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2-(4-Fluorobenzylidene)propanedinitrile: monoclinic polymorph

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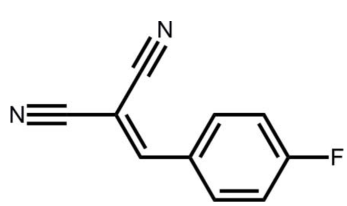
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.060; wR factor = 0.190; data-to-parameter ratio = 16.6.

The title compound, $C_{10}H_5FN_2$, is a monoclinic ($P2_1/c$) polymorph of the previously reported triclinic ($P\bar{1}$) form [Antipin *et al.* (2003). *J. Mol. Struct.* **650**, 1–20]. The 13 non-H atoms in the title polymorph are almost coplanar (r.m.s. deviation = 0.020 Å); a small twist between the fluorobenzene and dinitrile groups [C—C—C—C torsion angle = 175.49 (16)°] is evident in the triclinic polymorph. In the crystal, C—H⋯N interactions lead to supramolecular layers parallel to ($\bar{1}$01); these are connected by C—F⋯π interactions.

Related literature

For background to the chemistry and biological activity of 4*H*-pyran derivatives, see: El-Agrody *et al.* (2011); Sabry *et al.* (2011). For the structure of the triclinic polymorph, see: Antipin *et al.* (2003); Ng & Tiekink (2013).



Experimental

Crystal data

$C_{10}H_5FN_2$ | M_r = 172.16
Monoclinic, $P2_1/c$
a = 9.1491 (9) Å
b = 12.7961 (14) Å
c = 7.5828 (11) Å
β = 106.317 (13)°
V = 851.98 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 295 K
0.35 × 0.15 × 0.05 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
T_{min} = 0.892, T_{max} = 1.000
7732 measured reflections
1957 independent reflections
1149 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.190
S = 1.09
1957 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the C1–C6 ring.

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C3—H3⋯N1^{i}	0.93	2.61	3.478 (4)	155
C7—H7⋯N2^{ii}	0.93	2.51	3.424 (3)	167
C4—F1⋯*Cg*1^{iii}	1.35 (1)	3.59 (1)	3.573 (2)	79 (1)

Symmetry codes: (i) $x-1, y, z-1$; (ii) $-x+2, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x, -y-\frac{1}{2}, z-\frac{3}{2}$.

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012), *QMol* (Gans & Shalloway, 2001) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7051).

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S1. Comment

In continuation of a program on the chemistry of 4*H*-pyran derivatives (El-Agrody *et al.*, 2011; Sabry *et al.*, 2011), the title compound was isolated from a failed reaction, see Experimental, as a monoclinic polymorph ($P2_1/c$) of the previously reported triclinic ($P\overline{1}$) form (Antipin *et al.*, 2003). In fact, both forms were characterized from the same reaction product (Ng & Tiekink, 2013).

In (I), Fig. 1, the 13 non-hydrogen atoms are almost co-planar with a r.m.s. deviation of 0.020 Å, and with maximum deviations of 0.028 (2) Å for the C5 atom and -0.028 (2) Å for C2. This contrasts the small twist found in the triclinic form (r.m.s. deviation = 0.062 Å) as seen in the C2—C1—C7—C8 torsion angle of 175.49 (16)° (Ng & Tiekink, 2013), which compares to -180.0 (2)° in (I); this difference is emphasized in the overlay diagram shown in Fig. 2.

In the crystal, supramolecular layers mediated by C—H···N interactions are formed parallel to ($\overline{1}$ 0 1), Fig. 2 and Table 1, and these are connected into a three-dimensional array by C—F···*π* contacts, Fig. 4 and Table 1. This pattern of interactions is in stark contrast to that in the triclinic polymorph whereby C—H···N interactions, involving one N atom only, lead to supramolecular chains which are connected into double chains by weak *π*—*π* contacts (Ng & Tiekink, 2013).

S2. Experimental

A solution of 6-bromo-1-naphthol (0.01 mol) in EtOH (30 ml) was treated with 4-fluoro-1-(2,2-dicyanovinyl)benzene (0.01 mol) and piperidine (0.5 ml). The reaction mixture was heated until complete precipitation occurred (reaction time: 60 min). The solid product which formed was collected by filtration and recrystallized from ethanol to give the title compound, *i.e.* unreacted 4-fluoro-1-(2,2-dicyanovinyl)benzene, as both triclinic (Ng & Tiekink, 2013) and monoclinic (I) polymorphs. Both crystal forms have the appearance of yellow prisms.

S3. Refinement

The C-bound H atoms were geometrically placed (C–H = 0.93 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C).

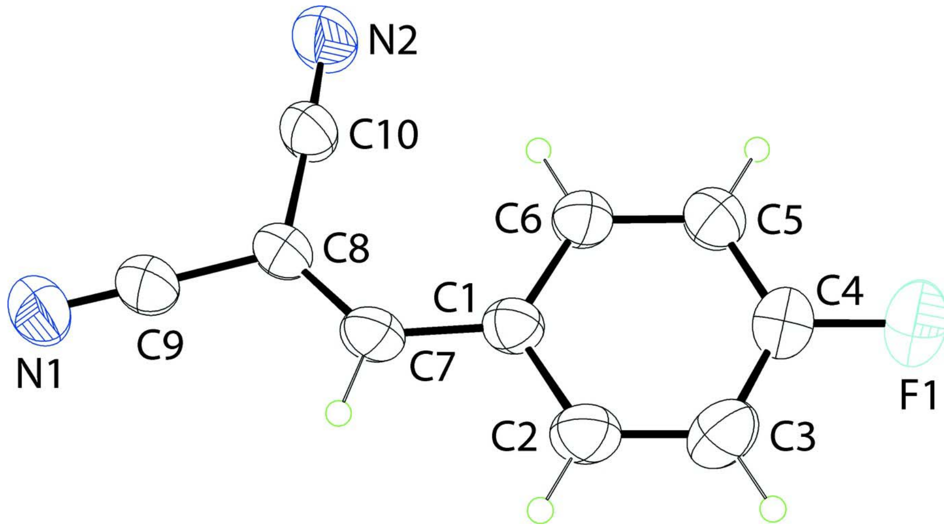

Figure 1

The molecular structures of (I) showing displacement ellipsoids at the 35% probability level.

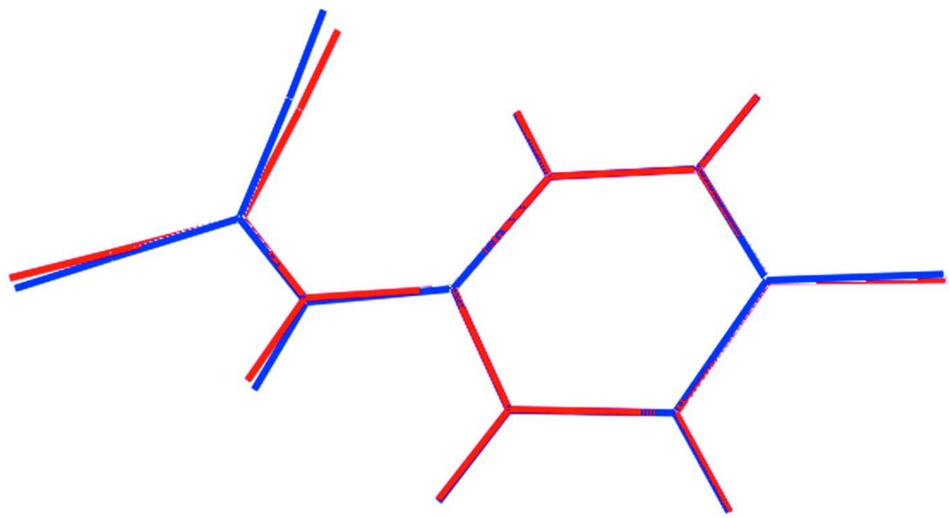

Figure 2

Overlay diagram of (I) (red) with the triclinic form (blue). The molecules are overlaid so that the benzene rings are superimposed.

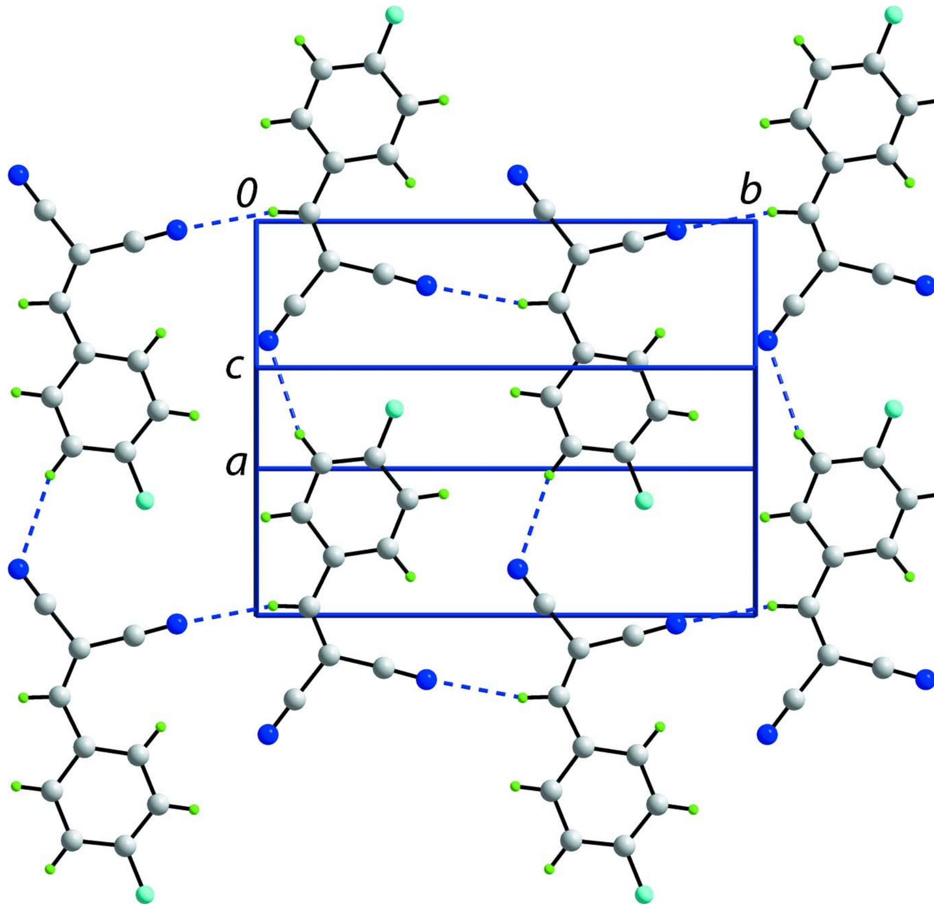

Figure 3

A view of the supramolecular layer parallel to ($\overline{1}$ 0 1) in (I) sustained by C—H···N interactions, shown as blue dashed lines.

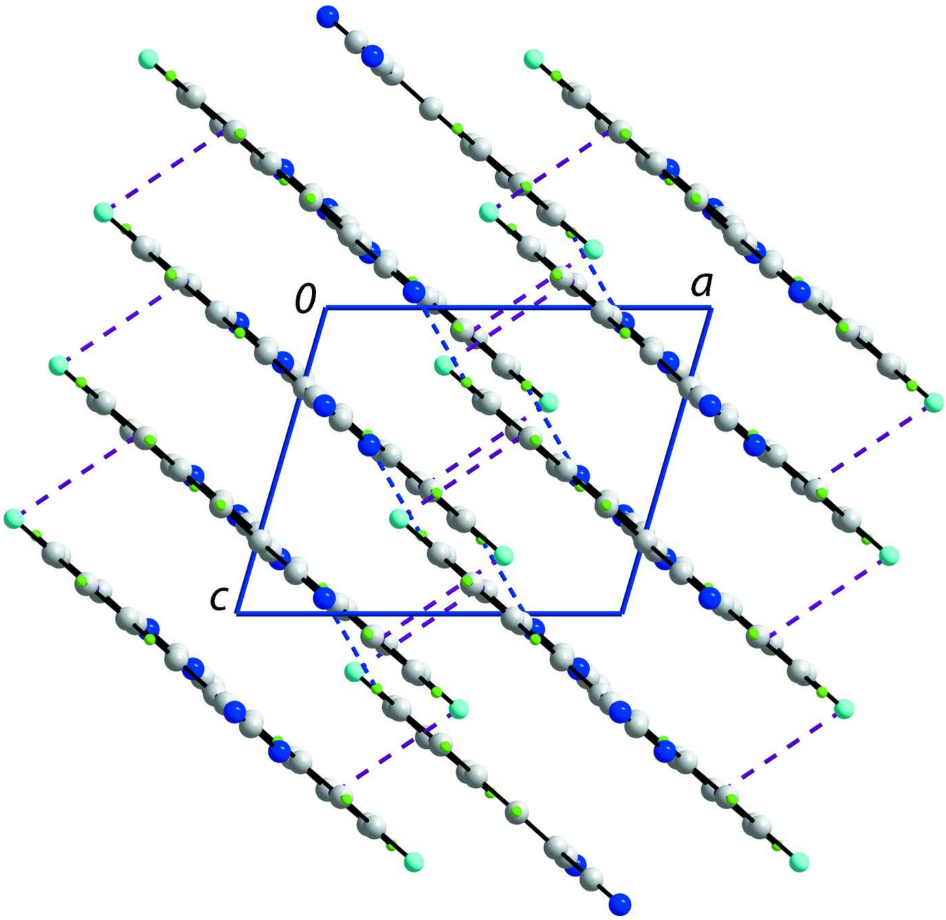

Figure 4

A view in projection down the *b* axis of the crystal packing in (I). The C—H···N and C—F···π interactions are shown as blue and purple dashed lines, respectively.

2-(4-Fluorobenzylidene)propanedinitrile

Crystal data

$C_{10}H_5FN_2$
M_r = 172.16
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 9.1491 (9) Å
b = 12.7961 (14) Å
c = 7.5828 (11) Å
β = 106.317 (13)°
V = 851.98 (18) Å^3
Z = 4
F(000) = 352
D_x = 1.342 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1835 reflections
θ = 3.1–27.5°
μ = 0.10 mm^{-1}
T = 295 K
Prism, yellow
0.35 × 0.15 × 0.05 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
T_{min} = 0.892, T_{max} = 1.000

7732 measured reflections
1957 independent reflections
1149 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.2°$
$h = -11 \rightarrow 11$
$k = -16 \rightarrow 14$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.190$
$S = 1.09$
1957 reflections
118 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0742P)^2 + 0.0917P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
F1	0.35050 (15)	0.22275 (14)	0.1895 (2)	0.0959 (6)
N1	1.2165 (3)	0.47627 (19)	0.9490 (4)	0.1097 (9)
N2	1.0706 (2)	0.15887 (19)	0.8205 (3)	0.0900 (8)
C1	0.7452 (2)	0.34429 (17)	0.5304 (3)	0.0566 (6)
C2	0.6299 (3)	0.4089 (2)	0.4263 (3)	0.0704 (7)
H2	0.6434	0.4809	0.4340	0.084*
C3	0.4968 (3)	0.3689 (2)	0.3126 (3)	0.0775 (7)
H3	0.4209	0.4129	0.2442	0.093*
C4	0.4797 (2)	0.2624 (2)	0.3035 (3)	0.0685 (7)
C5	0.5886 (2)	0.1964 (2)	0.4027 (3)	0.0651 (6)
H5	0.5733	0.1245	0.3937	0.078*
C6	0.7208 (2)	0.23631 (18)	0.5158 (3)	0.0606 (6)
H6	0.7952	0.1911	0.5836	0.073*
C7	0.8788 (2)	0.39364 (18)	0.6487 (3)	0.0627 (6)
H7	0.8763	0.4663	0.6451	0.075*
C8	1.0061 (2)	0.35367 (18)	0.7630 (3)	0.0611 (6)
C9	1.1232 (3)	0.4227 (2)	0.8665 (3)	0.0773 (7)
C10	1.0398 (2)	0.2445 (2)	0.7927 (3)	0.0648 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0668 (9)	0.1132 (15)	0.0951 (11)	−0.0038 (8)	0.0019 (8)	−0.0117 (9)
N1	0.1033 (16)	0.0584 (16)	0.131 (2)	−0.0124 (13)	−0.0271 (14)	0.0014 (13)
N2	0.0849 (14)	0.0549 (15)	0.1148 (19)	0.0018 (11)	0.0028 (12)	0.0019 (12)
C1	0.0644 (12)	0.0469 (14)	0.0582 (12)	0.0029 (9)	0.0169 (9)	0.0002 (9)
C2	0.0809 (15)	0.0528 (15)	0.0747 (14)	0.0089 (11)	0.0174 (12)	0.0037 (11)
C3	0.0714 (14)	0.080 (2)	0.0734 (15)	0.0198 (13)	0.0088 (11)	0.0048 (13)
C4	0.0595 (13)	0.080 (2)	0.0642 (14)	−0.0006 (11)	0.0139 (10)	−0.0066 (11)
C5	0.0686 (13)	0.0576 (15)	0.0684 (13)	−0.0032 (11)	0.0179 (11)	−0.0039 (10)
C6	0.0639 (12)	0.0516 (14)	0.0632 (12)	0.0033 (10)	0.0125 (10)	0.0013 (9)
C7	0.0760 (14)	0.0398 (13)	0.0702 (13)	0.0018 (10)	0.0174 (11)	0.0001 (9)
C8	0.0681 (12)	0.0434 (14)	0.0686 (13)	−0.0041 (10)	0.0141 (10)	−0.0015 (9)
C9	0.0803 (15)	0.0485 (16)	0.0899 (17)	−0.0003 (12)	0.0024 (13)	0.0028 (12)
C10	0.0638 (13)	0.0487 (15)	0.0766 (14)	−0.0028 (11)	0.0113 (10)	−0.0037 (11)

Geometric parameters (Å, º)

F1—C4	1.352 (3)	C3—H3	0.9300
N1—C9	1.135 (3)	C4—C5	1.361 (3)
N2—C10	1.136 (3)	C5—C6	1.370 (3)
C1—C6	1.399 (3)	C5—H5	0.9300
C1—C2	1.396 (3)	C6—H6	0.9300
C1—C7	1.443 (3)	C7—C8	1.342 (3)
C2—C3	1.378 (3)	C7—H7	0.9300
C2—H2	0.9300	C8—C10	1.435 (3)
C3—C4	1.372 (3)	C8—C9	1.440 (3)
C6—C1—C2	117.5 (2)	C4—C5—H5	120.2
C6—C1—C7	124.74 (19)	C6—C5—H5	120.2
C2—C1—C7	117.8 (2)	C5—C6—C1	120.7 (2)
C3—C2—C1	121.9 (3)	C5—C6—H6	119.6
C3—C2—H2	119.0	C1—C6—H6	119.6
C1—C2—H2	119.0	C8—C7—C1	131.6 (2)
C4—C3—C2	118.0 (2)	C8—C7—H7	114.2
C4—C3—H3	121.0	C1—C7—H7	114.2
C2—C3—H3	121.0	C7—C8—C10	125.6 (2)
F1—C4—C5	119.5 (3)	C7—C8—C9	119.7 (2)
F1—C4—C3	118.2 (2)	C10—C8—C9	114.7 (2)
C5—C4—C3	122.3 (2)	N1—C9—C8	179.3 (3)
C4—C5—C6	119.7 (2)	N2—C10—C8	177.8 (2)
C6—C1—C2—C3	−0.2 (3)	C4—C5—C6—C1	0.0 (3)
C7—C1—C2—C3	−178.55 (19)	C2—C1—C6—C5	0.2 (3)
C1—C2—C3—C4	−0.1 (4)	C7—C1—C6—C5	178.50 (19)
C2—C3—C4—F1	−178.78 (19)	C6—C1—C7—C8	1.7 (4)
C2—C3—C4—C5	0.3 (4)	C2—C1—C7—C8	−180.0 (2)

F1—C4—C5—C6	178.84 (19)	C1—C7—C8—C10	0.8 (4)
C3—C4—C5—C6	−0.2 (4)	C1—C7—C8—C9	−179.9 (2)

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···N1^{i}	0.93	2.61	3.478 (4)	155
C7—H7···N2^{ii}	0.93	2.51	3.424 (3)	167
C4—F1···*Cg*1^{iii}	1.35 (1)	3.59 (1)	3.573 (2)	79 (1)

Symmetry codes: (i) $x-1, y, z-1$; (ii) $-x+2, y+1/2, -z+3/2$; (iii) $x, -y-1/2, z-3/2$.