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2-(2,3,5,6-Tetrafluoro-4-iodoanilino)-ethanol

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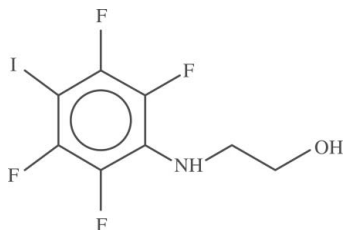
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Key indicators: single-crystal X-ray study; $T = 158$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.036; wR factor = 0.092; data-to-parameter ratio = 18.0.

The reaction of 2-aminoethanol and iodopentafluorobenzene in the presence of K_2CO_3 gave the title compound, $\text{C}_8\text{H}_6\text{F}_4\text{INO}$, in high yield. The structure is characterized by double layers of molecules linked by $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, and linear $\text{C}-\text{I}\cdots\text{F}$ [$\text{I}\cdots\text{F} = 3.049$ (2) Å] and bent $\text{C}-\text{I}\cdots\text{I}$ [$\text{I}\cdots\text{I} = 3.9388$ (7) Å] interactions between pairs of nearly parallel iodotetrafluorophenyl groups. No $\text{O}\cdots\text{I}$ or $\text{N}\cdots\text{I}$ halogen bonding is found.

Related literature

For related literature, see: Metrangolo & Resnati (2001); Metrangolo *et al.* (2005, 2007).



Experimental

Crystal data

$\text{C}_8\text{H}_6\text{F}_4\text{INO}$
 $M_r = 335.04$

Monoclinic, $C2/c$
 $a = 13.327$ (2) Å

$b = 17.663$ (3) Å
 $c = 8.3044$ (14) Å
 $\beta = 96.94$ (2)°
 $V = 1940.5$ (6) Å³
 $Z = 8$

Mo $K\alpha$ radiation
 $\mu = 3.33$ mm⁻¹
 $T = 158$ (2) K
 $0.24 \times 0.16 \times 0.08$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 1999)
 $T_{\min} = 0.691$, $T_{\max} = 1.000$
(expected range = 0.529–0.766)

8054 measured reflections
2924 independent reflections
2422 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.092$
 $S = 1.04$
2924 reflections
162 parameters

8 restraints
All H-atom parameters refined
 $\Delta\rho_{\max} = 1.65$ e Å⁻³
 $\Delta\rho_{\min} = -0.47$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^{\text{i}}$	0.87 (2)	2.23 (3)	3.046 (3)	157 (3)
$\text{O1}-\text{H1O}\cdots\text{O1}^{\text{ii}}$	0.82 (5)	1.90 (7)	2.715 (4)	175 (8)
$\text{O1}-\text{H2O}\cdots\text{O1}^{\text{iii}}$	0.82 (5)	1.99 (5)	2.781 (4)	162 (8)

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

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SIR2002 (Burla *et al.*, 2003); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: SHELXL97.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2167).

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supplementary materials

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2-(2,3,5,6-Tetrafluoro-4-iodoanilino)ethanol

P. Metrangolo, F. Meyer, T. Pilati and G. Resnati

Comment

Supramolecular architectures assembled by halogen bonding (XB) are our long-standing interest (Metrangolo & Resnati, 2001; Metrangolo *et al.*, 2005, 2007). As preliminary work, we need to design and to synthesize molecules showing functional and geometric properties adequate to give the supramolecular structures we wish to obtain. In the present study, we report the structure of 2-(2,3,5,6-tetrafluoro-4-iodo-phenylamino)ethanol, an intermediate in the synthesis of more complex molecules to be used in XB supramolecular engineering and in particular to cover gold surfaces with iodotetrafluorobenzene pendants. The molecular structure is shown in Figure 1. Containing alcohol and amino H atoms, the main interactions in this structure are O—H \cdots O and N—H \cdots O hydrogen bonds (HB) rather than O \cdots I or N \cdots I XB. In fact, we find the short distances O1 \cdots O1($-x, y, 1/2 - z$), O1 \cdots O1($-x, -y, 1 - z$) and N1 \cdots O1($x, -y, 1/2 + z$) of 2.715 (5), 2.781 (5), 3.046 (4) Å, respectively. These HBs generate one-dimensional sandwich ribbons; the distance between the centroids of a benzene ring and the mean plane through the nearest benzene ring in the sandwich is 3.393 Å. As shown in Figure 2 and 3, parallel one-dimensional ribbons are linked together by I1 \cdots I1($-x, 1 - y, 2 - z$) and I1 \cdots F3($x, 1 - y, 1/2 + x$) interactions, with length of 3.9388 (7) and 3.049 (2) Å, respectively, to form a two-dimensional sandwich layer. These are linked together only by residual forces; no distance below the sum of van der Waals radii is found between atoms in different sandwiches, as shown by the distance between the benzene centroid and the plane of the nearest benzene ring in a second two-dimensional layer, 0.074 Å larger than the intra-sandwich one (see Figure 4).

Experimental

500 mg (8.2 mmol) of ethanolamine, 1.64 ml (12.3 mmol) of iodopentafluorobenzene and 1.24 g (9.03 mmol) of K₂CO₃ were stirred in 15 ml of refluxing THF. After 5 h, water was added and the aqueous phase was extracted three times with dichloromethane. The combined organic phases were washed with saturated aqueous Na₂S₂O₃ solution and dried over Na₂SO₄. The residue was chromatographed on silica gel (240–400 mesh, eluent dichloromethane) to give the product in 86% yield. ¹H NMR (500 MHz, CDCl₃): δ 3.84 (2H, t, J = 5.2 Hz, CH₂), 3.56 (2H, tt, J = 1.4 Hz, J = 5.2 Hz, CH₂), 3.06 (2H, br s, OH and NH); ¹⁹F NMR (470 MHz, CDCl₃): δ -157.8 (2 F, m, CF—C), -124.3 (2 F, m, CF—Cl)

Refinement

Exchanging the atomic assignments of N1 and O1 worsens the results of the least-squares refinement, thus confirming that the tetrafluoroiodobenzene is bound to the amine group of ethanolamine. The H atoms were located in a difference map. For H of the hydroxyl group two possible positions were found. Both the positions (corresponding to H1O and H2O) are incompatible with a symmetry-equivalent position. In fact, the H1O \cdots H1O($-x, y, 1/2 - z$) distance is only 1.08 Å, and the H2O \cdots H2O($-x, -y, 1 - z$) distance is 1.24 Å. However, the distances H1O \cdots H2O($-x, y, 1/2 - z$) and H1O \cdots H2O($-x, -y, 1 - z$) are compatible with hydrogen bonding, being 2.17 and 2.32 Å, respectively. Any rotation of C—O—H around the C—O bond does not remedy the situation. The hydroxyl group is thus disordered. H atoms were refined with the following restraints: all the C—H distances are approximately equal, O—H is 0.82 (1) Å, and N—H is freely refined.

Figures

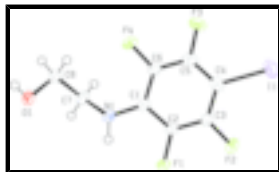


Fig. 1. Molecular structure with the numbering scheme and ADPs at the 50% probability level.

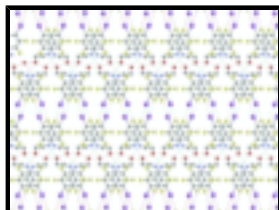


Fig. 2. A two-dimensional sandwich (see text) projected along the a^* axis, in Mercury ball and stick style. H atoms omitted.

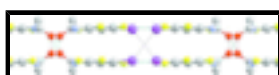


Fig. 3. The two-dimensional sandwich projected along the c axis.

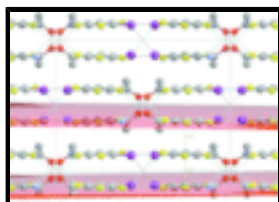


Fig. 4. The complete packing viewed along the c axis with the distances between the centroid of the benzene ring and the planes of the two nearest benzene rings.

2-(2,3,5,6-Tetrafluoro-4-iodoanilino)ethanol

Crystal data

$C_8H_6F_4INO$

$M_r = 335.04$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 13.327\ (2)\ \text{\AA}$

$b = 17.663\ (3)\ \text{\AA}$

$c = 8.3044\ (14)\ \text{\AA}$

$\beta = 96.94\ (2)^\circ$

$V = 1940.5\ (6)\ \text{\AA}^3$

$Z = 8$

$F_{000} = 1264$

$D_x = 2.294\ \text{Mg m}^{-3}$

Melting point: 343-348 K

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2964 reflections

$\theta = 3.0\text{--}29.1^\circ$

$\mu = 3.33\ \text{mm}^{-1}$

$T = 158\ (2)\ \text{K}$

Irregular table, colourless

$0.24 \times 0.16 \times 0.08\ \text{mm}$

Data collection

Bruker SMART CCD area-detector diffractometer

2924 independent reflections

Radiation source: fine-focus sealed tube

2422 reflections with $I > 2\sigma(I)$

Monochromator: graphite

$R_{\text{int}} = 0.026$

$T = 158\ (2)\ \text{K}$

$\theta_{\text{max}} = 31.3^\circ$

ω and φ scans

$\theta_{\text{min}} = 1.9^\circ$

Absorption correction: multi-scan

$h = -18 \rightarrow 18$

(SADABS; Bruker, 1999)

$T_{\min} = 0.691$, $T_{\max} = 1.000$

8054 measured reflections

$k = -16 \rightarrow 25$

$l = -11 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.092$

$S = 1.04$

2924 reflections

162 parameters

8 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 2.2775P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 1.65 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.47 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

Special details

Experimental. Data collection using an OXFORD low temperature device. Below 158 K the structure possibly shows a phase transition.

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
I1	0.116120 (18)	0.431723 (13)	1.05305 (3)	0.03962 (10)	
F1	0.11489 (13)	0.12833 (11)	1.03239 (18)	0.0310 (4)	
F2	0.10979 (15)	0.26089 (11)	1.1821 (2)	0.0341 (4)	
F3	0.13483 (15)	0.38552 (11)	0.6853 (2)	0.0375 (4)	
F4	0.13603 (14)	0.25397 (11)	0.53509 (19)	0.0320 (4)	
N1	0.12288 (19)	0.11508 (15)	0.7067 (3)	0.0256 (5)	
H1N	0.120 (3)	0.080 (2)	0.779 (5)	0.032 (10)*	
O1	0.06410 (19)	0.02423 (13)	0.3901 (2)	0.0322 (5)	
H1O	0.026 (5)	0.027 (5)	0.306 (6)	0.048*	0.50
H2O	0.022 (5)	0.020 (5)	0.454 (8)	0.048*	0.50
C1	0.12620 (19)	0.18530 (16)	0.7785 (3)	0.0211 (5)	
C2	0.1188 (2)	0.19221 (15)	0.9451 (3)	0.0220 (5)	

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C3	0.11614 (19)	0.26044 (18)	1.0212 (3)	0.0239 (5)
C4	0.1225 (2)	0.32801 (16)	0.9396 (4)	0.0257 (5)
C5	0.1307 (2)	0.32223 (17)	0.7744 (4)	0.0266 (6)
C6	0.13237 (19)	0.25369 (15)	0.6968 (3)	0.0235 (5)
C7	0.1830 (2)	0.0964 (2)	0.5749 (3)	0.0300 (6)
H7A	0.216 (3)	0.0485 (14)	0.602 (4)	0.031 (9)*
H7B	0.237 (2)	0.1319 (17)	0.573 (4)	0.032 (9)*
C8	0.1226 (3)	0.0915 (2)	0.4085 (3)	0.0303 (6)
H8A	0.079 (3)	0.136 (2)	0.388 (6)	0.072 (15)*
H8B	0.161 (2)	0.090 (2)	0.319 (3)	0.029 (9)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.04639 (16)	0.02605 (13)	0.04478 (15)	0.00436 (8)	-0.00122 (10)	-0.00885 (8)
F1	0.0507 (11)	0.0251 (9)	0.0177 (7)	-0.0040 (7)	0.0061 (7)	0.0048 (6)
F2	0.0505 (10)	0.0345 (10)	0.0177 (7)	0.0006 (8)	0.0052 (7)	-0.0053 (7)
F3	0.0486 (11)	0.0235 (9)	0.0403 (10)	-0.0035 (8)	0.0052 (8)	0.0108 (8)
F4	0.0446 (10)	0.0336 (10)	0.0190 (8)	-0.0037 (8)	0.0089 (7)	0.0048 (7)
N1	0.0371 (13)	0.0243 (12)	0.0161 (10)	-0.0009 (10)	0.0068 (9)	0.0017 (9)
O1	0.0516 (14)	0.0244 (10)	0.0190 (9)	0.0051 (10)	-0.0024 (9)	-0.0019 (8)
C1	0.0215 (11)	0.0249 (13)	0.0171 (11)	0.0001 (10)	0.0032 (9)	0.0004 (9)
C2	0.0263 (12)	0.0208 (12)	0.0192 (11)	-0.0011 (10)	0.0040 (9)	0.0039 (9)
C3	0.0248 (13)	0.0273 (14)	0.0195 (12)	-0.0018 (10)	0.0021 (10)	-0.0017 (10)
C4	0.0229 (12)	0.0215 (13)	0.0325 (14)	0.0001 (10)	0.0021 (10)	-0.0011 (11)
C5	0.0243 (12)	0.0253 (13)	0.0297 (14)	-0.0023 (11)	0.0020 (10)	0.0084 (11)
C6	0.0240 (12)	0.0275 (14)	0.0194 (12)	-0.0035 (11)	0.0045 (10)	0.0030 (10)
C7	0.0345 (15)	0.0359 (16)	0.0204 (12)	0.0057 (13)	0.0061 (11)	-0.0026 (11)
C8	0.0408 (16)	0.0331 (15)	0.0172 (12)	0.0021 (13)	0.0049 (11)	0.0016 (11)

Geometric parameters (\AA , $^\circ$)

I1—C4	2.067 (3)	C1—C6	1.393 (4)
F1—C2	1.345 (3)	C1—C2	1.404 (4)
F2—C3	1.349 (3)	C2—C3	1.363 (4)
F3—C5	1.346 (3)	C3—C4	1.380 (4)
F4—C6	1.350 (3)	C4—C5	1.392 (4)
N1—C1	1.375 (4)	C5—C6	1.373 (4)
N1—C7	1.470 (4)	C7—C8	1.516 (4)
N1—H1N	0.87 (4)	C7—H7A	0.97 (2)
O1—C8	1.419 (4)	C7—H7B	0.96 (2)
O1—H1O	0.818 (10)	C8—H8A	0.97 (2)
O1—H2O	0.818 (10)	C8—H8B	0.96 (2)
C1—N1—C7	122.3 (2)	F3—C5—C6	118.0 (3)
C1—N1—H1N	110 (3)	F3—C5—C4	119.6 (3)
C7—N1—H1N	115 (3)	C6—C5—C4	122.3 (3)
C8—O1—H1O	108 (6)	F4—C6—C5	117.9 (2)
C8—O1—H2O	115 (6)	F4—C6—C1	120.0 (2)

H1O—O1—H2O	99 (8)	C5—C6—C1	122.0 (2)
N1—C1—C6	124.9 (2)	N1—C7—C8	114.5 (3)
N1—C1—C2	120.3 (2)	N1—C7—H7A	107 (2)
C6—C1—C2	114.8 (2)	C8—C7—H7A	110 (2)
F1—C2—C3	119.1 (2)	N1—C7—H7B	110 (2)
F1—C2—C1	118.0 (2)	C8—C7—H7B	110 (2)
C3—C2—C1	122.9 (3)	H7A—C7—H7B	105 (3)
F2—C3—C2	118.2 (3)	O1—C8—C7	111.7 (3)
F2—C3—C4	119.8 (3)	O1—C8—H8A	110 (3)
C2—C3—C4	122.0 (3)	C7—C8—H8A	110 (3)
C3—C4—C5	115.9 (3)	O1—C8—H8B	104 (2)
C3—C4—I1	122.3 (2)	C7—C8—H8B	116 (2)
C5—C4—I1	121.8 (2)	H8A—C8—H8B	105 (4)
C7—N1—C1—C6	38.5 (4)	C3—C4—C5—F3	178.5 (2)
C7—N1—C1—C2	-144.3 (3)	I1—C4—C5—F3	0.1 (4)
N1—C1—C2—F1	4.1 (4)	C3—C4—C5—C6	0.0 (4)
C6—C1—C2—F1	-178.5 (2)	I1—C4—C5—C6	-178.3 (2)
N1—C1—C2—C3	-176.3 (3)	F3—C5—C6—F4	-1.3 (4)
C6—C1—C2—C3	1.1 (4)	C4—C5—C6—F4	177.1 (2)
F1—C2—C3—F2	0.1 (4)	F3—C5—C6—C1	-178.6 (2)
C1—C2—C3—F2	-179.5 (2)	C4—C5—C6—C1	-0.1 (4)
F1—C2—C3—C4	178.3 (2)	N1—C1—C6—F4	-0.3 (4)
C1—C2—C3—C4	-1.3 (4)	C2—C1—C6—F4	-177.6 (2)
F2—C3—C4—C5	178.8 (2)	N1—C1—C6—C5	176.9 (3)
C2—C3—C4—C5	0.7 (4)	C2—C1—C6—C5	-0.4 (4)
F2—C3—C4—I1	-2.8 (4)	C1—N1—C7—C8	-106.0 (3)
C2—C3—C4—I1	179.0 (2)	N1—C7—C8—O1	-74.4 (4)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1N...O1 ⁱ	0.87 (2)	2.23 (3)	3.046 (3)	157 (3)
O1—H1O...O1 ⁱⁱ	0.82 (5)	1.90 (7)	2.715 (4)	175 (8)
O1—H2O...O1 ⁱⁱⁱ	0.82 (5)	1.99 (5)	2.781 (4)	162 (8)

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

Fig. 1

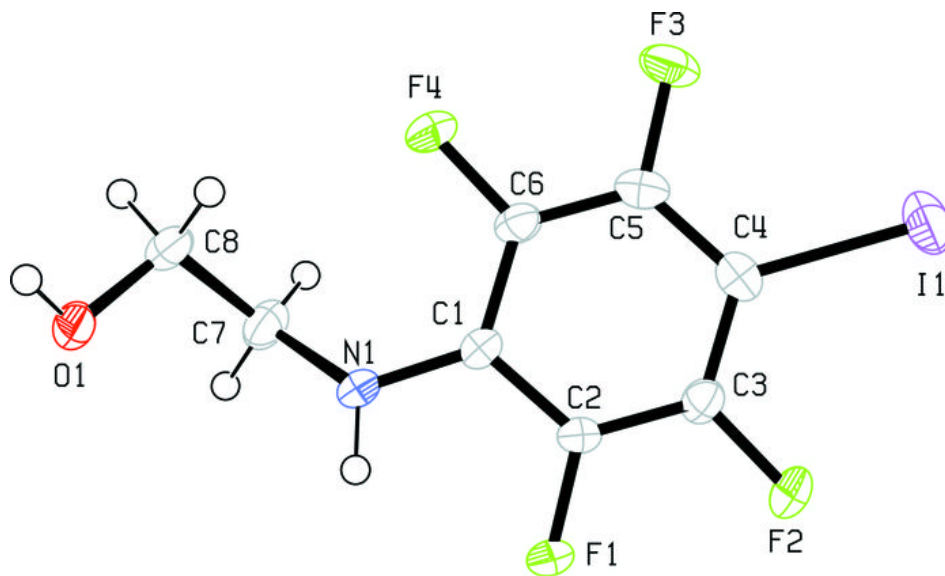


Fig. 2

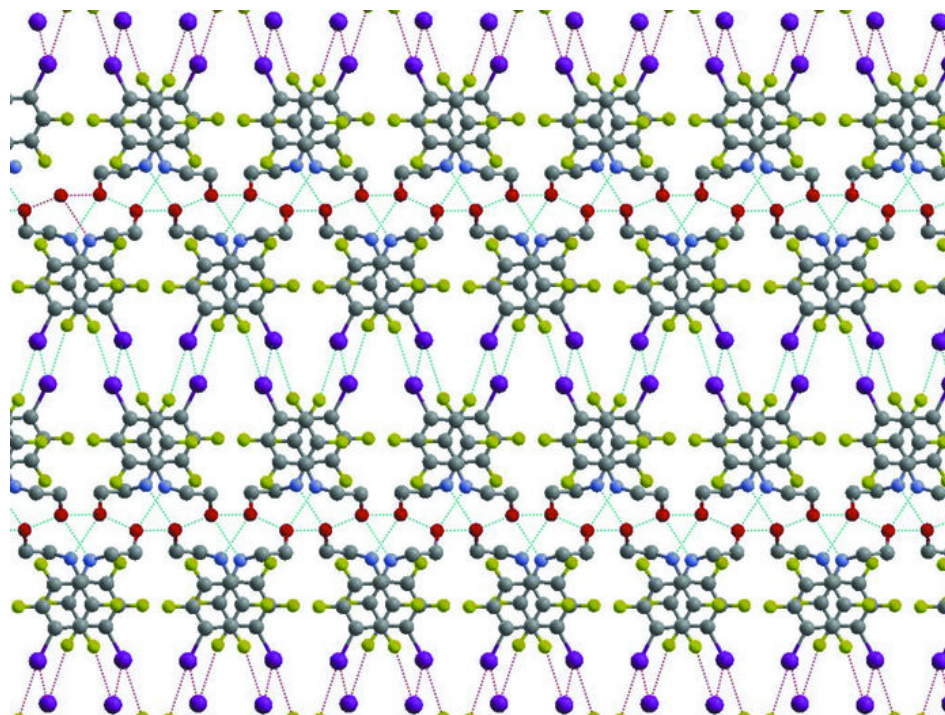


Fig. 3

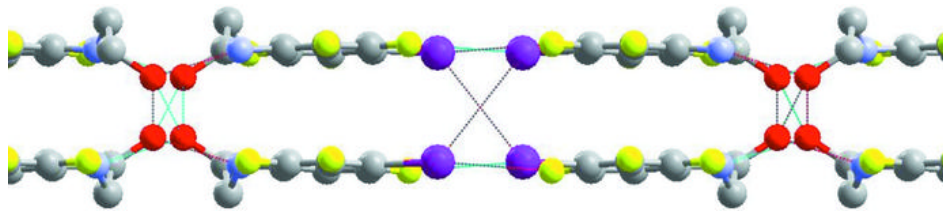


Fig. 4

