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2-Hydroxyethylammonium [2-(2,6-dichloroanilino)-phenyl]acetate monohydrate

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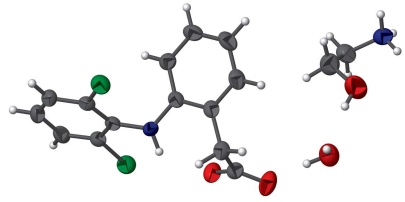
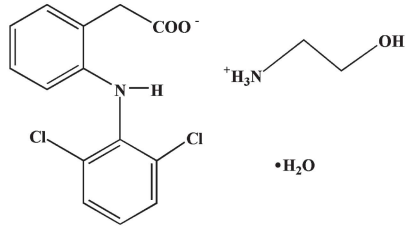
In the solid-state structure of the title compound derived from diclofenac, $C_2H_8NO^+\cdot C_{14}H_{10}Cl_2NO_2^-\cdot H_2O$, the asymmetric unit contains one cation, one anion and a water molecule, all in general positions. A complex network of hydrogen bonds is present in the crystal structure.

3D view



Chemical scheme



Structure description

The pharmaceutical diclofenac (D) is widely used as a non-steroidal anti-inflammatory drug, to treat pain and inflammatory diseases (Skoutakis *et al.*, 1988; Moser *et al.*, 1990). The Cambridge Structural Database (CSD version 5.42, last update February 2021; Groom *et al.*, 2016) includes crystallographic data for 50 entries with the term 'diclofenac'. Among them, there are 21 entries where diclofenac is present in the form of a salt, and in three entries, diclofenac forms salts with aliphatic amines: with (*R*) and (*S*)-phenylethylammonium (Lemmerer *et al.*, 2010), with diethyl ammonium (Castellari *et al.*, 2001) and with tris(2-ammonioethyl)amine (Lynch *et al.*, 2003). In this article, we present another complex in the form of a diclofenac salt with an amino-containing compound, namely monoethanolamine. Ethanolamine is always present in significant quantities in the human and animal body with a complete protein diet. Its formation occurs during the decarboxylation of serine, and in one of the metabolic variants, it turns into glycine (the simplest aliphatic amino acid; Wishart *et al.*, 2007). In addition, monoethanolamine is used in some cosmetic products (Knaak *et al.*, 1997). Therefore, the interaction of these compounds seems to be interesting for investigation.

The crystal structure of the title compound has one monoethanolamine (MEA) cation, one 2-(2,6-dichloroanilino)phenyl acetic acid or diclofenac (D) anion, and one water molecule in the asymmetric unit, and crystallizes in space group $P2_1/c$ (Fig. 1). The diclofenac anion is stabilized by one intramolecular hydrogen bond between the amino group and atom O1 of the carboxylic group: N1—H1···O1 [2.884 (3) Å, 128.9°; see

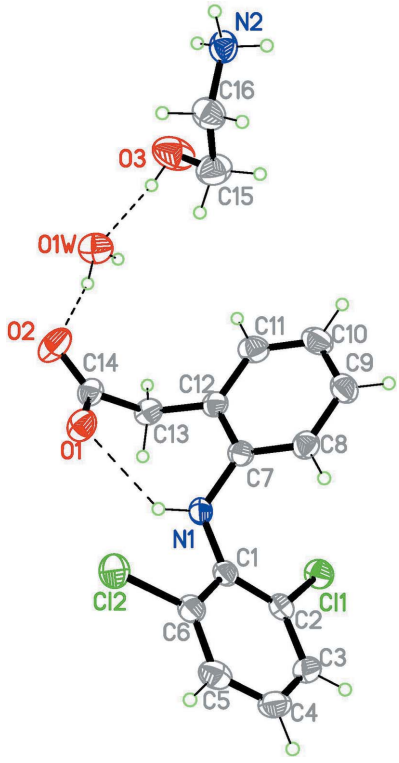


Figure 1
Perspective view of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at 40% probability level. The dashed lines represent hydrogen bonds within the asymmetric unit.

Table 1], which forms a seven-membered ring with graph-set notation $S(7)$ (Etter, 1990). The dihedral angle between the two benzene rings in D is 60.2 (2)°.

The ionic form of the title compound serves as a building block for the supramolecular architecture. In the crystal, the building blocks form screw-like chains along the *b*-axis direction, due to the crystallographic twofold screw axis, *via* N2—H2*B*···O1*W*^{ii} hydrogen bond [2.947 (4) Å, symmetry code: (ii) $-x, y-\frac{1}{2}, -z+\frac{3}{2}$; Fig. 2 and Table 1]. The chains are further consolidated into two-dimensional layers through N—H···O and O—H···O hydrogen bonds. These layers propagate parallel to the (100) plane, where the chains are related by the glide plane *c* [O1*W*···O1^{iv}, symmetry code: (iv) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; 2.809 (3) Å] and the inversion centre [N2... O2^{i}, symmetry code: (i) $-x, -y+1, -z+1$, 2.811 (4) Å, Fig. 2]. The layers are linked by Y—*X*···*Cg* π–ring interactions, for C3—H3 and C7—Cl1 bonds, for which the *X*···*Cg* separations and γ angles range from 3.533 to 3.958 Å and from 25.03 to 28.79°.

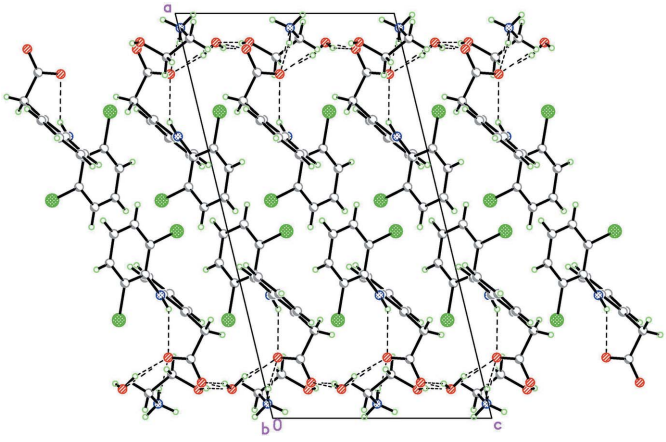


Figure 2
Packing diagram of the title compound, viewed down *b* axis. The hydrogen bonds are shown as dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.86	2.27	2.884 (3)	129
N2—H2*A*···O2^{i}	0.89	1.96	2.811 (4)	160
N2—H2*B*···O1*W*^{ii}	0.89	2.15	2.947 (4)	148
N2—H2*C*···O1^{iii}	0.89	1.92	2.802 (3)	169
O3—H3*A*···O1*W*	0.82	1.96	2.770 (4)	168
O1*W*—H1*WB*···O2	0.85	1.87	2.690 (3)	161
O1*W*—H1*WA*···O1^{iv}	0.85	2.00	2.809 (3)	158

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

In order to visualize the intermolecular interactions in the crystal of the title compound, a Hirshfeld surface analysis was carried out using *Crystal Explorer 17.5* (Turner *et al.*, 2017). The Hirshfeld surface mapped over d_{norm} shows the expected bright-red spots near atoms O1 and O2, involved in the O—H···O and N—H···O hydrogen-bonding interactions (Fig. 3). Fingerprint plots (Fig. 4) reveal that H···H, H···C/C···H, H···Cl/Cl···H and H···O/O···H interactions make the greatest contributions to the surface contacts (Table 1), while H···N/N···H, C···C and O···O contacts are much less significant.

Synthesis and crystallization

To a solution of 0.1 g (0.52 mmol) of D in 4 ml of ethanol, 32 µ*L* of monoethanolamine was added. The mixture was kept

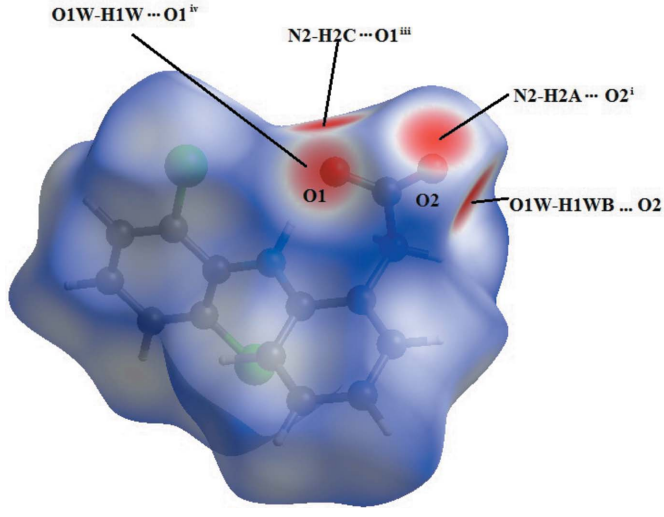


Figure 3
The Hirshfeld surface analysis indicates that the most important contributions to the crystal packing are from H···H (31.0%), H···C/C···H (26.3%) and H···Cl/Cl···H (25%) interactions.

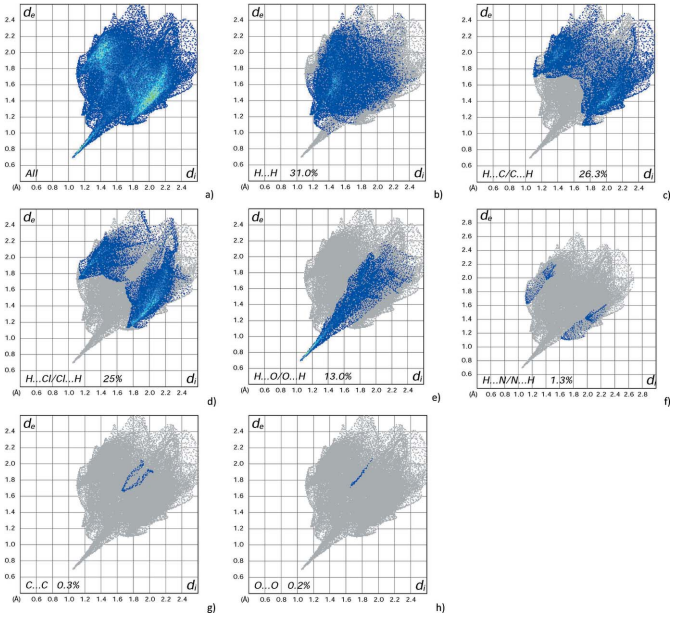


Figure 4
Full two-dimensional fingerprint plots for the title compound, showing all interactions (*a*), and delineated into (*b*) H···H, (*c*) H···C/C···H, (*d*) H···Cl/Cl···H, (*e*) H···O/O···H, (*f*) H···N/N···H, (*g*) C···C and (*h*) O···O interactions. The d_i and d_e values are the closest internal and external distances (in Å) from a given point on the Hirshfeld surface depicted in Fig. 3.

in an ultrasonic bath (30 kHz) at 298 K for 5 min. The solution was then placed in a loosely closed bottle and kept at 298 K for 10 days. The precipitated prismatic crystals were selected for the single-crystal X-ray diffraction analysis.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_2H_8NO^+·C_{14}H_{10}Cl_2NO_2^-·H_2O$
M_r	375.24
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	19.1257 (10), 9.3864 (5), 10.0502 (6)
β (°)	103.546 (6)
V (Å^3)	1754.05 (17)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	3.53
Crystal size (mm)	0.31 × 0.28 × 0.1
Data collection	
Diffractometer	Agilent Technologies Xcalibur, Ruby
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.356, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12416, 3621, 2431
R_{int}	0.078
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.631
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.051, 0.134, 1.01
No. of reflections	3621
No. of parameters	222
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.35

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *XP* (Siemens, 1994), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

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full crystallographic data

IUCrData (2022). **7**, x220441 [https://doi.org/10.1107/S2414314622004412]

2-Hydroxyethylammonium [2-(2,6-dichloroanilino)phenyl]acetate monohydrate

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Crystal data

$C_2H_8NO^+·C_{14}H_{10}Cl_2NO_2^-·H_2O$
M_r = 375.24
Monoclinic, $P2_1/c$
a = 19.1257 (10) Å
b = 9.3864 (5) Å
c = 10.0502 (6) Å
β = 103.546 (6)°
V = 1754.05 (17) Å^3
Z = 4
$F(000)$ = 784
D_x = 1.421 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 2166 reflections
θ = 4.7–73.9°
μ = 3.53 mm^{-1}
T = 293 K
Prism, colourless
0.31 × 0.28 × 0.1 mm

Data collection

Agilent Technologies Xcalibur, Ruby diffractometer
Radiation source: fine-focus sealed tube
/w scans
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2014)
T_{min} = 0.356, T_{max} = 1.000
12416 measured reflections
3621 independent reflections
2431 reflections with $I > 2\sigma(I)$
R_{int} = 0.078
θ_{max} = 76.5°, θ_{min} = 4.8°
h = −23→24
k = −11→6
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.134
S = 1.01
3621 reflections
222 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Special details

Refinement. All hydrogen atoms were placed in idealized positions and refined as riding to their carrier atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.46202 (4)	0.95109 (8)	0.76764 (8)	0.0441 (2)
Cl2	0.24250 (4)	1.20690 (9)	0.40514 (9)	0.0500 (2)

O1	0.15000 (11)	0.9926 (2)	0.5897 (2)	0.0472 (6)
O1W	0.07192 (13)	0.6316 (3)	0.8457 (2)	0.0558 (6)
H1WA	0.103624	0.612215	0.917982	0.084*
H1WB	0.085392	0.710654	0.818732	0.084*
N1	0.30390 (13)	0.9959 (2)	0.6226 (3)	0.0379 (6)
H1	0.269351	1.041181	0.645204	0.045*
O2	0.08825 (11)	0.8748 (3)	0.7146 (2)	0.0592 (7)
N2	0.03573 (14)	0.1808 (3)	0.4921 (3)	0.0446 (6)
H2A	0.003371	0.148791	0.419647	0.054*
H2B	0.015651	0.187499	0.563284	0.054*
H2C	0.072670	0.120586	0.512028	0.054*
O3	0.07091 (17)	0.3857 (3)	0.6928 (3)	0.0762 (9)
H3A	0.073883	0.464400	0.729146	0.114*
C2	0.42880 (15)	1.0634 (3)	0.6305 (3)	0.0327 (6)
C14	0.14604 (15)	0.9162 (3)	0.6906 (3)	0.0371 (7)
C1	0.35476 (15)	1.0754 (3)	0.5751 (3)	0.0322 (6)
C12	0.26283 (14)	0.7823 (3)	0.7158 (3)	0.0342 (6)
C7	0.30532 (15)	0.8463 (3)	0.6360 (3)	0.0342 (6)
C8	0.34720 (15)	0.7613 (3)	0.5707 (3)	0.0382 (7)
H8	0.374195	0.803323	0.515422	0.046*
C6	0.33381 (16)	1.1786 (3)	0.4739 (3)	0.0359 (6)
C13	0.21653 (15)	0.8708 (3)	0.7865 (3)	0.0383 (7)
H13A	0.243062	0.955166	0.824815	0.046*
H13B	0.206067	0.816317	0.861507	0.046*
C9	0.34871 (16)	0.6146 (3)	0.5877 (3)	0.0447 (8)
H9	0.377768	0.559121	0.546037	0.054*
C5	0.38298 (18)	1.2604 (3)	0.4257 (3)	0.0430 (7)
H5	0.366991	1.328721	0.358405	0.052*
C4	0.45510 (18)	1.2404 (3)	0.4774 (3)	0.0459 (8)
H4	0.488229	1.292653	0.442936	0.055*
C3	0.47873 (16)	1.1423 (3)	0.5811 (3)	0.0408 (7)
H3	0.527698	1.129421	0.617432	0.049*
C11	0.26455 (17)	0.6344 (3)	0.7292 (3)	0.0436 (8)
H11	0.236429	0.590723	0.781473	0.052*
C10	0.30743 (18)	0.5508 (3)	0.6661 (3)	0.0488 (8)
H10	0.308209	0.452307	0.676839	0.059*
C16	0.06160 (19)	0.3234 (3)	0.4609 (4)	0.0515 (8)
H16A	0.020800	0.385304	0.426179	0.062*
H16B	0.088673	0.314490	0.390895	0.062*
C15	0.1082 (2)	0.3869 (4)	0.5871 (4)	0.0593 (10)
H15A	0.152356	0.332453	0.614785	0.071*
H15B	0.120699	0.483999	0.568925	0.071*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0413 (4)	0.0418 (4)	0.0442 (4)	0.0030 (3)	0.0002 (3)	0.0044 (3)
Cl2	0.0424 (4)	0.0481 (5)	0.0552 (5)	0.0075 (3)	0.0025 (4)	0.0094 (4)

O1	0.0363 (12)	0.0534 (14)	0.0497 (13)	0.0053 (10)	0.0057 (10)	0.0195 (11)
O1W	0.0600 (15)	0.0558 (16)	0.0496 (15)	−0.0060 (12)	0.0087 (12)	0.0047 (11)
N1	0.0349 (13)	0.0309 (13)	0.0525 (15)	0.0053 (10)	0.0196 (12)	0.0055 (11)
O2	0.0325 (12)	0.0799 (18)	0.0625 (16)	−0.0032 (11)	0.0054 (11)	0.0235 (13)
N2	0.0409 (14)	0.0497 (16)	0.0404 (15)	0.0072 (12)	0.0037 (12)	−0.0030 (12)
O3	0.106 (2)	0.0641 (18)	0.0689 (18)	−0.0190 (17)	0.0403 (17)	−0.0214 (14)
C2	0.0376 (15)	0.0276 (14)	0.0331 (15)	0.0030 (12)	0.0087 (12)	−0.0018 (12)
C14	0.0317 (15)	0.0414 (17)	0.0364 (17)	0.0012 (13)	0.0043 (13)	−0.0003 (13)
C1	0.0351 (15)	0.0267 (14)	0.0347 (15)	0.0014 (11)	0.0080 (12)	−0.0029 (11)
C12	0.0283 (14)	0.0355 (16)	0.0351 (16)	0.0000 (12)	−0.0003 (12)	0.0050 (12)
C7	0.0300 (14)	0.0310 (15)	0.0377 (16)	0.0010 (11)	0.0002 (12)	0.0027 (12)
C8	0.0328 (15)	0.0415 (17)	0.0389 (17)	0.0001 (13)	0.0056 (13)	−0.0017 (13)
C6	0.0373 (15)	0.0322 (15)	0.0374 (16)	0.0013 (12)	0.0071 (13)	−0.0013 (12)
C13	0.0352 (15)	0.0428 (17)	0.0351 (16)	−0.0017 (13)	0.0044 (13)	0.0068 (13)
C9	0.0415 (17)	0.0377 (17)	0.0498 (19)	0.0045 (14)	0.0004 (15)	−0.0099 (15)
C5	0.057 (2)	0.0333 (16)	0.0410 (18)	−0.0019 (14)	0.0157 (16)	0.0027 (13)
C4	0.0494 (19)	0.0401 (18)	0.053 (2)	−0.0111 (15)	0.0226 (17)	−0.0036 (15)
C3	0.0346 (16)	0.0380 (17)	0.0505 (19)	−0.0056 (13)	0.0115 (14)	−0.0067 (14)
C11	0.0441 (18)	0.0387 (17)	0.0447 (19)	−0.0035 (14)	0.0039 (15)	0.0110 (14)
C10	0.052 (2)	0.0313 (16)	0.058 (2)	−0.0001 (14)	0.0025 (17)	0.0041 (15)
C16	0.059 (2)	0.0442 (19)	0.049 (2)	0.0028 (16)	0.0073 (17)	0.0046 (15)
C15	0.055 (2)	0.064 (2)	0.058 (2)	−0.0107 (18)	0.0104 (18)	−0.0099 (19)

Geometric parameters (Å, º)

Cl1—C2	1.734 (3)	C7—C8	1.398 (4)
Cl2—C6	1.741 (3)	C8—C9	1.387 (4)
O1—C14	1.259 (3)	C8—H8	0.9300
O1W—H1WA	0.8501	C6—C5	1.387 (4)
O1W—H1WB	0.8504	C13—H13A	0.9700
N1—C1	1.396 (3)	C13—H13B	0.9700
N1—C7	1.410 (3)	C9—C10	1.377 (4)
N1—H1	0.8600	C9—H9	0.9300
O2—C14	1.248 (3)	C5—C4	1.368 (5)
N2—C16	1.486 (4)	C5—H5	0.9300
N2—H2A	0.8900	C4—C3	1.384 (4)
N2—H2B	0.8900	C4—H4	0.9300
N2—H2C	0.8900	C3—H3	0.9300
O3—C15	1.412 (4)	C11—C10	1.391 (5)
O3—H3A	0.8200	C11—H11	0.9300
C2—C3	1.389 (4)	C10—H10	0.9300
C2—C1	1.400 (4)	C16—C15	1.494 (5)
C14—C13	1.523 (4)	C16—H16A	0.9700
C1—C6	1.394 (4)	C16—H16B	0.9700
C12—C11	1.395 (4)	C15—H15A	0.9700
C12—C7	1.403 (4)	C15—H15B	0.9700
C12—C13	1.508 (4)		

H1WA—O1W—H1WB	104.5	C14—C13—H13A	109.0
C1—N1—C7	124.4 (2)	C12—C13—H13B	109.0
C1—N1—H1	117.8	C14—C13—H13B	109.0
C7—N1—H1	117.8	H13A—C13—H13B	107.8
C16—N2—H2A	109.5	C10—C9—C8	120.3 (3)
C16—N2—H2B	109.5	C10—C9—H9	119.8
H2A—N2—H2B	109.5	C8—C9—H9	119.8
C16—N2—H2C	109.5	C4—C5—C6	119.8 (3)
H2A—N2—H2C	109.5	C4—C5—H5	120.1
H2B—N2—H2C	109.5	C6—C5—H5	120.1
C15—O3—H3A	109.5	C5—C4—C3	120.0 (3)
C3—C2—C1	122.1 (3)	C5—C4—H4	120.0
C3—C2—Cl1	117.0 (2)	C3—C4—H4	120.0
C1—C2—Cl1	120.9 (2)	C4—C3—C2	119.5 (3)
O2—C14—O1	123.9 (3)	C4—C3—H3	120.2
O2—C14—C13	118.9 (3)	C2—C3—H3	120.2
O1—C14—C13	117.2 (3)	C10—C11—C12	121.4 (3)
C6—C1—N1	121.1 (3)	C10—C11—H11	119.3
C6—C1—C2	115.9 (3)	C12—C11—H11	119.3
N1—C1—C2	122.8 (3)	C9—C10—C11	119.6 (3)
C11—C12—C7	118.5 (3)	C9—C10—H10	120.2
C11—C12—C13	120.4 (3)	C11—C10—H10	120.2
C7—C12—C13	121.1 (3)	N2—C16—C15	110.0 (3)
C8—C7—C12	119.7 (3)	N2—C16—H16A	109.7
C8—C7—N1	121.6 (3)	C15—C16—H16A	109.7
C12—C7—N1	118.7 (3)	N2—C16—H16B	109.7
C9—C8—C7	120.4 (3)	C15—C16—H16B	109.7
C9—C8—H8	119.8	H16A—C16—H16B	108.2
C7—C8—H8	119.8	O3—C15—C16	109.2 (3)
C5—C6—C1	122.5 (3)	O3—C15—H15A	109.8
C5—C6—Cl2	118.4 (2)	C16—C15—H15A	109.8
C1—C6—Cl2	119.1 (2)	O3—C15—H15B	109.8
C12—C13—C14	112.7 (2)	C16—C15—H15B	109.8
C12—C13—H13A	109.0	H15A—C15—H15B	108.3

C7—N1—C1—C6	131.3 (3)	C2—C1—C6—Cl2	−176.9 (2)
C7—N1—C1—C2	−52.7 (4)	C11—C12—C13—C14	−100.2 (3)
C3—C2—C1—C6	−4.4 (4)	C7—C12—C13—C14	80.1 (3)
Cl1—C2—C1—C6	174.1 (2)	O2—C14—C13—C12	117.9 (3)
C3—C2—C1—N1	179.4 (3)	O1—C14—C13—C12	−61.2 (4)
Cl1—C2—C1—N1	−2.1 (4)	C7—C8—C9—C10	1.8 (4)
C11—C12—C7—C8	0.9 (4)	C1—C6—C5—C4	0.3 (5)
C13—C12—C7—C8	−179.4 (3)	Cl2—C6—C5—C4	−179.8 (2)
C11—C12—C7—N1	−179.8 (3)	C6—C5—C4—C3	−2.4 (5)
C13—C12—C7—N1	−0.1 (4)	C5—C4—C3—C2	0.9 (5)
C1—N1—C7—C8	−16.6 (4)	C1—C2—C3—C4	2.6 (4)
C1—N1—C7—C12	164.1 (3)	Cl1—C2—C3—C4	−176.0 (2)
C12—C7—C8—C9	−2.0 (4)	C7—C12—C11—C10	0.3 (4)

N1—C7—C8—C9	178.7 (3)	C13—C12—C11—C10	−179.3 (3)
N1—C1—C6—C5	179.3 (3)	C8—C9—C10—C11	−0.5 (5)
C2—C1—C6—C5	3.0 (4)	C12—C11—C10—C9	−0.5 (5)
N1—C1—C6—Cl2	−0.6 (4)	N2—C16—C15—O3	−53.2 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.86	2.27	2.884 (3)	129
N2—H2*A*···O2^{i}	0.89	1.96	2.811 (4)	160
N2—H2*B*···O1*W*^{ii}	0.89	2.15	2.947 (4)	148
N2—H2*C*···O1^{iii}	0.89	1.92	2.802 (3)	169
O3—H3*A*···O1*W*	0.82	1.96	2.770 (4)	168
O1*W*—H1*WB*···O2	0.85	1.87	2.690 (3)	161
O1*W*—H1*WA*···O1^{iv}	0.85	2.00	2.809 (3)	158

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