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Hexane-1,6-diaminium bis[3,4,5,6-tetrachloro-2-(methoxycarbonyl)benzoate]

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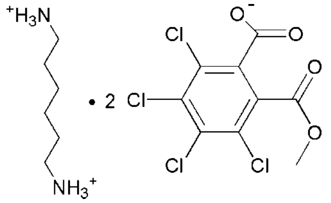
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.057; wR factor = 0.154; data-to-parameter ratio = 14.7.

In the anion of the title salt, $C_6H_{18}N_2^{2+}\cdot 2C_9H_3Cl_4O_4^-$, the methoxycarbonyl and carboxyl groups are aligned at dihedral angles of 71.0 (3) and 100.9 (3)°, respectively, with the aromatic ring. The asymmetric unit contains half a cation and one anion. In the crystal, intermolecular N—H⋯O, C—H⋯Cl and C—H⋯O hydrogen bonds link the components into a three-dimensional network.

Related literature

For related structures, see: Li (2011); Liang (2008).



Experimental

Crystal data

$C_6H_{18}N_2^{2+}\cdot 2C_9H_3Cl_4O_4^-$
M_r = 752.05
Monoclinic, $C2/c$
a = 31.236 (3) Å
b = 5.8911 (4) Å
c = 18.3762 (18) Å
β = 107.118 (1)°
V = 3231.7 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.74 mm^{-1}
T = 298 K
0.37 × 0.28 × 0.15 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.770, T_{max} = 0.897
7618 measured reflections
2829 independent reflections
1817 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.154
S = 1.04
2829 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1A⋯O4	0.89	1.90	2.770 (5)	165
N1—H1B⋯O3^i	0.89	1.87	2.757 (5)	171
C9—H9B⋯Cl4^{ii}	0.96	2.75	3.677 (9)	161
C10—H10B⋯O2	0.97	2.58	3.208 (7)	122

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5475).

References

Bruker (1997). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Li, J. (2011). *Acta Cryst.* E**67**, o200.
Liang, Z.-P. (2008). *Acta Cryst.* E**64**, o2416.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2011). E**67**, o901 [doi:10.1107/S1600536811008506]

Hexane-1,6-diaminium bis[3,4,5,6-tetrachloro-2-(methoxycarbonyl)benzoate]

Jian Li

S1. Comment

In the present work, the reaction of 3,4,5,6-tetrachloro-2-(methoxycarbonyl)benzoic acid and hexane-1,6-diamine in methanol is expected to yield 4,5,6,7-tetrachloro-2-[6-(4,5,6,7- tetrachloro-1,3-dioxoisoindolin-2-yl)hexyl]-isoindoline-1,3-dione. However, the product is hexane-1,6-diaminium 3,4,5,6-tetrachloro-2-(methoxycarbonyl)benzoate (Scheme I, Fig. 1), this may be the reason of a shorter time and cooler temperature in the reaction. The asymmetric unit of the title compound (I) contains half a hexane-1,6-diaminium cation and one 3,4,5,6-tetrachloro-2-(methoxycarbonyl)-benzoate anion (Fig. 1). In the anion of the title salt, the methoxycarbonyl and carboxyl groups are aligned at dihedral angles of 71.0 (3) and 100.9 (3) °, respectively, with the aromatic ring. The bond lengths and angles are in agreement with those in ethylammonium 2-(methoxycarbonyl)-3,4,5,6-tetrabromobenzoate methanol solvate (Li, 2011) and in ethane-1,2-diammonium bis(2-(methoxycarbonyl)-3,4,5,6-tetrabromobenzoate) methanol solvate (Liang, 2008). In the crystal structure, intermolecular N—H···O, C—H···Cl and C—H···O hydrogen bonds link the components of the structure into three-dimensional network (Fig. 2 and Table 1).

S2. Experimental

A mixture of 4,5,6,7-tetrachloroisobenzofuran-1,3-dione (2.86 g, 0.01 mol) and methanol (15 ml) was refluxed for 0.5 h. And then hexane-1,6-diamine (0.58 g, 0.005 mol) was added to the above solution, being mixed round for 20 min at room temperature. And then the solution was kept at room temperature for 6 d. Natural evaporation gave colourless single crystals of the title compound, suitable for X-ray analysis.

S3. Refinement

H atoms were initially located from difference maps and then refined in a riding model with C—H = 0.96–0.97 Å, N—H = 0.89 Å, and $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(O, N, methyl C).

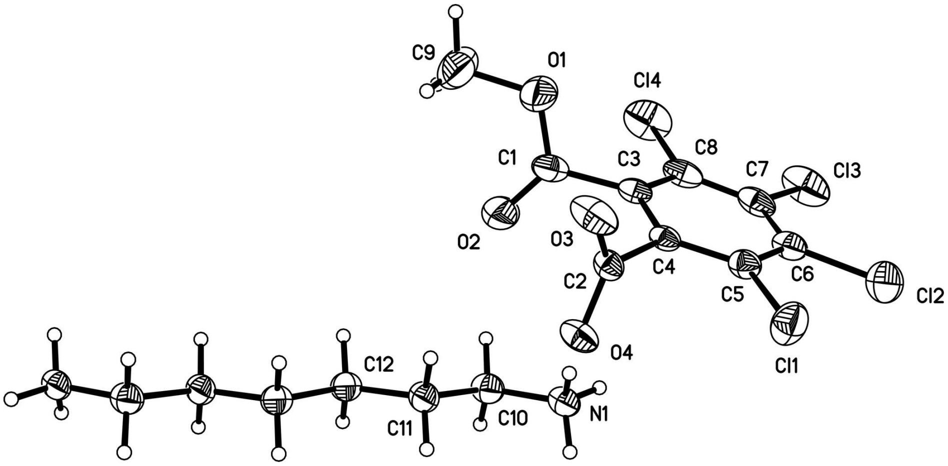

Figure 1

The molecular structure of (I), drawn with 30% probability ellipsoids.

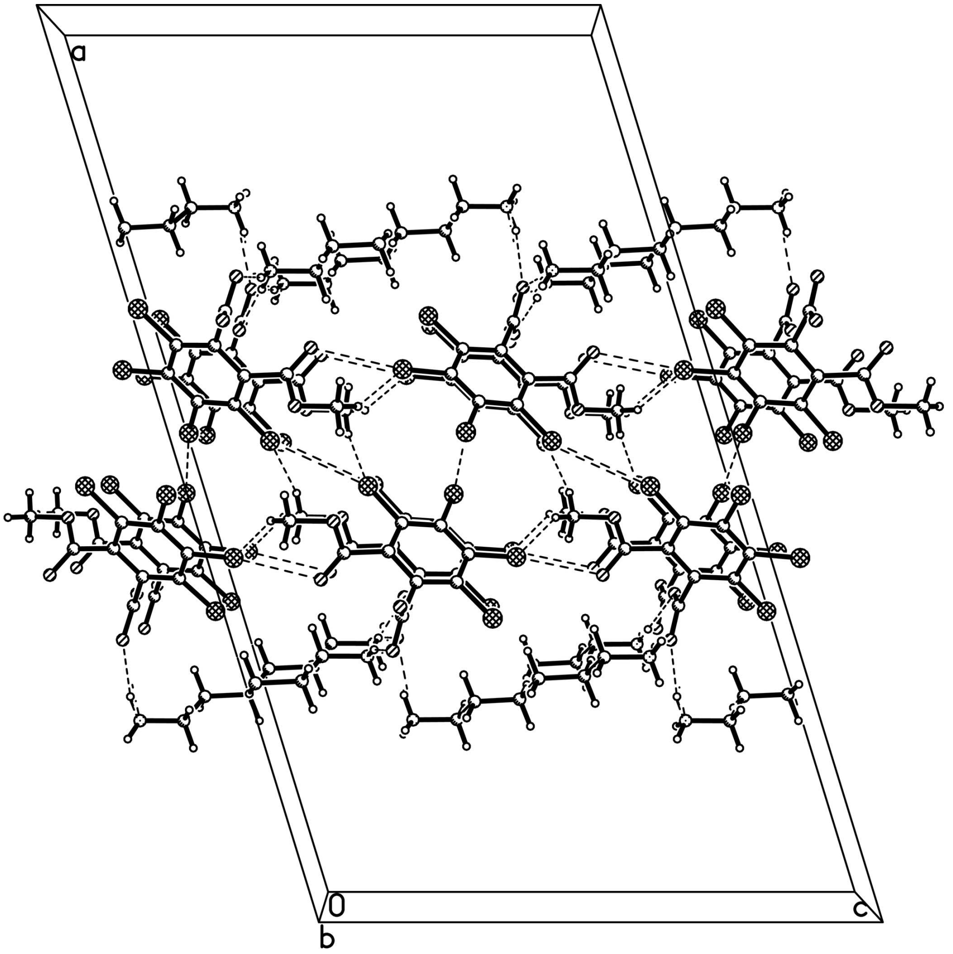

Figure 2

The crystal packing of (I), viewed along *b* axis. Hydrogen bonds are indicated by dashed lines.

Hexane-1,6-diaminium bis[3,4,5,6-tetrachloro-2-(methoxycarbonyl)benzoate]

Crystal data

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Monoclinic, $C2/c$
$a = 31.236\ (3)$ Å
$b = 5.8911\ (4)$ Å
$c = 18.3762\ (18)$ Å
$\beta = 107.118\ (1)°$
$V = 3231.7\ (5)$ Å^3
$Z = 4$
$F(000) = 1528$
$D_x = 1.546$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2053 reflections
$\theta = 2.7–26.1°$
$\mu = 0.74$ mm^{-1}
$T = 298$ K
Block, colorless
0.37 × 0.28 × 0.15 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{min} = 0.770$, $T_{max} = 0.897$
7618 measured reflections
2829 independent reflections
1817 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.3°$
$h = -36 \rightarrow 29$
$k = -6 \rightarrow 7$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.154$
$S = 1.04$
2829 reflections
192 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
$w = 1/[\sigma^2(F_o^2) + (0.0489P)^2 + 12.9951P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ 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	$U_{iso}*/U_{eq}$
Cl1	0.16469 (4)	0.8320 (3)	0.02548 (7)	0.0738 (5)
Cl2	0.10240 (5)	0.4349 (3)	−0.04960 (8)	0.0845 (5)
Cl3	0.03378 (5)	0.2434 (2)	0.02771 (10)	0.0912 (6)
Cl4	0.02472 (5)	0.4714 (3)	0.17526 (10)	0.0918 (6)
N1	0.21033 (11)	0.4754 (6)	0.26915 (19)	0.0485 (9)

H1A	0.2012	0.6131	0.2512	0.073*
H1B	0.1918	0.3715	0.2417	0.073*
H1C	0.2379	0.4510	0.2662	0.073*
O1	0.06089 (14)	0.9497 (7)	0.2473 (2)	0.0906 (13)
O2	0.12386 (14)	0.7821 (10)	0.3033 (2)	0.1124 (18)
O3	0.14859 (11)	1.1892 (6)	0.1748 (2)	0.0769 (12)
O4	0.19786 (10)	0.9160 (5)	0.21430 (17)	0.0537 (8)
C1	0.09506 (15)	0.8248 (9)	0.2481 (3)	0.0558 (12)
C2	0.16017 (13)	0.9909 (7)	0.1788 (2)	0.0400 (10)
C3	0.09403 (13)	0.7360 (7)	0.1712 (2)	0.0432 (10)
C4	0.12605 (12)	0.8139 (6)	0.1380 (2)	0.0373 (9)
C5	0.12705 (13)	0.7243 (7)	0.0688 (2)	0.0458 (10)
C6	0.09835 (14)	0.5502 (7)	0.0339 (3)	0.0511 (12)
C7	0.06762 (15)	0.4685 (8)	0.0678 (3)	0.0559 (13)
C8	0.06449 (14)	0.5651 (8)	0.1346 (3)	0.0542 (12)
C9	0.0599 (3)	1.0419 (13)	0.3203 (3)	0.122 (3)
H9A	0.0605	0.9197	0.3552	0.184*
H9B	0.0330	1.1290	0.3134	0.184*
H9C	0.0855	1.1377	0.3405	0.184*
C10	0.21042 (15)	0.4596 (8)	0.3498 (3)	0.0531 (12)
H10A	0.2253	0.3210	0.3722	0.064*
H10B	0.1798	0.4547	0.3523	0.064*
C11	0.23434 (15)	0.6622 (8)	0.3940 (2)	0.0498 (11)
H11A	0.2645	0.6693	0.3894	0.060*
H11B	0.2188	0.7997	0.3718	0.060*
C12	0.23681 (14)	0.6522 (8)	0.4771 (2)	0.0517 (11)
H12A	0.2507	0.5102	0.4983	0.062*
H12B	0.2066	0.6541	0.4816	0.062*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0691 (9)	0.0891 (11)	0.0710 (8)	−0.0250 (7)	0.0329 (7)	−0.0234 (7)
Cl2	0.0825 (10)	0.0811 (11)	0.0783 (9)	−0.0055 (8)	0.0057 (8)	−0.0407 (8)
Cl3	0.0688 (9)	0.0500 (8)	0.1278 (13)	−0.0243 (7)	−0.0131 (9)	−0.0073 (8)
Cl4	0.0664 (9)	0.0909 (12)	0.1210 (13)	−0.0240 (8)	0.0322 (9)	0.0288 (10)
N1	0.044 (2)	0.033 (2)	0.060 (2)	0.0003 (16)	0.0021 (17)	−0.0084 (17)
O1	0.112 (3)	0.090 (3)	0.067 (2)	0.053 (3)	0.021 (2)	0.007 (2)
O2	0.094 (3)	0.181 (5)	0.058 (2)	0.071 (3)	0.016 (2)	0.010 (3)
O3	0.063 (2)	0.035 (2)	0.105 (3)	0.0031 (16)	−0.019 (2)	−0.0100 (18)
O4	0.0404 (17)	0.0431 (18)	0.0658 (19)	−0.0031 (14)	−0.0025 (15)	0.0026 (15)
C1	0.041 (3)	0.066 (3)	0.061 (3)	0.012 (2)	0.015 (2)	0.018 (3)
C2	0.038 (2)	0.035 (3)	0.043 (2)	−0.0057 (19)	0.0049 (18)	−0.0034 (19)
C3	0.034 (2)	0.037 (2)	0.054 (3)	0.0065 (19)	0.0067 (19)	0.010 (2)
C4	0.031 (2)	0.027 (2)	0.047 (2)	0.0021 (16)	0.0018 (17)	−0.0004 (18)
C5	0.037 (2)	0.041 (3)	0.055 (3)	−0.0035 (19)	0.007 (2)	−0.008 (2)
C6	0.041 (2)	0.036 (3)	0.065 (3)	0.002 (2)	−0.002 (2)	−0.011 (2)
C7	0.040 (3)	0.036 (3)	0.075 (3)	−0.004 (2)	−0.008 (2)	−0.003 (2)

C8	0.035 (2)	0.045 (3)	0.076 (3)	−0.003 (2)	0.007 (2)	0.019 (3)
C9	0.171 (7)	0.127 (6)	0.072 (4)	0.084 (6)	0.042 (4)	0.010 (4)
C10	0.048 (3)	0.046 (3)	0.063 (3)	−0.001 (2)	0.012 (2)	−0.003 (2)
C11	0.049 (3)	0.042 (3)	0.053 (3)	0.002 (2)	0.007 (2)	−0.007 (2)
C12	0.045 (3)	0.052 (3)	0.057 (3)	0.001 (2)	0.014 (2)	−0.007 (2)

Geometric parameters (Å, º)

Cl1—C5	1.723 (5)	C4—C5	1.386 (6)
Cl2—C6	1.716 (5)	C5—C6	1.389 (6)
Cl3—C7	1.721 (4)	C6—C7	1.376 (7)
Cl4—C8	1.719 (5)	C7—C8	1.382 (7)
N1—C10	1.484 (5)	C9—H9A	0.9600
N1—H1A	0.8900	C9—H9B	0.9600
N1—H1B	0.8900	C9—H9C	0.9600
N1—H1C	0.8900	C10—C11	1.511 (6)
O1—C1	1.293 (5)	C10—H10A	0.9700
O1—C9	1.456 (7)	C10—H10B	0.9700
O2—C1	1.168 (5)	C11—C12	1.507 (6)
O3—C2	1.218 (5)	C11—H11A	0.9700
O4—C2	1.247 (5)	C11—H11B	0.9700
C1—C3	1.499 (6)	C12—C12^{i}	1.519 (9)
C2—C4	1.521 (5)	C12—H12A	0.9700
C3—C4	1.394 (6)	C12—H12B	0.9700
C3—C8	1.397 (6)		
C10—N1—H1A	109.5	C7—C8—C3	121.0 (4)
C10—N1—H1B	109.5	C7—C8—Cl4	120.2 (4)
H1A—N1—H1B	109.5	C3—C8—Cl4	118.8 (4)
C10—N1—H1C	109.5	O1—C9—H9A	109.5
H1A—N1—H1C	109.5	O1—C9—H9B	109.5
H1B—N1—H1C	109.5	H9A—C9—H9B	109.5
C1—O1—C9	116.3 (4)	O1—C9—H9C	109.5
O2—C1—O1	123.7 (5)	H9A—C9—H9C	109.5
O2—C1—C3	122.7 (4)	H9B—C9—H9C	109.5
O1—C1—C3	113.6 (4)	N1—C10—C11	110.0 (4)
O3—C2—O4	126.1 (4)	N1—C10—H10A	109.7
O3—C2—C4	118.3 (4)	C11—C10—H10A	109.7
O4—C2—C4	115.6 (4)	N1—C10—H10B	109.7
C4—C3—C8	119.0 (4)	C11—C10—H10B	109.7
C4—C3—C1	118.6 (4)	H10A—C10—H10B	108.2
C8—C3—C1	122.2 (4)	C12—C11—C10	112.5 (4)
C5—C4—C3	119.3 (4)	C12—C11—H11A	109.1
C5—C4—C2	120.8 (4)	C10—C11—H11A	109.1
C3—C4—C2	120.0 (4)	C12—C11—H11B	109.1
C4—C5—C6	121.3 (4)	C10—C11—H11B	109.1
C4—C5—Cl1	119.1 (3)	H11A—C11—H11B	107.8
C6—C5—Cl1	119.6 (3)	C11—C12—C12^{i}	113.0 (5)

C7—C6—C5	119.4 (4)	C11—C12—H12A	109.0
C7—C6—Cl2	120.7 (3)	C12^{i}—C12—H12A	109.0
C5—C6—Cl2	119.9 (4)	C11—C12—H12B	109.0
C6—C7—C8	120.0 (4)	C12^{i}—C12—H12B	109.0
C6—C7—Cl3	119.9 (4)	H12A—C12—H12B	107.8
C8—C7—Cl3	120.1 (4)		

C9—O1—C1—O2	1.0 (9)	C4—C5—C6—C7	1.5 (6)
C9—O1—C1—C3	−179.9 (5)	Cl1—C5—C6—C7	−178.4 (3)
O2—C1—C3—C4	−68.0 (7)	C4—C5—C6—Cl2	−177.0 (3)
O1—C1—C3—C4	112.9 (5)	Cl1—C5—C6—Cl2	3.1 (5)
O2—C1—C3—C8	106.7 (6)	C5—C6—C7—C8	2.3 (6)
O1—C1—C3—C8	−72.4 (6)	Cl2—C6—C7—C8	−179.3 (3)
C8—C3—C4—C5	1.6 (6)	C5—C6—C7—Cl3	−176.7 (3)
C1—C3—C4—C5	176.5 (4)	Cl2—C6—C7—Cl3	1.7 (5)
C8—C3—C4—C2	−176.8 (4)	C6—C7—C8—C3	−4.1 (7)
C1—C3—C4—C2	−1.8 (5)	Cl3—C7—C8—C3	174.9 (3)
O3—C2—C4—C5	101.7 (5)	C6—C7—C8—Cl4	177.5 (3)
O4—C2—C4—C5	−79.0 (5)	Cl3—C7—C8—Cl4	−3.6 (5)
O3—C2—C4—C3	−79.9 (5)	C4—C3—C8—C7	2.1 (6)
O4—C2—C4—C3	99.4 (4)	C1—C3—C8—C7	−172.6 (4)
C3—C4—C5—C6	−3.4 (6)	C4—C3—C8—Cl4	−179.4 (3)
C2—C4—C5—C6	174.9 (4)	C1—C3—C8—Cl4	5.9 (6)
C3—C4—C5—Cl1	176.5 (3)	N1—C10—C11—C12	−178.2 (4)
C2—C4—C5—Cl1	−5.2 (5)	C10—C11—C12—C12^{i}	176.6 (5)

Symmetry code: (i) −*x*+1/2, −*y*+3/2, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O4	0.89	1.90	2.770 (5)	165
N1—H1*B*···O3^{ii}	0.89	1.87	2.757 (5)	171
C9—H9*B*···Cl4^{iii}	0.96	2.75	3.677 (9)	161
C10—H10*B*···O2	0.97	2.58	3.208 (7)	122

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