weissenberg data. The average C—C bond in the phenyl ring is 1.389 Å. The molecule is non-planar; the angle between the phenyl rings is 36.3 (0.6)° and the

acid is rotated about its C—C bond by $3.7(0.5)^\circ$. There is also distortion in the bond angles at the C atom bonded to F, with the internal angle $124.3(1.2)^\circ$; the C—F bond is $1.363(13)$ Å and makes an angle of $1.4(0.4)^\circ$ with the phenyl plane. Dimers formed from the molecules by an O—H...O hydrogen bond of $2.623(13)$ Å pack in a herring-bone arrangement with the F atoms lying in sheets parallel to (001).

Introduction. The structure determination of 3'-fluorobiphenyl-4-carboxylic acid forms part of an investigation into liquid-crystal compounds and their chemical precursors.

Experimental. D_m measured by flotation using aqueous cadmium *n*-dodecatungstaborate. Colourless transparent crystals used in data collection about **a** had dimensions $0.45 \times 0.03 \times 0.15$ mm and about **b** $0.2 \times 0.2 \times 0.4$ mm. Data for 1102 reflexions measured on a Joyce-Loebl flying-spot densitometer from multiple-film Weissenberg photographs using Cu $K\alpha$ radiation; range of hkl : $0 \leq h \leq 3$; $0 \leq k \leq 9$; $-37 \leq l \leq 35$. Data merged to give 904 unique observed reflexions; $R_{\text{int}} = 0.07$. Structure solved by Patterson synthesis and refined by block diagonal least squares with anisotropic temperature factors for the non-hydrogen atoms. H-atom positions, initially obtained from a difference synthesis and placed at geometrically reasonable positions, were refined with isotropic thermal parameters. Five reflexions (102, 104, $11\bar{3}$, $11\bar{1}$ and 212) appeared to suffer from extinction and were removed from the refinement. Final $R = 0.087$ and $wR = 0.1$ for 899 reflexions, $w = 1/[2|F_{\text{min}}| + |F_o| + 2|F_o|^2/|F_{\text{max}}| + 5|F_o|^3/|F_{\text{max}}|^2]$, $(\Delta/\sigma)_{\text{max}}$ in final refinement cycle 0.1 for positional and 0.3 for thermal parameters, max. and min. height in final $\Delta\rho$ map $+0.32$ and -0.48 e Å $^{-3}$. Scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Table 1* gives atomic parameters and Table 2 bond lengths and angles. The atomic numbering is shown in Fig. 1.

The phenyl rings are planar to within ± 0.019 Å, with an average C—C bond of 1.389 Å. In several of the 2' and 3' halogen-substituted biphenyls the halogen atom has been found to be displaced out of the ring plane; the F atom in the present structure is displaced by $0.041(9)$ Å, the C(3)—F bond being inclined at $1.4(0.4)^\circ$ to the phenyl ring; C(7) is displaced from the ring C(1)—C(6) by $0.043(12)$ Å. The C(3)—F bond,

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, intermolecular contact distances and details of mean-plane calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38681 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{Å}^2 \times 10^3$) with e.s.d.'s in parentheses

$$U_{\text{eq}} = (U_{11}U_{22}U_{33})^{1/3}.$$

	x	y	z	U_{eq}
C(1)	1170 (30)	-1687 (14)	1528 (3)	41 (6)
C(2)	2642 (31)	-1440 (15)	1952 (3)	43 (6)
C(3)	2287 (32)	-2695 (16)	2228 (4)	47 (7)
C(4)	525 (33)	-4149 (16)	2115 (4)	51 (7)
C(5)	-972 (34)	-4380 (15)	1693 (4)	51 (7)
C(6)	-594 (31)	-3148 (16)	1405 (4)	48 (7)
C(7)	1646 (29)	-373 (14)	1219 (3)	41 (6)
C(8)	2107 (30)	-837 (14)	820 (3)	39 (6)
C(9)	2743 (32)	357 (15)	541 (3)	44 (6)
C(10)	2814 (30)	2051 (14)	648 (3)	40 (6)
C(11)	2297 (32)	2521 (15)	1042 (3)	45 (6)
C(12)	1632 (31)	1312 (15)	1323 (3)	43 (6)
C(13)	3650 (31)	3327 (15)	364 (4)	44 (6)
O(1)	4287 (24)	2865 (11)	10 (3)	60 (5)
O(2)	3829 (26)	4829 (11)	470 (3)	60 (5)
F	3825 (22)	-2466 (10)	2639 (2)	63 (5)

Table 2. Bond lengths (Å) and bond angles ($^\circ$) with e.s.d.'s in parentheses

C(1)—C(2)	1.397 (15)	C(7)—C(8)	1.396 (16)
C(1)—C(6)	1.381 (17)	C(8)—C(9)	1.377 (16)
C(1)—C(7)	1.495 (16)	C(9)—C(10)	1.405 (16)
C(2)—C(3)	1.376 (17)	C(10)—C(11)	1.389 (16)
C(3)—F	1.363 (13)	C(10)—C(13)	1.461 (17)
C(3)—C(4)	1.370 (18)	C(11)—C(12)	1.394 (17)
C(4)—C(5)	1.392 (17)	C(13)—O(1)	1.281 (15)
C(5)—C(6)	1.391 (18)	C(13)—O(2)	1.254 (15)
C(7)—C(12)	1.397 (17)		
C(1)—C(2)—C(3)	117.6 (1.1)	C(8)—C(7)—C(12)	119.5 (1.0)
C(2)—C(3)—C(4)	124.3 (1.2)	C(7)—C(8)—C(9)	120.0 (1.0)
C(2)—C(3)—F	117.2 (1.1)	C(8)—C(9)—C(10)	120.7 (1.1)
C(4)—C(3)—F	118.5 (1.1)	C(9)—C(10)—C(11)	119.5 (1.1)
C(3)—C(4)—C(5)	117.9 (1.2)	C(10)—C(11)—C(12)	119.7 (1.1)
C(4)—C(5)—C(6)	119.2 (1.2)	C(11)—C(12)—C(7)	120.5 (1.1)
C(5)—C(6)—C(1)	121.7 (1.1)	C(9)—C(10)—C(13)	121.4 (1.0)
C(6)—C(1)—C(2)	119.3 (1.1)	C(11)—C(10)—C(13)	119.0 (1.0)
C(6)—C(1)—C(7)	122.0 (1.0)	C(10)—C(13)—O(1)	118.2 (1.0)
C(2)—C(1)—C(7)	118.7 (1.0)	C(10)—C(13)—O(2)	120.7 (1.1)
C(1)—C(7)—C(8)	119.5 (1.0)	O(1)—C(13)—O(2)	121.1 (1.1)
C(1)—C(7)—C(12)	121.1 (1.0)		

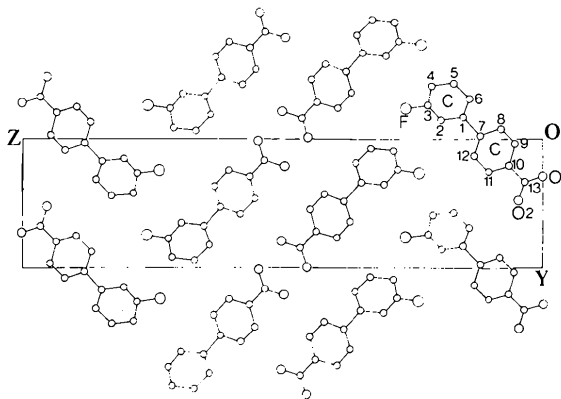


Fig. 1. The arrangement of the molecules in the unit cell viewed along a.

1.363 (13) Å, is in good agreement with the value of 1.369 (8) Å determined in 4-acetyl-2'-fluorobiphenyl (Young, Tollin & Sutherland, 1968). The distribution of bond angles around the C atom bonded to F is also similar in the two compounds. In the present structure C(2)–C(3)–C(4) is 124.3 (1.2), C(2)–C(3)–F is 117.2 (1.1) and C(4)–C(3)–F is 118.5 (1.1)° and these compare with 124.3 (0.6), 117.7 (0.7) and 118.0 (0.5)° for the comparable angles in 4-acetyl-2'-fluorobiphenyl.

The angle between the phenyl rings is 36.3 (0.6)° and in common with other halogen-substituted biphenyls the axis of the molecule defined by C(1), C(4), C(7), C(10) deviates significantly from collinearity. Not only is there a rotation by an angle ϕ_1 about the central C(1)–C(7) bond but also two other rotations: $\phi_2 = 1.6$ (0.5)° of the ring C(1)–C(6) about an axis in its plane, passing through C(1) perpendicular to C(1)–C(7); and $\phi_3 = 2.6$ (0.5)°, the corresponding angle of rotation for ring C(7)–C(12). Similar rotations have been found in a number of substituted biphenyls including 1.2 and 4.1° in 4-acetyl-3'-bromobiphenyl (Sutherland & Hoy, 1969), and 2.5 and 1.4° in 2,2'-dibromooctafluorobiphenyl (Hamor & Hamor, 1980).

The carboxyl group is rotated about the C(10)–C(13) bond through an angle ϕ_4 of 3.7 (0.5)°. Associated with this is a second rotation $\phi_5 = 3.1$ (0.5)° of the group C(10), C(13), O(1) and O(2)

about an axis in its plane passing through C(10) perpendicular to C(7)–C(10).

The various values of ϕ are larger than the corresponding values in 3'-iodobiphenyl-4-carboxylic acid (Sutherland & Mottram, 1972). These reductions are probably due to intermolecular forces since the molecules in that compound lie in a series of chains whereas they adopt a herring-bone configuration in the present structure (Fig. 1).

The O–H...O hydrogen bond of 2.623 (13) Å, the shortest intermolecular distance, is similar to the value of 2.61 Å quoted for 3'-iodobiphenyl-4-carboxylic acid.

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Iopamidol, C₁₇H₂₂I₃N₃O₈

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Abstract. $M_r = 777.088$, triclinic, $P\bar{1}$, $a = 12.478$ (4), $b = 11.233$ (3), $c = 9.241$ (3) Å, $\alpha = 104.49$ (8), $\beta = 92.63$ (7), $\gamma = 108.63$ (8)°, $V = 1177$ (1) Å³, $Z = 2$, $D_x = 2.19$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 3.986$ mm⁻¹, $F(000) = 736$, $T = 295$ K. Final $R =$

0.0544 for 2381 observed reflections. The title compound, a new, non-ionic, hydrosoluble contrast medium, shows rotational disorder at the asymmetric C atom C(8). Two I atoms deviate slightly from the benzene ring plane due to steric hindrance. The molecules are packed together by hydrogen-bond interactions between amide O atoms of symmetry-related molecules and by van der Waals forces.

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