

3-(Trifluoromethyl)anilinium 2-carboxy-4,5-dichlorobenzoate¹

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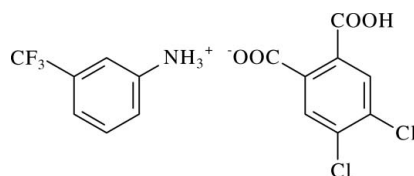
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.036; wR factor = 0.099; data-to-parameter ratio = 14.3.

The crystal structure of the title compound, $\text{C}_7\text{H}_7\text{F}_3\text{N}^+\cdot\text{C}_8\text{H}_3\text{Cl}_2\text{O}_4^-$, is stabilized by five $\text{N}-\text{H}\cdots\text{O}$, one $\text{O}-\text{H}\cdots\text{O}$, two $\text{C}-\text{H}\cdots\text{O}$ and three $\text{C}-\text{H}\cdots\text{X}$ ($\text{X} = \text{F}, \text{Cl}$) hydrogen bonds. Intramolecular $\text{O}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{X}$ hydrogen bonds generate edge-fused $[\text{S}(7)\text{R}_4^4(24)\text{S}(7)\text{R}_2^2(10)\text{S}(7)\text{R}_4^4(24)-\text{S}(7)]$ motifs. The cations and anions are linked to each other by $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming a chain along the c axis. This chain and the other hydrogen bonds form $[\text{R}_3^2(6)\text{R}_1^2(4)\text{S}(7)\text{R}_1^2(4)\text{R}_4^4(18)\text{R}_5^5(24)\text{R}_4^4(18)\text{R}_1^2(4)\text{R}_1^2(4)\text{S}(7)-\text{R}_1^2(4)\text{R}_3^2(6)]$ motifs. The dihedral angle between the planes of the aromatic rings in the cation and anion is 34.86 (1)°.

Related literature

For general background, see: Rigby *et al.* (1986); Buckingham (1993); Mas-Torrent *et al.* (2003); Setifi *et al.* (2002); Bryce & Petty (1995); Peng *et al.* (1998); Rombaut *et al.* (2001); Bredas *et al.* (1994); Etter (1990). For related structures, see: Odabaşoğlu & Büyükgüngör (2007a,b, 2006a,b); Odabaşoğlu *et al.* (2003); Yeşilel *et al.* (2006).



Experimental

Crystal data

$\text{C}_7\text{H}_7\text{F}_3\text{N}^+\cdot\text{C}_8\text{H}_3\text{Cl}_2\text{O}_4^-$ $b = 14.5270$ (9) Å
 $M_r = 396.14$ $c = 7.3292$ (3) Å
 Monoclinic, $P2_1/c$ $\beta = 99.887$ (4)°
 $a = 15.0048$ (7) Å $V = 1573.85$ (14) Å³

¹ Secondary interactions in organic halogen compounds. II. For Part I, see Odabaşoğlu & Büyükgüngör (2007b).

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.47$ mm⁻¹

$T = 296$ K
 $0.58 \times 0.45 \times 0.25$ mm

Data collection

Stoe IPDS 2 diffractometer
 Absorption correction: integration
 ($X\text{-RED32}$; Stoe & Cie, 2002)
 $T_{\min} = 0.776$, $T_{\max} = 0.895$
 28359 measured reflections
 3740 independent reflections
 2826 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.099$
 $S = 1.06$
 3740 reflections
 262 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.21$ e Å⁻³
 $\Delta\rho_{\min} = -0.27$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O3}^{\text{i}}$	0.88 (2)	1.92 (2)	2.788 (2)	169 (2)
$\text{N1}-\text{H1B}\cdots\text{O3}^{\text{ii}}$	0.95 (3)	2.72 (2)	3.360 (2)	125.8 (17)
$\text{N1}-\text{H1B}\cdots\text{O4}^{\text{iii}}$	0.95 (3)	1.86 (3)	2.789 (2)	168 (2)
$\text{N1}-\text{H1C}\cdots\text{O2}^{\text{iii}}$	0.88 (2)	1.89 (2)	2.7717 (18)	173.2 (19)
$\text{N1}-\text{H1C}\cdots\text{O1}^{\text{iii}}$	0.88 (2)	2.54 (2)	3.0518 (18)	117.8 (15)
$\text{O1}-\text{H1}\cdots\text{O4}$	0.82	1.57	2.3908 (17)	174
$\text{C3}-\text{H3}\cdots\text{F1}$	0.94 (2)	2.75 (2)	3.637 (2)	157.2 (15)
$\text{C15}-\text{H15}\cdots\text{F2}^{\text{iii}}$	0.95 (2)	2.76 (2)	3.582 (2)	144.7 (15)
$\text{C13}-\text{H13}\cdots\text{O3}^{\text{i}}$	0.97 (2)	2.95 (2)	3.571 (2)	122.7 (14)
$\text{C13}-\text{H13}\cdots\text{O3}^{\text{ii}}$	0.97 (2)	2.60 (2)	3.107 (2)	112.5 (14)
$\text{C13}-\text{H13}\cdots\text{Cl}^{\text{iv}}$	0.97 (2)	2.85 (2)	3.5693 (17)	131.4 (14)

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

Data collection: $X\text{-AREA}$ (Stoe & Cie, 2002); cell refinement: $X\text{-AREA}$; data reduction: $X\text{-RED32}$ (Stoe & Cie, 2002); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: $\text{ORTEP-3 for Windows}$ (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2370).

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supplementary materials

Acta Cryst. (2007). E63, o4374-o4375 [doi:10.1107/S1600536807051124]

3-(Trifluoromethyl)anilinium 2-carboxy-4,5-dichlorobenzoate

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Comment

Secondary interactions are proposed to be important in the biological activities of molecules involved in biochemical processes in the living cell. Molecular interactions find their origin in the dispersion, polarization, electrostatic, charge transfer and exchange forces (Rigby *et al.*, 1986; Buckingham, 1993). There is considerable current interest in the preparation of compounds constructed from two molecular networks as they provide a source of novel materials with highly anisotropic electrical, optical and magnetic properties (Mas-Torrent *et al.*, 2003; Setifi *et al.*, 2002; Bryce & Petty, 1995; Peng *et al.*, 1998; Rombaut *et al.*, 2001; Bredas *et al.*, 1994).

We have been interested in hydrogen-bonding systems formed by organic amines and carboxylic acids (Odabaşoğlu & Büyükgüngör, 2007a,b,c; 2006a,b; Odabaşoğlu *et al.*, 2003; Yeşilel *et al.*, 2006). The present work is part of a structural study of compounds of organic ammonium systems with hydrogen and halogen-bond donors and we report here the molecular and supramolecular structure of (I) (Fig. 1).

The C2–C7 ring and O1–C1–O2 and O3–C8–O4 planes are approximately coplanar with the dihedral angles between C2–C7 ring and O–C–O planes of 0.96 (2)° and 5.38 (1)°, respectively. The C1–C2 and C8–C7 bonds are approximately equal. This suggests some delocalization between the C2–C7 ring and the –COO[–] and –COOH groups that form an intramolecular hydrogen bond. Furthermore, these bonds, together with the C8–O3 and C8–O4 lengths indicate delocalization of the negative charge over the O3, C8 and O4 atoms. In the 4,5-dichlorohydrogenphthalate anion, the O1 and O4 distance is 2.3908 (17) Å and this distance is approximately 0.65 Å less than the sum of the van der Waals radii (3.04 Å) due to the presence of a strong intramolecular hydrogen bond in the anion (Table 1). The dihedral angle between the planes of the aromatic rings in the cation and anion is 34.86 (1)°.

Intramolecular O–H···O and C–H···F hydrogen bonds generate edge-fused [S(7)R₄⁴(24)S(7)R₂²(10)S(7)R₄⁴(24)S(7)] motifs (Etter, 1990) (Fig. 2). The ions are linked to each other by C–H···O hydrogen bonds chain along the *z*-axis (Fig. 3). This chain and other hydrogen bonds form [R₃²(6)R₁²(4)S(7)R₁²(4)R₄⁴(18)R₅⁵(24) R₄⁴(18)R₁²(4)R₁²(4)S(7)R₁²(4)R₃²(6)] motifs (Fig. 4).

Experimental

The title compound was prepared according to the method described by Odabaşoğlu & Büyükgüngör (2007c), using 3-(trifluoromethyl)aniline and 4,5-dichlorophthalic acid as starting materials (yield 95%; m.p. 465–466 K). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol–water (1:1) solution at room temperature.

Refinement

All H atoms bound to C and N were refined freely with isotropic displacement parameters. The O-bound H atom was refined using the riding model approximation with $d(\text{C}-\text{O}) = 0.82$ and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$

Figures

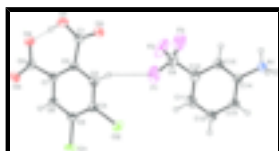


Fig. 1. A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bonds are drawn as dashed lines.

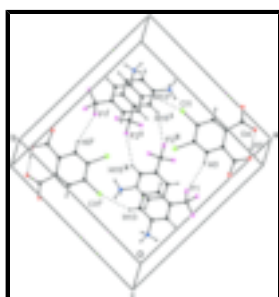


Fig. 2. Part of the crystal structure of (I), showing the formation of $S(7)R_4^4(24)S(7)R_2^2(10)S(7)R_4^4(24)S(7)$ motifs. H atoms not involved in hydrogen bonds (dashed lines) have been omitted for clarity. [Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $1-x, y+1/2, 1/2-z$; (iii) $x, 1-y, z-1/2$].

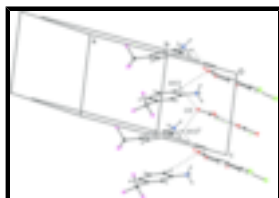


Fig. 3. Part of the crystal structure of (I), showing the linking of cations and anions by $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds into chains along the z -axis. Hydrogen bonds are drawn as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry code: (i) $x, 1/2-y, 1-z$].

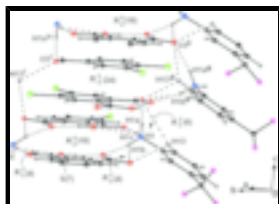


Fig. 4. Part of the crystal structure of (I), showing the formation of $[R_3^2(6)R_1^2(4)S(7)R_1^2(4)R_4^4(18)R_5^5(24)R_4^4(18)R_1^2(4)R_1^2(4)S(7)R_1^2(4)R_3^2(6)]$ motifs. Hydrogen bonds are drawn as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) $-x, y-1/2, 1/2-z$; (ii) $1-x, y-1/2, 1/2-z$; (iii) $x, y-1/2, z-1/2$; (iv) $-x, -y, -z$].

3-(Trifluoromethyl)anilinium 2-carboxy-4,5-dichlorobenzoate

Crystal data

$\text{C}_7\text{H}_7\text{F}_3\text{N}^+\cdot\text{C}_8\text{H}_3\text{Cl}_2\text{O}_4^-$

$M_r = 396.14$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 15.0048$ (7) Å

$b = 14.5270$ (9) Å

$c = 7.3292$ (3) Å

$\beta = 99.887$ (4)°

$F_{000} = 800$

$D_x = 1.672$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 28359 reflections

$\theta = 2.0$ – 28.0 °

$\mu = 0.47$ mm⁻¹

$T = 296$ K

Prism, colourless

$V = 1573.85 (14) \text{ \AA}^3$
 $Z = 4$ $0.58 \times 0.45 \times 0.25 \text{ mm}$

Data collection

Stoe IPDS 2 diffractometer	3740 independent reflections
Monochromator: plane graphite	2826 reflections with $I > 2\sigma(I)$
Detector resolution: 6.67 pixels mm^{-1}	$R_{\text{int}} = 0.032$
$T = 296 \text{ K}$	$\theta_{\text{max}} = 27.9^\circ$
ω scans	$\theta_{\text{min}} = 2.0^\circ$
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	$h = -19 \rightarrow 19$
$T_{\text{min}} = 0.776, T_{\text{max}} = 0.895$	$k = -19 \rightarrow 19$
28359 measured reflections	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.099$	$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 0.1939P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
3740 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
262 parameters	$\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$
	Extinction correction: none

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 $F^2 > 2\sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
C8	1.03044 (10)	0.23067 (10)	0.5787 (2)	0.0418 (3)
C7	0.93692 (9)	0.26142 (9)	0.6066 (2)	0.0379 (3)

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C6	0.92664 (11)	0.35648 (10)	0.6057 (2)	0.0466 (4)
C5	0.84683 (11)	0.39823 (10)	0.6262 (2)	0.0492 (4)
C4	0.77395 (11)	0.34493 (11)	0.6513 (2)	0.0478 (4)
C3	0.78183 (11)	0.25045 (11)	0.6524 (2)	0.0444 (3)
C2	0.86192 (9)	0.20676 (9)	0.6303 (2)	0.0378 (3)
C1	0.85570 (10)	0.10270 (10)	0.6377 (2)	0.0438 (3)
C9	0.51139 (13)	0.12618 (18)	0.6703 (3)	0.0723 (6)
C10	0.42647 (11)	0.17517 (13)	0.5905 (2)	0.0517 (4)
C11	0.42547 (12)	0.26998 (14)	0.5840 (3)	0.0574 (4)
C12	0.34774 (12)	0.31557 (13)	0.5041 (3)	0.0557 (4)
C13	0.27120 (11)	0.26640 (11)	0.4323 (3)	0.0475 (4)
C14	0.27297 (10)	0.17171 (10)	0.4429 (2)	0.0413 (3)
C15	0.34971 (11)	0.12457 (12)	0.5200 (2)	0.0466 (4)
N1	0.19072 (9)	0.12074 (10)	0.3714 (2)	0.0460 (3)
O3	1.08795 (8)	0.28955 (8)	0.5704 (2)	0.0615 (3)
O4	1.04647 (8)	0.14594 (8)	0.5608 (2)	0.0614 (4)
O2	0.78483 (8)	0.06732 (8)	0.6604 (2)	0.0600 (3)
O1	0.92388 (9)	0.05241 (8)	0.6222 (2)	0.0709 (4)
H1	0.9667	0.0853	0.6089	0.106*
F1	0.56882 (9)	0.17904 (13)	0.7817 (3)	0.1085 (6)
F2	0.55617 (10)	0.09759 (15)	0.5410 (3)	0.1219 (7)
F3	0.49697 (10)	0.05389 (11)	0.7687 (3)	0.1119 (6)
Cl1	0.84144 (4)	0.51638 (3)	0.62076 (9)	0.0786 (2)
Cl2	0.67383 (3)	0.39467 (4)	0.68542 (9)	0.07518 (19)
H1A	0.1623 (15)	0.1449 (15)	0.267 (3)	0.066 (6)*
H1B	0.1476 (16)	0.1275 (16)	0.451 (3)	0.071 (6)*
H1C	0.2018 (13)	0.0619 (15)	0.355 (3)	0.057 (5)*
H6	0.9769 (14)	0.3918 (13)	0.588 (3)	0.056 (5)*
H3	0.7311 (13)	0.2155 (13)	0.670 (3)	0.055 (5)*
H11	0.4770 (15)	0.3036 (14)	0.637 (3)	0.063 (6)*
H12	0.3451 (14)	0.3819 (15)	0.495 (3)	0.068 (6)*
H13	0.2163 (14)	0.2967 (13)	0.373 (3)	0.060 (5)*
H15	0.3498 (13)	0.0592 (14)	0.524 (3)	0.056 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C8	0.0378 (7)	0.0417 (7)	0.0445 (9)	-0.0005 (6)	0.0035 (6)	0.0002 (6)
C7	0.0376 (7)	0.0334 (6)	0.0406 (8)	0.0016 (5)	0.0010 (6)	-0.0012 (6)
C6	0.0444 (8)	0.0349 (7)	0.0579 (10)	-0.0024 (6)	0.0014 (7)	-0.0004 (7)
C5	0.0525 (9)	0.0321 (7)	0.0582 (10)	0.0059 (6)	-0.0039 (7)	-0.0068 (7)
C4	0.0421 (8)	0.0443 (8)	0.0538 (10)	0.0093 (6)	-0.0007 (7)	-0.0096 (7)
C3	0.0392 (7)	0.0417 (8)	0.0511 (9)	0.0003 (6)	0.0042 (6)	-0.0063 (7)
C2	0.0376 (7)	0.0333 (7)	0.0407 (8)	0.0010 (5)	0.0021 (6)	-0.0024 (6)
C1	0.0429 (8)	0.0339 (7)	0.0535 (9)	-0.0007 (6)	0.0050 (6)	-0.0018 (6)
C9	0.0433 (9)	0.0919 (16)	0.0801 (15)	0.0032 (10)	0.0062 (9)	0.0066 (12)
C10	0.0404 (8)	0.0640 (10)	0.0521 (10)	-0.0006 (7)	0.0115 (7)	0.0016 (8)
C11	0.0471 (9)	0.0653 (11)	0.0615 (12)	-0.0156 (8)	0.0144 (8)	-0.0071 (9)

C12	0.0563 (10)	0.0439 (9)	0.0700 (12)	-0.0097 (7)	0.0196 (9)	-0.0047 (8)
C13	0.0458 (8)	0.0388 (8)	0.0601 (11)	0.0005 (6)	0.0155 (7)	0.0015 (7)
C14	0.0379 (7)	0.0389 (7)	0.0493 (9)	-0.0023 (6)	0.0136 (6)	-0.0007 (6)
C15	0.0409 (8)	0.0442 (8)	0.0562 (10)	0.0030 (6)	0.0127 (7)	0.0034 (7)
N1	0.0370 (7)	0.0354 (7)	0.0658 (10)	0.0003 (5)	0.0095 (7)	0.0004 (6)
O3	0.0456 (6)	0.0535 (7)	0.0879 (10)	-0.0113 (5)	0.0187 (6)	-0.0086 (6)
O4	0.0463 (6)	0.0402 (6)	0.1027 (11)	0.0069 (5)	0.0264 (7)	0.0044 (6)
O2	0.0459 (6)	0.0378 (6)	0.0974 (10)	-0.0050 (5)	0.0154 (6)	-0.0002 (6)
O1	0.0536 (7)	0.0325 (6)	0.1329 (13)	0.0017 (5)	0.0339 (8)	0.0008 (7)
F1	0.0599 (8)	0.1288 (13)	0.1226 (13)	-0.0077 (8)	-0.0243 (8)	0.0022 (10)
F2	0.0727 (9)	0.1784 (18)	0.1183 (13)	0.0540 (10)	0.0266 (9)	-0.0015 (12)
F3	0.0657 (8)	0.1102 (12)	0.1504 (15)	0.0109 (8)	-0.0083 (8)	0.0569 (11)
Cl1	0.0804 (3)	0.0313 (2)	0.1167 (5)	0.0096 (2)	-0.0044 (3)	-0.0089 (2)
Cl2	0.0498 (2)	0.0661 (3)	0.1078 (4)	0.0182 (2)	0.0082 (2)	-0.0247 (3)

Geometric parameters (Å, °)

C8—O3	1.2239 (19)	C9—F1	1.326 (3)
C8—O4	1.2653 (19)	C9—C10	1.489 (3)
C8—C7	1.520 (2)	C10—C11	1.378 (3)
C7—C6	1.389 (2)	C10—C15	1.389 (2)
C7—C2	1.412 (2)	C11—C12	1.381 (3)
C6—C5	1.374 (2)	C11—H11	0.94 (2)
C6—H6	0.94 (2)	C12—C13	1.378 (2)
C5—C4	1.378 (2)	C12—H12	0.97 (2)
C5—Cl1	1.7184 (15)	C13—C14	1.378 (2)
C4—C3	1.377 (2)	C13—H13	0.97 (2)
C4—Cl2	1.7235 (16)	C14—C15	1.375 (2)
C3—C2	1.393 (2)	C14—N1	1.4574 (19)
C3—H3	0.94 (2)	C15—H15	0.95 (2)
C2—C1	1.5161 (19)	N1—H1A	0.88 (2)
C1—O2	1.2180 (19)	N1—H1B	0.95 (3)
C1—O1	1.2781 (19)	N1—H1C	0.88 (2)
C9—F3	1.313 (3)	O1—H1	0.8200
C9—F2	1.319 (3)		
O3—C8—O4	121.76 (14)	F2—C9—C10	112.05 (19)
O3—C8—C7	118.47 (13)	F1—C9—C10	112.8 (2)
O4—C8—C7	119.75 (13)	C11—C10—C15	120.84 (16)
C6—C7—C2	118.00 (13)	C11—C10—C9	119.66 (17)
C6—C7—C8	113.31 (13)	C15—C10—C9	119.49 (17)
C2—C7—C8	128.69 (12)	C10—C11—C12	119.82 (16)
C5—C6—C7	122.44 (15)	C10—C11—H11	120.1 (12)
C5—C6—H6	120.7 (12)	C12—C11—H11	120.1 (12)
C7—C6—H6	116.9 (12)	C13—C12—C11	120.08 (17)
C6—C5—C4	119.56 (14)	C13—C12—H12	118.0 (13)
C6—C5—Cl1	118.49 (13)	C11—C12—H12	121.9 (13)
C4—C5—Cl1	121.95 (13)	C14—C13—C12	119.30 (16)
C3—C4—C5	119.44 (14)	C14—C13—H13	119.0 (11)
C3—C4—Cl2	119.53 (13)	C12—C13—H13	121.7 (11)

supplementary materials

C5—C4—C12	121.02 (12)	C15—C14—C13	121.80 (15)
C4—C3—C2	121.88 (15)	C15—C14—N1	119.50 (14)
C4—C3—H3	117.9 (11)	C13—C14—N1	118.69 (14)
C2—C3—H3	120.2 (11)	C14—C15—C10	118.14 (15)
C3—C2—C7	118.67 (13)	C14—C15—H15	120.5 (12)
C3—C2—C1	112.98 (13)	C10—C15—H15	121.3 (12)
C7—C2—C1	128.35 (13)	C14—N1—H1A	111.4 (14)
O2—C1—O1	120.13 (14)	C14—N1—H1B	110.6 (14)
O2—C1—C2	119.09 (14)	H1A—N1—H1B	102 (2)
O1—C1—C2	120.77 (14)	C14—N1—H1C	111.9 (13)
F3—C9—F2	107.2 (2)	H1A—N1—H1C	110.1 (19)
F3—C9—F1	106.0 (2)	H1B—N1—H1C	110.2 (19)
F2—C9—F1	105.51 (19)	C1—O1—H1	109.5
F3—C9—C10	112.80 (17)		
O3—C8—C7—C6	4.5 (2)	C3—C2—C1—O2	0.3 (2)
O4—C8—C7—C6	-173.96 (15)	C7—C2—C1—O2	179.50 (16)
O3—C8—C7—C2	-176.27 (16)	C3—C2—C1—O1	-179.04 (16)
O4—C8—C7—C2	5.2 (3)	C7—C2—C1—O1	0.2 (3)
C2—C7—C6—C5	-0.2 (3)	F3—C9—C10—C11	141.3 (2)
C8—C7—C6—C5	179.10 (15)	F2—C9—C10—C11	-97.7 (3)
C7—C6—C5—C4	0.9 (3)	F1—C9—C10—C11	21.2 (3)
C7—C6—C5—C11	-179.42 (13)	F3—C9—C10—C15	-40.1 (3)
C6—C5—C4—C3	-1.0 (3)	F2—C9—C10—C15	81.0 (3)
C11—C5—C4—C3	179.28 (14)	F1—C9—C10—C15	-160.16 (19)
C6—C5—C4—C12	177.78 (14)	C15—C10—C11—C12	-0.9 (3)
C11—C5—C4—C12	-1.9 (2)	C9—C10—C11—C12	177.70 (19)
C5—C4—C3—C2	0.5 (3)	C10—C11—C12—C13	0.5 (3)
C12—C4—C3—C2	-178.33 (13)	C11—C12—C13—C14	0.6 (3)
C4—C3—C2—C7	0.2 (2)	C12—C13—C14—C15	-1.3 (3)
C4—C3—C2—C1	179.50 (15)	C12—C13—C14—N1	178.19 (16)
C6—C7—C2—C3	-0.4 (2)	C13—C14—C15—C10	0.8 (3)
C8—C7—C2—C3	-179.51 (15)	N1—C14—C15—C10	-178.66 (16)
C6—C7—C2—C1	-179.54 (15)	C11—C10—C15—C14	0.3 (3)
C8—C7—C2—C1	1.3 (3)	C9—C10—C15—C14	-178.33 (17)

Hydrogen-bond geometry (\AA , $^\circ$)

<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—H1A...O3 ⁱ	0.88 (2)	1.92 (2)	2.788 (2)	169 (2)
N1—H1B...O3 ⁱⁱ	0.95 (3)	2.72 (2)	3.360 (2)	125.8 (17)
N1—H1B...O4 ⁱⁱ	0.95 (3)	1.86 (3)	2.789 (2)	168 (2)
N1—H1C...O2 ⁱⁱⁱ	0.88 (2)	1.89 (2)	2.7717 (18)	173.2 (19)
N1—H1C...O1 ⁱⁱⁱ	0.88 (2)	2.54 (2)	3.0518 (18)	117.8 (15)
O1—H1...O4	0.82	1.57	2.3908 (17)	174
C3—H3...F1	0.94 (2)	2.75 (2)	3.637 (2)	157.2 (15)
C15—H15...F2 ⁱⁱⁱ	0.95 (2)	2.76 (2)	3.582 (2)	144.7 (15)
C13—H13...O3 ⁱ	0.97 (2)	2.95 (2)	3.571 (2)	122.7 (14)

C13—H13...O3 ⁱⁱ	0.97 (2)	2.60 (2)	3.107 (2)	112.5 (14)
C13—H13...Cl1 ^{iv}	0.97 (2)	2.85 (2)	3.5693 (17)	131.4 (14)

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

Fig. 1

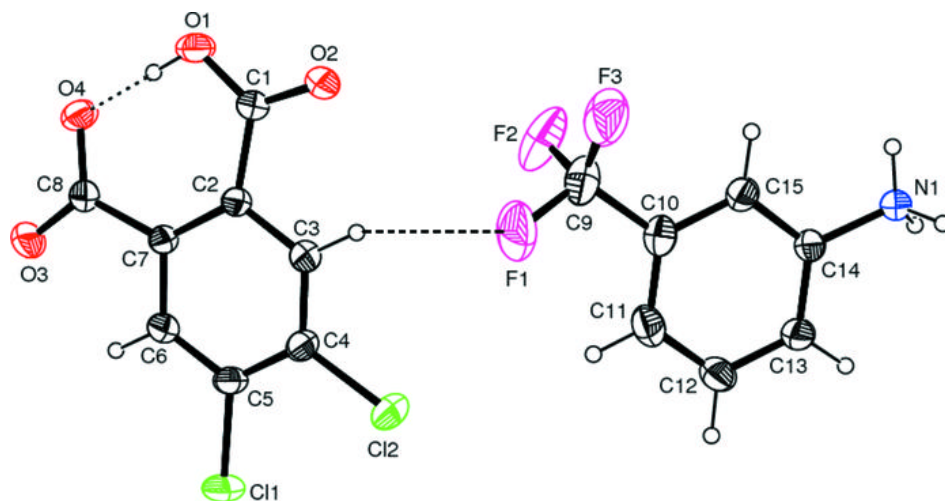


Fig. 2

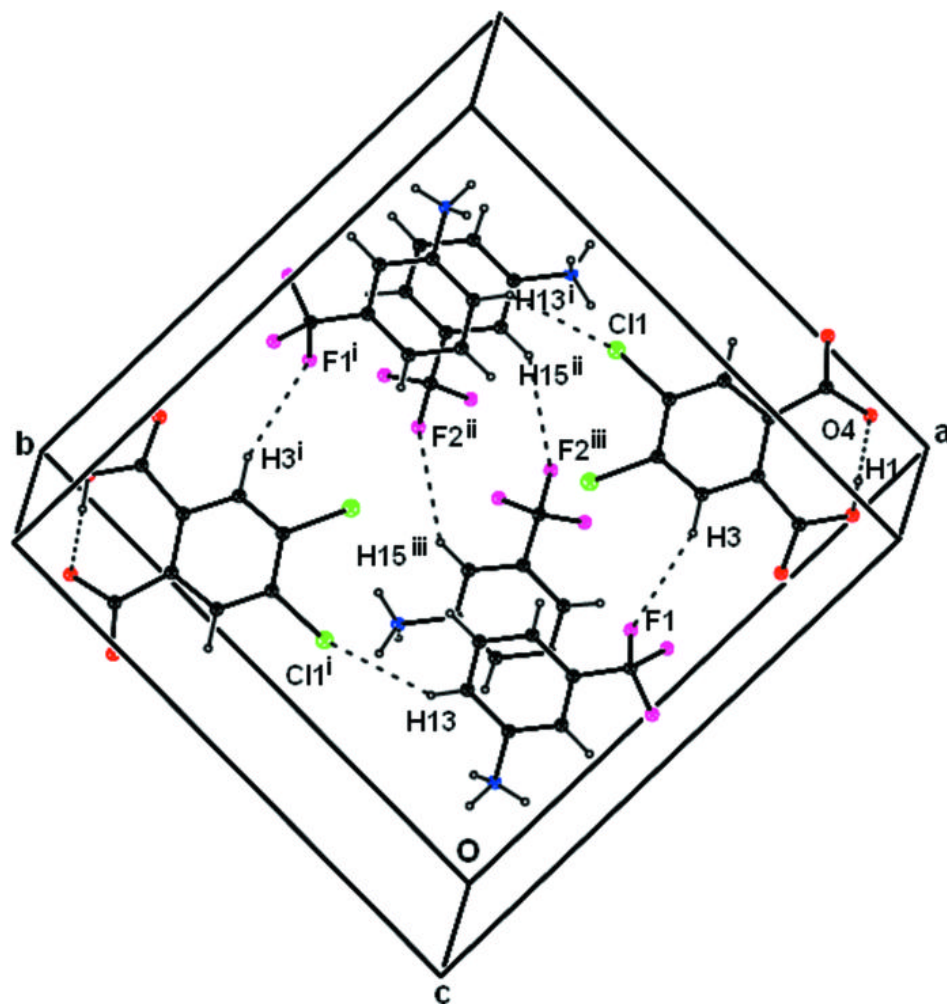


Fig. 3

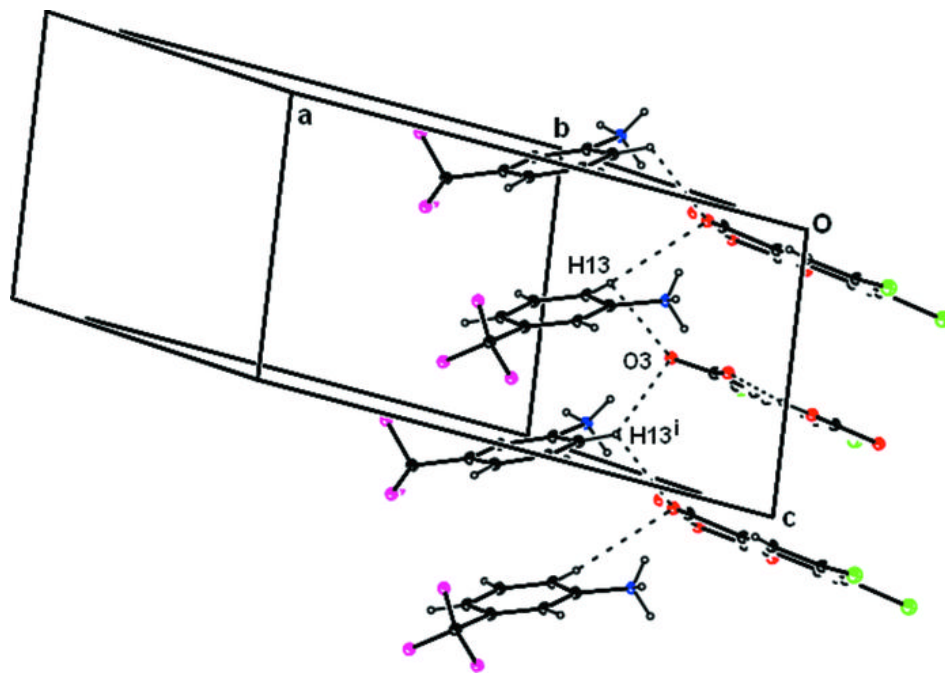


Fig. 4

