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3,3′-(5-Chloro-2-hydroxy-*m*-phenylenedimethylene)diimidazolium oxalate

Xiao-Yu Su,[a] Wen-Hai Wang,[a] Jing-Bo Lan,[a] Zhi-Hua Mao[b] and Ru-Gang Xie[a]*

[a]College of Chemistry, Sichuan University, Chengdu 610064, People's Republic of China, and [b]Analytical and Testing Centre, Sichuan University, Chengdu 610064, People's Republic of China
Correspondence e-mail: orgxie@scu.edu.cn

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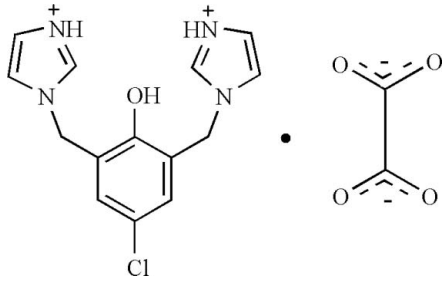
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.069; wR factor = 0.201; data-to-parameter ratio = 12.5.

The title compound, $C_{14}H_{15}ClN_4O^{2+}\cdot C_2O_4^{2-}$, was obtained as a 1:1 binary ionic compound. Two cations and two anions are connected *via* ionic N—H···O hydrogen bonds to form an annulus with internal dimensions of about 8.5 × 9.5 Å, and these units are connected through O—H···O hydrogen bonds to form a two-dimensional porous layer. Weak C—H···π and C—Cl···π [Cl···centroid = 3.575 (2) and 3.598 (2) Å] interactions, together with strong π–π stacking interactions [centroid-to-centroid distance = 3.429 (3) Å], contribute to the stability of the structure.

Related literature

For related literature, see: Aakeröy *et al.* (2006); Aakeröy & Seddon (1993); Bhogala (2003); Corna *et al.* (2004); Dobrzanska *et al.* (2006); MacGillivray & Atwood (1997); Van Roey *et al.* (1991); Sarkar & Biradha (2006); Wang *et al.* (2006, 2007); Zou *et al.* (2006). For structure interpretation tools, see: Spek (2003).



Experimental

Crystal data

$C_{14}H_{15}ClN_4O^{2+}\cdot C_2O_4^{2-}$
M_r = 378.77
Monoclinic, $P2_1/c$
a = 7.3158 (18) Å
b = 19.824 (4) Å
c = 11.901 (2) Å
β = 95.18 (2)°
V = 1719.0 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 294 (2) K
0.30 × 0.26 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
4162 measured reflections
3122 independent reflections
1789 reflections with $I > 2\sigma(I)$
R_{int} = 0.007
3 standard reflections every 300 reflections
intensity decay: 3.7%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.201
S = 0.96
3122 reflections
249 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

Cg2 is the centroid of the imidazole ring containing N3 and N4.

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···O4^{i}	0.79 (5)	1.81 (5)	2.597 (4)	174 (5)
N2—H2N···O2^{ii}	0.80 (5)	1.86 (6)	2.647 (4)	166 (6)
N2—H2N···O5^{ii}	0.80 (5)	2.53 (6)	3.012 (5)	119 (5)
N4—H4N···O3^{iii}	0.87 (5)	2.33 (5)	2.861 (5)	120 (4)
N4—H4N···O4^{iii}	0.87 (5)	1.88 (5)	2.710 (4)	160 (4)
C8—H8···*Cg2*	0.93	2.83	3.664 (4)	149

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

Data collection: *DIFRAC* (Gabe & White, 1993); cell refinement: *DIFRAC*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *Mercury* (Version 1.2; Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2146).

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supplementary materials

Acta Cryst. (2007). E**63**, o4513-o4514 [**doi:10.1107/S160053680704809X**]

3,3'-(5-Chloro-2-hydroxy-*m*-phenylenedimethylene)diimidazolium oxalate

X.-Y. Su, W.-H. Wang, J.-B. Lan, Z.-H. Mao and R.-G. Xie

Comment

The construction of organic crystals based on the hydrogen-bond interactions of organic ligands and various hydrogen bonding donors has been rapidly developed because of their fascinating structural diversity and potential applications for functional materials (MacGillivray & Atwood, 1997; Corna *et al.*, 2004). In particular, intermolecular hydrogen bonds have been proven to be ideal and efficient tools in the design and construction of organic crystals because of their strength and directional properties (Aakeröy & Seddon, 1993). Dicarboxylic acids which can form strong and directional hydrogen bonds are frequently chosen as building blocks for crystal engineering, and a variety of cocrystals have been synthesized by assembling dicarboxylic acids and organic ligands bearing N-donors, such as pyridine-based ligands (Bhogala, 2003; Sarkar & Biradha, 2006). Imidazoles, which are also to N-donor compounds, have attracted attentions in the construction of some metal–organic frameworks in recent years (Dobrzanska *et al.*, 2006; Zou *et al.*, 2006; Wang *et al.*, 2006), but only a few reports describing organic crystals composed of dicarboxylic acids and diimidazole compounds have appeared in the literature to date (Aakeröy *et al.*, 2006; Van Roey *et al.*, 1991; Wang *et al.*, 2007). In further development of such interesting hydrogen-bonding supramolecular systems and as a continuation of our research in this area, we report here the crystal structure of an imidazolium oxalate salt, *viz*. the title compound, (I).

As depicted in Figure 1, the carboxyl protons H2N and H4N of oxalic acid have completely transferred to N2 and N4 of BICP, resulting in the formation of an imidazolium oxalate salt. Two cations and two anions form an annulus with internal dimensions of about 8.5 Å × 9.5 Å *via* N—H···O hydrogen bonds as illustrated in Figure 2 and Table 1. These units are further connected by O—H···O hydrogen bonds involving the hydroxy groups of the cation and the carbonyl group of oxalate to result in a 2-D porous layer as shown in Figure 3.

A network of intermolecular π–π and C—H···π interactions, as well as C–Cl···π interactions (Spek, 2003), provide strong packing forces in the structure of (I). A comparatively strong π–π interaction between an imidazole ring and another symmetry-related imidazole ring at (2 − *x*, −*y*, −1 − *z*), with their centroids separated by 3.429 (3) Å, plays an important part in the connection of adjacent porous layers. Weak C—H···π interactions also contribute to the interaction of neighboring layers. In addition, chlorine is involved in two separate C—Cl···π interactions, with C1···*Cg*1 = 4.623 (5) Å, Cl1···*Cg*1 = 3.575 (2) Å and C1—Cl1···*Cg*1 = 116°, and C1···*Cg*3 = 4.103 (4) Å, Cl1···*Cg*3 = 3.598 (2) Å and C1—Cl1···*Cg*3 = 94°, where *Cg*1 is the centroid of the imidazole ring of the molecule at (2 − *x*, −*y*, −*z*) and *Cg*3 is the centroid of the phenyl ring of the molecule at (1 − *x*, −*y*, −*z*).

Experimental

2,6-Bis[(imidazol-1-yl)methyl]-4-chlorophenol (0.05 mmol, 14.4 mg) in methanol (8 ml) and oxalic acid (0.05 mmol, 4.5 mg) in methanol (2 ml) and water (0.5 ml) were mixed and left to stand at room temperature. Colorless block crystals were obtained by slow evaporation of the solution after 8 days. Yield, 60%; ^{1}H NMR (400 MHz, DMSO-d6): δ 5.25 (s, 4H), 7.08 (s, 2H), 7.10 (s, 2H), 7.30 (s, 2H), 8.09 (s, 2H).

Refinement

H1O, H2N and H4N were observed in difference electron density maps and refined with isotropic displacement parameters. All other hydrogen atoms were positioned geometrically and refined in the riding model approximation with C—H = 0.93 or 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C).

Figures

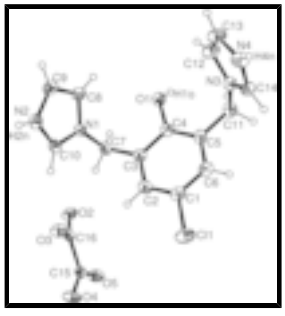

Fig. 1. The asymmetric unit of (I), showing 30% probability displacement ellipsoids and the atomic numbering.

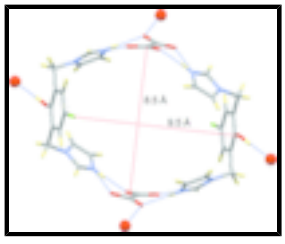

Fig. 2. The annulus structure of (I) formed *via* ionic hydrogen bonds.

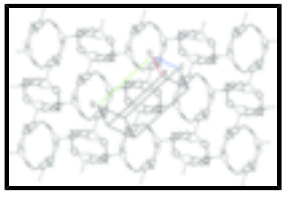

Fig. 3. A 2-D porous layer containing many annular units. Dashed lines indicate hydrogen bonds. H atoms have been omitted for clarity.

3,3'-(5-Chloro-2-hydroxy-*m*-phenylenedimethylene)diimidazolium oxalate

Crystal data

$C_{14}H_{15}ClN_4O^{2+}\cdot C_2O_4^{2-}$	F_{000} = 784
M_r = 378.77	D_x = 1.464 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 28 reflections
a = 7.3158 (18) Å	θ = 4.6–9.5º
b = 19.824 (4) Å	μ = 0.26 mm^{-1}
c = 11.901 (2) Å	T = 294 (2) K
β = 95.18 (2)º	Block, colourless
V = 1719.0 (6) Å^3	0.30 × 0.26 × 0.20 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.007
Radiation source: fine-focus sealed tube	θ_{max} = 25.5º
Monochromator: graphite	θ_{min} = 2.0º
T = 294(2) K	h = −8→8
ω/2θ scans	k = 0→23
Absorption correction: none	l = −7→14
4162 measured reflections	3 standard reflections
3122 independent reflections	every 300 reflections
1789 reflections with $I > 2\sigma(I)$	intensity decay: 3.7%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.070	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.201	Calculated $w = 1/[\sigma^2(F_o^2) + (0.1334P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$?
S = 0.96	$(\Delta/\sigma)_{max} < 0.001$
3122 reflections	$\Delta\rho_{max}$ = 0.53 e Å^{-3}
249 parameters	$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Primary atom site location: structure-invariant direct methods	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	0.74430 (17)	0.05960 (6)	0.08437 (11)	0.0619 (4)
O1	0.7766 (4)	−0.17156 (15)	−0.2216 (2)	0.0429 (7)
O2	0.4825 (4)	0.08021 (14)	−0.4268 (3)	0.0468 (8)
O3	0.7050 (3)	0.13828 (14)	−0.3313 (3)	0.0482 (8)

O4	0.4533 (4)	0.22842 (13)	−0.2732 (3)	0.0503 (8)
O5	0.2327 (4)	0.15468 (14)	−0.3234 (3)	0.0510 (8)
N1	0.9367 (4)	−0.03705 (14)	−0.3488 (3)	0.0328 (7)
N2	1.1907 (5)	0.00811 (19)	−0.3871 (3)	0.0427 (9)
N3	0.9597 (4)	−0.21898 (15)	0.0491 (3)	0.0364 (8)
N4	1.2236 (5)	−0.22666 (19)	0.1424 (3)	0.0481 (10)
C1	0.7455 (5)	−0.0099 (2)	−0.0058 (4)	0.0406 (9)
C2	0.7426 (5)	0.0001 (2)	−0.1197 (3)	0.0395 (9)
H2	0.7378	0.0438	−0.1484	0.047*
C3	0.7466 (4)	−0.05428 (18)	−0.1930 (3)	0.0329 (9)
C4	0.7551 (4)	−0.12011 (19)	−0.1478 (3)	0.0322 (8)
C5	0.7528 (5)	−0.1295 (2)	−0.0318 (3)	0.0358 (9)
C6	0.7471 (5)	−0.0742 (2)	0.0388 (4)	0.0417 (10)
H6	0.7443	−0.0803	0.1161	0.050*
C7	0.7489 (5)	−0.04358 (19)	−0.3167 (3)	0.0358 (9)
H71	0.6892	−0.0813	−0.3568	0.043*
H72	0.6804	−0.0030	−0.3386	0.043*
C8	1.0552 (6)	−0.0889 (2)	−0.3687 (4)	0.0451 (10)
H8	1.0305	−0.1349	−0.3652	0.054*
C9	1.2137 (6)	−0.0604 (2)	−0.3942 (4)	0.0470 (10)
H9	1.3183	−0.0828	−0.4131	0.056*
C10	1.0253 (5)	0.02073 (19)	−0.3585 (3)	0.0368 (9)
H10	0.9776	0.0634	−0.3469	0.044*
C11	0.7680 (5)	−0.1998 (2)	0.0192 (4)	0.0433 (10)
H11A	0.7019	−0.2012	0.0863	0.052*
H11B	0.7115	−0.2321	−0.0344	0.052*
C12	1.0751 (6)	−0.2505 (2)	−0.0185 (4)	0.0507 (11)
H12	1.0452	−0.2660	−0.0916	0.061*
C13	1.2387 (6)	−0.2548 (2)	0.0404 (4)	0.0562 (12)
H13	1.3442	−0.2737	0.0157	0.067*
C14	1.0532 (6)	−0.2052 (2)	0.1469 (3)	0.0409 (10)
H14	1.0070	−0.1842	0.2082	0.049*
C15	0.3939 (5)	0.17246 (18)	−0.3160 (3)	0.0325 (8)
C16	0.5419 (5)	0.12589 (17)	−0.3614 (3)	0.0318 (8)
H1O	0.711 (7)	−0.203 (3)	−0.226 (4)	0.063 (16)*
H2N	1.270 (8)	0.035 (3)	−0.394 (5)	0.079 (18)*
H4N	1.312 (7)	−0.222 (2)	0.196 (4)	0.055 (13)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0567 (7)	0.0605 (8)	0.0677 (8)	0.0019 (6)	0.0008 (6)	−0.0262 (6)
O1	0.0453 (17)	0.0360 (16)	0.0483 (18)	−0.0147 (14)	0.0097 (13)	−0.0047 (13)
O2	0.0307 (15)	0.0506 (17)	0.0604 (19)	−0.0080 (13)	0.0115 (13)	−0.0209 (14)
O3	0.0237 (14)	0.0511 (17)	0.069 (2)	0.0019 (12)	−0.0003 (12)	−0.0134 (15)
O4	0.0319 (15)	0.0374 (16)	0.079 (2)	0.0125 (12)	−0.0116 (14)	−0.0118 (14)
O5	0.0290 (15)	0.0453 (16)	0.079 (2)	0.0083 (13)	0.0075 (14)	−0.0029 (15)
N1	0.0313 (16)	0.0319 (16)	0.0346 (18)	−0.0020 (13)	0.0006 (13)	0.0008 (13)

N2	0.037 (2)	0.045 (2)	0.046 (2)	−0.0123 (17)	0.0044 (15)	−0.0011 (16)
N3	0.0323 (17)	0.0364 (17)	0.0395 (19)	−0.0050 (14)	−0.0019 (14)	−0.0009 (14)
N4	0.0336 (19)	0.051 (2)	0.057 (2)	−0.0017 (16)	−0.0115 (17)	−0.0116 (18)
C1	0.0270 (19)	0.044 (2)	0.050 (3)	−0.0021 (17)	−0.0012 (17)	−0.0095 (19)
C2	0.0286 (19)	0.037 (2)	0.052 (3)	−0.0009 (16)	−0.0033 (17)	0.0011 (18)
C3	0.0141 (16)	0.038 (2)	0.046 (2)	0.0003 (14)	−0.0025 (15)	0.0015 (17)
C4	0.0160 (16)	0.037 (2)	0.043 (2)	−0.0090 (14)	−0.0020 (14)	−0.0018 (16)
C5	0.0228 (18)	0.046 (2)	0.037 (2)	−0.0080 (16)	−0.0034 (15)	0.0052 (17)
C6	0.034 (2)	0.052 (2)	0.039 (2)	−0.0021 (18)	−0.0012 (17)	−0.0040 (18)
C7	0.0267 (19)	0.036 (2)	0.043 (2)	−0.0021 (15)	−0.0029 (16)	0.0076 (17)
C8	0.042 (2)	0.036 (2)	0.058 (3)	0.0021 (18)	0.0097 (19)	−0.0064 (19)
C9	0.038 (2)	0.054 (3)	0.050 (3)	0.0035 (19)	0.0081 (19)	−0.001 (2)
C10	0.040 (2)	0.033 (2)	0.037 (2)	−0.0086 (18)	−0.0006 (17)	0.0017 (16)
C11	0.028 (2)	0.053 (2)	0.048 (2)	−0.0075 (18)	−0.0022 (17)	0.005 (2)
C12	0.043 (2)	0.056 (3)	0.054 (3)	−0.002 (2)	0.003 (2)	−0.017 (2)
C13	0.036 (2)	0.058 (3)	0.074 (3)	0.005 (2)	0.006 (2)	−0.011 (2)
C14	0.043 (2)	0.042 (2)	0.037 (2)	−0.0015 (18)	0.0009 (18)	−0.0073 (18)
C15	0.0278 (19)	0.034 (2)	0.035 (2)	0.0053 (16)	−0.0002 (15)	0.0072 (16)
C16	0.0242 (18)	0.0286 (18)	0.043 (2)	0.0010 (15)	0.0024 (15)	0.0041 (16)

Geometric parameters (Å, °)

Cl1—C1	1.747 (4)	C2—C3	1.389 (5)
O1—C4	1.364 (5)	C2—H2	0.930
O1—H1O	0.79 (5)	C3—C4	1.411 (5)
O2—C16	1.247 (4)	C3—C7	1.489 (5)
O3—C16	1.239 (4)	C4—C5	1.395 (5)
O4—C15	1.280 (4)	C5—C6	1.385 (6)
O5—C15	1.227 (4)	C5—C11	1.520 (5)
N1—C10	1.326 (4)	C6—H6	0.930
N1—C8	1.380 (5)	C7—H71	0.970
N1—C7	1.464 (5)	C7—H72	0.970
N2—C10	1.310 (5)	C8—C9	1.349 (6)
N2—C9	1.372 (6)	C8—H8	0.930
N2—H2N	0.80 (5)	C9—H9	0.930
N3—C14	1.325 (5)	C10—H10	0.930
N3—C12	1.369 (5)	C11—H11A	0.970
N3—C11	1.465 (5)	C11—H11B	0.970
N4—C14	1.323 (5)	C12—C13	1.334 (6)
N4—C13	1.348 (6)	C12—H12	0.930
N4—H4N	0.87 (5)	C13—H13	0.930
C1—C2	1.369 (6)	C14—H14	0.930
C1—C6	1.380 (6)	C15—C16	1.556 (5)
C4—O1—H1O	122 (4)	N1—C7—H72	109.3
C10—N1—C8	108.0 (3)	C3—C7—H72	109.3
C10—N1—C7	125.2 (3)	H71—C7—H72	108.0
C8—N1—C7	126.7 (3)	C9—C8—N1	107.0 (4)
C10—N2—C9	109.0 (4)	C9—C8—H8	126.5
C10—N2—H2N	126 (4)	N1—C8—H8	126.5

C9—N2—H2N	124 (4)	C8—C9—N2	106.8 (4)
C14—N3—C12	108.3 (3)	C8—C9—H9	126.6
C14—N3—C11	124.5 (4)	N2—C9—H9	126.6
C12—N3—C11	127.1 (3)	N2—C10—N1	109.1 (4)
C14—N4—C13	108.9 (4)	N2—C10—H10	125.4
C14—N4—H4N	125 (3)	N1—C10—H10	125.4
C13—N4—H4N	126 (3)	N3—C11—C5	111.6 (3)
C2—C1—C6	120.9 (4)	N3—C11—H11A	109.3
C2—C1—Cl1	119.6 (3)	C5—C11—H11A	109.3
C6—C1—Cl1	119.5 (3)	N3—C11—H11B	109.3
C1—C2—C3	120.6 (4)	C5—C11—H11B	109.3
C1—C2—H2	119.7	H11A—C11—H11B	108.0
C3—C2—H2	119.7	C13—C12—N3	107.0 (4)
C2—C3—C4	118.8 (4)	C13—C12—H12	126.5
C2—C3—C7	120.8 (3)	N3—C12—H12	126.5
C4—C3—C7	120.3 (3)	C12—C13—N4	107.7 (4)
O1—C4—C5	123.3 (3)	C12—C13—H13	126.1
O1—C4—C3	116.7 (3)	N4—C13—H13	126.1
C5—C4—C3	119.8 (3)	N4—C14—N3	108.2 (4)
C6—C5—C4	119.9 (4)	N4—C14—H14	125.9
C6—C5—C11	119.3 (4)	N3—C14—H14	125.9
C4—C5—C11	120.8 (3)	O5—C15—O4	124.5 (3)
C1—C6—C5	119.8 (4)	O5—C15—C16	119.9 (3)
C1—C6—H6	120.1	O4—C15—C16	115.5 (3)
C5—C6—H6	120.1	O3—C16—O2	126.6 (3)
N1—C7—C3	111.4 (3)	O3—C16—C15	117.6 (3)
N1—C7—H71	109.3	O2—C16—C15	115.7 (3)
C3—C7—H71	109.3		
C6—C1—C2—C3	−1.9 (6)	C7—N1—C8—C9	−179.2 (3)
Cl1—C1—C2—C3	178.9 (3)	N1—C8—C9—N2	1.3 (5)
C1—C2—C3—C4	−0.4 (5)	C10—N2—C9—C8	0.0 (5)
C1—C2—C3—C7	−178.2 (3)	C9—N2—C10—N1	−1.3 (5)
C2—C3—C4—O1	−173.9 (3)	C8—N1—C10—N2	2.1 (4)
C7—C3—C4—O1	3.9 (4)	C7—N1—C10—N2	179.3 (3)
C2—C3—C4—C5	2.3 (5)	C14—N3—C11—C5	87.6 (5)
C7—C3—C4—C5	−180.0 (3)	C12—N3—C11—C5	−89.1 (5)
O1—C4—C5—C6	174.1 (3)	C6—C5—C11—N3	−87.1 (4)
C3—C4—C5—C6	−1.7 (5)	C4—C5—C11—N3	89.2 (4)
O1—C4—C5—C11	−2.1 (5)	C14—N3—C12—C13	−0.5 (5)
C3—C4—C5—C11	−178.0 (3)	C11—N3—C12—C13	176.6 (4)
C2—C1—C6—C5	2.5 (6)	N3—C12—C13—N4	0.3 (5)
Cl1—C1—C6—C5	−178.4 (3)	C14—N4—C13—C12	0.0 (5)
C4—C5—C6—C1	−0.6 (5)	C13—N4—C14—N3	−0.3 (5)
C11—C5—C6—C1	175.7 (3)	C12—N3—C14—N4	0.5 (5)
C10—N1—C7—C3	−93.7 (4)	C11—N3—C14—N4	−176.7 (3)
C8—N1—C7—C3	83.0 (5)	O5—C15—C16—O3	−164.8 (3)
C2—C3—C7—N1	88.7 (4)	O4—C15—C16—O3	15.6 (5)
C4—C3—C7—N1	−89.1 (4)	O5—C15—C16—O2	16.5 (5)
C10—N1—C8—C9	−2.1 (5)	O4—C15—C16—O2	−163.0 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O···O4^{i}	0.79 (5)	1.81 (5)	2.597 (4)	174 (5)
N2—H2N···O2^{ii}	0.80 (5)	1.86 (6)	2.647 (4)	166 (6)
N2—H2N···O5^{ii}	0.80 (5)	2.53 (6)	3.012 (5)	119 (5)
N4—H4N···O3^{iii}	0.87 (5)	2.33 (5)	2.861 (5)	120 (4)
N4—H4N···O4^{iii}	0.87 (5)	1.88 (5)	2.710 (4)	160 (4)
C8—H8···Cg2	0.93	2.83	3.664 (4)	149

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

Fig. 1

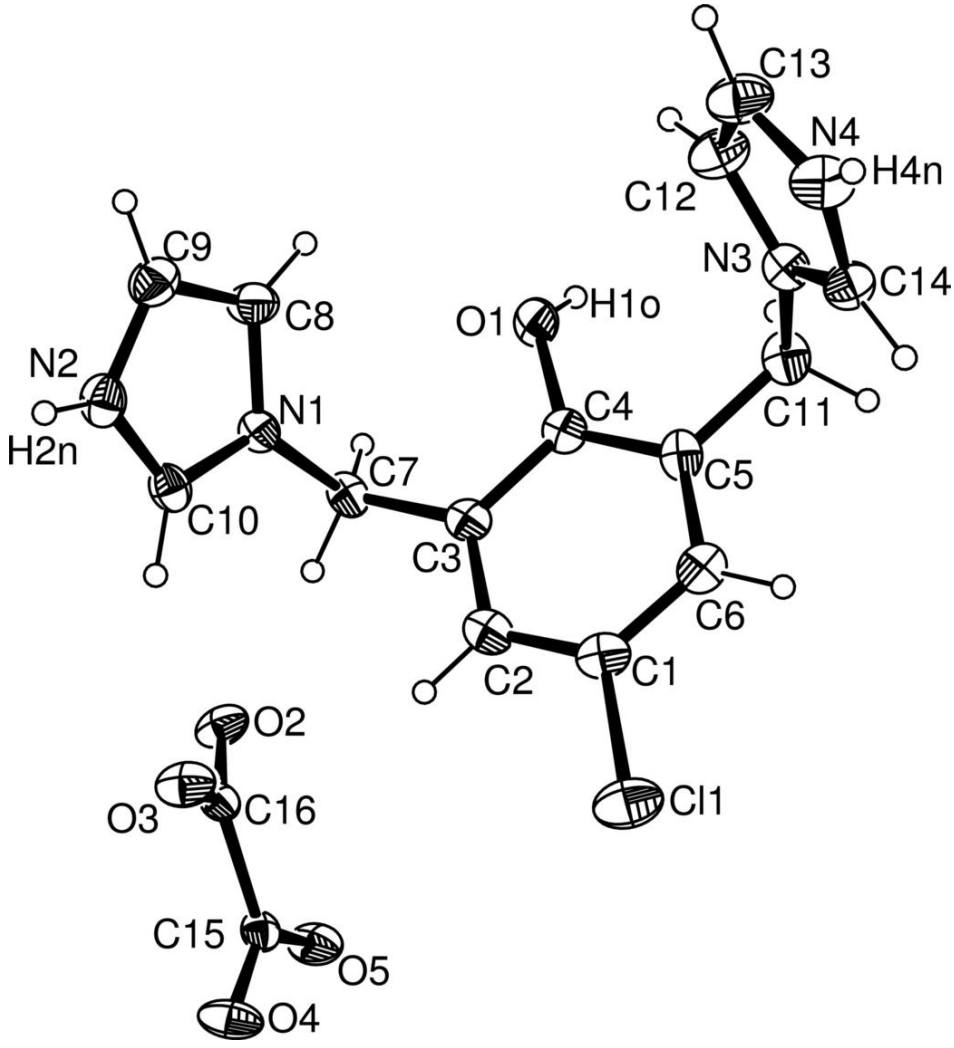
C13
N4
H4n
C12
N3
C14
C9
C8
O1
H1o
N2
N1
C4
C11
H2n
C7
C5
C10
C3
C6
C2
C1
O2
O3
C16
Cl1
C15
O5
O4

Fig. 2

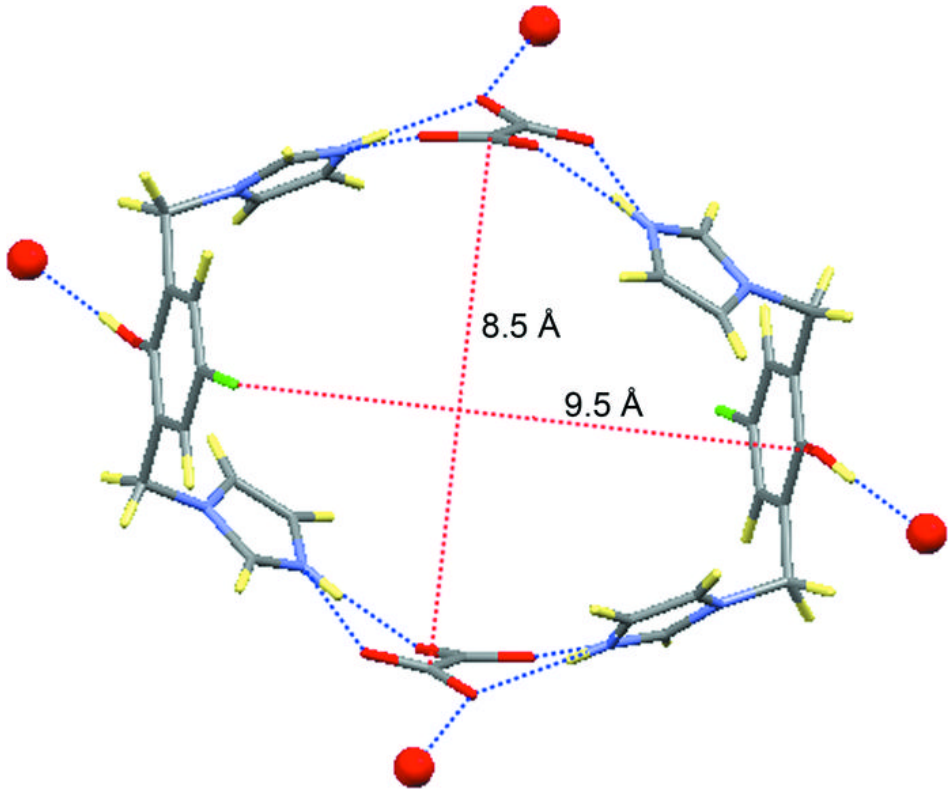
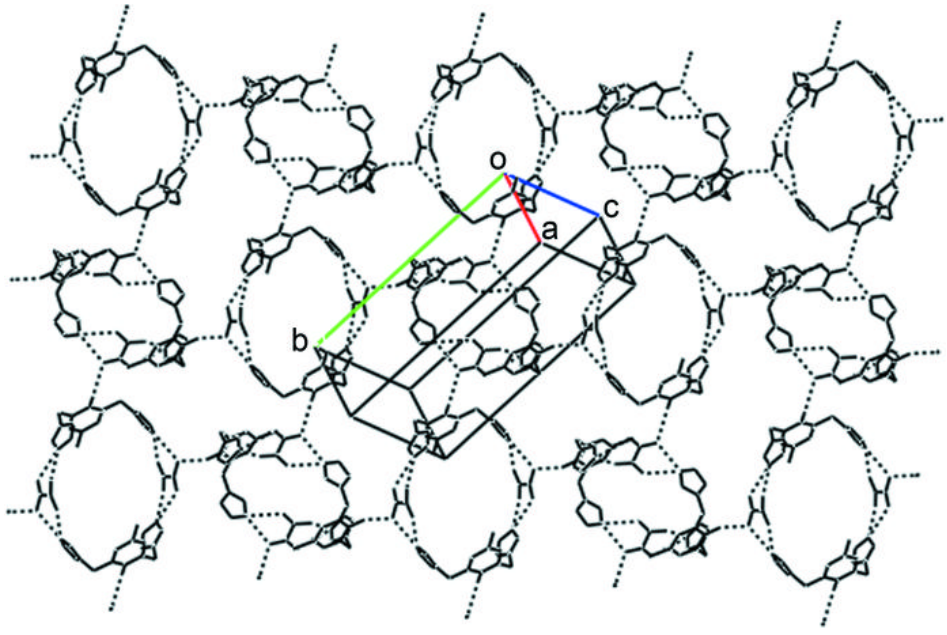
Fig. 3