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Bis(4-ethylanilinium) 4,5-dichlorophthalate[1]

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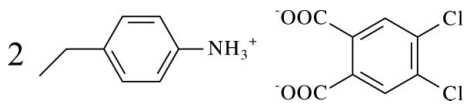
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.052; wR factor = 0.145; data-to-parameter ratio = 14.6.

The crystal structure of the title compound, $2C_8H_{12}N^+\cdot C_8H_3Cl_2O_4^{2-}$, is stabilized by nine N—H···O and four C—H···O hydrogen bonds, and also by C—H···π interactions. Intramolecular C—H···O hydrogen bonds form $C(6)$ chains along the c axis. This chain and the other hydrogen bonds generate edge-fused $[R_1^2(6)R_1^2(4)R_4^3(10)R_1^2(4)R_3^2(9)]$ motifs in a three-dimensional network. The dihedral angles between the carboxylate anion and the cation aromatic ring planes are 75.90 (2) and 68.15 (2)°, and the dihedral angles between the carboxylate groups and the anion aromatic ring plane are 13.72 (3) and 84.64 (3)°.

Related literature

For related literature, see: Bozkurt *et al.* (2006); Braga *et al.* (2002); Büyükgüngör & Odabaşoğlu (2002); Büyükgüngör & Odabaşoğlu (2003); Büyükgüngör & Odabaşoğlu (2006*a*,*b*); Ersanlı *et al.* (2004); Etter (1990); Goswami *et al.* (1998); Goswami & Ghosh (1997); Joesten & Schaad (1974); Lam & Mak (2000); Mulliken & Person (1969); Odabaşoğlu & Büyükgüngör (2006*a*,*b*,*c*,*d*); Odabaşoğlu & Büyükgüngör (2007*a*,*b*,*c*); Odabaşoğlu *et al.* (2003*a*,*b*); Pimentel & McClellan (1960); Scheiner (1997*a*); Scheiner (1997*b*); Temel *et al.* (2007); Yeşilel *et al.* (2006).



Experimental

Crystal data

$2C_8H_{12}N^+\cdot C_8H_3Cl_2O_4^{2-}$
M_r = 478.38
Monoclinic, $P2_1/c$
a = 16.9642 (16) Å
b = 11.8744 (8) Å
c = 11.8783 (11) Å
β = 103.133 (8)°
V = 2330.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 296 K
0.78 × 0.45 × 0.23 mm

Data collection

Stoe IPDS2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.828, T_{max} = 0.932
16318 measured reflections
4585 independent reflections
2666 reflections with $I > 2\sigma(I)$
R_{int} = 0.069

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.145
S = 0.92
4585 reflections
314 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 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
N1—H1*B*···O1^{i}	0.99 (4)	2.67 (3)	3.442 (3)	134 (2)
N1—H1*C*···O1^{ii}	0.86 (4)	1.90 (4)	2.747 (4)	172 (3)
N1—H1*B*···O2^{i}	0.99 (4)	1.77 (4)	2.746 (3)	166 (3)
N1—H1*A*···O3^{iii}	0.93 (4)	2.24 (4)	3.021 (3)	140 (3)
N1—H1*A*···O4^{iii}	0.93 (4)	2.15 (4)	3.013 (4)	154 (3)
N2—H2*C*···O2^{i}	0.84 (5)	2.20 (5)	2.784 (4)	126 (4)
N2—H2*A*···O3^{iv}	0.98 (5)	1.94 (5)	2.913 (5)	174 (4)
N2—H2*C*···O4^{i}	0.84 (5)	2.58 (5)	3.350 (5)	152 (4)
N2—H2*B*···O4^{v}	0.92 (4)	1.83 (4)	2.739 (4)	172 (3)
C6—H6···O1^{vi}	0.93	2.52	3.388 (3)	154
C13—H13···O3^{i}	0.93	2.54	3.402 (4)	154
C15—H15···O1^{ii}	0.93	2.55	3.254 (4)	133
C23—H23···O3^{iv}	0.93	2.93	3.641 (4)	134
C23—H23···*Cg*1	0.93	2.69	3.389 (4)	133

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *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: AT2423).

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supplementary materials

Acta Cryst. (2007). E**63**, o4376-o4377 [**doi:10.1107/S1600536807050490**]

Bis(4-ethylanilinium) 4,5-dichlorophthalate

O. Büyükgüngör and M. Odabasoglu

Comment

We have been interested in hydrogen-bonding systems formed by organic amines and carboxylic acids (Odabaşoğlu & Büyükgüngör, 2007*a*-c; 2006*a*-d; Temel *et al.*, 2007; Odabaşoğlu *et al.*, 2003; Büyükgüngör & Odabaşoğlu, 2006*a*,b; Büyükgüngör & Odabaşoğlu, 2003; Büyükgüngör & Odabaşoğlu, 2002; Ersanlı *et al.*, 2004; Bozkurt *et al.*, 2006; 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) (Figure 1).

In the phthalate anion, O1—C1—O2—C2 and O3—C8—O4—C7 planes and the plane of C2–C7 ring are not the same plane. The dihedral angles between the C2–C7 ring and O1—C1—O2—C2, O3—C8—O4—C7 planes are 13.72 (3)° and 84,64 (3)°, respectively. The dihedral angles between the aromatic C2–C7 (A), C11–C16 (B) and C19–C24 (C) rings planes are 75.90 (2)° (A/B), 68.15 (2)° (A/C), and 9.01 (2)° (B/C).

The ions are linked to each other by C6—H6···O1 hydrogen bonds forming C(6) chain along the *z*-axis (Fig. 2). Other hydrogen bons form [$R_1^2(6)R_1^2(4)R_4^3(10)R_1^2(4)R_3^2(9)$] (Etter, 1990) motifs (Fig. 3). The C—H···π interaction and hydrogen bonds properties are given in Table1.

Experimental

The title compound was prepared according to the method described by Odabaşoğlu & Büyükgüngör (2007*c*), using 3-ethylaniline and 4,5-dichlorophthalic acid as starting materials (yield 95%; m.p. 458–459 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 except bounded to N were refined using the riding model approximation with d(C—H) = 0.93 for aromatic, d(C—H) = 0.97 for methylene, d(C—H) = 0.96 for methyl and d(C—O) = 0.82 for hydroxyl H. (U_{iso}(H) = (1.2–1.5)U_{eq}(parent atom)]. N-bound H atoms were located in Fourier difference map and refined freely.

Figures

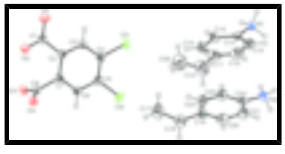
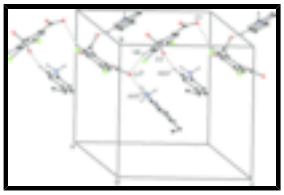
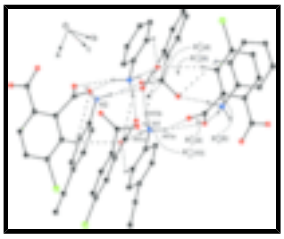
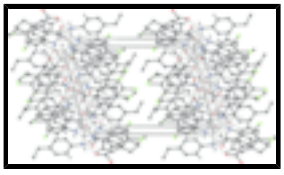


Figure 1. A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level..

Figure 2. Part of the crystal structure of (I), showing the C(6) chain along the *c* axis. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) 1 − *x*, 1 − *y*, 1 − *z*; (ii) *x*, 3/2 − *y*, 1 − *z*].

Figure 3. Part of the crystal structure of (I), showing the hydrogen bonding $R_1^2(6)R_1^2(4)R_4^3(10)$ $R_1^2(4)R_3^2(9)$ motif. H atoms not involved in hydrogen bonds have been omitted for clarity.

Bis(4-ethylanilinium) 4,5-dichlorophthalate

Crystal data

$2C_7H_7F_3N^+·C_8H_3Cl_2O_4^{2-}$	F_{000} = 1004
M_r = 478.38	D_x = 1.364 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 16318 reflections
a = 16.9642 (16) Å	θ = 2.1–27.9º
b = 11.8744 (8) Å	μ = 0.31 mm^{-1}
c = 11.8783 (11) Å	T = 296 K
β = 103.133 (8)º	Prism, colourless
V = 2330.2 (3) Å^3	0.78 × 0.45 × 0.23 mm
Z = 4	

Data collection

Stoe IPDS2 diffractometer	4585 independent reflections
Monochromator: plane graphite	2666 reflections with $I > 2\sigma(I)$
Detector resolution: 6.67 pixels mm^{-1}	R_{int} = 0.069
T = 296 K	θ_{max} = 26.0º
ω scans	θ_{min} = 2.1º
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	h = −20→20
T_{min} = 0.828, T_{max} = 0.932	k = −14→14
16318 measured reflections	l = −14→14

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.052$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.145$	$w = 1/[\sigma^2(F_o^2) + (0.0797P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.92$	$(\Delta/\sigma)_{max} < 0.001$
4585 reflections	$\Delta\rho_{max} = 0.62$ e Å^{-3}
314 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
3 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.89830 (17)	0.9151 (2)	0.7758 (2)	0.0419 (6)
C2	0.83587 (16)	0.8676 (2)	0.6751 (2)	0.0393 (6)
C3	0.75484 (17)	0.8843 (2)	0.6729 (2)	0.0446 (7)
H3	0.7405	0.9267	0.7310	0.054*
C4	0.69517 (16)	0.8395 (3)	0.5866 (2)	0.0468 (7)
C5	0.71630 (17)	0.7758 (3)	0.5007 (2)	0.0468 (7)
C6	0.79687 (17)	0.7593 (2)	0.5006 (2)	0.0452 (7)
H6	0.8107	0.7178	0.4415	0.054*
C7	0.85740 (16)	0.8040 (2)	0.5880 (2)	0.0386 (6)
C8	0.94313 (17)	0.7689 (3)	0.5895 (2)	0.0450 (7)
C10	0.3417 (3)	0.8015 (5)	0.3264 (6)	0.1243 (19)
H10A	0.3462	0.7593	0.2581	0.149*
H10B	0.3283	0.8787	0.3027	0.149*
C11	0.2713 (3)	0.7511 (3)	0.3748 (4)	0.0775 (11)
C12	0.1952 (3)	0.7812 (4)	0.3238 (4)	0.0795 (11)
H12	0.1865	0.8332	0.2637	0.095*

C13	0.1310 (2)	0.7375 (3)	0.3581 (3)	0.0655 (9)
H13	0.0788	0.7594	0.3215	0.079*
C14	0.14248 (18)	0.6603 (3)	0.4476 (2)	0.0470 (7)
C15	0.21790 (19)	0.6257 (3)	0.5022 (3)	0.0611 (9)
H15	0.2254	0.5734	0.5619	0.073*
C16	0.2846 (2)	0.6721 (4)	0.4650 (4)	0.0795 (11)
H16	0.3371	0.6502	0.5002	0.095*
C17	0.4675 (3)	0.4776 (5)	0.3165 (5)	0.1188 (18)
H17A	0.5224	0.4905	0.3111	0.178*
H17B	0.4594	0.3986	0.3264	0.178*
H17C	0.4570	0.5181	0.3814	0.178*
C18	0.4113 (2)	0.5174 (4)	0.2085 (4)	0.0857 (12)
H18A	0.4242	0.4774	0.1437	0.103*
H18B	0.4219	0.5967	0.1986	0.103*
C19	0.3213 (2)	0.5033 (3)	0.2024 (3)	0.0582 (8)
C20	0.2659 (3)	0.5503 (3)	0.1126 (3)	0.0707 (10)
H20	0.2844	0.5928	0.0581	0.085*
C21	0.1838 (2)	0.5367 (3)	0.1003 (3)	0.0666 (9)
H21	0.1476	0.5698	0.0386	0.080*
C22	0.15610 (18)	0.4730 (3)	0.1807 (3)	0.0490 (7)
C23	0.2101 (2)	0.4279 (3)	0.2713 (3)	0.0625 (9)
H23	0.1918	0.3866	0.3269	0.075*
C24	0.2915 (2)	0.4426 (3)	0.2815 (3)	0.0661 (9)
H24	0.3276	0.4105	0.3440	0.079*
C26	0.4134 (4)	0.8002 (7)	0.4016 (6)	0.155 (3)
H26A	0.4537	0.8321	0.3661	0.233*
H26B	0.4278	0.7240	0.4243	0.233*
H26C	0.4101	0.8437	0.4685	0.233*
N1	0.07153 (15)	0.6147 (3)	0.4812 (2)	0.0472 (6)
N2	0.07019 (19)	0.4498 (3)	0.1670 (3)	0.0636 (8)
O1	0.87227 (13)	0.9484 (2)	0.86003 (19)	0.0649 (6)
H1	0.9093	0.9490	0.9180	0.097*
O2	0.97009 (11)	0.91689 (17)	0.76807 (17)	0.0506 (5)
O3	0.96912 (12)	0.68350 (18)	0.64711 (18)	0.0552 (5)
O4	0.98173 (12)	0.8219 (2)	0.52755 (18)	0.0606 (6)
Cl1	0.59467 (5)	0.86080 (9)	0.58955 (8)	0.0726 (3)
Cl2	0.64334 (5)	0.71200 (9)	0.39448 (7)	0.0705 (3)
H1A	0.048 (3)	0.671 (4)	0.521 (4)	0.094 (14)*
H1B	0.032 (2)	0.592 (3)	0.410 (3)	0.059 (9)*
H1C	0.085 (2)	0.562 (3)	0.531 (3)	0.055 (10)*
H2A	0.061 (3)	0.405 (4)	0.233 (4)	0.099 (15)*
H2B	0.053 (2)	0.412 (3)	0.099 (3)	0.064 (10)*
H2C	0.042 (3)	0.508 (4)	0.154 (4)	0.094 (16)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0444 (16)	0.0418 (15)	0.0400 (15)	−0.0017 (13)	0.0109 (13)	0.0024 (12)

C2	0.0373 (14)	0.0427 (15)	0.0385 (14)	0.0007 (12)	0.0098 (11)	0.0037 (12)
C3	0.0404 (14)	0.0529 (17)	0.0413 (15)	0.0054 (13)	0.0107 (12)	−0.0009 (13)
C4	0.0346 (14)	0.0601 (18)	0.0463 (16)	0.0020 (13)	0.0102 (12)	0.0059 (14)
C5	0.0408 (15)	0.0549 (18)	0.0423 (15)	−0.0059 (13)	0.0042 (12)	0.0019 (14)
C6	0.0464 (16)	0.0531 (17)	0.0375 (14)	−0.0001 (13)	0.0123 (13)	−0.0009 (13)
C7	0.0379 (13)	0.0425 (15)	0.0366 (13)	0.0008 (12)	0.0114 (11)	0.0029 (12)
C8	0.0418 (15)	0.0593 (19)	0.0337 (14)	0.0011 (14)	0.0085 (12)	−0.0067 (14)
C10	0.090 (4)	0.117 (4)	0.173 (6)	−0.019 (3)	0.043 (4)	−0.026 (4)
C11	0.097 (3)	0.066 (2)	0.084 (3)	−0.027 (2)	0.052 (2)	−0.018 (2)
C12	0.091 (3)	0.080 (3)	0.073 (2)	−0.017 (2)	0.029 (2)	0.003 (2)
C13	0.069 (2)	0.069 (2)	0.059 (2)	−0.0064 (18)	0.0153 (17)	0.0029 (17)
C14	0.0458 (16)	0.0553 (18)	0.0421 (15)	−0.0014 (13)	0.0148 (13)	−0.0089 (14)
C15	0.0466 (18)	0.070 (2)	0.069 (2)	0.0003 (16)	0.0174 (16)	−0.0007 (18)
C16	0.0445 (18)	0.088 (3)	0.109 (3)	−0.0009 (18)	0.022 (2)	−0.025 (2)
C17	0.064 (3)	0.145 (5)	0.139 (5)	−0.011 (3)	0.005 (3)	−0.013 (4)
C18	0.070 (3)	0.091 (3)	0.099 (3)	−0.014 (2)	0.026 (2)	−0.016 (2)
C19	0.059 (2)	0.0532 (19)	0.064 (2)	−0.0058 (16)	0.0174 (17)	−0.0085 (17)
C20	0.087 (3)	0.064 (2)	0.066 (2)	−0.007 (2)	0.028 (2)	0.0127 (18)
C21	0.074 (2)	0.064 (2)	0.059 (2)	0.0117 (18)	0.0100 (18)	0.0138 (17)
C22	0.0506 (17)	0.0477 (17)	0.0496 (17)	0.0036 (14)	0.0131 (14)	−0.0100 (14)
C23	0.065 (2)	0.068 (2)	0.0549 (19)	−0.0063 (17)	0.0145 (17)	0.0163 (17)
C24	0.058 (2)	0.071 (2)	0.065 (2)	−0.0010 (18)	0.0043 (17)	0.0144 (18)
C26	0.106 (5)	0.217 (8)	0.144 (5)	−0.024 (5)	0.030 (4)	−0.011 (5)
N1	0.0397 (13)	0.0626 (18)	0.0404 (14)	0.0060 (13)	0.0112 (12)	0.0018 (14)
N2	0.0565 (18)	0.069 (2)	0.065 (2)	0.0073 (16)	0.0134 (16)	−0.0157 (18)
O1	0.0560 (13)	0.0947 (18)	0.0461 (12)	−0.0116 (13)	0.0156 (11)	−0.0244 (12)
O2	0.0368 (11)	0.0623 (13)	0.0521 (12)	−0.0064 (9)	0.0091 (9)	−0.0079 (10)
O3	0.0502 (12)	0.0549 (13)	0.0599 (13)	0.0133 (10)	0.0112 (10)	0.0027 (11)
O4	0.0424 (11)	0.0909 (17)	0.0525 (12)	0.0032 (11)	0.0194 (10)	0.0124 (12)
Cl1	0.0358 (4)	0.1121 (8)	0.0693 (6)	0.0081 (4)	0.0108 (4)	0.0000 (5)
Cl2	0.0515 (5)	0.0935 (7)	0.0599 (5)	−0.0144 (4)	−0.0014 (4)	−0.0156 (5)

Geometric parameters (Å, °)

C1—O2	1.242 (3)	C16—H16	0.9300
C1—O1	1.246 (3)	C17—C18	1.491 (7)
C1—C2	1.514 (4)	C17—H17A	0.9600
C2—C3	1.383 (4)	C17—H17B	0.9600
C2—C7	1.395 (4)	C17—H17C	0.9600
C3—C4	1.374 (4)	C18—C19	1.521 (5)
C3—H3	0.9300	C18—H18A	0.9700
C4—C5	1.381 (4)	C18—H18B	0.9700
C4—Cl1	1.732 (3)	C19—C20	1.370 (5)
C5—C6	1.381 (4)	C19—C24	1.370 (5)
C5—Cl2	1.729 (3)	C20—C21	1.376 (5)
C6—C7	1.389 (4)	C20—H20	0.9300
C6—H6	0.9300	C21—C22	1.382 (5)
C7—C8	1.509 (4)	C21—H21	0.9300
C8—O3	1.246 (3)	C22—C23	1.356 (4)

C8—O4	1.260 (4)	C22—N2	1.456 (4)
C10—C26	1.335 (8)	C23—C24	1.370 (5)
C10—C11	1.557 (7)	C23—H23	0.9300
C10—H10A	0.9700	C24—H24	0.9300
C10—H10B	0.9700	C26—H26A	0.9600
C11—C12	1.345 (6)	C26—H26B	0.9600
C11—C16	1.403 (6)	C26—H26C	0.9600
C12—C13	1.350 (5)	N1—H1A	0.96 (5)
C12—H12	0.9300	N1—H1B	0.99 (3)
C13—C14	1.383 (5)	N1—H1C	0.86 (4)
C13—H13	0.9300	N2—H2A	0.98 (5)
C14—C15	1.360 (4)	N2—H2B	0.91 (4)
C14—N1	1.456 (4)	N2—H2C	0.84 (5)
C15—C16	1.417 (5)	O1—H1	0.8200
C15—H15	0.9300		
O2—C1—O1	125.9 (3)	C18—C17—H17B	109.5
O2—C1—C2	118.0 (2)	H17A—C17—H17B	109.5
O1—C1—C2	116.1 (3)	C18—C17—H17C	109.5
C3—C2—C7	119.3 (2)	H17A—C17—H17C	109.5
C3—C2—C1	118.4 (2)	H17B—C17—H17C	109.5
C7—C2—C1	122.2 (2)	C17—C18—C19	116.4 (4)
C4—C3—C2	121.3 (3)	C17—C18—H18A	108.2
C4—C3—H3	119.4	C19—C18—H18A	108.2
C2—C3—H3	119.4	C17—C18—H18B	108.2
C3—C4—C5	119.5 (3)	C19—C18—H18B	108.2
C3—C4—Cl1	119.3 (2)	H18A—C18—H18B	107.3
C5—C4—Cl1	121.1 (2)	C20—C19—C24	117.0 (3)
C4—C5—C6	120.1 (2)	C20—C19—C18	119.8 (3)
C4—C5—Cl2	121.1 (2)	C24—C19—C18	123.3 (3)
C6—C5—Cl2	118.8 (2)	C19—C20—C21	122.3 (3)
C5—C6—C7	120.6 (3)	C19—C20—H20	118.9
C5—C6—H6	119.7	C21—C20—H20	118.9
C7—C6—H6	119.7	C20—C21—C22	119.1 (3)
C6—C7—C2	119.2 (3)	C20—C21—H21	120.5
C6—C7—C8	116.7 (2)	C22—C21—H21	120.5
C2—C7—C8	123.7 (2)	C23—C22—C21	119.4 (3)
O3—C8—O4	124.2 (3)	C23—C22—N2	119.5 (3)
O3—C8—C7	116.8 (3)	C21—C22—N2	121.1 (3)
O4—C8—C7	118.8 (3)	C22—C23—C24	120.4 (3)
C26—C10—C11	114.0 (6)	C22—C23—H23	119.8
C26—C10—H10A	108.8	C24—C23—H23	119.8
C11—C10—H10A	108.8	C23—C24—C19	121.9 (3)
C26—C10—H10B	108.8	C23—C24—H24	119.0
C11—C10—H10B	108.8	C19—C24—H24	119.0
H10A—C10—H10B	107.7	C10—C26—H26A	109.5
C12—C11—C16	119.5 (4)	C10—C26—H26B	109.5
C12—C11—C10	118.0 (5)	H26A—C26—H26B	109.5
C16—C11—C10	122.4 (5)	C10—C26—H26C	109.5
C11—C12—C13	121.4 (4)	H26A—C26—H26C	109.5

C11—C12—H12	119.3	H26B—C26—H26C	109.5
C13—C12—H12	119.3	C14—N1—H1A	110 (2)
C12—C13—C14	120.2 (4)	C14—N1—H1B	107.7 (19)
C12—C13—H13	119.9	H1A—N1—H1B	109 (3)
C14—C13—H13	119.9	C14—N1—H1C	111 (2)
C15—C14—C13	121.3 (3)	H1A—N1—H1C	104 (3)
C15—C14—N1	120.2 (3)	H1B—N1—H1C	115 (3)
C13—C14—N1	118.4 (3)	C22—N2—H2A	110 (3)
C14—C15—C16	117.7 (3)	C22—N2—H2B	107 (2)
C14—C15—H15	121.1	H2A—N2—H2B	112 (3)
C16—C15—H15	121.1	C22—N2—H2C	112 (3)
C11—C16—C15	119.8 (4)	H2A—N2—H2C	114 (4)
C11—C16—H16	120.1	H2B—N2—H2C	100 (4)
C15—C16—H16	120.1	C1—O1—H1	109.5
C18—C17—H17A	109.5		
O2—C1—C2—C3	−168.0 (3)	C26—C10—C11—C12	158.2 (6)
O1—C1—C2—C3	12.5 (4)	C26—C10—C11—C16	−25.0 (8)
O2—C1—C2—C7	14.8 (4)	C16—C11—C12—C13	0.4 (6)
O1—C1—C2—C7	−164.7 (3)	C10—C11—C12—C13	177.4 (4)
C7—C2—C3—C4	0.0 (4)	C11—C12—C13—C14	0.2 (6)
C1—C2—C3—C4	−177.3 (3)	C12—C13—C14—C15	−0.7 (5)
C2—C3—C4—C5	0.5 (4)	C12—C13—C14—N1	−179.5 (3)
C2—C3—C4—Cl1	178.8 (2)	C13—C14—C15—C16	0.5 (5)
C3—C4—C5—C6	−1.2 (4)	N1—C14—C15—C16	179.3 (3)
Cl1—C4—C5—C6	−179.5 (2)	C12—C11—C16—C15	−0.6 (6)
C3—C4—C5—Cl2	176.8 (2)	C10—C11—C16—C15	−177.4 (4)
Cl1—C4—C5—Cl2	−1.6 (4)	C14—C15—C16—C11	0.1 (5)
C4—C5—C6—C7	1.6 (4)	C17—C18—C19—C20	171.9 (4)
Cl2—C5—C6—C7	−176.5 (2)	C17—C18—C19—C24	−9.8 (6)
C5—C6—C7—C2	−1.1 (4)	C24—C19—C20—C21	−0.9 (6)
C5—C6—C7—C8	172.1 (3)	C18—C19—C20—C21	177.5 (4)
C3—C2—C7—C6	0.4 (4)	C19—C20—C21—C22	−0.3 (6)
C1—C2—C7—C6	177.5 (2)	C20—C21—C22—C23	1.6 (5)
C3—C2—C7—C8	−172.4 (3)	C20—C21—C22—N2	−175.7 (3)
C1—C2—C7—C8	4.7 (4)	C21—C22—C23—C24	−1.7 (5)
C6—C7—C8—O3	−90.4 (3)	N2—C22—C23—C24	175.7 (3)
C2—C7—C8—O3	82.5 (3)	C22—C23—C24—C19	0.5 (6)
C6—C7—C8—O4	85.0 (3)	C20—C19—C24—C23	0.8 (5)
C2—C7—C8—O4	−102.1 (3)	C18—C19—C24—C23	−177.5 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1B···O1^{i}	0.99 (4)	2.67 (3)	3.442 (3)	134 (2)
N1—H1C···O1^{ii}	0.86 (4)	1.90 (4)	2.747 (4)	172 (3)
N1—H1B···O2^{i}	0.99 (4)	1.77 (4)	2.746 (3)	166 (3)
N1—H1A···O3^{iii}	0.93 (4)	2.24 (4)	3.021 (3)	140 (3)
N1—H1A···O4^{iii}	0.93 (4)	2.15 (4)	3.013 (4)	154 (3)

N2—H2C···O2^{i}	0.84 (5)	2.20 (5)	2.784 (4)	126 (4)
N2—H2A···O3^{iv}	0.98 (5)	1.94 (5)	2.913 (5)	174 (4)
N2—H2C···O4^{i}	0.84 (5)	2.58 (5)	3.350 (5)	152 (4)
N2—H2B···O4^{v}	0.92 (4)	1.83 (4)	2.739 (4)	172 (3)
C6—H6···O1^{vi}	0.93	2.52	3.388 (3)	154
C13—H13···O3^{i}	0.93	2.54	3.402 (4)	154
C15—H15···O1^{ii}	0.93	2.55	3.254 (4)	133
C23—H23···O3^{iv}	0.93	2.93	3.641 (4)	134
C23—H23···Cg1	0.93	2.69	3.389 (4)	133

Symmetry codes: (i) *x*−1, −*y*+3/2, *z*−1/2; (ii) −*x*+1, *y*−1/2, −*z*+3/2; (iii) *x*−1, *y*, *z*; (iv) −*x*+1, −*y*+1, −*z*+1; (v) −*x*+1, *y*−1/2, −*z*+1/2; (vi) *x*, −*y*+3/2, *z*−1/2.

Fig. 1

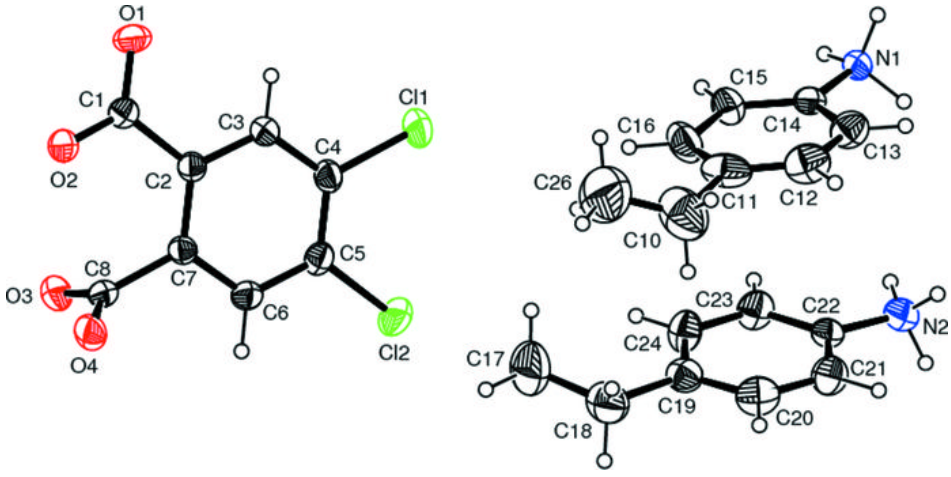

O1
C1
O2
C2
C3
C4
Cl1
C8
O3
O4
C7
C6
C5
Cl2
N1
C15
C16
C14
C13
C26
C11
C12
C10
C23
C22
N2
C24
C17
C21
C19
C18
C20

Fig. 2

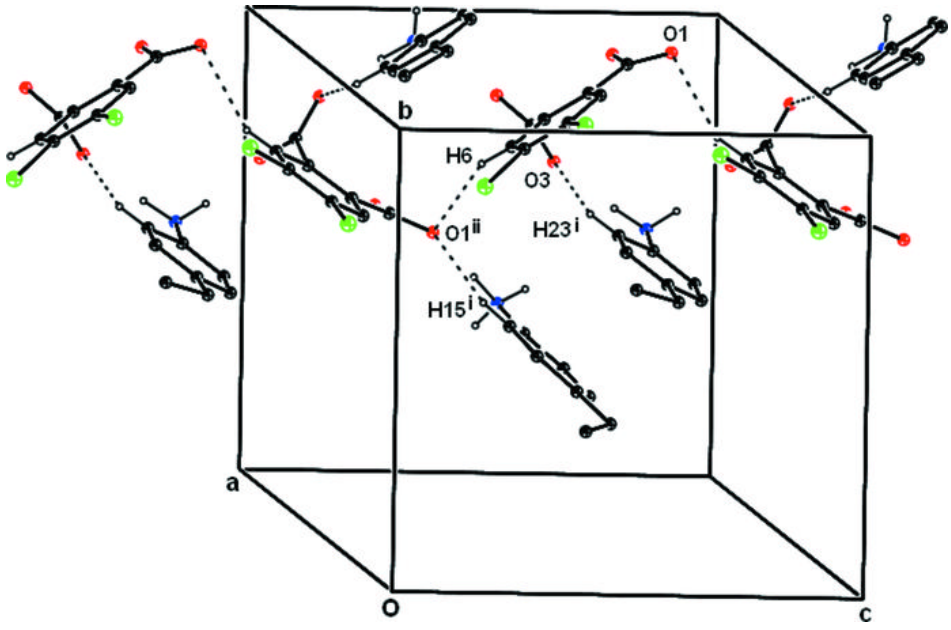


Fig. 3

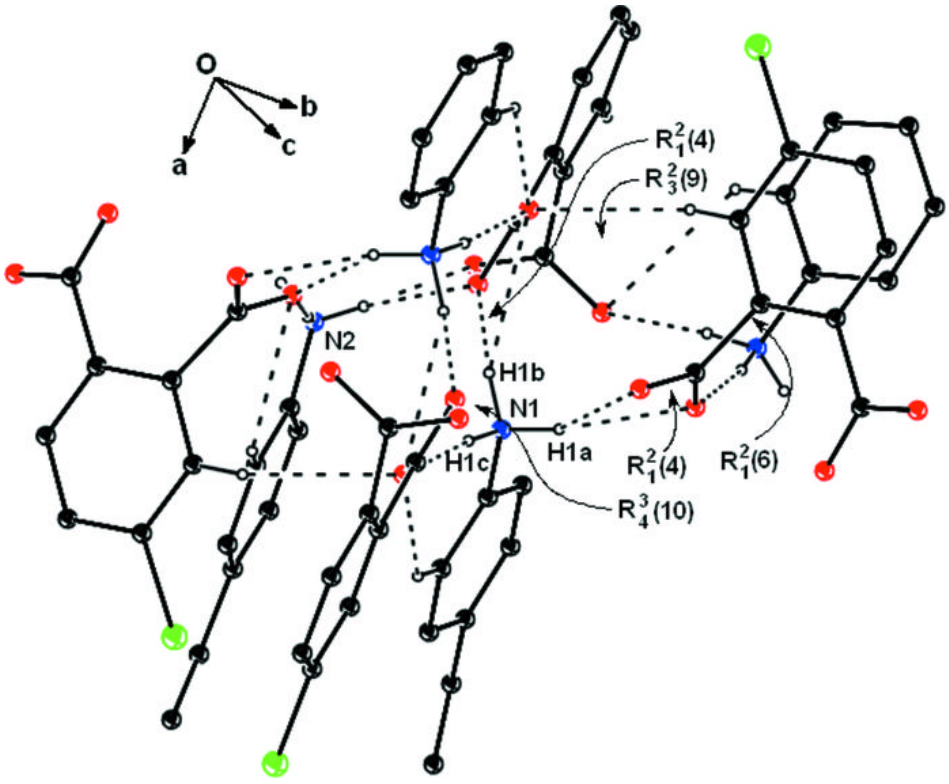


Fig. 4