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tert-Butylaminium 2-carboxy-4,5-dichlorobenzoate

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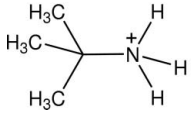
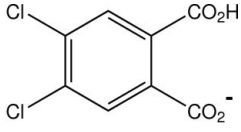
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.033; wR factor = 0.094; data-to-parameter ratio = 14.5.

In the structure of the title anhydrous salt, $C_4H_{12}N^+\cdot C_8H_3Cl_2O_4^-$, the 4,5-dichlorophthalate monoanions have the common 'planar' conformation with the carboxyl groups close to coplanar with the benzene ring and with a short intramolecular carboxylic acid O—H···O hydrogen bond. In the crystal, a two-dimensional sheet structure is formed through aminium N—H···$O_{carboxyl}$ hydrogen-bonding associations.

Related literature

For structures of 1:1 salts of 4,5-dichlorophthalic acid with acyclic aliphatic amines, see: Mattes & Dorau (1986); Bozkurt *et al.* (2006); Smith & Wermuth (2010*a*,*b*,*c*).





Experimental

Crystal data

$C_4H_{12}N^+\cdot C_8H_3Cl_2O_4^-$
M_r = 308.15
Monoclinic, $P2_1/n$
a = 6.1778 (2) Å
b = 12.7158 (4) Å
c = 17.7125 (7) Å
β = 96.784 (4)°
V = 1381.68 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.48 mm^{-1}
T = 200 K
0.45 × 0.26 × 0.18 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.977, T_{max} = 0.990
8677 measured reflections
2719 independent reflections
2307 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.094
S = 0.90
2719 reflections
188 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1A—H11A···O21	0.89 (2)	2.02 (2)	2.883 (2)	164 (2)
N1A—H12A···O11^{i}	0.91 (2)	1.88 (2)	2.784 (2)	174 (2)
N1A—H13A···O12^{ii}	0.89 (2)	1.99 (2)	2.861 (2)	167 (2)
O21—H21···O12	0.94 (4)	1.47 (4)	2.4021 (19)	173 (4)

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) within *WinGX* (Farrugia, 1999); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5620).

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supporting information

Acta Cryst. (2011). E**67**, o2461 [doi:10.1107/S1600536811034131]

tert-Butylaminium 2-carboxy-4,5-dichlorobenzoate

Graham Smith and Urs D. Wermuth

S1. Comment

4,5-Dichlorophthalic acid (DCPA) commonly forms 1:1 salts with the acyclic aliphatic amine analogues and with these, low-dimensional hydrogen-bonded structures are usually found, featuring the 'planar′ hydrogen phthalate anion e.g with isopropylamine (Smith & Wermuth, 2010*a*), diisopropylamine (Smith & Wermuth, 2010*b*), diethylamine, triethylamine and *n*-butylamine (Smith & Wermuth, 2010*c*), the ammonium and tetra(*n*butyl)ammonium salts (Mattes & Dorau, 1986) and the tetramethylammonium salt (Bozkurt *et al.*, 2006). Our 1:1 stoichiometric reaction of DCPA with *t*-butylamine also gave a 1:1 salt $C_4H_{12}N^+$ $C_8H_3Cl_2O_4^-$, the title compound and the structure is reported here.

In this structure the common 'planar′ DCPA anion is found (Fig. 1) and has the previously described (Smith & Wermuth, 2010*c*) short intramolecular carboxylic acid *O*—*H*···$O_{carboxy}$ hydrogen bond (Table 1) (torsion angles C1–C2–C21–O22 and C2–C1–C11–O11: 176.59 (18) and 175.26 (17) Å respectively). Other structural features common to this 'planar′ monoanion are a lengthening of the C1—C11 and C2—C21 bond lengths [1.522 (2) and 1.528 (3) Å] and distortion of the external bond angles at C1 and C2 [C1—C2—C21, 129.57 (15)° and C2—C1—C11, 128.84 (15)°].

Intermolecular aminium *N*—*H*···*O*(carboxyl) hydrogen bonds (Table 1) link the DCPA monoanions across *b* as well as down the *a* axis, forming a two-dimensional sheet structure (Fig. 2).

S2. Experimental

The title compound was synthesized by heating together for 10 min under reflux, 1 mmol quantities of 4,5-dichlorophthalic acid and *t*-butylamine in 50 ml of 50% ethanol–water. Partial evaporation of the solvent gave colourless crystalline plates from which a specimen was cleaved for the X-ray analysis..

S3. Refinement

Hydrogen atoms potentially involved in hydrogen-bonding interactions were located by difference methods and their positional and isotropic displacement parameters were refined. Other H atoms were included at calculated positions [C—H (aromatic) = 0.93 Å or C—H (methyl) = 0.97 Å] and treated as riding, with U_{iso}(H) = 1.2U_{eq}C(aromatic) or 1.5U_{eq} *C*(methyl).

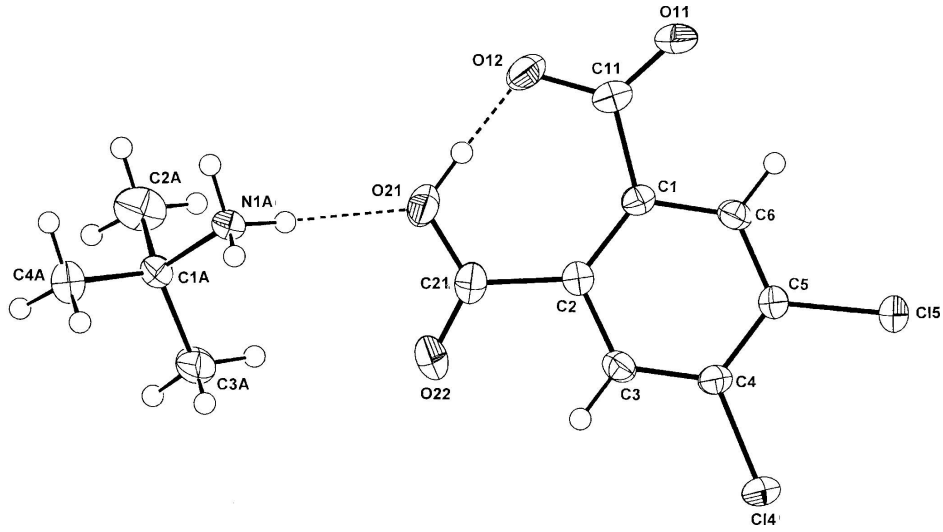

Figure 1

Molecular conformation and atom-numbering scheme for the title salt, with the hydrogen bonds shown as a dashed lines. Non-H atoms are shown as 50% probability displacement ellipsoids.

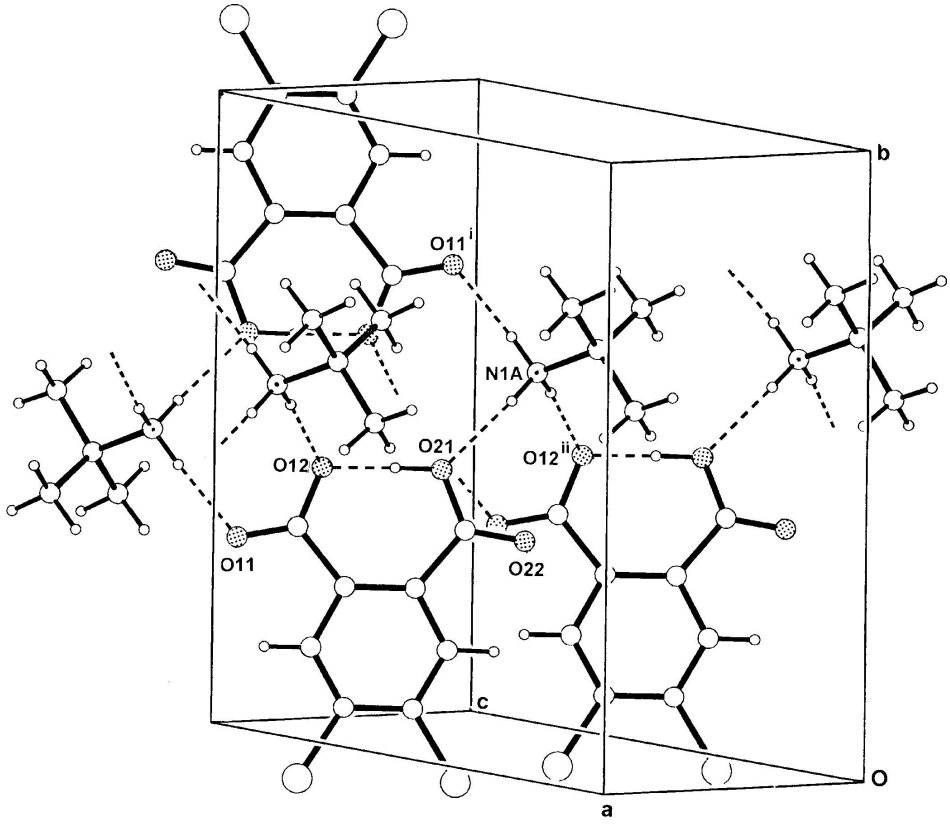

Figure 2

A perspective view the two-dimensional sheet structure looking down the sheet, showing hydrogen-bonding associations as dashed lines.

tert-Butylaminium 2-carboxy-4,5-dichlorobenzoate

Crystal data

$C_4H_{12}N^+·C_8H_3Cl_2O_4^-$
M_r = 308.15
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 6.1778 (2) Å
b = 12.7158 (4) Å
c = 17.7125 (7) Å
β = 96.784 (4)°
V = 1381.68 (8) Å^3
Z = 4
$F(000)$ = 640
D_x = 1.481 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4272 reflections
θ = 3.3–28.8°
μ = 0.48 mm^{-1}
T = 200 K
Plate, colourless
0.45 × 0.26 × 0.18 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.077 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.977, T_{max} = 0.990
8677 measured reflections
2719 independent reflections
2307 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 26.0°, θ_{min} = 3.4°
$h = -7 \rightarrow 7$
$k = -15 \rightarrow 15$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.094
S = 0.90
2719 reflections
188 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0608P)^2 + 0.4558P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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	0.77566 (7)	−0.08494 (3)	0.92701 (3)	0.0313 (1)
Cl2	0.29591 (7)	−0.09103 (3)	0.84148 (3)	0.0370 (2)
O11	0.8896 (2)	0.29012 (10)	1.01297 (8)	0.0394 (4)
O12	0.6289 (2)	0.40012 (9)	0.97033 (8)	0.0353 (4)

O21	0.2615 (2)	0.39679 (10)	0.90835 (10)	0.0452 (5)
O22	0.0363 (2)	0.28642 (11)	0.84456 (9)	0.0475 (5)
C1	0.5836 (3)	0.21454 (12)	0.93969 (9)	0.0218 (5)
C2	0.3705 (3)	0.21275 (13)	0.90024 (9)	0.0230 (5)
C3	0.2876 (3)	0.11656 (13)	0.87218 (10)	0.0239 (5)
C4	0.4066 (3)	0.02459 (12)	0.87963 (9)	0.0233 (5)
C5	0.6169 (3)	0.02693 (12)	0.91703 (9)	0.0221 (5)
C6	0.7012 (3)	0.12081 (13)	0.94635 (9)	0.0236 (5)
C11	0.7102 (3)	0.30722 (13)	0.97701 (9)	0.0268 (5)
C21	0.2089 (3)	0.30285 (14)	0.88234 (11)	0.0311 (6)
N1A	−0.0938 (3)	0.54459 (13)	0.90362 (9)	0.0257 (5)
C1A	−0.2152 (3)	0.58740 (13)	0.83097 (10)	0.0255 (5)
C2A	−0.0614 (3)	0.66054 (18)	0.79557 (12)	0.0450 (7)
C3A	−0.2819 (3)	0.49408 (16)	0.77986 (11)	0.0380 (6)
C4A	−0.4120 (3)	0.64663 (15)	0.85216 (12)	0.0373 (6)
H3	0.14630	0.11430	0.84740	0.0290*
H6	0.84200	0.12160	0.97160	0.0280*
H21	0.400 (6)	0.399 (3)	0.936 (2)	0.109 (12)*
H11A	0.016 (3)	0.5023 (18)	0.8953 (12)	0.039 (6)*
H12A	−0.036 (4)	0.5995 (18)	0.9325 (13)	0.044 (6)*
H13A	−0.185 (4)	0.5080 (18)	0.9289 (13)	0.042 (6)*
H21A	0.06120	0.62120	0.78220	0.0670*
H22A	−0.01140	0.71450	0.83140	0.0670*
H23A	−0.13660	0.69220	0.75070	0.0670*
H31A	−0.37940	0.44980	0.80390	0.0570*
H32A	−0.15460	0.45460	0.77120	0.0570*
H33A	−0.35380	0.51880	0.73220	0.0570*
H41A	−0.50720	0.59880	0.87410	0.0560*
H42A	−0.48830	0.67810	0.80740	0.0560*
H43A	−0.36510	0.70060	0.88840	0.0560*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0293 (2)	0.0232 (2)	0.0394 (3)	0.0066 (2)	−0.0041 (2)	−0.0007 (2)
Cl2	0.0298 (3)	0.0252 (2)	0.0534 (3)	−0.0039 (2)	−0.0061 (2)	−0.0113 (2)
O11	0.0394 (8)	0.0314 (7)	0.0433 (8)	−0.0070 (6)	−0.0116 (6)	−0.0082 (6)
O12	0.0367 (7)	0.0208 (6)	0.0486 (8)	−0.0044 (5)	0.0059 (6)	−0.0061 (5)
O21	0.0321 (8)	0.0228 (7)	0.0798 (11)	0.0057 (6)	0.0025 (8)	−0.0041 (7)
O22	0.0377 (8)	0.0409 (8)	0.0591 (10)	0.0146 (6)	−0.0148 (7)	−0.0028 (7)
C1	0.0250 (8)	0.0205 (8)	0.0198 (8)	−0.0024 (6)	0.0028 (7)	−0.0010 (6)
C2	0.0228 (8)	0.0233 (8)	0.0231 (8)	0.0017 (6)	0.0031 (7)	0.0010 (6)
C3	0.0180 (8)	0.0274 (8)	0.0257 (8)	−0.0004 (7)	0.0004 (7)	−0.0007 (7)
C4	0.0231 (8)	0.0216 (8)	0.0249 (8)	−0.0033 (6)	0.0012 (7)	−0.0026 (7)
C5	0.0223 (8)	0.0213 (8)	0.0225 (8)	0.0018 (6)	0.0018 (6)	0.0016 (6)
C6	0.0204 (8)	0.0265 (8)	0.0228 (8)	−0.0016 (6)	−0.0020 (7)	−0.0007 (7)
C11	0.0311 (9)	0.0247 (9)	0.0251 (9)	−0.0068 (7)	0.0049 (8)	−0.0032 (7)
C21	0.0316 (10)	0.0258 (9)	0.0361 (10)	0.0067 (7)	0.0053 (8)	0.0027 (8)

N1A	0.0236 (8)	0.0251 (8)	0.0268 (8)	0.0001 (7)	−0.0037 (7)	−0.0033 (6)
C1A	0.0245 (9)	0.0262 (9)	0.0246 (9)	0.0016 (7)	−0.0021 (7)	−0.0001 (7)
C2A	0.0437 (12)	0.0542 (13)	0.0374 (11)	−0.0117 (10)	0.0066 (9)	0.0081 (10)
C3A	0.0385 (11)	0.0404 (11)	0.0315 (10)	0.0050 (9)	−0.0108 (8)	−0.0097 (8)
C4A	0.0328 (10)	0.0329 (10)	0.0451 (11)	0.0079 (8)	0.0004 (9)	0.0001 (9)

Geometric parameters (Å, º)

Cl1—C5	1.7251 (17)	C4—C5	1.387 (3)
Cl2—C4	1.7253 (16)	C5—C6	1.379 (2)
O11—C11	1.231 (2)	C3—H3	0.9300
O12—C11	1.284 (2)	C6—H6	0.9300
O21—C21	1.307 (2)	C1A—C2A	1.517 (3)
O22—C21	1.208 (2)	C1A—C3A	1.520 (3)
O21—H21	0.94 (4)	C1A—C4A	1.515 (3)
N1A—C1A	1.512 (2)	C2A—H21A	0.9600
N1A—H11A	0.89 (2)	C2A—H22A	0.9600
N1A—H13A	0.89 (2)	C2A—H23A	0.9600
N1A—H12A	0.91 (2)	C3A—H31A	0.9600
C1—C11	1.522 (2)	C3A—H32A	0.9600
C1—C6	1.394 (2)	C3A—H33A	0.9600
C1—C2	1.416 (3)	C4A—H41A	0.9600
C2—C3	1.395 (2)	C4A—H42A	0.9600
C2—C21	1.528 (3)	C4A—H43A	0.9600
C3—C4	1.379 (2)		
Cl1···Cl2	3.1661 (7)	O11···C21^{v}	3.217 (2)
Cl1···O11^{i}	3.4197 (14)	O11···Cl1^{i}	3.4197 (14)
Cl1···C3^{ii}	3.6461 (18)	O11···N1A^{vi}	2.784 (2)
Cl2···Cl1	3.1661 (7)	O12···C21	3.120 (2)
Cl1···H43A^{iii}	2.9200	O12···O12^{vi}	3.2387 (17)
Cl1···H6^{i}	2.8300	O12···O21	2.4021 (19)
Cl2···H22A^{iv}	3.1100	O12···N1A^{v}	2.861 (2)
O11···O22^{v}	3.219 (2)	O21···C11	3.109 (2)
C21—O21—H21	113 (2)	C4—C3—H3	119.00
C1A—N1A—H11A	112.8 (14)	C1—C6—H6	119.00
C1A—N1A—H12A	108.9 (14)	C5—C6—H6	119.00
H11A—N1A—H12A	107 (2)	C3A—C1A—C4A	111.52 (15)
H11A—N1A—H13A	108 (2)	N1A—C1A—C2A	107.49 (15)
C1A—N1A—H13A	109.6 (15)	N1A—C1A—C3A	107.35 (14)
H12A—N1A—H13A	110 (2)	N1A—C1A—C4A	107.46 (15)
C2—C1—C11	128.84 (15)	C2A—C1A—C3A	111.81 (16)
C6—C1—C11	112.93 (15)	C2A—C1A—C4A	110.97 (15)
C2—C1—C6	118.24 (15)	C1A—C2A—H21A	109.00
C1—C2—C21	129.57 (15)	C1A—C2A—H22A	109.00
C1—C2—C3	118.09 (15)	C1A—C2A—H23A	109.00
C3—C2—C21	112.35 (16)	H21A—C2A—H22A	109.00

C2—C3—C4	122.71 (17)	H21A—C2A—H23A	109.00
Cl2—C4—C5	120.72 (12)	H22A—C2A—H23A	109.00
Cl2—C4—C3	120.19 (14)	C1A—C3A—H31A	110.00
C3—C4—C5	119.08 (15)	C1A—C3A—H32A	109.00
Cl1—C5—C4	121.35 (12)	C1A—C3A—H33A	109.00
Cl1—C5—C6	119.36 (14)	H31A—C3A—H32A	109.00
C4—C5—C6	119.28 (15)	H31A—C3A—H33A	109.00
C1—C6—C5	122.58 (17)	H32A—C3A—H33A	109.00
O11—C11—C1	118.25 (15)	C1A—C4A—H41A	109.00
O11—C11—O12	121.94 (16)	C1A—C4A—H42A	109.00
O12—C11—C1	119.82 (15)	C1A—C4A—H43A	109.00
O21—C21—C2	118.87 (16)	H41A—C4A—H42A	110.00
O21—C21—O22	121.30 (17)	H41A—C4A—H43A	109.00
O22—C21—C2	119.83 (16)	H42A—C4A—H43A	109.00
C2—C3—H3	119.00		

C6—C1—C2—C3	1.9 (2)	C1—C2—C21—O21	−3.2 (3)
C6—C1—C2—C21	−177.99 (17)	C1—C2—C21—O22	176.59 (18)
C11—C1—C2—C3	−178.68 (16)	C3—C2—C21—O21	176.90 (17)
C11—C1—C2—C21	1.4 (3)	C3—C2—C21—O22	−3.3 (2)
C2—C1—C6—C5	−0.9 (2)	C2—C3—C4—Cl2	−178.43 (14)
C11—C1—C6—C5	179.57 (15)	C2—C3—C4—C5	0.5 (3)
C2—C1—C11—O11	175.26 (17)	Cl2—C4—C5—Cl1	−0.1 (2)
C2—C1—C11—O12	−4.8 (3)	Cl2—C4—C5—C6	179.48 (13)
C6—C1—C11—O11	−5.3 (2)	C3—C4—C5—Cl1	−179.02 (13)
C6—C1—C11—O12	174.65 (15)	C3—C4—C5—C6	0.5 (2)
C1—C2—C3—C4	−1.8 (3)	Cl1—C5—C6—C1	179.23 (13)
C21—C2—C3—C4	178.14 (16)	C4—C5—C6—C1	−0.3 (3)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H11*A*···O21	0.89 (2)	2.02 (2)	2.883 (2)	164 (2)
N1*A*—H12*A*···O11^{vi}	0.91 (2)	1.88 (2)	2.784 (2)	174 (2)
N1*A*—H13*A*···O12^{vii}	0.89 (2)	1.99 (2)	2.861 (2)	167 (2)
O21—H21···O12	0.94 (4)	1.47 (4)	2.4021 (19)	173 (4)
C3—H3···O22	0.93	2.29	2.671 (2)	104
C6—H6···O11	0.93	2.27	2.657 (2)	104

Symmetry codes: (vi) −*x*+1, −*y*+1, −*z*+2; (vii) *x*−1, *y*, *z*.