Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Zu Pei Liang

Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, People's Republic of China
Correspondence e-mail: lzpwfu@163.com

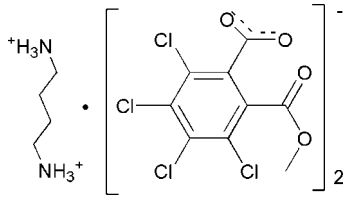
Received 29 April 2011; accepted 3 May 2011

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.036; wR factor = 0.096; data-to-parameter ratio = 14.2.

In the title salt, $C_4H_{14}N_2^{+}\cdot 2C_9H_3Cl_4O_4^{-}$, the cation lies on an inversion center. In the anion, the mean planes of methoxycarbonyl and carboxylate groups form dihedral angles of 64.9 (3) and 58.5 (3)°, respectively, with the benzene ring. In the crystal, intermolecular N—H···O hydrogen bonds connect the components into sheets parallel to (100).

Related literature

For a related structure, see: Li (2011).



Experimental

Crystal data

$C_4H_{14}N_2^{2+}\cdot 2C_9H_3Cl_4O_4^{-}$
M_r = 724.00
Monoclinic, $P2_1/c$
a = 14.4243 (13) Å
b = 6.1041 (6) Å
c = 16.9653 (15) Å
β = 97.056 (1)°
V = 1482.4 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.81 mm^{-1}
T = 298 K
0.46 × 0.43 × 0.40 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.708, T_{max} = 0.738
7307 measured reflections
2603 independent reflections
1908 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.096
S = 1.05
2603 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1C···O3^{i}	0.89	1.85	2.735 (3)	172
N1—H1B···O4^{ii}	0.89	1.94	2.823 (3)	174
N1—H1A···O4	0.89	1.88	2.761 (3)	169

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

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) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL*.

The author thanks Shandong Provincial Natural Science Foundation, China (ZR2010BM033) for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5244).

References

Bruker (1997). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Li, J. (2011). *Acta Cryst.* E**67**, o901.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

Acta Cryst. (2011). E**67**, o1357 [doi:10.1107/S1600536811016795]

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

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S1. Comment

In the present work, the reaction of 3,4,5,6-tetrachloro-2-(methoxycarbonyl)benzoic acid and butane-1,4-diamine in methanol was expected to yield 4,5,6,7-tetrachloro-2-[4-(4,5,6,7- tetrachloro-1,3-dioxoisoindolin-2-yl)butanyl]isoindoline-1,3-dione. However, the product was the title compound and this may have occurred because of the reduced time and temperature of the reaction. The asymmetric unit of the title compound (I) contains one half of a butane-1,4-diaminium cation and one 3,4,5,6-tetrachloro-2-(methoxycarbonyl)benzoate anions (Fig. 1). In the anion, the mean planes of the methoxycarbonyl and carboxyl groups are aligned at dihedral angles of 64.9 (3) and 58.5 (3) °, respectively with the benzene ring. The bond lengths and angles are in agreement with those which are related in hexane-1,6-diaminium bis[3,4,5,6-tetrachloro-2-(methoxycarbonyl)benzoate](Li, 2011). In the crystal, intermolecular N—H···O hydrogen bonds connect the components into two-dimensional sheets parallel to (100) (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. Then butane-1,4-diamine (0.44 g, 0.005 mol) was added to the above solution and mixed for 20 min at room temperature. The solution was kept at room temperature for 5 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 approximation with C—H = 0.96–0.97 Å and N—H = 0.89 Å U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(N, methyl C).

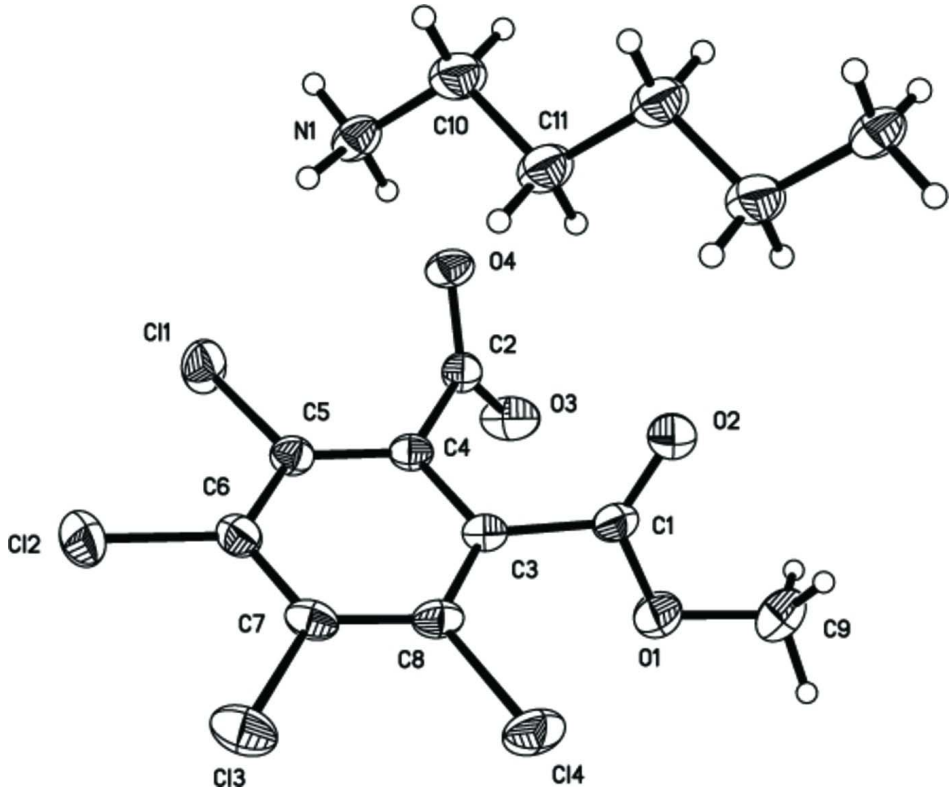

Figure 1

The asymmetric unit of (I), drawn with 30% probability ellipsoids. The symmetry complete cation is shown with unlabeled atoms related by the symmetry operator (-x+1, -y+2, -z+1).

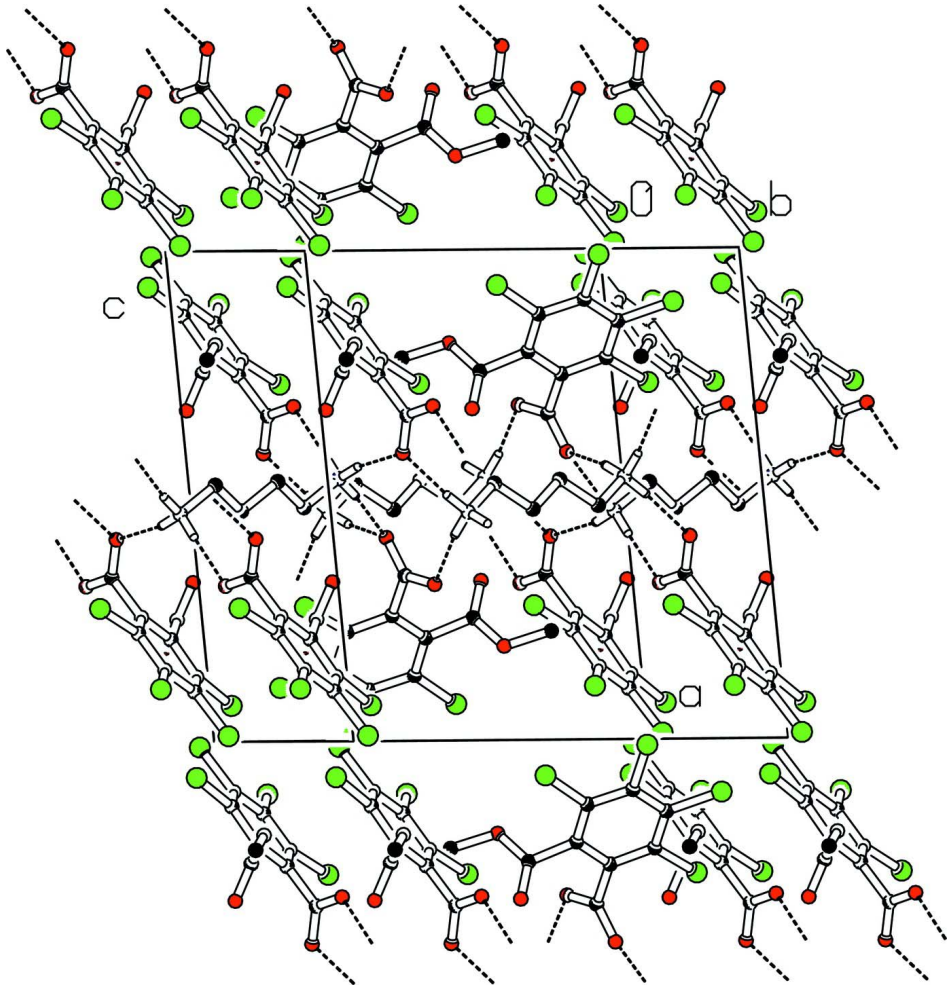

Figure 2

The crystal packing of (I) with hydrogen bonds shown as dashed lines. Only H atoms involved in hydrogen bonds are shown.

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

Crystal data

$C_4H_{14}N_2^{2+}\cdot 2C_9H_3Cl_4O_4^-$
$M_r = 724.00$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 14.4243\ (13)$ Å
$b = 6.1041\ (6)$ Å
$c = 16.9653\ (15)$ Å
$\beta = 97.056\ (1)°$
$V = 1482.4\ (2)$ Å^3
$Z = 2$
$F(000) = 732$
$D_x = 1.622$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2612 reflections
$\theta = 2.4–26.5°$
$\mu = 0.81$ mm^{-1}
$T = 298$ K
Block, colorless
0.46 × 0.43 × 0.40 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} = 0.708, T_{max} = 0.738
7307 measured reflections
2603 independent reflections
1908 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 25.0°, θ_{min} = 2.4°
$h = -9 \rightarrow 17$
$k = -7 \rightarrow 7$
$l = -18 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.096
S = 1.05
2603 reflections
183 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
$w = 1/[\sigma^2(F_o^2) + (0.0373P)^2 + 1.0059P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.27338 (6)	0.79357 (15)	0.17344 (4)	0.0617 (3)
Cl2	0.10875 (6)	1.10088 (15)	0.19656 (5)	0.0705 (3)
Cl3	0.01193 (5)	1.07644 (14)	0.34929 (5)	0.0642 (3)
Cl4	0.07322 (6)	0.72291 (15)	0.47687 (5)	0.0630 (3)
N1	0.46526 (16)	0.9951 (4)	0.31490 (13)	0.0465 (6)
H1A	0.4416	0.8603	0.3128	0.070*
H1B	0.5002	1.0132	0.2756	0.070*
H1C	0.4189	1.0923	0.3099	0.070*
O1	0.19109 (14)	0.3094 (3)	0.46777 (12)	0.0559 (5)
O2	0.32465 (15)	0.4957 (4)	0.48905 (13)	0.0675 (6)
O3	0.31815 (14)	0.2789 (3)	0.31198 (14)	0.0626 (6)
O4	0.41355 (12)	0.5599 (3)	0.30166 (11)	0.0459 (5)
C1	0.24890 (19)	0.4667 (5)	0.45324 (15)	0.0415 (6)
C2	0.33628 (19)	0.4761 (4)	0.30956 (14)	0.0379 (6)
C3	0.20896 (17)	0.6149 (4)	0.38674 (15)	0.0376 (6)
C4	0.25510 (17)	0.6303 (4)	0.31904 (15)	0.0358 (6)

C5	0.22239 (18)	0.7791 (4)	0.26073 (15)	0.0390 (6)
C6	0.14757 (18)	0.9170 (4)	0.26928 (16)	0.0428 (7)
C7	0.10285 (18)	0.9025 (4)	0.33718 (17)	0.0430 (7)
C8	0.13250 (18)	0.7475 (5)	0.39497 (16)	0.0415 (7)
C9	0.2212 (2)	0.1710 (6)	0.53489 (19)	0.0662 (9)
H9A	0.2630	0.0610	0.5195	0.099*
H9B	0.1679	0.1018	0.5529	0.099*
H9C	0.2529	0.2581	0.5770	0.099*
C10	0.5233 (2)	1.0276 (6)	0.39168 (18)	0.0619 (9)
H10A	0.5461	1.1772	0.3950	0.074*
H10B	0.5770	0.9309	0.3947	0.074*
C11	0.4702 (2)	0.9828 (6)	0.46041 (17)	0.0578 (8)
H11A	0.4477	0.8330	0.4572	0.069*
H11B	0.4163	1.0791	0.4572	0.069*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0654 (5)	0.0794 (6)	0.0435 (4)	0.0200 (4)	0.0191 (4)	0.0101 (4)
Cl2	0.0592 (5)	0.0752 (6)	0.0774 (6)	0.0234 (5)	0.0093 (4)	0.0269 (5)
Cl3	0.0443 (4)	0.0644 (5)	0.0856 (6)	0.0176 (4)	0.0143 (4)	−0.0125 (4)
Cl4	0.0541 (5)	0.0812 (6)	0.0597 (5)	0.0003 (4)	0.0309 (4)	−0.0073 (4)
N1	0.0478 (14)	0.0430 (13)	0.0528 (14)	0.0035 (11)	0.0233 (11)	0.0023 (11)
O1	0.0553 (13)	0.0521 (12)	0.0602 (13)	−0.0123 (11)	0.0065 (10)	0.0088 (10)
O2	0.0502 (13)	0.0832 (16)	0.0665 (14)	−0.0169 (12)	−0.0032 (11)	0.0221 (12)
O3	0.0513 (13)	0.0355 (12)	0.1034 (18)	0.0027 (10)	0.0198 (12)	−0.0069 (11)
O4	0.0376 (11)	0.0456 (11)	0.0581 (12)	0.0035 (9)	0.0206 (9)	−0.0009 (9)
C1	0.0385 (16)	0.0471 (17)	0.0407 (15)	−0.0064 (13)	0.0117 (13)	−0.0019 (12)
C2	0.0390 (16)	0.0385 (16)	0.0372 (14)	0.0043 (13)	0.0087 (12)	−0.0014 (11)
C3	0.0320 (14)	0.0382 (15)	0.0433 (14)	−0.0038 (12)	0.0076 (11)	−0.0036 (12)
C4	0.0319 (14)	0.0339 (14)	0.0424 (14)	−0.0005 (11)	0.0077 (11)	−0.0051 (11)
C5	0.0352 (15)	0.0435 (15)	0.0393 (14)	0.0011 (12)	0.0083 (11)	−0.0043 (12)
C6	0.0357 (15)	0.0415 (16)	0.0502 (16)	0.0029 (13)	0.0014 (12)	0.0003 (13)
C7	0.0308 (14)	0.0415 (16)	0.0572 (17)	0.0040 (12)	0.0074 (13)	−0.0096 (13)
C8	0.0335 (14)	0.0487 (17)	0.0448 (15)	−0.0025 (13)	0.0144 (12)	−0.0093 (13)
C9	0.077 (2)	0.062 (2)	0.063 (2)	−0.0074 (19)	0.0209 (18)	0.0136 (17)
C10	0.0485 (19)	0.082 (2)	0.057 (2)	−0.0095 (17)	0.0134 (15)	0.0004 (17)
C11	0.0532 (19)	0.067 (2)	0.0557 (18)	−0.0077 (17)	0.0179 (15)	0.0026 (16)

Geometric parameters (Å, º)

Cl1—C5	1.735 (3)	C3—C8	1.389 (4)
Cl2—C6	1.711 (3)	C3—C4	1.400 (3)
Cl3—C7	1.719 (3)	C4—C5	1.382 (4)
Cl4—C8	1.725 (3)	C5—C6	1.390 (4)
N1—C10	1.473 (4)	C6—C7	1.390 (4)
N1—H1A	0.8900	C7—C8	1.391 (4)
N1—H1B	0.8900	C9—H9A	0.9600

N1—H1C	0.8900	C9—H9B	0.9600
O1—C1	1.314 (3)	C9—H9C	0.9600
O1—C9	1.441 (4)	C10—C11	1.497 (4)
O2—C1	1.196 (3)	C10—H10A	0.9700
O3—C2	1.234 (3)	C10—H10B	0.9700
O4—C2	1.248 (3)	C11—C11^{i}	1.518 (6)
C1—C3	1.504 (4)	C11—H11A	0.9700
C2—C4	1.526 (3)	C11—H11B	0.9700
C10—N1—H1A	109.5	C6—C7—C8	119.6 (2)
C10—N1—H1B	109.5	C6—C7—Cl3	119.9 (2)
H1A—N1—H1B	109.5	C8—C7—Cl3	120.6 (2)
C10—N1—H1C	109.5	C3—C8—C7	120.3 (2)
H1A—N1—H1C	109.5	C3—C8—Cl4	120.6 (2)
H1B—N1—H1C	109.5	C7—C8—Cl4	119.1 (2)
C1—O1—C9	116.1 (2)	O1—C9—H9A	109.5
O2—C1—O1	125.0 (3)	O1—C9—H9B	109.5
O2—C1—C3	122.2 (3)	H9A—C9—H9B	109.5
O1—C1—C3	112.7 (2)	O1—C9—H9C	109.5
O3—C2—O4	126.8 (3)	H9A—C9—H9C	109.5
O3—C2—C4	115.5 (2)	H9B—C9—H9C	109.5
O4—C2—C4	117.7 (2)	N1—C10—C11	112.0 (2)
C8—C3—C4	120.5 (2)	N1—C10—H10A	109.2
C8—C3—C1	120.7 (2)	C11—C10—H10A	109.2
C4—C3—C1	118.6 (2)	N1—C10—H10B	109.2
C5—C4—C3	118.5 (2)	C11—C10—H10B	109.2
C5—C4—C2	121.9 (2)	H10A—C10—H10B	107.9
C3—C4—C2	119.6 (2)	C10—C11—C11^{i}	112.1 (3)
C4—C5—C6	121.6 (2)	C10—C11—H11A	109.2
C4—C5—Cl1	120.1 (2)	C11^{i}—C11—H11A	109.2
C6—C5—Cl1	118.3 (2)	C10—C11—H11B	109.2
C5—C6—C7	119.5 (2)	C11^{i}—C11—H11B	109.2
C5—C6—Cl2	120.8 (2)	H11A—C11—H11B	107.9
C7—C6—Cl2	119.7 (2)		
C9—O1—C1—O2	3.3 (4)	C4—C5—C6—C7	−1.5 (4)
C9—O1—C1—C3	−175.2 (2)	Cl1—C5—C6—C7	177.3 (2)
O2—C1—C3—C8	−112.4 (3)	C4—C5—C6—Cl2	179.6 (2)
O1—C1—C3—C8	66.2 (3)	Cl1—C5—C6—Cl2	−1.6 (3)
O2—C1—C3—C4	62.3 (4)	C5—C6—C7—C8	−1.2 (4)
O1—C1—C3—C4	−119.2 (3)	Cl2—C6—C7—C8	177.8 (2)
C8—C3—C4—C5	−0.4 (4)	C5—C6—C7—Cl3	178.6 (2)
C1—C3—C4—C5	−175.1 (2)	Cl2—C6—C7—Cl3	−2.5 (3)
C8—C3—C4—C2	−177.3 (2)	C4—C3—C8—C7	−2.2 (4)
C1—C3—C4—C2	8.0 (4)	C1—C3—C8—C7	172.4 (2)
O3—C2—C4—C5	−119.9 (3)	C4—C3—C8—Cl4	178.0 (2)
O4—C2—C4—C5	61.3 (3)	C1—C3—C8—Cl4	−7.5 (3)
O3—C2—C4—C3	56.9 (3)	C6—C7—C8—C3	3.0 (4)

O4—C2—C4—C3	−121.9 (3)	Cl3—C7—C8—C3	−176.8 (2)
C3—C4—C5—C6	2.3 (4)	C6—C7—C8—Cl4	−177.2 (2)
C2—C4—C5—C6	179.1 (2)	Cl3—C7—C8—Cl4	3.1 (3)
C3—C4—C5—Cl1	−176.52 (19)	N1—C10—C11—C11^{i}	−179.8 (3)
C2—C4—C5—Cl1	0.3 (4)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*C*···O3^{ii}	0.89	1.85	2.735 (3)	172
N1—H1*B*···O4^{iii}	0.89	1.94	2.823 (3)	174
N1—H1*A*···O4	0.89	1.88	2.761 (3)	169

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