Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4,4′-Bipiperidinediium bis(hydrogen chloranilate)

Kazuma Gotoh, Rie Ishikawa and Hiroyuki Ishida*

Department of Chemistry, Faculty of Science, Okayama University, Okayama 700-8530, Japan
Correspondence e-mail: ishidah@cc.okayama-u.ac.jp

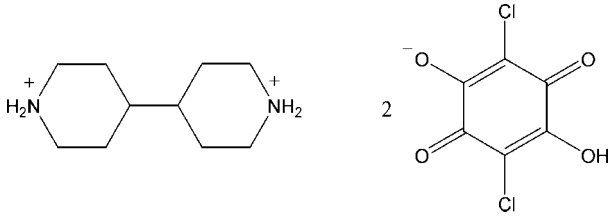
Received 27 October 2007; accepted 28 October 2007

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.088; data-to-parameter ratio = 20.0.

In the crystal structure of the title 1:2 salt, $C_{10}H_{22}N_2^{2+}\cdot 2C_6HCl_2O_4^-$, the 4,4′-bipiperidinium dication and the two hydrogen chloranilate anions are held together by N—H···O and C—H···O hydrogen bonds. Two 1:2 units are connected by O—H···O hydrogen bonds, forming a centrosymmetric molecular ring. The rings are further linked by O—H···O, N—H···O and C—H···O hydrogen bonds, leading to a three-dimensional hydrogen-bonding network.

Related literature

For related literature, see: Allen (2002); Ishida (2004); Refat *et al.* (2006).



Experimental

Crystal data

$C_{10}H_{22}N_2^{2+}\cdot 2C_6HCl_2O_4^-$
M_r = 586.25
Monoclinic, $P2_1/n$
a = 17.4716 (10) Å
b = 7.7132 (4) Å
c = 19.1252 (11) Å
β = 111.5434 (19)°
V = 2397.3 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.55 mm^{-1}
T = 100 (2) K
0.25 × 0.17 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.850, T_{max} = 0.937
26002 measured reflections
6982 independent reflections
5848 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.088
S = 1.11
6982 reflections
349 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1···O3	0.87 (3)	2.18 (2)	2.6479 (16)	113.6 (19)
O2—H1···O8^{i}	0.87 (3)	2.17 (3)	2.9113 (18)	142 (2)
O6—H2···O3^{ii}	0.82 (3)	2.12 (3)	2.7290 (18)	132 (2)
O6—H2···O7	0.82 (3)	2.11 (2)	2.6093 (16)	119 (2)
N1—H3···O1	0.90 (2)	1.97 (2)	2.7921 (19)	152 (2)
N1—H3···O3^{iii}	0.90 (2)	2.48 (2)	2.9412 (19)	112.7 (17)
N1—H4···O8^{iv}	0.86 (2)	2.03 (2)	2.8269 (17)	156 (2)
N2—H14···O5	0.90 (2)	2.15 (2)	2.9223 (19)	143 (2)
N2—H14···O8	0.90 (2)	2.50 (2)	3.200 (2)	135.7 (18)
N2—H15···O2^{i}	0.88 (2)	2.55 (2)	3.0332 (18)	115.1 (17)
N2—H15···O4^{v}	0.88 (2)	1.98 (2)	2.8158 (18)	159 (2)
C14—H7···O1	0.98	2.49	3.211 (2)	130
C14—H8···O6^{vi}	0.98	2.48	3.2561 (19)	136
C16—H10···O5^{vii}	0.98	2.59	3.531 (2)	161
C18—H17···O7^{viii}	0.98	2.39	3.193 (2)	138

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

Data collection: *PROCESS-AUTO* (Rigaku/MSC, 2004); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure* and *PLATON* (Spek, 2003).

This work was supported by a Grant-in-Aid for Scientific Research (C) (No. 19550018) from the Japan Society for the Promotion of Science.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2333).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Ishida, H. (2004). *Acta Cryst.* E**60**, o1674–o1676.
Refat, M. S., Ahmed, H. A.-D., El-Zayat, L. A., Fukunaga, T. & Ishida, H. (2006). *Acta Cryst.* E**62**, o1886–o1887.
Rigaku/MSC (2004). *PROCESS-AUTO* and *CrystalStructure* (Version 3.7.0). Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

  doi:10.1107/S160053680705386X

supplementary materials

Acta Cryst. (2007). E**63**, o4518 [**doi:10.1107/S160053680705386X**]

4,4'-Bipiperidinediium bis(hydrogen chloranilate)

K. Gotoh, R. Ishikawa and H. Ishida

Comment

The title compound, (I), was prepared in order to extend our study on D—H···A hydrogen bonding (*D* = N, O, or C; *A* = N, O or Cl) in amine – chloranilic acid (1/1) and (2/1) systems (Ishida, 2004; Refat *et al.*, 2006). To our knowledge, this is the first crystallographic report of the amine – chloranilic acid (1/2) system; no structural data of compounds of the (1/2) system are recorded in the Cambridge Structural Database, version 5.28 (2007 Release; Allen, 2002).

The asymmetric unit in the title compound contains one 4,4'-bipiperidinium dication and two hydrogen chloranilate anions, which are connected by N—H···O, bifurcated N—H···O and C—H···O hydrogen bonds (Table 1) to form a 1:2 unit (Fig. 1). The two units related by an inversion center are held together through O—H···O hydrogen bonds, forming a macro molecular ring (Fig. 2). The rings are further connected by O—H···O, N—H···O and C—H···O hydrogen bonds, leading to a three-dimensional hydrogen-bonding network.

Experimental

Single crystals were obtained by slow evaporation from a methanol solution of chloranilic acid (63 mg) with 4,4'-bipiperidyl dihydrochloride (36 mg).

Refinement

N– and O-bound H atoms were located in a difference Fourier map and refined isotropically (refined distances are given in Table 1). Other H atoms were positioned geometrically (C—H = 0.98 or 0.99 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

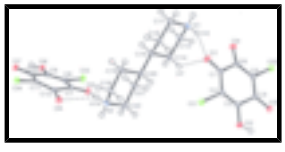


Fig. 1. The molecular structure of the title compound, with the atom numbering. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level. N—H···O and C—H···O hydrogen bonds are indicated by dashed lines.

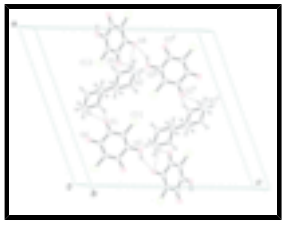


Fig. 2. A partial packing diagram, viewed approximately along the *b* axis, showing the centrosymmetric molecular ring formed by O—H···O, N—H···O and C—H···O hydrogen bonds (dashed lines) [symmetry code (i) is as given in Table 1].

4,4'-Bipiperidinium bis(hydrogen chloranilate)

Crystal data

$C_{10}H_{22}N_2^{2+}\cdot 2C_6HCl_2O_4^-$	F_{000} = 1208.00
M_r = 586.25	D_x = 1.624 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71075 Å
Hall symbol: -P 2yn	Cell parameters from 27745 reflections
a = 17.4716 (10) Å	θ = 3.2–30.1°
b = 7.7132 (4) Å	μ = 0.55 mm^{-1}
c = 19.1252 (11) Å	T = 100 (2) K
β = 111.5434 (19)°	Needle, purple
V = 2397.3 (2) Å^3	0.25 × 0.17 × 0.12 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID diffractometer	6982 independent reflections
Detector resolution: 10.00 pixels mm^{-1}	5848 reflections with $I > 2\sigma(I)$
T = 100(2) K	R_{int} = 0.035
ω scans	θ_{max} = 30.0°
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	h = −24→23
T_{min} = 0.850, T_{max} = 0.937	k = −10→10
26002 measured reflections	l = −26→26

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.040	$w = 1/[\sigma^2(F_o^2) + (0.0399P)^2 + 1.34P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.088	$(\Delta/\sigma)_{max}$ = <0.001
S = 1.11	$\Delta\rho_{max}$ = 0.43 e Å^{-3}
6982 reflections	$\Delta\rho_{min}$ = −0.29 e Å^{-3}
349 parameters	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.40240 (2)	0.05182 (5)	0.45568 (2)	0.01654 (8)
Cl2	0.10315 (2)	0.00510 (6)	0.14248 (2)	0.01867 (9)
Cl3	0.79278 (2)	0.95757 (6)	0.30125 (2)	0.02012 (9)
Cl4	1.08946 (2)	0.66212 (5)	0.58222 (2)	0.01466 (8)
O1	0.40238 (7)	−0.07957 (16)	0.31131 (7)	0.0174 (2)
O2	0.22655 (7)	0.13356 (15)	0.42519 (6)	0.0139 (2)
O3	0.10072 (7)	0.08933 (15)	0.29685 (6)	0.0143 (2)
O4	0.28393 (7)	−0.05474 (16)	0.17399 (6)	0.0165 (2)
O5	0.78585 (7)	0.73421 (16)	0.42744 (6)	0.0163 (2)
O6	0.97184 (7)	1.01381 (15)	0.33987 (6)	0.0141 (2)
O7	1.09316 (7)	0.88325 (15)	0.45278 (6)	0.0151 (2)
O8	0.90753 (7)	0.66571 (16)	0.55947 (6)	0.0163 (2)
N1	0.48752 (8)	−0.08134 (18)	0.21307 (7)	0.0127 (2)
N2	0.72848 (8)	0.49927 (18)	0.51771 (8)	0.0137 (3)
C1	0.33488 (9)	−0.0260 (2)	0.30686 (8)	0.0120 (3)
C2	0.31958 (9)	0.0360 (2)	0.37204 (8)	0.0120 (3)
C3	0.24259 (9)	0.07760 (19)	0.36626 (8)	0.0109 (3)
C4	0.16933 (9)	0.05946 (19)	0.29323 (8)	0.0112 (3)
C5	0.18512 (9)	0.0135 (2)	0.22824 (8)	0.0132 (3)
C6	0.26422 (9)	−0.0220 (2)	0.22931 (8)	0.0119 (3)
C7	0.85514 (9)	0.7831 (2)	0.43545 (8)	0.0119 (3)
C8	0.87308 (9)	0.8865 (2)	0.37990 (8)	0.0127 (3)
C9	0.95195 (9)	0.9208 (2)	0.38904 (8)	0.0115 (3)
C10	1.02423 (9)	0.84710 (19)	0.45370 (8)	0.0111 (3)
C11	1.00710 (9)	0.7486 (2)	0.50857 (8)	0.0115 (3)
C12	0.92737 (9)	0.72772 (19)	0.50780 (8)	0.0116 (3)
C13	0.48474 (10)	0.1099 (2)	0.19929 (9)	0.0160 (3)
H5	0.4285	0.1439	0.1682	0.019*
H6	0.5205	0.1386	0.1717	0.019*
C14	0.51248 (10)	0.2110 (2)	0.27274 (8)	0.0138 (3)
H7	0.4736	0.1906	0.2980	0.017*
H8	0.5119	0.3353	0.2618	0.017*
C15	0.59924 (9)	0.1584 (2)	0.32548 (8)	0.0113 (3)
H9	0.6384	0.1855	0.3005	0.014*
C16	0.59974 (9)	−0.0392 (2)	0.33798 (8)	0.0123 (3)
H10	0.6557	−0.0760	0.3689	0.015*
H11	0.5638	−0.0665	0.3656	0.015*
C17	0.57093 (10)	−0.1400 (2)	0.26450 (9)	0.0146 (3)
H12	0.6104	−0.1239	0.2396	0.018*
H13	0.5690	−0.2639	0.2751	0.018*

C18	0.73772 (10)	0.3062 (2)	0.52769 (9)	0.0148 (3)
H16	0.7031	0.2651	0.5547	0.018*
H17	0.7950	0.2787	0.5583	0.018*
C19	0.71351 (9)	0.2121 (2)	0.45278 (9)	0.0140 (3)
H18	0.7172	0.0867	0.4616	0.017*
H19	0.7522	0.2429	0.4284	0.017*
C20	0.62581 (9)	0.25870 (19)	0.40037 (8)	0.0107 (3)
H20	0.5879	0.2268	0.4260	0.013*
C21	0.62210 (9)	0.4567 (2)	0.38979 (8)	0.0129 (3)
H21	0.6600	0.4914	0.3652	0.015*
H22	0.5664	0.4904	0.3571	0.015*
C22	0.64503 (10)	0.5509 (2)	0.46465 (9)	0.0147 (3)
H23	0.6440	0.6763	0.4561	0.018*
H24	0.6043	0.5243	0.4872	0.018*
H1	0.1747 (15)	0.161 (3)	0.4119 (13)	0.032 (6)*
H2	1.0218 (16)	1.013 (3)	0.3529 (14)	0.034 (7)*
H3	0.4514 (13)	−0.111 (3)	0.2344 (12)	0.018 (5)*
H4	0.4730 (13)	−0.133 (3)	0.1706 (12)	0.016 (5)*
H14	0.7663 (13)	0.542 (3)	0.5008 (12)	0.019 (5)*
H15	0.7399 (13)	0.543 (3)	0.5629 (12)	0.016 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01155 (15)	0.02349 (19)	0.01206 (16)	0.00183 (14)	0.00137 (13)	−0.00107 (14)
Cl2	0.01405 (16)	0.0287 (2)	0.01102 (16)	−0.00135 (15)	0.00191 (13)	−0.00284 (14)
Cl3	0.01221 (16)	0.0314 (2)	0.01461 (17)	0.00159 (15)	0.00244 (13)	0.00730 (15)
Cl4	0.01224 (15)	0.01595 (17)	0.01420 (16)	0.00208 (13)	0.00299 (13)	0.00227 (13)
O1	0.0131 (5)	0.0216 (6)	0.0191 (6)	0.0026 (5)	0.0079 (4)	−0.0011 (5)
O2	0.0126 (5)	0.0195 (6)	0.0107 (5)	0.0015 (4)	0.0055 (4)	−0.0016 (4)
O3	0.0116 (5)	0.0166 (5)	0.0158 (5)	0.0000 (4)	0.0065 (4)	−0.0001 (4)
O4	0.0188 (5)	0.0202 (6)	0.0128 (5)	0.0025 (5)	0.0086 (4)	−0.0007 (4)
O5	0.0109 (5)	0.0209 (6)	0.0173 (5)	−0.0021 (4)	0.0055 (4)	0.0020 (5)
O6	0.0130 (5)	0.0166 (5)	0.0148 (5)	−0.0004 (4)	0.0075 (4)	0.0030 (4)
O7	0.0112 (5)	0.0180 (6)	0.0176 (5)	−0.0010 (4)	0.0069 (4)	0.0003 (4)
O8	0.0148 (5)	0.0212 (6)	0.0145 (5)	−0.0008 (5)	0.0074 (4)	0.0045 (4)
N1	0.0135 (6)	0.0140 (6)	0.0102 (6)	−0.0026 (5)	0.0038 (5)	−0.0022 (5)
N2	0.0137 (6)	0.0147 (6)	0.0124 (6)	−0.0038 (5)	0.0043 (5)	−0.0030 (5)
C1	0.0129 (6)	0.0113 (7)	0.0128 (7)	−0.0007 (5)	0.0058 (5)	0.0006 (5)
C2	0.0109 (6)	0.0147 (7)	0.0094 (6)	−0.0002 (5)	0.0025 (5)	−0.0002 (5)
C3	0.0130 (6)	0.0100 (6)	0.0109 (6)	0.0001 (5)	0.0057 (5)	0.0008 (5)
C4	0.0112 (6)	0.0105 (6)	0.0123 (6)	−0.0012 (5)	0.0048 (5)	0.0005 (5)
C5	0.0116 (6)	0.0172 (7)	0.0096 (6)	−0.0007 (6)	0.0024 (5)	−0.0013 (5)
C6	0.0133 (6)	0.0111 (6)	0.0120 (6)	−0.0005 (5)	0.0054 (5)	−0.0008 (5)
C7	0.0126 (6)	0.0129 (7)	0.0113 (6)	0.0008 (5)	0.0057 (5)	−0.0003 (5)
C8	0.0114 (6)	0.0161 (7)	0.0095 (6)	0.0009 (6)	0.0027 (5)	0.0011 (5)
C9	0.0141 (6)	0.0109 (6)	0.0113 (6)	0.0000 (5)	0.0069 (5)	−0.0001 (5)
C10	0.0122 (6)	0.0105 (6)	0.0111 (6)	−0.0001 (5)	0.0047 (5)	−0.0024 (5)

C11	0.0107 (6)	0.0119 (7)	0.0106 (6)	0.0012 (5)	0.0023 (5)	0.0001 (5)
C12	0.0125 (6)	0.0101 (6)	0.0124 (6)	−0.0006 (5)	0.0049 (5)	−0.0010 (5)
C13	0.0194 (7)	0.0137 (7)	0.0109 (7)	−0.0008 (6)	0.0007 (6)	0.0017 (6)
C14	0.0160 (7)	0.0107 (6)	0.0109 (6)	0.0008 (6)	0.0004 (5)	0.0000 (5)
C15	0.0118 (6)	0.0110 (6)	0.0105 (6)	−0.0003 (5)	0.0035 (5)	0.0003 (5)
C16	0.0129 (6)	0.0108 (7)	0.0115 (6)	0.0009 (5)	0.0023 (5)	−0.0007 (5)
C17	0.0158 (7)	0.0133 (7)	0.0130 (7)	0.0020 (6)	0.0033 (6)	−0.0008 (5)
C18	0.0141 (7)	0.0129 (7)	0.0138 (7)	−0.0003 (6)	0.0011 (6)	−0.0001 (6)
C19	0.0121 (6)	0.0143 (7)	0.0126 (7)	0.0015 (6)	0.0011 (5)	−0.0028 (6)
C20	0.0109 (6)	0.0107 (6)	0.0097 (6)	−0.0004 (5)	0.0030 (5)	0.0003 (5)
C21	0.0141 (7)	0.0110 (6)	0.0121 (7)	−0.0007 (5)	0.0031 (5)	−0.0001 (5)
C22	0.0153 (7)	0.0133 (7)	0.0144 (7)	0.0010 (6)	0.0039 (6)	−0.0007 (6)

Geometric parameters (Å, °)

Cl1—C2	1.7227 (15)	C9—C10	1.517 (2)
Cl2—C5	1.7379 (15)	C10—C11	1.414 (2)
Cl3—C8	1.7267 (15)	C11—C12	1.397 (2)
Cl4—C11	1.7350 (15)	C13—C14	1.522 (2)
O1—C1	1.2226 (18)	C13—H5	0.9800
O2—C3	1.3293 (18)	C13—H6	0.9800
O2—H1	0.87 (2)	C14—C15	1.535 (2)
O3—C4	1.2474 (18)	C14—H7	0.9800
O4—C6	1.2527 (18)	C14—H8	0.9800
O5—C7	1.2228 (18)	C15—C20	1.542 (2)
O6—C9	1.3264 (18)	C15—C16	1.543 (2)
O6—H2	0.82 (3)	C15—H9	0.9900
O7—C10	1.2425 (18)	C16—C17	1.521 (2)
O8—C12	1.2557 (18)	C16—H10	0.9800
N1—C13	1.496 (2)	C16—H11	0.9800
N1—C17	1.498 (2)	C17—H12	0.9800
N1—H3	0.90 (2)	C17—H13	0.9800
N1—H4	0.86 (2)	C18—C19	1.521 (2)
N2—C22	1.493 (2)	C18—H16	0.9800
N2—C18	1.502 (2)	C18—H17	0.9800
N2—H14	0.90 (2)	C19—C20	1.534 (2)
N2—H15	0.88 (2)	C19—H18	0.9800
C1—C2	1.449 (2)	C19—H19	0.9800
C1—C6	1.543 (2)	C20—C21	1.539 (2)
C2—C3	1.347 (2)	C20—H20	0.9900
C3—C4	1.515 (2)	C21—C22	1.522 (2)
C4—C5	1.414 (2)	C21—H21	0.9800
C5—C6	1.402 (2)	C21—H22	0.9800
C7—C8	1.452 (2)	C22—H23	0.9800
C7—C12	1.551 (2)	C22—H24	0.9800
C8—C9	1.350 (2)		
C3—O2—H1	110.6 (16)	H5—C13—H6	108.0
C9—O6—H2	107.9 (18)	C13—C14—C15	111.96 (13)
C13—N1—C17	111.98 (12)	C13—C14—H7	109.2

C13—N1—H3	110.4 (14)	C15—C14—H7	109.2
C17—N1—H3	107.6 (14)	C13—C14—H8	109.2
C13—N1—H4	108.4 (14)	C15—C14—H8	109.2
C17—N1—H4	111.0 (14)	H7—C14—H8	107.9
H3—N1—H4	107.4 (19)	C14—C15—C20	111.45 (12)
C22—N2—C18	112.55 (12)	C14—C15—C16	108.11 (12)
C22—N2—H14	108.7 (14)	C20—C15—C16	111.67 (12)
C18—N2—H14	110.6 (14)	C14—C15—H9	108.5
C22—N2—H15	112.2 (14)	C20—C15—H9	108.5
C18—N2—H15	106.2 (14)	C16—C15—H9	108.5
H14—N2—H15	106.3 (19)	C17—C16—C15	112.39 (12)
O1—C1—C2	121.98 (14)	C17—C16—H10	109.1
O1—C1—C6	118.99 (13)	C15—C16—H10	109.1
C2—C1—C6	119.02 (13)	C17—C16—H11	109.1
C3—C2—C1	120.14 (14)	C15—C16—H11	109.1
C3—C2—Cl1	122.14 (12)	H10—C16—H11	107.9
C1—C2—Cl1	117.71 (11)	N1—C17—C16	111.37 (13)
O2—C3—C2	121.52 (14)	N1—C17—H12	109.4
O2—C3—C4	116.22 (13)	C16—C17—H12	109.4
C2—C3—C4	122.25 (13)	N1—C17—H13	109.4
O3—C4—C5	126.65 (14)	C16—C17—H13	109.4
O3—C4—C3	115.82 (13)	H12—C17—H13	108.0
C5—C4—C3	117.52 (13)	N2—C18—C19	111.94 (13)
C6—C5—C4	123.08 (14)	N2—C18—H16	109.2
C6—C5—Cl2	118.23 (11)	C19—C18—H16	109.2
C4—C5—Cl2	118.68 (11)	N2—C18—H17	109.2
O4—C6—C5	127.04 (14)	C19—C18—H17	109.2
O4—C6—C1	115.89 (13)	H16—C18—H17	107.9
C5—C6—C1	117.05 (13)	C18—C19—C20	111.67 (13)
O5—C7—C8	123.20 (14)	C18—C19—H18	109.3
O5—C7—C12	118.06 (13)	C20—C19—H18	109.3
C8—C7—C12	118.72 (13)	C18—C19—H19	109.3
C9—C8—C7	119.82 (14)	C20—C19—H19	109.3
C9—C8—Cl3	120.98 (12)	H18—C19—H19	107.9
C7—C8—Cl3	119.16 (11)	C19—C20—C21	107.58 (12)
O6—C9—C8	122.33 (14)	C19—C20—C15	112.17 (12)
O6—C9—C10	115.10 (13)	C21—C20—C15	113.08 (12)
C8—C9—C10	122.47 (13)	C19—C20—H20	107.9
O7—C10—C11	126.98 (14)	C21—C20—H20	107.9
O7—C10—C9	115.18 (13)	C15—C20—H20	107.9
C11—C10—C9	117.83 (13)	C22—C21—C20	111.48 (12)
C12—C11—C10	122.41 (13)	C22—C21—H21	109.3
C12—C11—Cl4	119.40 (12)	C20—C21—H21	109.3
C10—C11—Cl4	118.08 (11)	C22—C21—H22	109.3
O8—C12—C11	126.71 (14)	C20—C21—H22	109.3
O8—C12—C7	115.95 (13)	H21—C21—H22	108.0
C11—C12—C7	117.33 (13)	N2—C22—C21	111.48 (13)
N1—C13—C14	111.34 (12)	N2—C22—H23	109.3
N1—C13—H5	109.4	C21—C22—H23	109.3

C14—C13—H5	109.4	N2—C22—H24	109.3
N1—C13—H6	109.4	C21—C22—H24	109.3
C14—C13—H6	109.4	H23—C22—H24	108.0
O1—C1—C2—C3	−173.35 (15)	O6—C9—C10—C11	179.20 (13)
C6—C1—C2—C3	7.5 (2)	C8—C9—C10—C11	−4.3 (2)
O1—C1—C2—Cl1	6.1 (2)	O7—C10—C11—C12	174.38 (15)
C6—C1—C2—Cl1	−173.04 (11)	C9—C10—C11—C12	−4.5 (2)
C1—C2—C3—O2	179.49 (14)	O7—C10—C11—Cl4	−1.7 (2)
Cl1—C2—C3—O2	0.1 (2)	C9—C10—C11—Cl4	179.40 (11)
C1—C2—C3—C4	1.0 (2)	C10—C11—C12—O8	−168.50 (15)
Cl1—C2—C3—C4	−178.42 (11)	Cl4—C11—C12—O8	7.5 (2)
O2—C3—C4—O3	−4.0 (2)	C10—C11—C12—C7	12.7 (2)
C2—C3—C4—O3	174.57 (15)	Cl4—C11—C12—C7	−171.22 (11)
O2—C3—C4—C5	175.08 (14)	O5—C7—C12—O8	−13.5 (2)
C2—C3—C4—C5	−6.3 (2)	C8—C7—C12—O8	167.88 (14)
O3—C4—C5—C6	−178.45 (15)	O5—C7—C12—C11	165.34 (15)
C3—C4—C5—C6	2.6 (2)	C8—C7—C12—C11	−13.2 (2)
O3—C4—C5—Cl2	3.0 (2)	C17—N1—C13—C14	−55.38 (17)
C3—C4—C5—Cl2	−175.97 (11)	N1—C13—C14—C15	56.83 (17)
C4—C5—C6—O4	−176.08 (15)	C13—C14—C15—C20	−178.69 (13)
Cl2—C5—C6—O4	2.5 (2)	C13—C14—C15—C16	−55.60 (16)
C4—C5—C6—C1	5.6 (2)	C14—C15—C16—C17	55.13 (16)
Cl2—C5—C6—C1	−175.86 (11)	C20—C15—C16—C17	178.08 (12)
O1—C1—C6—O4	−8.5 (2)	C13—N1—C17—C16	54.67 (17)
C2—C1—C6—O4	170.67 (14)	C15—C16—C17—N1	−55.48 (17)
O1—C1—C6—C5	170.03 (15)	C22—N2—C18—C19	52.57 (17)
C2—C1—C6—C5	−10.8 (2)	N2—C18—C19—C20	−55.52 (17)
O5—C7—C8—C9	−173.27 (15)	C18—C19—C20—C21	57.38 (16)
C12—C7—C8—C9	5.2 (2)	C18—C19—C20—C15	−177.66 (13)
O5—C7—C8—Cl3	4.5 (2)	C14—C15—C20—C19	−178.13 (12)
C12—C7—C8—Cl3	−176.98 (11)	C16—C15—C20—C19	60.84 (16)
C7—C8—C9—O6	179.66 (14)	C14—C15—C20—C21	−56.25 (16)
Cl3—C8—C9—O6	1.9 (2)	C16—C15—C20—C21	−177.29 (12)
C7—C8—C9—C10	3.4 (2)	C19—C20—C21—C22	−58.14 (16)
Cl3—C8—C9—C10	−174.37 (11)	C15—C20—C21—C22	177.44 (12)
O6—C9—C10—O7	0.2 (2)	C18—N2—C22—C21	−53.18 (17)
C8—C9—C10—O7	176.71 (15)	C20—C21—C22—N2	56.98 (17)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1···O3	0.87 (3)	2.18 (2)	2.6479 (16)	113.6 (19)
O2—H1···O8^{i}	0.87 (3)	2.17 (3)	2.9113 (18)	142 (2)
O6—H2···O3^{ii}	0.82 (3)	2.12 (3)	2.7290 (18)	132 (2)
O6—H2···O7	0.82 (3)	2.11 (2)	2.6093 (16)	119 (2)
N1—H3···O1	0.90 (2)	1.97 (2)	2.7921 (19)	152 (2)
N1—H3···O3^{iii}	0.90 (2)	2.48 (2)	2.9412 (19)	112.7 (17)
N1—H4···O8^{iv}	0.86 (2)	2.03 (2)	2.8269 (17)	156 (2)

N2—H14···O5	0.90 (2)	2.15 (2)	2.9223 (19)	143 (2)
N2—H14···O8	0.90 (2)	2.50 (2)	3.200 (2)	135.7 (18)
N2—H15···O2^{i}	0.88 (2)	2.55 (2)	3.0332 (18)	115.1 (17)
N2—H15···O4^{v}	0.88 (2)	1.98 (2)	2.8158 (18)	159 (2)
C14—H7···O1	0.98	2.49	3.211 (2)	130
C14—H8···O6^{vi}	0.98	2.48	3.2561 (19)	136
C16—H10···O5^{vii}	0.98	2.59	3.531 (2)	161
C18—H17···O7^{viii}	0.98	2.39	3.193 (2)	138

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

Fig. 1

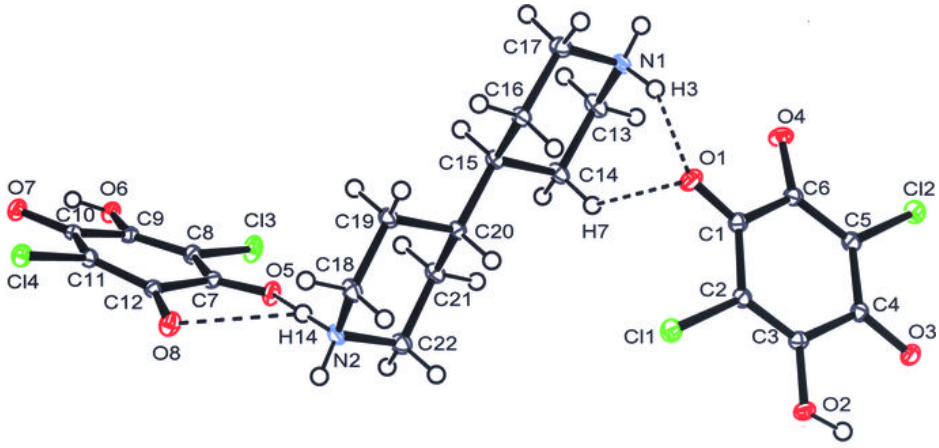

C17
N1
H3
C16
C13
O4
C15
C14
O1
C6
Cl2
O7
O6
C10
C9
C8
Cl3
C19
C20
H7
C1
C5
Cl4
C11
C12
C7
O5
C18
C21
C2
C4
Cl1
C3
O3
O8
H14
N2
C22
O2

Fig. 2