Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(benzylaminium) 4,5-dichlorobenzene-1,2-dicarboxylate monohydrate

Graham Smith* and Urs D. Wermuth

Science and Engineering Faculty, Queensland University of Technology, GPO Box 2434, Brisbane, Queensland 4001, Australia
Correspondence e-mail: g.smith@qut.edu.au

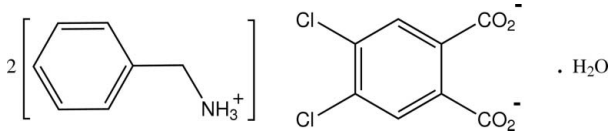
Received 21 May 2012; accepted 22 May 2012

Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.007 Å; R factor = 0.068; wR factor = 0.189; data-to-parameter ratio = 12.3.

In the structure of the title salt, $2C_7H_{10}N^+\cdot C_8H_2Cl_2O_4^{2-}\cdot H_2O$, the two benzylaminium anions have different conformations, one being essentially planar and the other having the side chain rotated out of the benzene plane [minimum ring to side-chain C—C—C—N torsion angles = −3.6 (6) and 50.1 (5)°, respectively]. In the 4,5-dichlorophthalate dianion, the carboxylate groups make dihedral angles of 23.0 (2) and 76.5 (2)° with the benzene ring. In the crystal, aminium N—H···O and water O—H···O hydrogen-bonding associations with carboxylate O-atom acceptors give a two-dimensional duplex sheet structure which extends along the (011) plane. Weak π–π interactions are also present between the benzene ring of the dianion and one of the cation rings [minimum ring centroid separation = 2.749 (3) Å].

Related literature

For the crystal structures of some 1:1 Lewis base salts of 4,5-dichlorophthalic acid, see: Mattes & Dorau (1986); Smith *et al.* (2008). For crystal structures having dianionic 4,5-dichlorophthalate species, see: Büyükgüngör & Odabaşoğlu (2007); Smith & Wermuth (2010, 2011).



Experimental

Crystal data

$2C_7H_{10}N^+\cdot C_8H_2Cl_2O_4^{2-}\cdot H_2O$
M_r = 467.33
Monoclinic, $P2_1/c$
a = 17.3005 (16) Å
b = 10.0084 (7) Å
c = 13.6990 (12) Å
β = 112.641 (11)°
V = 2189.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 200 K
0.33 × 0.22 × 0.12 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.86, T_{max} = 0.98
12311 measured reflections
3842 independent reflections
3019 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.189
S = 1.19
3842 reflections
312 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.79 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N11A—H11A···O21	0.87	2.01	2.866 (4)	168
N11A—H12A···O12^{i}	0.90	1.90	2.801 (5)	180
N11A—H13A···O22^{ii}	0.88	1.94	2.816 (4)	175
N11B—H11B···O11^{i}	0.88	1.96	2.823 (4)	167
N11B—H12B···O21	0.96	1.85	2.770 (4)	160
N11B—H13B···O22^{iii}	0.82	1.99	2.803 (4)	172
O1W—H11W···O11^{i}	0.85	1.94	2.789 (4)	179
O1W—H12W···O12	0.76	2.28	2.980 (4)	154

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); 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*.

The authors acknowledge financial support from the Australian Research Council, the Science and Engineering Faculty and the University Library, Queensland University of Technology.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2476).

References

Büyükgüngör, O. & Odabaşoğlu, M. (2007). *Acta Cryst.* E**63**, o4376–o4377.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Mattes, R. & Dorau, A. (1986). *Z. Naturforsch. Teil B*, **41**, 808–814.
Oxford Diffraction (2010). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Smith, G. & Wermuth, U. D. (2010). *Acta Cryst.* E**66**, o235.
Smith, G. & Wermuth, U. D. (2011). *Acta Cryst.* E**67**, o1645.
Smith, G., Wermuth, U. D. & White, J. M. (2008). *Acta Cryst.* C**64**, o532–o536.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supplementary materials

Acta Cryst. (2012). E**68**, o1928 [doi:10.1107/S1600536812023458]

Bis(benzylaminium) 4,5-dichlorobenzene-1,2-dicarboxylate monohydrate

Graham Smith and Urs D. Wermuth

Comment

4,5-Dichlorophthalic acid (DCPA) most commonly forms 1:1 salts with the Lewis bases, often giving low-dimensional hydrogen-bonded structures (Mattes & Dorau, 1986; Smith *et al.*, 2008). The structures of the 2:1 Lewis base salts of DCPA are less common; the bis(4-ethylaminium) salt (Büyükgüngör & Odabaşoğlu, 2007) and the bis(guanidinium) salt (Smith & Wermuth, 2011) are among these while the DCPA dianion is also found in the ethylenediaminium salt (Smith & Wermuth, 2010). However, our 1:1 stoichiometric reaction of DCPA with benzylamine gave unexpectedly a 2:1 salt $2(C_7H_{10}N^+)\,C_8H_2Cl_2O_4^{2-}.\,H_2O$, the title compound, and the structure is reported here.

In this structure (Fig. 1), the two benzylaminium cations (*A* and *B*) have very different conformations, one being essentially planar the other having the side-chain rotated out of the benzene plane [minimum ring to side-chain C—C—C—N torsion angles = -3.6 (6)° (*A*) and 50.1 (5)° *B*]. In the 4,5-dichlorophthalate dianion the carboxyl groups make dihedral angles of 23.0 (2) and 76.5 (2)° with the benzene ring, corresponding to torsion angles C1—C2—C21—O21 and C2—C1—C11—O11 of -157.7 (4) and 78.1 (5)°. Aminium N—H···O and water O—H···O hydrogen-bonding associations with carboxyl O-atom acceptors (Table 1) give a two-dimensional duplex-sheet structure which extends along the (011) plane (Fig. 2). Weak π–π interactions are also present between the benzene ring of the DCPA dianion and one of the cation rings (*A*) [minimum ring centroid separation, 2.749 (3) Å].

Experimental

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

Refinement

Hydrogen atoms potentially involved in hydrogen-bonding interactions were located by difference methods and their positional and isotropic displacement parameters were initially refined. However, in the final refinement cycles these were set invariant with the displacement parameters riding on the parent atom [with U_{iso}(H) = 1.2U_{eq}(N) or 1.5U_{eq}(O)]. Other H atoms were included at calculated positions [C—H (aromatic) = 0.93 Å or C—H (methylene) = 0.97 Å] and allowed to ride, with U_{iso}(H) = 1.2U_{eq}(C).

Computing details

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2010); data reduction: *CrysAlis PRO* (Oxford Diffraction, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); 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* (Spek, 2009).

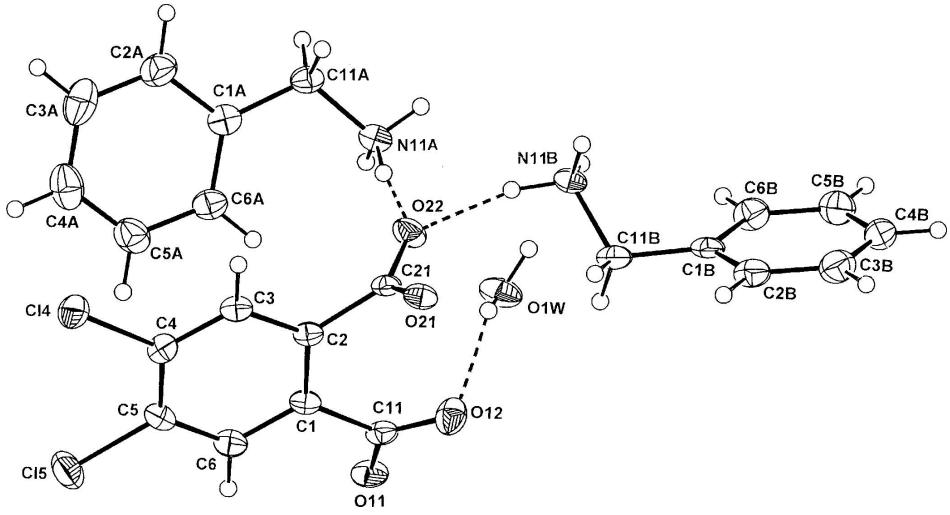


Figure 1

Structure of the two cations (*A* and *B*), the dianion and the water molecule of solvation in the asymmetric unit of the title salt, with the inter-species hydrogen bonds shown as dashed lines. Non-H atoms are shown as 40% probability displacement ellipsoids. Hydrogen atoms are shown as spheres of arbitrary radius.

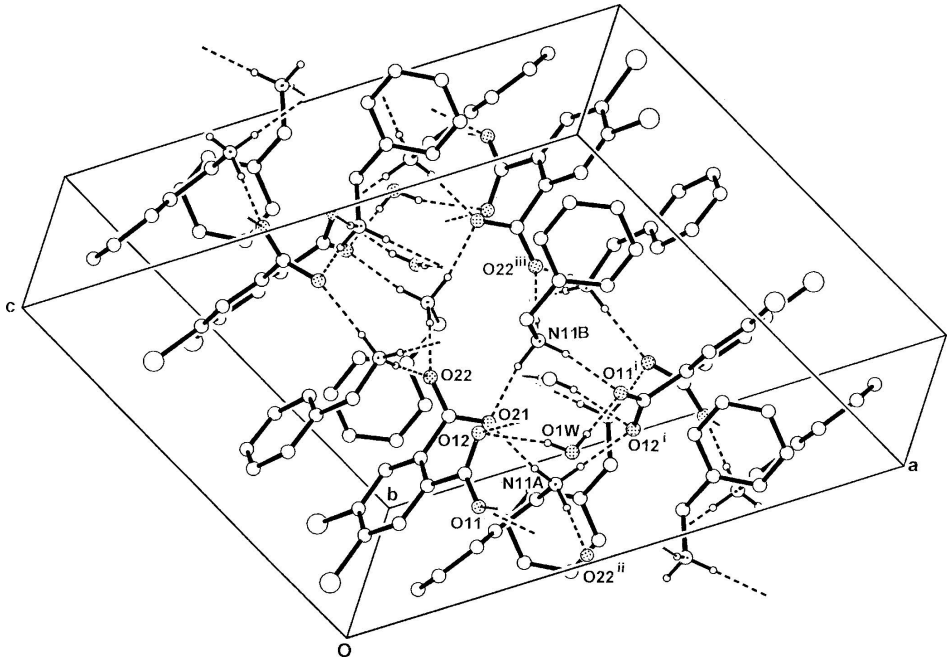


Figure 2

A perspective view of part of the two-dimensional duplex-sheet structure in the unit cell, showing hydrogen-bonding associations as dashed lines. Non-associative H-atoms are omitted. For symmetry codes, see Table 1.

Bis(benzylaminium) 4,5-dichlorobenzene-1,2-dicarboxylate monohydrate

Crystal data

$2C_7H_{10}N^+\cdot C_8H_2Cl_2O_4^{2-}\cdot H_2O$
$M_r = 467.33$

Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc

a = 17.3005 (16) Å
b = 10.0084 (7) Å
c = 13.6990 (12) Å
β = 112.641 (11)°
V = 2189.2 (4) Å^3
Z = 4
F(000) = 976
D_x = 1.418 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6279 reflections
θ = 3.2–28.7°
μ = 0.33 mm^{-1}
T = 200 K
Plate, colourless
0.33 × 0.22 × 0.12 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} = 0.86, T_{max} = 0.98

12311 measured reflections
3842 independent reflections
3019 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 25.0°, θ_{min} = 3.2°
h = −20→20
k = −11→11
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.189
S = 1.19
3842 reflections
312 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
$w = 1/[\sigma^2(F_o^2) + (0.0794P)^2 + 3.4869P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.79 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 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}
N11A	0.4026 (2)	0.4021 (3)	0.0987 (2)	0.0293 (11)
C1A	0.2474 (3)	0.4407 (4)	0.0233 (3)	0.0307 (11)
C2A	0.1790 (3)	0.5252 (5)	−0.0185 (4)	0.0460 (17)
C3A	0.0984 (3)	0.4801 (6)	−0.0447 (4)	0.0571 (19)
C4A	0.0838 (3)	0.3462 (6)	−0.0318 (4)	0.0515 (19)
C5A	0.1508 (3)	0.2619 (5)	0.0077 (4)	0.0443 (16)
C6A	0.2319 (3)	0.3065 (4)	0.0357 (3)	0.0353 (14)
C11A	0.3333 (3)	0.4980 (4)	0.0506 (3)	0.0322 (12)
N11B	0.5631 (2)	0.4462 (3)	0.4310 (2)	0.0270 (10)
C1B	0.6924 (3)	0.3655 (4)	0.5777 (3)	0.0295 (11)

C2B	0.7238 (3)	0.3664 (4)	0.6877 (3)	0.0378 (14)
C3B	0.8081 (3)	0.3812 (5)	0.7454 (4)	0.0446 (16)
C4B	0.8630 (3)	0.3963 (4)	0.6953 (4)	0.0431 (16)
C5B	0.8322 (3)	0.3971 (5)	0.5861 (4)	0.0443 (17)
C6B	0.7479 (3)	0.3822 (5)	0.5275 (4)	0.0392 (16)
C11B	0.6004 (3)	0.3454 (4)	0.5161 (3)	0.0329 (12)
Cl4	0.05397 (7)	0.26123 (11)	0.21573 (9)	0.0404 (4)
Cl5	0.06311 (7)	−0.04167 (12)	0.15221 (10)	0.0496 (4)
O11	0.38460 (19)	−0.0979 (3)	0.2378 (2)	0.0332 (9)
O12	0.45046 (18)	0.0453 (3)	0.3688 (2)	0.0394 (10)
O21	0.40882 (18)	0.3398 (3)	0.3058 (2)	0.0353 (9)
O22	0.39728 (17)	0.3096 (3)	0.4609 (2)	0.0285 (8)
C1	0.3039 (2)	0.0669 (4)	0.2786 (3)	0.0243 (11)
C2	0.2997 (2)	0.1997 (4)	0.3108 (3)	0.0232 (11)
C3	0.2221 (2)	0.2560 (4)	0.2924 (3)	0.0259 (11)
C4	0.1492 (2)	0.1842 (4)	0.2424 (3)	0.0283 (12)
C5	0.1534 (3)	0.0524 (4)	0.2126 (3)	0.0302 (12)
C6	0.2302 (3)	−0.0051 (4)	0.2304 (3)	0.0293 (12)
C11	0.3866 (3)	0.0005 (4)	0.2974 (3)	0.0278 (12)
C21	0.3755 (2)	0.2872 (4)	0.3636 (3)	0.0240 (11)
O1W	0.5248 (2)	0.1641 (3)	0.2253 (2)	0.0437 (11)
H2A	0.18780	0.61480	−0.02910	0.0550*
H3A	0.05360	0.53930	−0.07100	0.0690*
H4A	0.02950	0.31450	−0.04970	0.0620*
H5A	0.14150	0.17180	0.01590	0.0530*
H6A	0.27630	0.24680	0.06290	0.0420*
H11A	0.40280	0.37170	0.15800	0.0350*
H12A	0.44980	0.44770	0.10900	0.0350*
H13A	0.40080	0.33970	0.05300	0.0350*
H14A	0.33700	0.53380	−0.01330	0.0390*
H15A	0.34070	0.57180	0.09930	0.0390*
H2B	0.68730	0.35690	0.72240	0.0450*
H3B	0.82830	0.38090	0.81890	0.0540*
H4B	0.92010	0.40590	0.73450	0.0520*
H5B	0.86890	0.40780	0.55180	0.0530*
H6B	0.72790	0.38340	0.45400	0.0470*
H11B	0.58300	0.44510	0.38110	0.0320*
H12B	0.50500	0.42810	0.39110	0.0320*
H13B	0.57000	0.52010	0.45910	0.0320*
H14B	0.59160	0.25700	0.48470	0.0390*
H15B	0.57160	0.34930	0.56430	0.0390*
H3	0.21900	0.34330	0.31390	0.0310*
H6	0.23260	−0.09300	0.20990	0.0350*
H11W	0.55210	0.23660	0.23670	0.0650*
H12W	0.50510	0.15860	0.26570	0.0650*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11A	0.037 (2)	0.0257 (18)	0.0298 (17)	−0.0037 (15)	0.0179 (15)	−0.0026 (14)

C1A	0.040 (2)	0.030 (2)	0.0254 (19)	0.0025 (19)	0.0164 (18)	−0.0033 (16)
C2A	0.048 (3)	0.036 (3)	0.052 (3)	0.008 (2)	0.017 (2)	0.002 (2)
C3A	0.040 (3)	0.069 (4)	0.058 (3)	0.018 (3)	0.014 (2)	−0.002 (3)
C4A	0.039 (3)	0.079 (4)	0.040 (3)	−0.008 (3)	0.019 (2)	−0.004 (3)
C5A	0.045 (3)	0.050 (3)	0.041 (2)	−0.009 (2)	0.020 (2)	0.003 (2)
C6A	0.043 (3)	0.036 (2)	0.031 (2)	0.002 (2)	0.0188 (19)	0.0054 (18)
C11A	0.041 (2)	0.023 (2)	0.033 (2)	0.0014 (18)	0.0148 (18)	−0.0012 (17)
N11B	0.0371 (19)	0.0191 (16)	0.0293 (16)	−0.0034 (14)	0.0179 (15)	−0.0041 (13)
C1B	0.038 (2)	0.0147 (18)	0.041 (2)	0.0021 (17)	0.0210 (19)	0.0040 (16)
C2B	0.045 (3)	0.031 (2)	0.042 (2)	0.002 (2)	0.022 (2)	0.0070 (19)
C3B	0.049 (3)	0.040 (3)	0.041 (2)	0.002 (2)	0.013 (2)	0.004 (2)
C4B	0.034 (3)	0.027 (2)	0.064 (3)	0.0046 (19)	0.014 (2)	−0.001 (2)
C5B	0.041 (3)	0.039 (3)	0.062 (3)	0.004 (2)	0.030 (2)	0.003 (2)
C6B	0.042 (3)	0.040 (3)	0.041 (2)	0.004 (2)	0.022 (2)	0.005 (2)
C11B	0.041 (2)	0.021 (2)	0.043 (2)	0.0023 (18)	0.023 (2)	0.0063 (17)
Cl4	0.0307 (6)	0.0397 (6)	0.0530 (7)	0.0075 (5)	0.0185 (5)	0.0053 (5)
Cl5	0.0350 (6)	0.0422 (7)	0.0676 (8)	−0.0120 (5)	0.0155 (6)	−0.0114 (6)
O11	0.0445 (18)	0.0204 (14)	0.0431 (16)	0.0051 (13)	0.0262 (14)	−0.0038 (12)
O12	0.0333 (17)	0.0378 (17)	0.0430 (17)	0.0084 (14)	0.0103 (14)	−0.0079 (14)
O21	0.0378 (17)	0.0388 (17)	0.0327 (15)	−0.0107 (14)	0.0173 (13)	0.0040 (13)
O22	0.0405 (16)	0.0210 (14)	0.0276 (14)	−0.0035 (12)	0.0171 (12)	−0.0039 (11)
C1	0.035 (2)	0.0204 (19)	0.0226 (18)	−0.0011 (16)	0.0169 (16)	0.0002 (15)
C2	0.031 (2)	0.0222 (19)	0.0221 (17)	0.0015 (16)	0.0164 (16)	0.0029 (15)
C3	0.036 (2)	0.0210 (19)	0.0255 (19)	0.0025 (17)	0.0173 (17)	−0.0015 (15)
C4	0.030 (2)	0.032 (2)	0.029 (2)	0.0059 (18)	0.0180 (17)	0.0040 (17)
C5	0.035 (2)	0.025 (2)	0.032 (2)	−0.0073 (18)	0.0146 (18)	−0.0015 (16)
C6	0.038 (2)	0.024 (2)	0.031 (2)	−0.0005 (18)	0.0189 (18)	−0.0018 (16)
C11	0.034 (2)	0.023 (2)	0.032 (2)	0.0036 (17)	0.0188 (18)	0.0075 (17)
C21	0.029 (2)	0.0151 (18)	0.031 (2)	0.0028 (15)	0.0150 (17)	0.0032 (15)
O1W	0.059 (2)	0.0254 (16)	0.0577 (19)	−0.0065 (15)	0.0345 (17)	−0.0072 (14)

Geometric parameters (Å, º)

Cl4—C4	1.726 (4)	C6A—H6A	0.9300
Cl5—C5	1.737 (5)	C11A—H15A	0.9700
O11—C11	1.271 (5)	C11A—H14A	0.9700
O12—C11	1.244 (5)	C1B—C2B	1.391 (5)
O21—C21	1.260 (5)	C1B—C11B	1.501 (7)
O22—C21	1.258 (5)	C1B—C6B	1.390 (7)
O1W—H11W	0.8500	C2B—C3B	1.373 (7)
O1W—H12W	0.7600	C3B—C4B	1.378 (8)
N11A—C11A	1.478 (6)	C4B—C5B	1.381 (7)
N11A—H13A	0.8800	C5B—C6B	1.376 (8)
N11A—H11A	0.8700	C2B—H2B	0.9300
N11A—H12A	0.9000	C3B—H3B	0.9300
N11B—C11B	1.488 (5)	C4B—H4B	0.9300
N11B—H12B	0.9600	C5B—H5B	0.9300
N11B—H13B	0.8200	C6B—H6B	0.9300
N11B—H11B	0.8800	C11B—H15B	0.9700
C1A—C6A	1.393 (6)	C11B—H14B	0.9700

C1A—C2A	1.386 (7)	C1—C11	1.507 (6)
C1A—C11A	1.500 (7)	C1—C2	1.411 (6)
C2A—C3A	1.375 (8)	C1—C6	1.390 (6)
C3A—C4A	1.388 (8)	C2—C21	1.510 (5)
C4A—C5A	1.366 (8)	C2—C3	1.387 (5)
C5A—C6A	1.379 (8)	C3—C4	1.382 (5)
C2A—H2A	0.9300	C4—C5	1.391 (6)
C3A—H3A	0.9300	C5—C6	1.381 (7)
C4A—H4A	0.9300	C3—H3	0.9300
C5A—H5A	0.9300	C6—H6	0.9300

H11W—O1W—H12W	108.00	C4B—C5B—C6B	120.8 (5)
C11A—N11A—H13A	110.00	C1B—C6B—C5B	120.2 (5)
C11A—N11A—H11A	111.00	N11B—C11B—C1B	113.3 (4)
H12A—N11A—H13A	105.00	C1B—C2B—H2B	120.00
H11A—N11A—H13A	114.00	C3B—C2B—H2B	120.00
H11A—N11A—H12A	111.00	C4B—C3B—H3B	120.00
C11A—N11A—H12A	106.00	C2B—C3B—H3B	120.00
C11B—N11B—H11B	115.00	C3B—C4B—H4B	120.00
H12B—N11B—H13B	112.00	C5B—C4B—H4B	120.00
C11B—N11B—H13B	108.00	C4B—C5B—H5B	120.00
H11B—N11B—H13B	110.00	C6B—C5B—H5B	120.00
C11B—N11B—H12B	111.00	C5B—C6B—H6B	120.00
H11B—N11B—H12B	101.00	C1B—C6B—H6B	120.00
C6A—C1A—C11A	123.9 (4)	N11B—C11B—H15B	109.00
C2A—C1A—C11A	118.4 (4)	C1B—C11B—H14B	109.00
C2A—C1A—C6A	117.7 (5)	C1B—C11B—H15B	109.00
C1A—C2A—C3A	121.7 (5)	H14B—C11B—H15B	108.00
C2A—C3A—C4A	120.0 (5)	N11B—C11B—H14B	109.00
C3A—C4A—C5A	118.6 (5)	C2—C1—C6	119.2 (4)
C4A—C5A—C6A	121.9 (5)	C2—C1—C11	121.4 (3)
C1A—C6A—C5A	120.1 (4)	C6—C1—C11	119.3 (4)
N11A—C11A—C1A	114.8 (3)	C1—C2—C21	123.8 (3)
C3A—C2A—H2A	119.00	C3—C2—C21	116.9 (3)
C1A—C2A—H2A	119.00	C1—C2—C3	119.3 (4)
C2A—C3A—H3A	120.00	C2—C3—C4	120.9 (4)
C4A—C3A—H3A	120.00	Cl4—C4—C3	119.2 (3)
C3A—C4A—H4A	121.00	Cl4—C4—C5	121.0 (3)
C5A—C4A—H4A	121.00	C3—C4—C5	119.8 (4)
C4A—C5A—H5A	119.00	Cl5—C5—C6	119.1 (3)
C6A—C5A—H5A	119.00	C4—C5—C6	119.9 (4)
C1A—C6A—H6A	120.00	Cl5—C5—C4	121.0 (4)
C5A—C6A—H6A	120.00	C1—C6—C5	120.8 (4)
H14A—C11A—H15A	108.00	O11—C11—C1	116.3 (4)
N11A—C11A—H14A	109.00	O12—C11—C1	118.2 (4)
N11A—C11A—H15A	109.00	O11—C11—O12	125.5 (5)
C1A—C11A—H14A	109.00	O21—C21—C2	117.7 (3)
C1A—C11A—H15A	109.00	O22—C21—C2	117.5 (3)
C2B—C1B—C6B	118.6 (5)	O21—C21—O22	124.7 (4)

C2B—C1B—C11B	119.8 (4)	C2—C3—H3	120.00
C6B—C1B—C11B	121.6 (4)	C4—C3—H3	119.00
C1B—C2B—C3B	120.6 (5)	C1—C6—H6	120.00
C2B—C3B—C4B	120.5 (5)	C5—C6—H6	120.00
C3B—C4B—C5B	119.2 (5)		

C6A—C1A—C2A—C3A	1.6 (7)	C11—C1—C2—C3	−179.5 (4)
C11A—C1A—C2A—C3A	−179.5 (4)	C11—C1—C2—C21	1.4 (6)
C2A—C1A—C6A—C5A	−0.6 (6)	C2—C1—C6—C5	0.9 (6)
C11A—C1A—C6A—C5A	−179.4 (4)	C11—C1—C6—C5	179.6 (4)
C2A—C1A—C11A—N11A	177.6 (4)	C2—C1—C11—O11	−157.7 (4)
C6A—C1A—C11A—N11A	−3.6 (6)	C2—C1—C11—O12	22.4 (6)
C1A—C2A—C3A—C4A	−1.5 (8)	C6—C1—C11—O11	23.6 (6)
C2A—C3A—C4A—C5A	0.4 (8)	C6—C1—C11—O12	−156.3 (4)
C3A—C4A—C5A—C6A	0.6 (8)	C1—C2—C3—C4	−0.5 (6)
C4A—C5A—C6A—C1A	−0.5 (7)	C21—C2—C3—C4	178.6 (3)
C6B—C1B—C2B—C3B	−1.1 (6)	C1—C2—C21—O21	78.1 (5)
C11B—C1B—C2B—C3B	178.3 (4)	C1—C2—C21—O22	−106.7 (5)
C2B—C1B—C6B—C5B	1.0 (7)	C3—C2—C21—O21	−101.0 (4)
C11B—C1B—C6B—C5B	−178.4 (4)	C3—C2—C21—O22	74.2 (5)
C2B—C1B—C11B—N11B	130.6 (4)	C2—C3—C4—Cl4	−177.0 (3)
C6B—C1B—C11B—N11B	−50.1 (5)	C2—C3—C4—C5	1.8 (6)
C1B—C2B—C3B—C4B	0.4 (7)	Cl4—C4—C5—Cl5	−2.6 (5)
C2B—C3B—C4B—C5B	0.3 (7)	Cl4—C4—C5—C6	177.0 (3)
C3B—C4B—C5B—C6B	−0.4 (7)	C3—C4—C5—Cl5	178.6 (3)
C4B—C5B—C6B—C1B	−0.3 (7)	C3—C4—C5—C6	−1.8 (6)
C6—C1—C2—C3	−0.8 (6)	Cl5—C5—C6—C1	−180.0 (3)
C6—C1—C2—C21	−179.9 (4)	C4—C5—C6—C1	0.4 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11*A*—H11*A*···O21	0.87	2.01	2.866 (4)	168
N11*A*—H12*A*···O12^{i}	0.90	1.90	2.801 (5)	180
N11*A*—H13*A*···O22^{ii}	0.88	1.94	2.816 (4)	175
N11*B*—H11*B*···O11^{i}	0.88	1.96	2.823 (4)	167
N11*B*—H12*B*···O21	0.96	1.85	2.770 (4)	160
N11*B*—H13*B*···O22^{iii}	0.82	1.99	2.803 (4)	172
O1*W*—H11*W*···O11^{i}	0.85	1.94	2.789 (4)	179
O1*W*—H12*W*···O12	0.76	2.28	2.980 (4)	154

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