Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(guanidinium) 4,5-dichlorophthalate monohydrate

Graham Smith* and Urs D. Wermuth

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

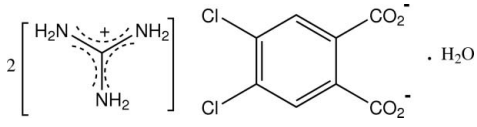
Received 30 May 2011; accepted 1 June 2011

Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.105; data-to-parameter ratio = 12.6.

In the structure of the title hydrated salt, $2CH_6N_3^+\cdot C_8H_2Cl_2O_4^{2-}\cdot H_2O$, the planes of the carboxylate groups of the dianion are rotated out of the plane of the benzene ring [dihedral angles = 48.42 (10) and 55.64 (9)°]. A duplex-sheet structure is formed through guanidinium–carboxylate N—H···O, guanidinium–water N—H···O and water–carboxylate O—H···O hydrogen-bonding associations.

Related literature

For the structures of 1:1 salts of 4,5-dichlorophthalate, see: Mallinson *et al.* (2003); Bozkurt *et al.* (2006); Smith *et al.* (2008, 2009); Smith & Wermuth (2010*a*,*d*). For 1:2 salts, see: Büyükgüngör & Odabaşoğlu (2007); Smith & Wermuth (2010*a*,*c*). For guanidinium salts of aromatic dicarboxylic acids, see: Krumbe & Haussuhl (1986); Smith & Wermuth (2010*b*).



Experimental

Crystal data

$2CH_6N_3^+\cdot C_8H_2Cl_2O_4^{2-}\cdot H_2O$
M_r = 371.19
Monoclinic, $P2_1/c$
a = 15.9797 (5) Å
b = 6.9432 (2) Å
c = 15.2266 (5) Å
β = 94.650 (3)°
V = 1683.84 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.42 mm^{-1}
T = 200 K
0.28 × 0.25 × 0.20 mm

Data collection

Oxford Diffraction Gemini-S CCD area-detector diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.933, T_{max} = 0.990
11236 measured reflections
3319 independent reflections
2627 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.105
S = 1.16
3319 reflections
264 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.63 e Å^{-3}
$\Delta\rho_{min}$ = −0.83 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1A—H11A···O1W^{i}	0.83 (2)	2.14 (2)	2.966 (2)	171 (2)
N1A—H12A···O12	0.89 (2)	2.07 (2)	2.914 (2)	156.8 (19)
N1B—H11B···O22^{ii}	0.88 (2)	2.07 (2)	2.936 (2)	166.3 (19)
N1B—H12B···O12^{iii}	0.85 (2)	2.09 (2)	2.904 (2)	162 (2)
N2A—H21A···O11	0.89 (3)	2.59 (3)	3.447 (2)	160 (2)
N2A—H21A···O12	0.89 (3)	2.35 (3)	3.125 (2)	145 (2)
N2A—H22A···O1W^{iv}	0.81 (3)	2.20 (3)	3.010 (2)	175 (2)
N2B—H21B···O22^{iii}	0.86 (3)	2.07 (3)	2.894 (2)	161 (3)
N2B—H22B···O11	0.91 (3)	2.09 (3)	2.880 (2)	144 (2)
N3A—H31A···O11^{v}	0.85 (3)	2.06 (3)	2.874 (2)	159 (2)
N3A—H32A···O22^{i}	0.84 (2)	2.19 (2)	2.923 (2)	147 (2)
N3B—H31B···O21	0.89 (2)	1.92 (2)	2.799 (2)	169 (2)
N3B—H32B···O11^{vi}	0.86 (2)	2.20 (2)	2.966 (2)	149.2 (19)
O1W—H11W···O21	0.83 (3)	1.97 (3)	2.789 (2)	169 (3)
O1W—H12W···O12^{vi}	0.88 (3)	1.90 (3)	2.7716 (19)	174 (3)

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

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*.

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

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5176).

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Bozkurt, E., Kartal, I., Odabaşoğlu, M. & Büyükgüngör, O. (2006). *Acta Cryst.* E**62**, o4258–o4260.
Büyükgüngör, O. & Odabaşoğlu, M. (2007). *Acta Cryst.* E**63**, o4376–o4377.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Krumbe, W. & Haussuhl, S. (1986). *Z. Kristallogr.* **179**, 267–279.
Mallinson, P. R., Smith, G. T., Wilson, C. C., Grech, E. & Wozniak, K. (2003). *J. Am. Chem. Soc.* **125**, 4259–4270.
Oxford Diffraction (2010). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, Oxfordshire, England.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Smith, G. & Wermuth, U. D. (2010*a*). *Acta Cryst.* C**66**, o374–o380.
Smith, G. & Wermuth, U. D. (2010*b*). *Acta Cryst.* C**66**, o575–o580.
Smith, G. & Wermuth, U. D. (2010*c*). *Acta Cryst.* E**66**, o235.
Smith, G. & Wermuth, U. D. (2010*d*). *J. Chem. Crystallogr.* **40**, 151–155.
Smith, G., Wermuth, U. D. & White, J. M. (2008). *Acta Cryst.* C**64**, o180–o183.
Smith, G., Wermuth, U. D. & White, J. M. (2009). *Acta Cryst.* C**65**, o103–o107.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supplementary materials

Acta Cryst. (2011). E**67**, o1645 [**doi:10.1107/S1600536811021192**]

Bis(guanidinium) 4,5-dichlorophthalate monohydrate

G. Smith and U. D. Wermuth

Comment

4,5-Dichlorophthalic acid (DCPA) forms 1:1 salts with a number of Lewis bases, having most commonly low-dimensional hydrogen-bonded structures featuring the `planar' hydrogen phthalate anion (Mallinson *et al.*, 2003; Bozkurt *et al.*, 2006; Smith *et al.*, 2008, 2009; Smith & Wermuth, 2010*a*,*d*). The `nonplanar' dianionic DCPA species is much less common among the known structures, examples being the 1:2 salts with 4-ethylaniline (Büyükgüngör & Odabaşoğlu, 2007), ethylenediamine (Smith & Wermuth, 2010*c*), *n*-butylamine and piperidine (Smith & Wermuth, 2010*a*). With the strong base guanidine, the formation of 1:2 salts with dicarboxylic acids is more common, *e.g.* with phthalic acid (Krumbe & Haussuhl, 1986) and terephthalic acids (Smith & Wermuth, 2010*b*) and our 1:1 stoichiometric reaction of DCPA with guanidine carbonate not unexpectedly gave the bis(guanidinium) salt hydrate, the title compound, $2(CH_6N_3^+)\ C_8H_2Cl_2O_4^{2-}.\ H_2O$ (I) (Fig. 1), and the structure is reported here.

In the structure of (I), the two guanidinium cations (*A* and *B*) and the water molecule of solvation provide hydrogen-bonding links between the `non-planar' DCPA dianions (Table 1). The planes of the carboxyl groups of the dianion are rotated out of the plane of the benzene ring [torsion angles C1—C2—C21–O22, -131.93 (17)°; C2—C1—C11–O11, -129.41 (16)°]. Duplex-sheet structures are formed, extending down the (011) planes in the unit cell (Fig. 2). Within these sheets there are guanidinium N—H···$O_{carboxyl}$, N—H···O_{water} and water O—H···$O_{carboxyl}$ associations.

Experimental

Compound (I) was synthesized by heating together for 10 min under reflux, 1 mmol quantities of 4,5-dichlorophthalic acid and guanidine carbonate in 50 ml of 50% ethanol–water. Total evaporation of solvent gave a white non-crystalline powder which on subsequent slow room-temperature evaporation of an aqueous solution gave colourless crystalline plates of (I) from which a specimen was cleaved for the X-ray analysis.

Refinement

H 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 = 0.93 Å) and treated as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

Fig. 1. Molecular conformation and atom-numbering scheme for the two guanidinium cations, the DCPA dianion and the water molecule of solvation in (I), with inter-species hydrogen bonds shown as dashed lines. Non-H atoms are shown as 40% probability displacement ellipsoids.

Fig. 2. A view the two-dimensional duplex-sheet structure in the unit cell of (I), viewed down the sheets, showing hydrogen-bonding associations as dashed lines. Non-associative H atoms are omitted.

Bis(guanidinium) 4,5-dichlorophthalate monohydrate

Crystal data

$2CH_6N_3^+ \cdot C_8H_2Cl_2O_4^{2-} \cdot H_2O$	$F(000) = 768$
$M_r = 371.19$	$D_x = 1.464$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 6093 reflections
$a = 15.9797$ (5) Å	$\theta = 3.2$–$28.6°$
$b = 6.9432$ (2) Å	$\mu = 0.42$ mm^{-1}
$c = 15.2266$ (5) Å	$T = 200$ K
$\beta = 94.650$ (3)°	Block, colourless
$V = 1683.84$ (9) Å^3	0.28 × 0.25 × 0.20 mm
$Z = 4$	

Data collection

Oxford Diffraction Gemini-S CCD area-detector diffractometer	3319 independent reflections
Radiation source: Enhance (Mo) X-ray source	2627 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.022$
Detector resolution: 16.077 pixels mm^{-1}	$\theta_{max} = 26.0°$, $\theta_{min} = 3.2°$
ω scans	$h = -18 \rightarrow 19$
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)	$k = -8 \rightarrow 8$
$T_{min} = 0.933$, $T_{max} = 0.990$	$l = -11 \rightarrow 18$
11236 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.039$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.105$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.16$	$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.1489P]$ where $P = (F_o^2 + 2F_c^2)/3$
3319 reflections	$(\Delta/\sigma)_{max} = 0.001$
264 parameters	$\Delta\rho_{max} = 0.63$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.83$ 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}
Cl4	1.05872 (4)	0.24406 (13)	0.56212 (5)	0.0786 (3)
Cl5	1.03264 (4)	−0.12369 (11)	0.67711 (5)	0.0676 (3)
O11	0.70520 (8)	0.02886 (19)	0.75803 (8)	0.0304 (4)
O12	0.66435 (8)	0.04844 (17)	0.61463 (8)	0.0260 (4)
O21	0.70448 (9)	0.47401 (19)	0.64857 (9)	0.0325 (4)
O22	0.73076 (9)	0.47465 (18)	0.50680 (9)	0.0313 (4)
C1	0.80565 (11)	0.1185 (2)	0.65803 (11)	0.0207 (5)
C2	0.81671 (11)	0.2830 (2)	0.60675 (11)	0.0221 (5)
C3	0.89502 (12)	0.3185 (3)	0.57737 (13)	0.0338 (6)
C4	0.96178 (12)	0.1945 (4)	0.59905 (14)	0.0397 (7)
C5	0.95069 (13)	0.0335 (3)	0.65047 (14)	0.0361 (7)
C6	0.87267 (12)	−0.0039 (3)	0.68007 (12)	0.0278 (6)
C11	0.71840 (11)	0.0626 (2)	0.67974 (12)	0.0206 (5)
C21	0.74498 (11)	0.4223 (2)	0.58557 (12)	0.0221 (5)
N1A	0.51848 (11)	0.2730 (3)	0.54869 (12)	0.0297 (5)
N2A	0.51734 (11)	0.2568 (3)	0.69927 (12)	0.0318 (5)
N3A	0.40865 (10)	0.4034 (2)	0.61686 (13)	0.0281 (5)
C1A	0.48092 (11)	0.3101 (3)	0.62137 (12)	0.0231 (5)
N1B	0.74241 (11)	0.6136 (3)	0.96559 (11)	0.0288 (5)
N2B	0.73486 (13)	0.3392 (3)	0.88218 (13)	0.0403 (6)
N3B	0.74928 (11)	0.6306 (3)	0.81514 (12)	0.0314 (6)
C1B	0.74182 (11)	0.5289 (3)	0.88781 (12)	0.0246 (6)
O1W	0.56692 (9)	0.7224 (2)	0.63168 (9)	0.0296 (4)
H3	0.90300	0.42630	0.54280	0.0410*
H6	0.86520	−0.11150	0.71490	0.0330*
H11A	0.4941 (13)	0.288 (3)	0.4989 (16)	0.029 (6)*
H12A	0.5663 (15)	0.206 (3)	0.5529 (14)	0.040 (6)*
H21A	0.5643 (18)	0.187 (4)	0.7004 (17)	0.056 (7)*
H22A	0.4931 (16)	0.254 (3)	0.7442 (18)	0.048 (7)*
H31A	0.3862 (16)	0.437 (3)	0.6634 (18)	0.049 (7)*
H32A	0.3855 (14)	0.436 (3)	0.5679 (16)	0.038 (7)*
H11B	0.7413 (13)	0.740 (3)	0.9689 (13)	0.033 (6)*
H12B	0.7272 (14)	0.546 (3)	1.0077 (15)	0.037 (6)*
H21B	0.7375 (16)	0.267 (4)	0.9278 (18)	0.055 (8)*

H22B	0.7359 (15)	0.281 (4)	0.8289 (18)	0.056 (8)*
H31B	0.7401 (14)	0.570 (3)	0.7640 (16)	0.039 (6)*
H32B	0.7451 (13)	0.753 (3)	0.8189 (14)	0.032 (6)*
H11W	0.6032 (18)	0.637 (4)	0.6354 (18)	0.058 (8)*
H12W	0.5946 (16)	0.831 (4)	0.6270 (17)	0.054 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl4	0.0240 (3)	0.1185 (7)	0.0961 (6)	0.0108 (3)	0.0218 (3)	0.0627 (5)
Cl5	0.0354 (3)	0.0869 (5)	0.0818 (5)	0.0326 (3)	0.0135 (3)	0.0423 (4)
O11	0.0375 (8)	0.0322 (7)	0.0229 (7)	−0.0065 (6)	0.0117 (6)	−0.0002 (6)
O12	0.0220 (7)	0.0265 (7)	0.0292 (7)	−0.0010 (5)	0.0003 (5)	0.0023 (5)
O21	0.0359 (8)	0.0334 (7)	0.0283 (7)	0.0100 (6)	0.0042 (6)	−0.0032 (6)
O22	0.0381 (8)	0.0279 (7)	0.0276 (7)	0.0084 (6)	0.0002 (6)	0.0072 (6)
C1	0.0228 (9)	0.0234 (9)	0.0160 (8)	0.0001 (7)	0.0024 (7)	0.0006 (7)
C2	0.0230 (9)	0.0242 (9)	0.0188 (9)	−0.0003 (7)	0.0007 (7)	0.0030 (7)
C3	0.0274 (10)	0.0399 (11)	0.0345 (11)	−0.0015 (9)	0.0050 (8)	0.0178 (9)
C4	0.0202 (10)	0.0607 (14)	0.0391 (12)	0.0016 (9)	0.0077 (9)	0.0193 (11)
C5	0.0254 (10)	0.0471 (13)	0.0360 (12)	0.0122 (9)	0.0029 (9)	0.0129 (10)
C6	0.0288 (10)	0.0299 (10)	0.0248 (10)	0.0038 (8)	0.0025 (8)	0.0082 (8)
C11	0.0241 (9)	0.0143 (8)	0.0241 (9)	0.0013 (7)	0.0059 (7)	−0.0002 (7)
C21	0.0246 (9)	0.0161 (8)	0.0251 (10)	−0.0023 (7)	−0.0004 (7)	0.0007 (7)
N1A	0.0270 (9)	0.0394 (10)	0.0230 (9)	0.0087 (8)	0.0038 (7)	0.0013 (7)
N2A	0.0275 (9)	0.0451 (10)	0.0232 (9)	0.0083 (8)	0.0051 (7)	0.0033 (8)
N3A	0.0216 (8)	0.0381 (9)	0.0248 (10)	0.0028 (7)	0.0036 (7)	−0.0012 (8)
C1A	0.0211 (9)	0.0228 (9)	0.0258 (10)	−0.0032 (7)	0.0046 (7)	0.0001 (7)
N1B	0.0426 (10)	0.0223 (9)	0.0216 (9)	−0.0005 (7)	0.0027 (7)	−0.0003 (7)
N2B	0.0696 (14)	0.0238 (9)	0.0275 (10)	−0.0036 (9)	0.0042 (9)	−0.0020 (8)
N3B	0.0434 (10)	0.0275 (10)	0.0236 (9)	0.0023 (8)	0.0048 (7)	0.0019 (7)
C1B	0.0251 (9)	0.0247 (10)	0.0237 (10)	−0.0003 (7)	0.0005 (7)	0.0011 (7)
O1W	0.0265 (7)	0.0258 (8)	0.0363 (8)	−0.0026 (6)	0.0021 (6)	0.0010 (6)

Geometric parameters (Å, °)

Cl4—C4	1.725 (2)	N2B—C1B	1.324 (3)
Cl5—C5	1.728 (2)	N3B—C1B	1.326 (3)
O11—C11	1.250 (2)	N1B—H12B	0.85 (2)
O12—C11	1.265 (2)	N1B—H11B	0.88 (2)
O21—C21	1.252 (2)	N2B—H21B	0.86 (3)
O22—C21	1.256 (2)	N2B—H22B	0.91 (3)
O1W—H12W	0.88 (3)	N3B—H31B	0.89 (2)
O1W—H11W	0.83 (3)	N3B—H32B	0.86 (2)
N1A—C1A	1.326 (3)	C1—C6	1.387 (3)
N2A—C1A	1.331 (3)	C1—C11	1.510 (2)
N3A—C1A	1.321 (2)	C1—C2	1.403 (2)
N1A—H12A	0.89 (2)	C2—C21	1.514 (2)
N1A—H11A	0.83 (2)	C2—C3	1.385 (3)
N2A—H22A	0.81 (3)	C3—C4	1.390 (3)

N2A—H21A	0.89 (3)	C4—C5	1.384 (3)
N3A—H32A	0.84 (2)	C5—C6	1.384 (3)
N3A—H31A	0.85 (3)	C3—H3	0.9300
N1B—C1B	1.322 (3)	C6—H6	0.9300
H11W—O1W—H12W	105 (3)	C2—C3—C4	120.62 (19)
H11A—N1A—H12A	118 (2)	C3—C4—C5	120.24 (19)
C1A—N1A—H12A	119.0 (14)	Cl4—C4—C3	119.43 (19)
C1A—N1A—H11A	121.8 (15)	Cl4—C4—C5	120.33 (17)
H21A—N2A—H22A	115 (2)	Cl5—C5—C4	120.90 (16)
C1A—N2A—H22A	123.6 (18)	Cl5—C5—C6	119.38 (16)
C1A—N2A—H21A	118.3 (17)	C4—C5—C6	119.71 (19)
C1A—N3A—H32A	120.1 (16)	C1—C6—C5	120.30 (18)
H31A—N3A—H32A	119 (2)	O12—C11—C1	115.63 (15)
C1A—N3A—H31A	121.1 (17)	O11—C11—O12	125.16 (16)
C1B—N1B—H12B	116.8 (15)	O11—C11—C1	119.18 (16)
H11B—N1B—H12B	120 (2)	O22—C21—C2	117.60 (15)
C1B—N1B—H11B	119.8 (13)	O21—C21—C2	116.69 (15)
C1B—N2B—H22B	119.6 (18)	O21—C21—O22	125.71 (16)
C1B—N2B—H21B	122.2 (19)	C2—C3—H3	120.00
H21B—N2B—H22B	118 (3)	C4—C3—H3	120.00
H31B—N3B—H32B	122 (2)	C5—C6—H6	120.00
C1B—N3B—H31B	117.4 (14)	C1—C6—H6	120.00
C1B—N3B—H32B	117.4 (14)	N1A—C1A—N2A	119.67 (18)
C6—C1—C11	119.89 (14)	N1A—C1A—N3A	120.31 (18)
C2—C1—C6	120.26 (16)	N2A—C1A—N3A	120.00 (18)
C2—C1—C11	119.44 (15)	N1B—C1B—N2B	119.68 (19)
C3—C2—C21	120.38 (15)	N1B—C1B—N3B	121.1 (2)
C1—C2—C21	120.74 (15)	N2B—C1B—N3B	119.24 (19)
C1—C2—C3	118.86 (16)		
C6—C1—C2—C3	1.2 (2)	C1—C2—C21—O21	47.5 (2)
C6—C1—C2—C21	−177.34 (16)	C1—C2—C21—O22	−131.93 (17)
C11—C1—C2—C3	−171.43 (16)	C3—C2—C21—O21	−131.02 (18)
C11—C1—C2—C21	10.0 (2)	C3—C2—C21—O22	49.6 (2)
C2—C1—C6—C5	−1.1 (3)	C2—C3—C4—Cl4	−179.47 (15)
C11—C1—C6—C5	171.54 (17)	C2—C3—C4—C5	0.0 (3)
C2—C1—C11—O11	−129.41 (16)	Cl4—C4—C5—Cl5	−1.3 (3)
C2—C1—C11—O12	52.6 (2)	Cl4—C4—C5—C6	179.63 (16)
C6—C1—C11—O11	57.9 (2)	C3—C4—C5—Cl5	179.30 (17)
C6—C1—C11—O12	−120.04 (17)	C3—C4—C5—C6	0.2 (3)
C1—C2—C3—C4	−0.7 (3)	Cl5—C5—C6—C1	−178.78 (15)
C21—C2—C3—C4	177.87 (18)	C4—C5—C6—C1	0.4 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1A—H11A···O1W^{i}	0.83 (2)	2.14 (2)	2.966 (2)	171 (2)
N1A—H12A···O12	0.89 (2)	2.07 (2)	2.914 (2)	156.8 (19)
N1B—H11B···O22^{ii}	0.88 (2)	2.07 (2)	2.936 (2)	166.3 (19)

N1B—H12B···O12^{iii}	0.85 (2)	2.09 (2)	2.904 (2)	162 (2)
N2A—H21A···O11	0.89 (3)	2.59 (3)	3.447 (2)	160 (2)
N2A—H21A···O12	0.89 (3)	2.35 (3)	3.125 (2)	145 (2)
N2A—H22A···O1W^{iv}	0.81 (3)	2.20 (3)	3.010 (2)	175 (2)
N2B—H21B···O22^{iii}	0.86 (3)	2.07 (3)	2.894 (2)	161 (3)
N2B—H22B···O11	0.91 (3)	2.09 (3)	2.880 (2)	144 (2)
N3A—H31A···O11^{v}	0.85 (3)	2.06 (3)	2.874 (2)	159 (2)
N3A—H32A···O22^{i}	0.84 (2)	2.19 (2)	2.923 (2)	147 (2)
N3B—H31B···O21	0.89 (2)	1.92 (2)	2.799 (2)	169 (2)
N3B—H32B···O11^{vi}	0.86 (2)	2.20 (2)	2.966 (2)	149.2 (19)
O1W—H11W···O21	0.83 (3)	1.97 (3)	2.789 (2)	169 (3)
O1W—H12W···O12^{vi}	0.88 (3)	1.90 (3)	2.7716 (19)	174 (3)

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

Fig. 1

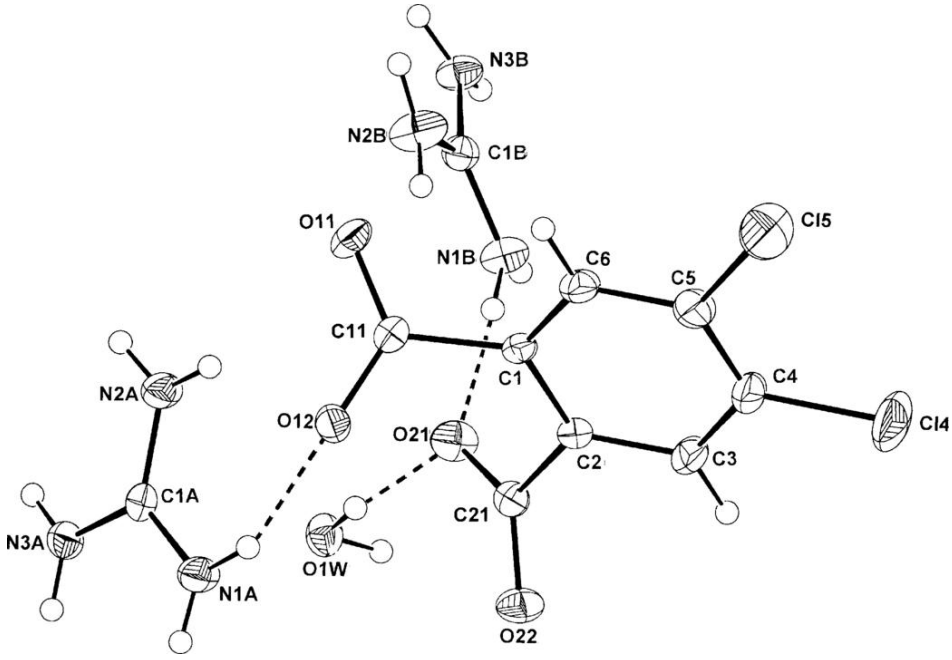
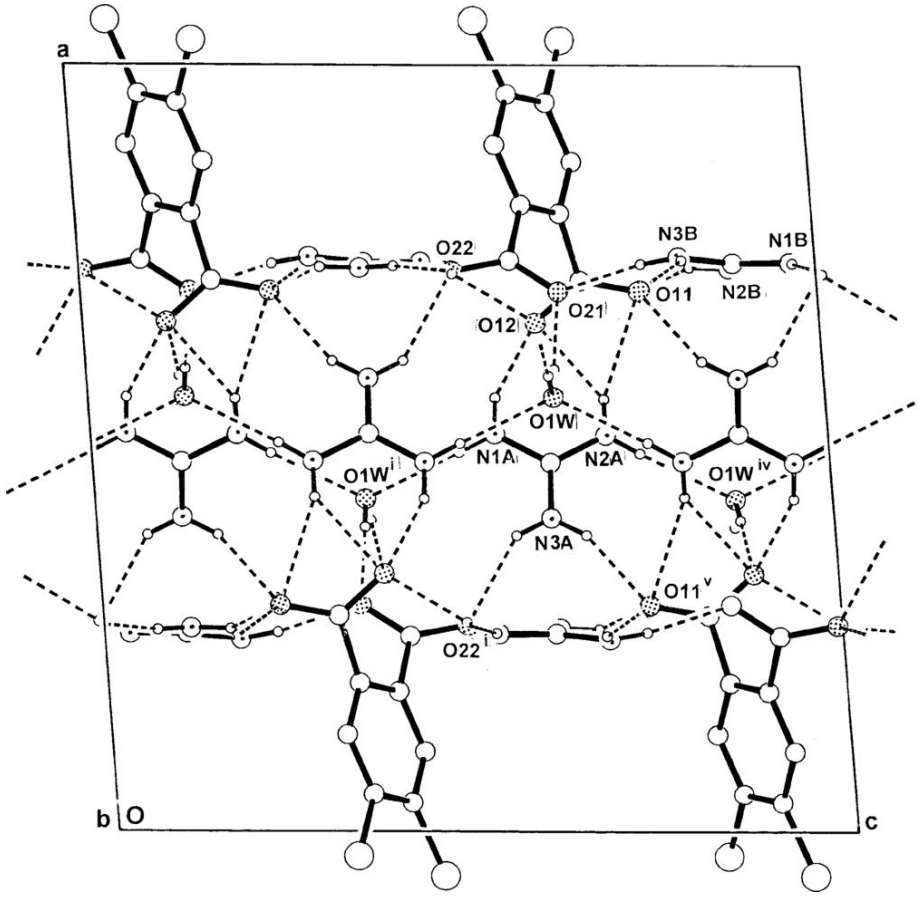

N3B
N2B
C1B
O11
N1B
C6
C5
Cl5
C11
C1
C4
N2A
O12
O21
C2
C3
Cl4
C1A
C21
N3A
O1W
N1A
O22

Fig. 2


a
b
O
c
O22
N3B
N1B
O11
N2B
O12
O21
O1W
O1W i
N1A
N2A
O1W iv
N3A
O11 v
O22 i