C6	0.7247 (7)	0.0684 (5)	0.4574 (6)	8.4 (2)
C11	0.7541 (5)	0.5274 (4)	0.4101 (4)	4.2 (1)
C12	0.6955 (5)	0.4011 (4)	0.4035 (4)	4.0 (1)
C13	0.6772 (5)	0.3772 (4)	0.3037 (4)	3.8 (1)
C14	0.7136 (4)	0.4712 (4)	0.2146 (4)	2.7 (1)
C15	0.7696 (4)	0.5973 (4)	0.2162 (4)	2.5 (1)
C16	0.7844 (5)	0.6190 (4)	0.3190 (4)	3.4 (1)
C21	0.7351 (5)	0.5168 (4)	0.0224 (4)	3.4 (1)
C22	0.7178 (5)	0.4660 (4)	−0.0686 (4)	5.1 (1)
C23	0.7610 (6)	0.5395 (4)	−0.1661 (4)	5.1 (1)
C24	0.8232 (5)	0.6625 (4)	−0.1748 (4)	3.8 (1)
C25	0.8397 (5)	0.7120 (4)	−0.0827 (4)	3.1 (1)
C26	0.7940 (4)	0.6424 (4)	0.0193 (4)	3.2 (1)
C31	0.7887 (4)	0.9397 (4)	0.1275 (4)	2.9 (1)
C32	0.7218 (5)	1.0746 (4)	0.1372 (4)	3.6 (1)
C33	0.5532 (5)	1.1042 (4)	0.1469 (4)	4.1 (1)
C34	0.4564 (4)	1.0016 (4)	0.1482 (4)	3.7 (1)
C35	0.5250 (4)	0.8678 (4)	0.1372 (4)	3.0 (1)
C36	0.6935 (4)	0.8383 (3)	0.1275 (4)	2.5 (1)

Table 2. *Selected geometric parameters* (Å, °)

O1—C14	1.385 (7)	C11—C16	1.358 (7)
O1—C21	1.367 (8)	C12—C13	1.369 (9)
O2—C1	1.372 (5)	C13—C14	1.363 (8)
O2—C2	1.487 (5)	C14—C15	1.400 (8)
O3—C1	1.204 (4)	C15—C16	1.390 (7)
O4—N2	1.213 (8)	C21—C22	1.37 (1)
O5—N2	1.213 (9)	C21—C26	1.395 (8)
N1—C3	1.456 (7)	C22—C23	1.350 (9)
N1—C5	1.465 (7)	C23—C24	1.374 (9)
N1—C12	1.372 (8)	C24—C25	1.379 (9)
N2—C24	1.466 (9)	C25—C26	1.385 (8)
C1—C31	1.471 (5)	C31—C32	1.376 (5)
C2—C15	1.476 (7)	C31—C36	1.371 (5)
C2—C26	1.499 (8)	C32—C33	1.379 (6)
C2—C36	1.510 (6)	C33—C34	1.386 (6)
C3—C4	1.456 (7)	C34—C35	1.372 (5)
C5—C6	1.481 (8)	C35—C36	1.378 (5)
C11—C12	1.427 (8)		
C14—O1—C21	120.9 (8)	C2—C15—C14	123.5 (7)
C1—O2—C2	111.0 (3)	C2—C15—C16	123.1 (7)
C3—N1—C5	116.3 (6)	C14—C15—C16	113.3 (7)
C3—N1—C12	123.5 (7)	C11—C16—C15	124.9 (7)
C5—N1—C12	120.0 (7)	O1—C21—C22	115.6 (7)
O4—N2—O5	125.7 (5)	O1—C21—C26	121.2 (9)
O4—N2—C24	117.7 (9)	C22—C21—C26	123.1 (4)
O5—N2—C24	116.5 (4)	C21—C22—C23	119.1 (9)
O2—C1—O3	121.0 (4)	C22—C23—C24	120.7 (8)
O2—C1—C31	107.9 (3)	N2—C24—C23	120.0 (9)
O3—C1—C31	131.1 (4)	N2—C24—C25	120.5 (9)
O2—C2—C15	109.5 (5)	C23—C24—C25	119.5 (4)
O2—C2—C26	107.3 (4)	C24—C25—C26	122.2 (9)
O2—C2—C36	102.4 (3)	C2—C26—C21	122.4 (8)
C15—C2—C26	110.9 (5)	C2—C26—C25	122.2 (7)
C15—C2—C36	113.1 (5)	C21—C26—C25	115.4 (9)
C26—C2—C36	113.1 (5)	C1—C31—C32	129.1 (4)
N1—C3—C4	113.7 (6)	C1—C31—C36	108.8 (3)
N1—C5—C6	114.3 (5)	C32—C31—C36	122.1 (4)
C12—C11—C16	119.2 (7)	C31—C32—C33	117.0 (4)
N1—C12···C11	120.2 (8)	C32—C33—C34	121.1 (4)
N1—C12—C13	122.2 (8)	C33—C34—C35	121.3 (4)
C11—C12—C13	117.5 (9)	C34—C35—C36	117.5 (4)
C12—C13—C14	120.8 (8)	C2—C36—C31	109.8 (3)
O1—C14—C13	115.7 (7)	C2—C36—C35	129.2 (3)
O1—C14—C15	120.1 (8)	C31—C36—C35	121.0 (3)
C13—C14—C15	124.3 (7)		

The structure was solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). All H atoms were found in $\Delta\rho$ maps. The structure was refined by full-matrix least-squares calculations with anisotropic displacement factors for non-H atoms and isotropic displacement factors for H atoms. Calculations were carried out on a VAX 3100 computer using *MolEN* (Fair, 1990).

This work was supported by the Key Discipline Fund of Tianjin Higher Education, People's Republic of China.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including H-atom geometry, and least-squares-planes data have been deposited with the IUCr (Reference: AB1138). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Phenyl 2-Fluorobenzoate, Phenyl 4-Fluorobenzoate and Phenyl Benzoate

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Abstract

On comparing the crystal structures of the title compounds, two isomers of $C_{13}H_9FO_2$, and $C_{13}H_{10}O_2$, it becomes clear that the differences in the molecular arrangements and conformations of the fluorinated phenyl benzoates compared with phenyl benzoate (PB) depend on the position of the F atom pendent on the aromatic nucleus of PB, as an F atom at C(2) does not affect either the molecular arrangement or conformation as much as an F atom at C(4).

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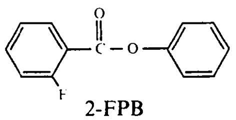
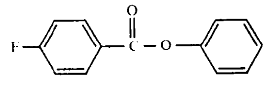
Comment

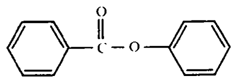
In crystal engineering, a useful method for controlling molecular arrangement is the addition of polar substituents to components. Over the past few decades a number of studies have been made on 'chlorosubstitution', in which a Cl atom pendent on an aromatic nucleus tends to 'steer' the crystal structure to the β-mode, characterized by a short axis of *circa* 4 Å (Sarma & Desiraju, 1986). In contrast, no crystallographic studies have been carried out on the use of an F atom to 'steer' the structure, even though F is also in Group VII. We have reported the crystal structure of phenyl 3-fluorobenzoate (3-FPB) previously, demonstrating that remarkable changes in both the molecular arrangement and conformation were seen compared with phenyl benzoate (PB) (Shibakami, Tamura, Arimura, Kurosawa & Sekiya, 1994). Here we wish to report the crystal structures of phenyl 2- and 4-fluorobenzoate (2-FPB and 4-FPB, respectively) and phenyl benozate (PB), the structure of which has also been reported by other workers (Adams & Morsi, 1976), demonstrating that an F atom at a particular position on an aromatic nucleus can 'steer' the molecular arrangement and conformation.



2-FPB



4-FPB



PB

The bond distances and angles in all three compounds are normal within experimental error. As shown in Figs. 1–3, the molecular conformations of the compounds are somewhat different. The dihedral angles between the two benzene rings are 122.5 (3), 56.7 (5) and 123.8 (3)° in 2-FPB, 4-FPB and PB, respectively. The corresponding angle in 3-FPB is 66.0 (5)°. The O(1)—C(7)—C(6)—C(1) dihedral angles, which represent the torsion between the C(1)—C(6) ring and the —COO group, are 163.0 (2), 177.8 (4) and 170.1 (2)° in 2-FPB, 4-FPB and PB, respectively, while the corresponding angle in 3-FPB is −171.9 (4)°. The C(7)—O(1)—C(8)—C(9) torsion angles, which represent the torsion between the C(8)–C(13) ring and the —COO group, are 76.0 (2), 62.5 (5) and 67.5 (2)° for 2-FPB, 4-FPB and PB, respectively, and the corresponding angle in 3-FPB is −72.9 (6)°. Thus, all these values, together with the structure of 3-FPB, make it clear that an F atom pendent on the aromatic nucleus of PB can 'steer' the molecular conformation, despite the slight difference in volume between the components. An F atom at

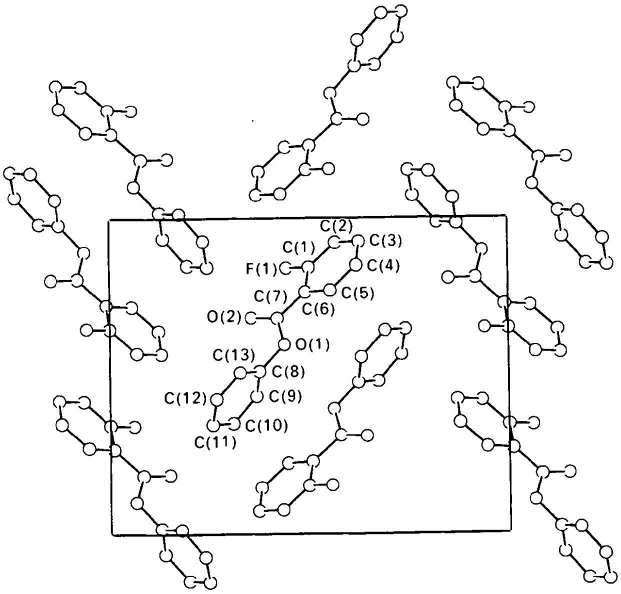


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the crystal structure of 2-FPB viewed along the *a* axis.

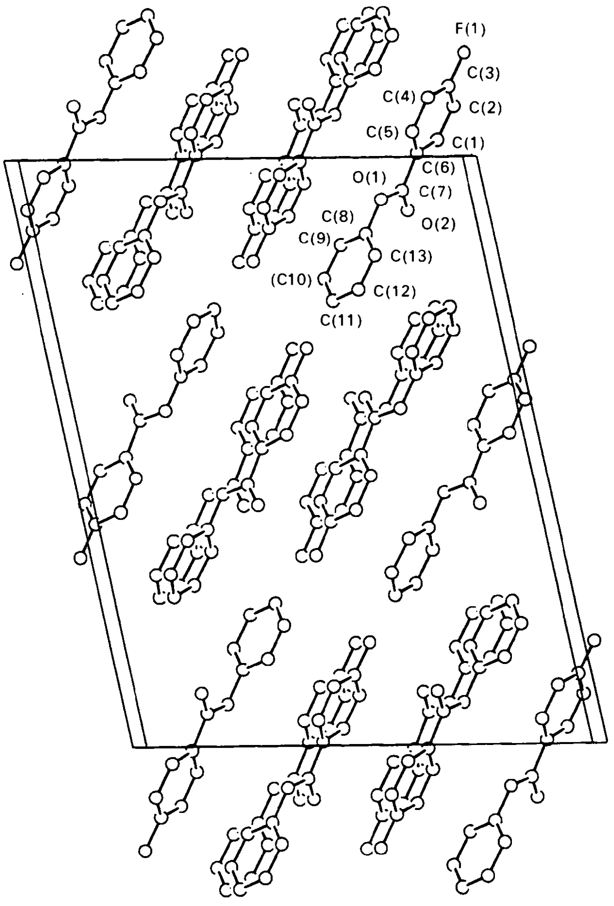


Fig. 2. *ORTEP*II (Johnson, 1976) drawing of the crystal structureof 4-FPB viewed along the *b* axis.

C(2) is exceptional in that it does not affect the conformation as much as an F atom at C(3) or C(4).

In each structure, including that of 3-FPB, it appears that two stack-related components constitute the crystal structure. The closest distances between the two components are 5.782 (1), 5.782 (1) and 5.689 (1) Å for 2-FPB, 4-FPB and PB, respectively. The corresponding distance in 3-FPB is 5.783 (1) Å. The most interesting structural feature is that the position of the F atom affects the arrangement of the molecules quite remarkably. Hence the arrangement in 4-FPB is quite different from that in PB, while in contrast 2-FPB has almost the same molecular arrangement as PB and crystallizes in the same space group. It may be worth mentioning, in passing, that the molecular arrangement in 3-FPB, which belongs to the orthorhombic space group $P2_12_12_1$, differs from that in PB, 2- and 4-FPB. In this way, we may say that an F atom at C(3) and/or C(4) of PB can indeed 'steer' the molecular arrangement, as well as the molecular conformation.

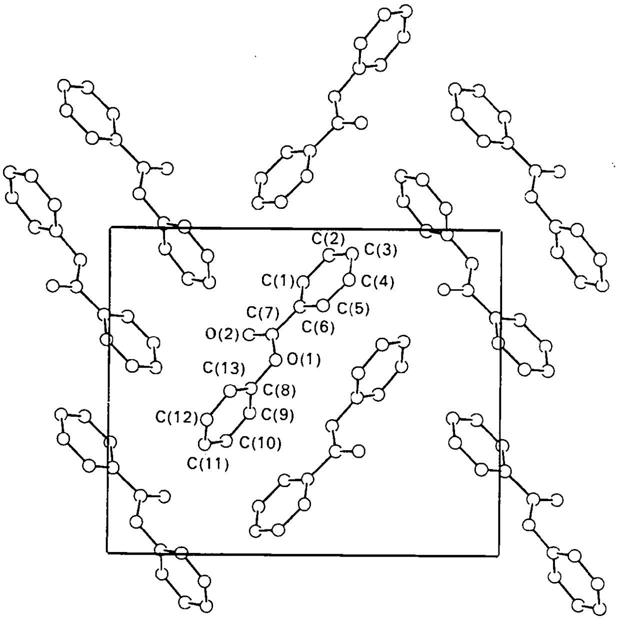


Fig. 3. *ORTEP*II (Johnson, 1976) drawing of the crystal structure of PB viewed along the *a* axis.

Experimental

2-FPB and 4-FPB were synthesized by the conventional Schotten–Baumann method (Schotten, 1884). PB was purchased from the Tokyo Chemical Industry Co., Ltd. The prismatic crystals of 2- and 4-FPB were grown from ethanol solutions of the compounds at room temperature.

2-FPB

Crystal data

$C_{13}H_9FO_2$
$M_r = 216.21$
Monoclinic
$P2_1/c$
$a = 5.782$ (1) Å
$b = 15.148$ (2) Å
$c = 12.064$ (2) Å
$\beta = 98.98$ (2)°
$V = 1043.8$ (3) Å^3
$Z = 4$
$D_x = 1.376$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 27.7$–28.5°
$\mu = 0.876$ mm^{-1}
$T = 298$ K
Prismatic
0.15 × 0.10 × 0.10 mm
Colourless

Data collection

Rigaku AFC-7*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF (Walker & Stuart, 1983)
1813 measured reflections
1636 independent reflections
1330 observed reflections $[I > 3\sigma(I)]$

$R_{\rm int} = 0.125$
$\theta_{\rm max} = 60.0$°
$h = 0 \rightarrow 6$
$k = 0 \rightarrow 16$
$l = -13 \rightarrow 13$
3 standard reflections monitored every 150 reflections intensity decay: 1.69%

Refinement

Refinement on *F*
$R = 0.046$
$wR = 0.063$
$S = 3.38$
1330 reflections
146 parameters
H-atom parameters not refined

$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{\rm max} = 0.01$
$\Delta\rho_{\rm max} = 0.15$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.17$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

4-FPB

Crystal data

$C_{13}H_9FO_2$
$M_r = 216.21$
Monoclinic
$C2/c$
$a = 16.966$ (2) Å
$b = 5.783$ (1) Å
$c = 22.038$ (1) Å
$\beta = 101.981$ (8)°
$V = 2115.0$ (5) Å^3
$Z = 8$
$D_x = 1.358$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 25.5$–28.4°
$\mu = 0.864$ mm^{-1}
$T = 298$ K
Prismatic
0.30 × 0.20 × 0.20 mm
Colourless

Data collection

Rigaku AFC-7*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF (Walker & Stuart, 1983)
2079 measured reflections
1999 independent reflections
906 observed reflections $[I > 3\sigma(I)]$

$R_{\rm int} = 0.067$
$\theta_{\rm max} = 60.0$°
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 6$
$l = -24 \rightarrow 24$
3 standard reflections monitored every 150 reflections intensity decay: 2.41%

Refinement

Refinement on *F*
$R = 0.064$
$wR = 0.072$
$S = 2.80$
906 reflections
146 parameters
H-atom parameters not refined

$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{\text{max}} = 0.01$
$\Delta\rho_{\text{max}} = 0.19$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.17$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

PB

Crystal data

$C_{13}H_{10}O_2$
$M_r = 198.22$
Monoclinic
$P2_1/c$
$a = 5.689$ (1) Å
$b = 14.734$ (1) Å
$c = 12.385$ (1) Å
$\beta = 101.06$ (1)°
$V = 1018.9$ (2) Å^3
$Z = 4$
$D_x = 1.292$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 27.4$–28.4°
$\mu = 0.662$ mm^{-1}
$T = 298$ K
Prismatic
0.11 × 0.06 × 0.05 mm
Colourless

Data collection

Rigaku AFC-7*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF (Walker & Stuart, 1983)
1774 measured reflections
1598 independent reflections
1316 observed reflections [$I > 3\sigma(I)$]

$R_{\text{int}} = 0.014$
$\theta_{\text{max}} = 60.0°$
$h = 0 \rightarrow 6$
$k = 0 \rightarrow 16$
$l = -13 \rightarrow 13$
3 standard reflections monitored every 150 reflections
intensity decay: 3.12%

Refinement

Refinement on *F*
$R = 0.036$
$wR = 0.054$
$S = 2.52$
1316 reflections
136 parameters
H-atom parameters not refined

$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{\text{max}} = 0.01$
$\Delta\rho_{\text{max}} = 0.10$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.12$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{\text{eq}} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}
2-FPB				
F(1)	1.3355 (2)	0.5577 (1)	0.8474 (1)	7.11 (8)
O(1)	0.7506 (3)	0.5628 (1)	0.6045 (1)	5.32 (7)
O(2)	1.0412 (3)	0.6464 (1)	0.6915 (1)	6.42 (8)
C(1)	1.1598 (4)	0.4993 (1)	0.8484 (2)	4.5 (1)
C(2)	1.1940 (4)	0.4324 (2)	0.9251 (2)	5.2 (1)
C(3)	1.0204 (4)	0.3714 (2)	0.9274 (2)	5.4 (1)
C(4)	0.8151 (4)	0.3779 (2)	0.8533 (2)	5.3 (1)
C(5)	0.7842 (4)	0.4451 (1)	0.7761 (2)	4.6 (1)
C(6)	0.9574 (3)	0.5082 (1)	0.7715 (2)	3.92 (8)
C(7)	0.9275 (4)	0.5804 (1)	0.6887 (2)	4.22 (9)
C(8)	0.6998 (4)	0.6257 (1)	0.5190 (2)	4.27 (9)
C(9)	0.8415 (4)	0.6336 (1)	0.4385 (2)	4.8 (1)
C(10)	0.7826 (4)	0.6935 (2)	0.3531 (2)	5.1 (1)
C(11)	0.5847 (4)	0.7445 (1)	0.3484 (2)	5.2 (1)
C(12)	0.4430 (4)	0.7344 (2)	0.4283 (2)	5.1 (1)
C(13)	0.4987 (4)	0.6750 (2)	0.5145 (2)	4.9 (1)
4-FPB				
F(1)	0.4618 (2)	−0.0311 (6)	0.3234 (1)	8.0 (2)
O(1)	0.7133 (2)	0.0522 (5)	0.5723 (1)	5.4 (1)
O(2)	0.6516 (2)	−0.2758 (7)	0.5913 (2)	7.2 (2)
C(1)	0.5540 (3)	−0.2616 (8)	0.4711 (2)	5.5 (2)
C(2)	0.5037 (3)	−0.242 (1)	0.4142 (2)	5.9 (3)
C(3)	0.5113 (3)	−0.050 (1)	0.3799 (2)	5.6 (3)
C(4)	0.5657 (3)	0.1212 (9)	0.3993 (2)	5.6 (2)
C(5)	0.6155 (3)	0.0997 (8)	0.4571 (2)	5.1 (2)
C(6)	0.6095 (3)	−0.0921 (8)	0.4932 (2)	4.5 (2)
C(7)	0.6593 (3)	−0.120 (1)	0.5568 (2)	5.0 (2)
C(8)	0.7639 (3)	0.0522 (9)	0.6317 (2)	4.7 (2)
C(9)	0.8178 (3)	−0.1227 (9)	0.6500 (2)	5.3 (2)
C(10)	0.8703 (3)	−0.1060 (9)	0.7065 (2)	5.9 (2)
C(11)	0.8671 (3)	0.083 (1)	0.7437 (2)	6.1 (3)
C(12)	0.8122 (3)	0.254 (1)	0.7252 (3)	5.9 (2)
C(13)	0.7595 (3)	0.2407 (9)	0.6683 (2)	5.5 (2)
PB				
O(1)	0.2552 (2)	0.07376 (8)	0.9002 (1)	4.96 (6)
O(2)	−0.0863 (3)	0.1435 (1)	0.8241 (1)	5.93 (7)
C(1)	−0.1752 (3)	0.0052 (1)	0.6611 (1)	4.48 (8)
C(2)	−0.2023 (4)	−0.0612 (1)	0.5816 (2)	5.21 (9)
C(3)	−0.0189 (4)	−0.1203 (1)	0.5770 (2)	5.01 (9)
C(4)	0.1934 (4)	−0.1135 (1)	0.6511 (2)	5.08 (9)
C(5)	0.2228 (3)	−0.0472 (1)	0.7316 (1)	4.37 (8)
C(6)	0.0371 (3)	0.0126 (1)	0.7367 (1)	3.76 (7)
C(7)	0.0552 (3)	0.0838 (1)	0.8223 (1)	4.08 (7)
C(8)	0.2973 (3)	0.1369 (1)	0.9878 (1)	4.00 (7)
C(9)	0.1535 (3)	0.1400 (1)	1.0641 (1)	4.60 (8)
C(10)	0.2093 (4)	0.1998 (1)	1.1515 (1)	4.92 (9)
C(11)	0.4083 (4)	0.2543 (1)	1.1608 (1)	4.79 (8)
C(12)	0.5528 (4)	0.2491 (1)	1.0840 (1)	4.77 (8)
C(13)	0.4977 (3)	0.1900 (1)	0.9963 (1)	4.49 (8)

Table 2. *Selected geometric parameters* (Å, °)

2-FPB			
F(1)—C(1)	1.348 (2)	C(5)—C(6)	1.392 (3)
O(1)—C(7)	1.351 (3)	C(6)—C(7)	1.474 (3)
O(1)—C(8)	1.402 (2)	C(8)—C(9)	1.369 (3)
O(2)—C(7)	1.193 (2)	C(8)—C(13)	1.376 (3)
C(1)—C(2)	1.366 (3)	C(9)—C(10)	1.376 (3)
C(1)—C(6)	1.382 (3)	C(10)—C(11)	1.374 (3)
C(2)—C(3)	1.367 (3)	C(11)—C(12)	1.367 (3)
C(3)—C(4)	1.373 (3)	C(12)—C(13)	1.376 (3)
C(4)—C(5)	1.373 (5)		
C(7)—O(1)—C(8)	117.8 (2)	O(1)—C(7)—O(2)	122.4 (2)
F(1)—C(1)—C(2)	117.6 (2)	O(1)—C(7)—C(6)	111.0 (2)
F(1)—C(1)—C(6)	119.3 (2)	O(2)—C(7)—C(6)	126.6 (2)
C(2)—C(1)—C(6)	123.1 (2)	O(1)—C(8)—C(9)	120.1 (2)
C(1)—C(2)—C(3)	119.0 (2)	O(1)—C(8)—C(13)	118.4 (2)
C(2)—C(3)—C(4)	120.1 (2)	C(9)—C(8)—C(13)	121.4 (2)
C(3)—C(4)—C(5)	120.1 (2)	C(8)—C(9)—C(10)	118.9 (2)
C(4)—C(5)—C(6)	121.2 (2)	C(9)—C(10)—C(11)	120.6 (2)
C(1)—C(6)—C(5)	116.5 (2)	C(10)—C(11)—C(12)	119.7 (2)
C(1)—C(6)—C(7)	121.7 (2)	C(11)—C(12)—C(13)	120.7 (2)
C(5)—C(6)—C(7)	121.8 (2)	C(8)—C(13)—C(12)	118.7 (2)
4-FPB			
F(1)—C(3)	1.355 (5)	C(5)—C(6)	1.381 (6)
O(1)—C(7)	1.347 (5)	C(6)—C(7)	1.488 (6)
O(1)—C(8)	1.409 (5)	C(8)—C(9)	1.366 (6)
O(2)—C(7)	1.204 (5)	C(8)—C(13)	1.366 (6)
C(1)—C(2)	1.369 (6)	C(9)—C(10)	1.377 (6)
C(1)—C(6)	1.376 (6)	C(10)—C(11)	1.372 (7)
C(2)—C(3)	1.364 (7)	C(11)—C(12)	1.365 (6)
C(3)—C(4)	1.361 (6)	C(12)—C(13)	1.384 (6)
C(4)—C(5)	1.381 (6)		

C(7)—O(1)—C(8)	119.2 (3)	O(1)—C(7)—O(2)	123.5 (4)
C(2)—C(1)—C(6)	121.1 (5)	O(1)—C(7)—C(6)	112.5 (4)
C(1)—C(2)—C(3)	117.5 (5)	O(2)—C(7)—C(6)	124.0 (5)
F(1)—C(3)—C(2)	117.6 (5)	O(1)—C(8)—C(9)	121.0 (4)
F(1)—C(3)—C(4)	118.7 (5)	O(1)—C(8)—C(13)	116.8 (4)
C(2)—C(3)—C(4)	123.7 (4)	C(9)—C(8)—C(13)	122.2 (4)
C(3)—C(4)—C(5)	118.0 (4)	C(8)—C(9)—C(10)	118.8 (4)
C(4)—C(5)—C(6)	120.0 (4)	C(9)—C(10)—C(11)	119.9 (5)
C(1)—C(6)—C(5)	119.7 (4)	C(10)—C(11)—C(12)	120.6 (4)
C(1)—C(6)—C(7)	118.0 (4)	C(11)—C(12)—C(13)	120.1 (5)
C(5)—C(6)—C(7)	122.3 (4)	C(8)—C(13)—C(12)	118.4 (5)

PB

O(1)—C(7)	1.351 (2)	C(5)—C(6)	1.386 (2)
O(1)—C(8)	1.415 (2)	C(6)—C(7)	1.481 (2)
O(2)—C(7)	1.195 (2)	C(8)—C(9)	1.364 (3)
C(1)—C(2)	1.376 (3)	C(8)—C(13)	1.371 (3)
C(1)—C(6)	1.383 (2)	C(9)—C(10)	1.384 (3)
C(2)—C(3)	1.368 (3)	C(10)—C(11)	1.375 (3)
C(3)—C(4)	1.373 (3)	C(11)—C(12)	1.373 (3)
C(4)—C(5)	1.382 (3)	C(12)—C(13)	1.381 (3)

C(7)—O(1)—C(8)	118.1 (1)	O(1)—C(7)—C(6)	111.5 (1)
C(2)—C(1)—C(6)	120.3 (2)	O(2)—C(7)—C(6)	125.4 (2)
C(1)—C(2)—C(3)	120.1 (2)	O(1)—C(8)—C(9)	121.2 (2)
C(2)—C(3)—C(4)	120.3 (2)	O(1)—C(8)—C(13)	116.8 (2)
C(3)—C(4)—C(5)	120.2 (2)	C(9)—C(8)—C(13)	121.9 (2)
C(4)—C(5)—C(6)	119.6 (2)	C(8)—C(9)—C(10)	119.0 (2)
C(1)—C(6)—C(5)	119.6 (2)	C(9)—C(10)—C(11)	119.9 (2)
C(1)—C(6)—C(7)	118.2 (2)	C(10)—C(11)—C(12)	120.1 (2)
C(5)—C(6)—C(7)	122.2 (2)	C(11)—C(12)—C(13)	120.3 (2)
O(1)—C(7)—O(2)	123.1 (2)	C(8)—C(13)—C(12)	118.7 (2)

The structures were solved by direct methods using *SAPI*91 (Fan, 1991) and expanded using *DIRDIF*92 (Beurskens *et al.*, 1992). H atoms were placed at calculated positions (C—H 0.95 Å). The displacement factors of the H atoms were kept fixed at the isotropic value of the parent atoms. All calculations were performed using the *TEXSAN* crystallographic software package (Molecular Structure Corporation, 1985).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, interatomic distances, bond distances involving H atoms and torsion angles have been deposited with the IUCr (Reference: AS1092). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3,4,5-Trimethoxybenzaldehyde Thiosemicarbazone Monohydrate

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Abstract

The structure of the title compound, $C_{11}H_{15}N_3O_3S.H_2O$ (III), has been determined. The molecule as a whole is not planar, though the phenyl ring is planar and makes a dihedral angle of 73.8 (2)° with the methoxy group at C(4). Molecules are linked through intermolecular N—H···S and N—H···O hydrogen bonds.

Comment

Thiosemicarbazones (TSCs) are derivatives of carbonyl compounds which exhibit wide ranging biological activities. These include antituberculosis, antiviral, antileukaemic and antitumour activities and are given below with their appropriate TSC. Combinations of metal complexes with pyridoxal thiosemicarbazone (Ferrari Belicchi, Fava Gasparri, Leporati, Pelizzi, Tarasconi & Tosi, 1986) possess interesting spectroscopic properties. Non-bonded interactions of thiophene derivatives, observed in 2-formylthiophene semicarbazone (Anna, Palenik & Palenik, 1988), include a novel intramolecular S···N attraction. 4-Formylpyridine thiosemicarbazone (4FPYTSC) possesses both antitumour (Hagenbach & Gysin, 1952) and antiviral activity (Jones, Slack, Squires & Woolridge, 1965), 2-formylpyridine thiosemicarbazone (French & Blanz, 1966) is a tumor inhibitor and *P. Colinalde* thiosemicarbazone (Brockman & Thomson, 1956) possesses anti-leukaemic activity. Extensive delocalization involving the heteroaromatic ring system and the thiosemicarbazide side chain (Palenik, Rendle & Carter, 1974) takes place and this is responsible for various