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N-(4-Fluorophenyl)-4-nitrophthalimide: tripartite hydrogen-bonded sheets

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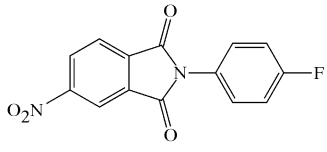
Molecules of the title compound, $C_{14}H_7FN_2O_4$, are linked by two C—H···O hydrogen bonds [H···O = 2.42 and 2.44 Å, C···O = 3.173 (9) and 3.313 (10) Å, and C—H···O = 134 and 157°] into deep tripartite sheets, where the central layer is built from hydrogen-bonded $R_6^6(24)$ rings and where the F atoms all lie on the exterior surfaces of the sheets.

Comment

The title compound, (I), was synthesized as part of a study of supramolecular interactions in substituted phthalimides and related compounds. We present here its molecular and crystal structure.



(I)

Within the molecule of (I) (Fig. 1), the dihedral angle between the planes of the heterocyclic rings and the fluorinated aryl ring is 50.5 (4)°, while the dihedral angle between the C—NO_2 plane and the adjacent carbocyclic ring is 10.3 (4)°. Consequently, the molecules of (I) have no internal symmetry in the solid state, and hence they are chiral in the solid state, although the bulk material in solution is racemic. Compound (I) crystallizes in the noncentrosymmetric space group $P2_1$. If the crystals form inversion twins, which is common in crystals having non-centrosymmetric space groups (Flack & Bernardinelli, 1999), then both enantiomers will be present in each such twinned crystal, although in the absence of such twinning, each crystal will contain only a single enantiomer, so that (I) would, in these circumstances, represent an example of conglomerate crystallization associated with spontaneous resolution. The indeterminate nature of the Flack (1983) parameter in (I) prevents any decision between these possibilities. The bond lengths and angles in (I) present no unusual features.

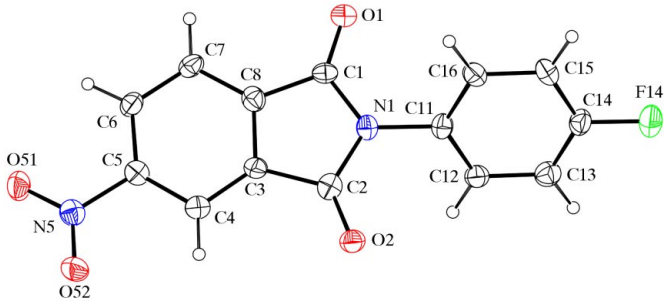


Figure 1
The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

The molecules of (I) are linked into thick sheets by means of two C—H···O hydrogen bonds (Table 1). Atom C6 in the molecule at (x, y, z) acts as hydrogen-bond donor to nitro atom O51 in the molecule at ($1-x, \frac{1}{2}+y, 1-z$), so producing a zigzag $C(5)$ chain (Bernstein *et al.*, 1995) running parallel to the [010] direction and generated by the 2_1 screw axis along ($\frac{1}{2}, y, \frac{1}{2}$) (Fig. 2). At the same time, adjacent atom C7 in the molecule at (x, y, z) acts as hydrogen-bond donor to the other nitro O atom, O52, in the molecule at ($x-1, 1+y, z$), so generating by translation a $C(6)$ chain running parallel to the [$\bar{1}$10] direction (Fig. 3). The combination of these [010] and [$\bar{1}$10] chains generates a tripartite sheet occupying the entire

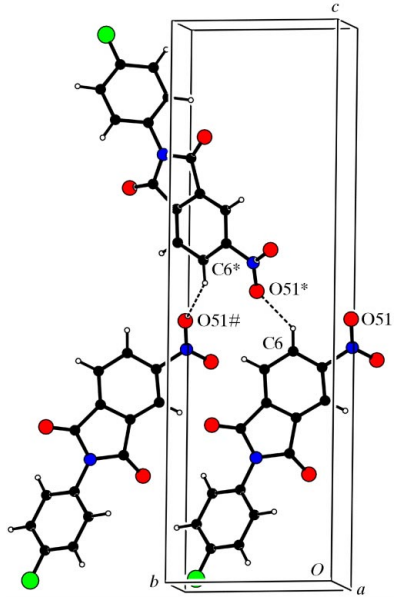


Figure 2
Part of the crystal structure of (I), showing the formation of a $C(5)$ chain along [010]. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions ($1-x, y-\frac{1}{2}, 1-z$) and ($x, 1+y, z$), respectively.

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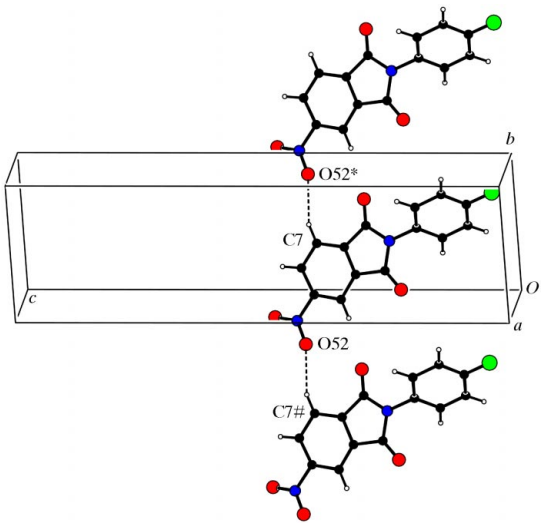

Figure 3
Part of the crystal structure of (I), showing the formation of a $C(6)$ chain along [$\bar{1}$10]. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions ($x-1, 1+y, z$) and ($1+x, y-1, z$), respectively.

domain of z, in which a central hydrogen-bonded layer built from a single type of $R_6^6(24)$ ring lies between two outer layers of aryl rings, with the F substituents on the outside surfaces of the layer (Fig. 4).

There are neither C—H···π(arene) hydrogen bonds nor aromatic π–π stacking interactions in the structure of (I). The only possible direction-specific interaction between the sheets, involving a C—H bond in one layer and an F substituent in the adjacent sheet, in fact, has an H···F distance (Table 1) far in excess of those associated with non-trivial interaction energies in C—H···F hydrogen bonds (Howard *et al.*, 1996). Accordingly, any possible structural significance of this H···F contact can be discounted, so that the hydrogen-bonded supramolecular structure of (I) is strictly two-dimensional.

The form of the supramolecular structure, as deep sheets partially coated with F, is reflected in the macroscopic behaviour of the crystals, which form very thin flakes which are highly hydrophobic and which when rubbed between the fingers leave a distinctly greasy coating.

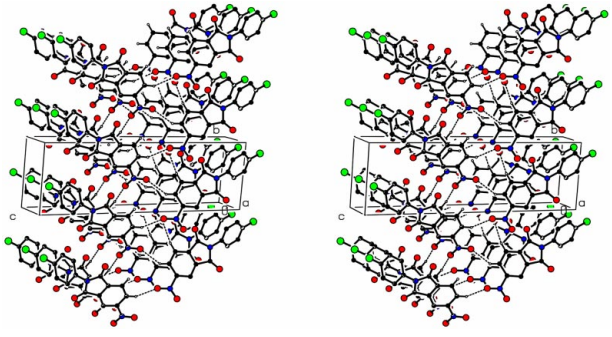

Figure 4
A stereoview of part of the crystal structure of (I), showing the formation of an (001) sheet built from $R_6^6(24)$ rings. For the sake of clarity, the H atoms not involved in the motifs shown have been omitted.

Experimental

An equimolar mixture of very finely divided 4-fluoroaniline and 4-nitrophthalimide was heated on an electric hotplate, in the absence of solvent, until the evolution of water had ceased. The reaction product was cooled and dissolved in 1,2-dichloroethane. Activated charcoal was added, and the mixture was then filtered. Evaporation of the solvent gave compound (I). After repeated attempts to obtain crystals suitable for single-crystal X-ray diffraction, some very thin flakes of rather indifferent quality and markedly waxy consistency were finally obtained from ethanol. A number of these crystals were investigated before any satisfactory diffraction data were obtained.

Crystal data

$C_{14}H_7FN_2O_4$
$M_r = 286.22$
Monoclinic, $P2_1$
$a = 3.7492\ (13)$ Å
$b = 6.9376\ (14)$ Å
$c = 23.099\ (8)$ Å
$\beta = 90.118\ (11)^\circ$
$V = 600.8\ (3)$ Å^3
$Z = 2$
$D_x = 1.582$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1099 reflections
$\theta = 3.5$–25.0°
$\mu = 0.13$ mm^{-1}
$T = 120\ (2)$ K
Plate, yellow
0.15 × 0.08 × 0.02 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.976$, $T_{max} = 0.997$
4455 measured reflections
1099 independent reflections
757 reflections with $I > 2\sigma(I)$
$R_{int} = 0.114$
$\theta_{max} = 25.0^\circ$
$h = -4 \rightarrow 4$
$k = -7 \rightarrow 7$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.070$
$wR(F^2) = 0.154$
$S = 1.06$
1099 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0761P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Table 1
Geometry of hydrogen bonds and short intermolecular contacts (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O51^{i}	0.95	2.44	3.173 (9)	134
C7—H7···O52^{ii}	0.95	2.42	3.313 (10)	157
C13—H13···F14^{iii}	0.95	2.54	3.438 (9)	157

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

The systematic absences permitted $P2_1$ and $P2_1/m$ as possible space groups; space group $P2_1$ was selected and confirmed by the subsequent structure analysis. All H atoms were located from difference maps and then treated as riding atoms, with C—H distances of 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. In the absence of significant anomalous scattering, the Flack (1983) parameter was indeterminate (Flack & Bernardinelli, 2000). Accordingly, the Friedel-equivalent reflections were merged prior to the final refinement.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS*97 (Sheldrick, 1997);

program(s) used to refine structure: *OSCAIL* and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA* (Ferguson, 1999).

The X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton, England; the authors thank the staff for all their help and advice. JNL thanks NCR Self-Service, Dundee, for grants which have provided computing facilities for this work. JLW thanks CNPq and FAPERJ for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1736). Services for accessing these data are described at the back of the journal.

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supporting information

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$c = 23.099\ (8)$ Å
$\beta = 90.118\ (11)°$
$V = 600.8\ (3)$ Å^3
$Z = 2$
$F(000) = 292$
$D_x = 1.582$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1099 reflections
$\theta = 3.5–25.0°$
$\mu = 0.13$ mm^{-1}
$T = 120$ K
Plate, yellow
0.15 × 0.08 × 0.02 mm

Data collection

Bruker-Nonius 95mm CCD camera on κ-goniostat diffractometer
Radiation source: Bruker-Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.976$, $T_{max} = 0.997$
4455 measured reflections
1099 independent reflections
757 reflections with $I > 2\sigma(I)$
$R_{int} = 0.114$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.5°$
$h = -4\rightarrow4$
$k = -7\rightarrow7$
$l = -27\rightarrow27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.070$
$wR(F^2) = 0.154$
$S = 1.06$
1099 reflections
190 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0761P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.8373 (15)	0.5621 (8)	0.2351 (2)	0.0324 (14)
C1	0.693 (2)	0.6393 (11)	0.2848 (3)	0.0344 (17)
O1	0.5499 (14)	0.7969 (7)	0.2890 (2)	0.0426 (13)
C2	0.9774 (19)	0.3779 (12)	0.2444 (3)	0.0366 (19)
O2	1.1382 (14)	0.2786 (7)	0.2098 (2)	0.0411 (13)
C3	0.901 (2)	0.3314 (10)	0.3067 (3)	0.0381 (19)
C4	0.974 (2)	0.1666 (11)	0.3377 (3)	0.0356 (18)
C5	0.862 (2)	0.1736 (11)	0.3950 (3)	0.0354 (18)
N5	0.9341 (16)	0.0010 (9)	0.4310 (3)	0.0413 (16)
O51	0.7959 (16)	−0.0063 (9)	0.4793 (2)	0.0536 (15)
O52	1.1204 (16)	−0.1251 (7)	0.4115 (2)	0.0524 (15)
C6	0.703 (2)	0.3298 (11)	0.4214 (3)	0.040 (2)
C7	0.633 (2)	0.4936 (12)	0.3887 (3)	0.0397 (18)
C8	0.733 (2)	0.4914 (11)	0.3306 (3)	0.0400 (19)
C11	0.836 (2)	0.6548 (11)	0.1781 (3)	0.0364 (18)
C12	0.712 (2)	0.5539 (11)	0.1311 (3)	0.0379 (18)
C13	0.708 (2)	0.6435 (12)	0.0778 (3)	0.044 (2)
C14	0.833 (2)	0.8282 (12)	0.0739 (3)	0.043 (2)
F14	0.8358 (13)	0.9151 (6)	0.02062 (18)	0.0593 (14)
C15	0.958 (2)	0.9307 (11)	0.1198 (3)	0.046 (2)
C16	0.965 (2)	0.8390 (10)	0.1744 (3)	0.042 (2)
H1	1.0388	1.0597	0.1153	0.055*
H4	1.0912	0.0579	0.3214	0.043*
H6	0.6415	0.3254	0.4612	0.049*
H7	0.5217	0.6034	0.4053	0.048*
H12	0.6303	0.4249	0.1351	0.046*
H13	0.6212	0.5783	0.0445	0.053*
H16	1.0557	0.9033	0.2076	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.025 (3)	0.039 (4)	0.033 (3)	−0.002 (3)	0.001 (3)	0.005 (3)
C1	0.034 (4)	0.029 (4)	0.041 (4)	−0.004 (3)	−0.001 (3)	−0.007 (3)
O1	0.050 (4)	0.036 (3)	0.042 (3)	0.002 (3)	0.000 (2)	−0.004 (2)
C2	0.025 (4)	0.045 (5)	0.040 (4)	−0.007 (4)	0.001 (3)	0.000 (4)
O2	0.047 (3)	0.035 (3)	0.042 (3)	−0.003 (3)	0.007 (3)	−0.004 (2)
C3	0.048 (5)	0.028 (5)	0.038 (4)	−0.003 (3)	0.006 (4)	−0.002 (3)
C4	0.030 (5)	0.036 (4)	0.041 (5)	−0.004 (3)	0.003 (3)	−0.003 (4)
C5	0.025 (4)	0.038 (4)	0.044 (5)	−0.003 (3)	0.003 (3)	0.001 (4)
N5	0.039 (4)	0.040 (4)	0.045 (4)	−0.007 (3)	0.000 (3)	0.002 (3)
O51	0.063 (4)	0.057 (4)	0.042 (3)	0.007 (3)	0.014 (3)	0.012 (3)
O52	0.061 (4)	0.040 (3)	0.056 (4)	0.013 (3)	0.010 (3)	0.002 (3)
C6	0.045 (5)	0.047 (5)	0.029 (4)	0.007 (4)	0.000 (3)	−0.004 (3)
C7	0.037 (5)	0.048 (5)	0.035 (4)	0.007 (4)	0.004 (3)	−0.007 (4)

C8	0.042 (5)	0.039 (4)	0.039 (4)	−0.004 (4)	−0.008 (4)	0.003 (4)
C11	0.034 (5)	0.042 (5)	0.033 (4)	−0.001 (4)	0.003 (3)	0.004 (3)
C12	0.041 (5)	0.034 (4)	0.039 (4)	0.001 (3)	0.000 (3)	−0.003 (3)
C13	0.044 (5)	0.044 (5)	0.045 (5)	0.001 (4)	0.004 (4)	−0.003 (4)
C14	0.048 (5)	0.050 (6)	0.030 (4)	0.009 (4)	−0.003 (4)	0.003 (4)
F14	0.073 (4)	0.066 (3)	0.039 (3)	−0.002 (3)	0.003 (2)	0.011 (2)
C15	0.057 (6)	0.035 (5)	0.046 (5)	0.001 (4)	−0.001 (4)	0.011 (3)
C16	0.049 (6)	0.034 (5)	0.042 (4)	0.004 (3)	−0.004 (4)	0.003 (3)

Geometric parameters (Å, º)

N1—C1	1.378 (9)	C6—C7	1.389 (11)
N1—C2	1.398 (10)	C6—H6	0.95
N1—C11	1.465 (9)	C7—C8	1.392 (10)
C1—O1	1.221 (9)	C7—H7	0.95
C1—C8	1.482 (10)	C11—C16	1.369 (11)
C2—O2	1.216 (9)	C11—C12	1.373 (10)
C2—C3	1.502 (10)	C12—C13	1.379 (10)
C3—C4	1.377 (10)	C12—H12	0.95
C3—C8	1.392 (10)	C13—C14	1.366 (12)
C4—C5	1.391 (9)	C13—H13	0.95
C4—H4	0.95	C14—C15	1.360 (10)
C5—C6	1.379 (10)	C14—F14	1.372 (8)
C5—N5	1.483 (10)	C15—C16	1.412 (10)
N5—O52	1.208 (8)	C15—H1	0.95
N5—O51	1.233 (7)	C16—H16	0.95
C1—N1—C2	112.1 (6)	C6—C7—C8	117.6 (7)
C1—N1—C11	125.2 (6)	C6—C7—H7	121.2
C2—N1—C11	122.7 (6)	C8—C7—H7	121.2
O1—C1—N1	125.9 (7)	C7—C8—C3	121.0 (7)
O1—C1—C8	127.4 (7)	C7—C8—C1	130.8 (7)
N1—C1—C8	106.6 (6)	C3—C8—C1	108.2 (6)
O2—C2—N1	127.1 (7)	C16—C11—C12	123.0 (7)
O2—C2—C3	127.1 (7)	C16—C11—N1	117.7 (7)
N1—C2—C3	105.7 (6)	C12—C11—N1	119.2 (7)
C4—C3—C8	123.0 (7)	C11—C12—C13	118.6 (7)
C4—C3—C2	129.7 (7)	C11—C12—H12	120.7
C8—C3—C2	107.3 (6)	C13—C12—H12	120.7
C3—C4—C5	113.9 (7)	C14—C13—C12	118.6 (7)
C3—C4—H4	123.0	C14—C13—H13	120.7
C5—C4—H4	123.0	C12—C13—H13	120.7
C6—C5—C4	125.5 (7)	C15—C14—C13	123.9 (7)
C6—C5—N5	117.7 (6)	C15—C14—F14	117.8 (7)
C4—C5—N5	116.7 (7)	C13—C14—F14	118.3 (7)
O52—N5—O51	123.6 (6)	C14—C15—C16	117.7 (7)
O52—N5—C5	118.8 (6)	C14—C15—H1	121.1
O51—N5—C5	117.7 (6)	C16—C15—H1	121.1

C5—C6—C7	118.9 (7)	C11—C16—C15	118.1 (7)
C5—C6—H6	120.6	C11—C16—H16	120.9
C7—C6—H6	120.6	C15—C16—H16	120.9
C2—N1—C1—O1	−179.3 (7)	C6—C7—C8—C1	179.2 (7)
C11—N1—C1—O1	−1.6 (11)	C4—C3—C8—C7	1.0 (11)
C2—N1—C1—C8	−1.5 (7)	C2—C3—C8—C7	−178.9 (7)
C11—N1—C1—C8	176.2 (6)	C4—C3—C8—C1	−179.1 (7)
C1—N1—C2—O2	−175.7 (7)	C2—C3—C8—C1	1.0 (8)
C11—N1—C2—O2	6.6 (11)	O1—C1—C8—C7	−2.2 (13)
C1—N1—C2—C3	2.1 (7)	N1—C1—C8—C7	−179.9 (7)
C11—N1—C2—C3	−175.6 (6)	O1—C1—C8—C3	177.9 (7)
O2—C2—C3—C4	−3.9 (13)	N1—C1—C8—C3	0.2 (8)
N1—C2—C3—C4	178.3 (7)	C1—N1—C11—C16	52.6 (10)
O2—C2—C3—C8	175.9 (7)	C2—N1—C11—C16	−130.0 (7)
N1—C2—C3—C8	−1.9 (7)	C1—N1—C11—C12	−128.4 (8)
C8—C3—C4—C5	0.7 (10)	C2—N1—C11—C12	49.0 (10)
C2—C3—C4—C5	−179.5 (7)	C16—C11—C12—C13	−1.7 (12)
C3—C4—C5—C6	−2.5 (10)	N1—C11—C12—C13	179.3 (7)
C3—C4—C5—N5	−179.6 (7)	C11—C12—C13—C14	0.9 (11)
C6—C5—N5—O52	−168.8 (7)	C12—C13—C14—C15	−0.6 (13)
C4—C5—N5—O52	8.6 (9)	C12—C13—C14—F14	178.6 (7)
C6—C5—N5—O51	12.0 (10)	C13—C14—C15—C16	1.0 (13)
C4—C5—N5—O51	−170.6 (6)	F14—C14—C15—C16	−178.2 (7)
C4—C5—C6—C7	2.6 (11)	C12—C11—C16—C15	2.1 (12)
N5—C5—C6—C7	179.7 (7)	N1—C11—C16—C15	−178.9 (7)
C5—C6—C7—C8	−0.7 (11)	C14—C15—C16—C11	−1.7 (12)
C6—C7—C8—C3	−1.0 (10)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O51^{i}	0.95	2.44	3.173 (9)	134
C7—H7···O52^{ii}	0.95	2.42	3.313 (10)	157
C13—H13···F14^{iii}	0.95	2.54	3.438 (9)	157

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1; (ii) *x*−1, *y*+1, *z*; (iii) −*x*+1, *y*−1/2, −*z*.