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(2*R*,3*S*)-2-Ammonio-3-hydroxy-3-(4-nitrophenyl)propanoic acid chloride monohydrate

Vincent Gaumet,[a]* Valérie Weber,[b] Valery P. Zaitsev[c] and Michel Madesclaire[b]

[a]Laboratoire de Chimie Physique, EA 4231, Faculté de Pharmacie, Université d'Auvergne, 63001 Clermont-Ferrand, France, [b]Laboratoire de Chimie Thérapeutique, EA 4231, Faculté de Pharmacie, Université d'Auvergne, 63001 Clermont-Ferrand, France, and [c]Chimie Organique, Faculté de Chimie, 443011 Samara, Russian Federation
Correspondence e-mail: vincent.gaumet@u-clermont1.fr

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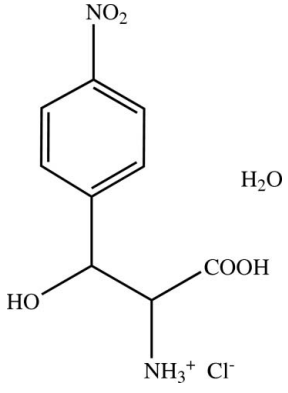
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.043; wR factor = 0.115; data-to-parameter ratio = 25.5.

The title compound, $C_9H_{11}N_2O_5^+\cdot Cl^-\cdot H_2O$, was synthesized from (1*S*,2*S*)-2-amino-1-(4-nitrophenyl)propane-1,3-diol in four steps. As demonstrated by this work, no racemization occurs during this synthetic procedure. The crystal structure displays many intermolecular hydrogen bonds between the acidic cation, chloride anions and the water molecules, forming a three-dimensional network. An intramolecular bond between the ammonium group and a hydroxyl O atom is also present.

Related literature

For related compounds see: Crich *et al.* (2007); Di Giovanni *et al.* (1996); Easton *et al.* (1996); Madesclaire *et al.* (2006, 2007); Steinreiber *et al.* (2007); Zaitsev *et al.* (1998).



Experimental

Crystal data

$C_9H_{11}N_2O_5^+\cdot Cl^-\cdot H_2O$
M_r = 280.66
Monoclinic, $P2_1$
a = 8.1286 (17) Å
b = 5.056 (3) Å
c = 15.848 (3) Å
β = 104.626 (17)°
V = 630.2 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 293 (2) K
0.49 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.872, T_{max} = 0.931
6026 measured reflections
5517 independent reflections
5058 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
3 standard reflections every 63 reflections
intensity decay: 3%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.115
S = 1.09
5517 reflections
216 parameters
1 restraint
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.63 e Å^{-3}
Absolute structure: Flack (1983), 2752 Friedel pairs
Flack parameter: 0.00 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N10—H101···O13^{i}	0.810 (17)	2.420 (16)	2.9259 (13)	121.5 (14)
N10—H101···Cl^{ii}	0.810 (17)	2.489 (16)	3.2298 (11)	152.7 (15)
N10—H102···O14	0.83 (2)	2.265 (19)	2.6759 (14)	110.8 (16)
N10—H102···Cl^{iii}	0.83 (2)	2.62 (2)	3.3409 (14)	145.5 (16)
N10—H103···Cl^{iv}	0.97 (3)	2.28 (3)	3.2435 (14)	176 (3)
O12—H12···O17^{v}	0.78 (5)	1.84 (5)	2.6168 (19)	171 (4)
O14—H14···Cl	0.82 (3)	2.25 (3)	3.0539 (13)	166 (3)
O17—H171···O16^{vi}	0.78 (3)	2.33 (3)	3.069 (2)	159 (4)
O17—H172···Cl^{vii}	0.76 (3)	2.45 (3)	3.2170 (13)	178 (3)

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CS2076).

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supporting information

Acta Cryst. (2008). E**64**, o1003–o1004 [doi:10.1107/S1600536808012750]

(2*R*,3*S*)-2-Ammonio-3-hydroxy-3-(4-nitrophenyl)propanoic acid chloride monohydrate

Vincent Gaumet, Valérie Weber, Valery P. Zaitsev and Michel Madesclaire

S1. Comment

Preparation of new α-amino acids are of constant interest. (1*S*,2*S*) and (1*R*,2*R*)-2-amino-1- (4-nitrophenyl)-1,3-propanediols, byproducts in the manufacture of the antibiotic Chloramphenicol (Zaitsev *et al.*, 1998), can be used as starting materials in the preparation of corresponding α-amino acid isomers. The title compound (V) was synthesized from (1*S*,2*S*)-2-amino-1- (4-nitrophenyl)-1,3-propanediol (I) in four steps according to Figure 1. The intermediate formation of the 2-oxazolidinone derivative (III) allowed to protect both the adjacent amino and hydroxyl groups (Di Giovanni *et al.*, 1996; Crich *et al.*, 2007). The absolute configuration, determined using anomalous dispersion by chlorine, confirms the *R* and *S* configurations respectively for C2 and C3 atoms, confirming that no racemization occurs during this synthetic route (note that the Cahn-Ingold-Prelog designation at the α-carbon of the hydroxyl group is reversed by comparison with that of the starting material due to the change in priority of the substituents). As expected, the phenyl ring is planar, r.m.s. deviation from the best plane is ca. 0.007Å. The nitro group is coplanar with the adjacent phenyl ring (O15—N11—C7—C8 = 0.2 (3)° and O16—N11—C7—C8 = 179.9 (2)°). However, the σ bond C7—N11 (1.4704 (17) Å) shows that there is no appreciable π delocalization in the bond between the sp^2 hybridized N11 and the phenyl ring.

S2. Experimental

Figure 1 summarizes the synthetic route used (Madesclaire *et al.*, 2006 and 2007). Crystals suitable for an X-ray diffraction study were obtained by slow evaporation of a water solution containing compound (V).

S3. Refinement

The structure was solved by direct methods and refined with anisotropic temperature factors for non-H atoms. All H atoms were found from difference Fourier maps. The H atoms were all refined isotropically with no constraints.

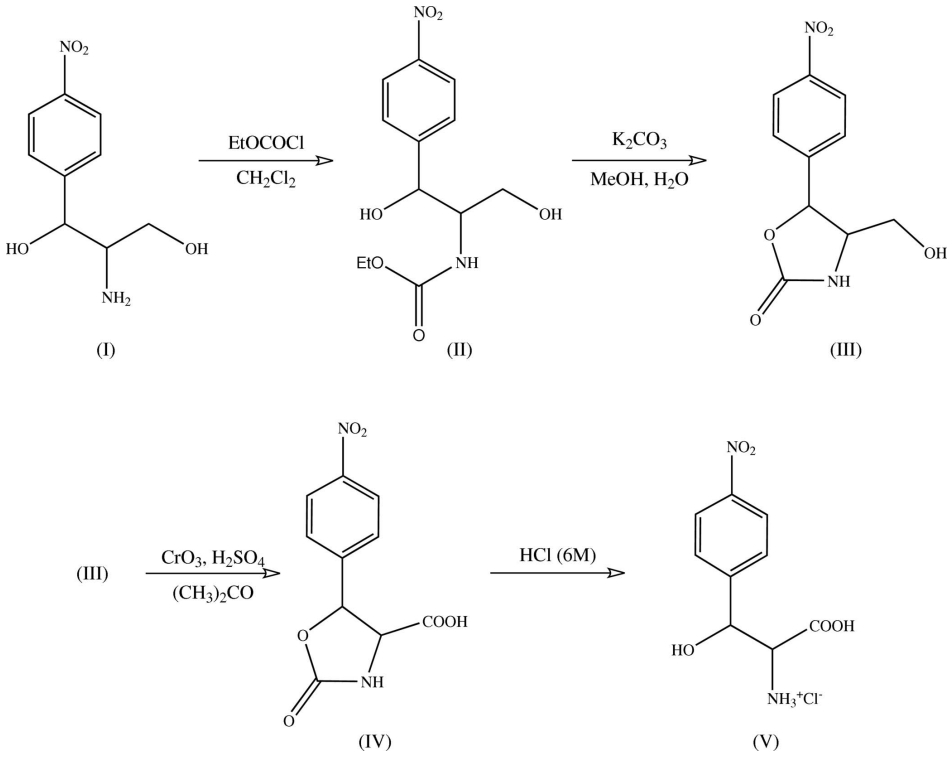

Figure 1

Synthesis of the title compound (V).

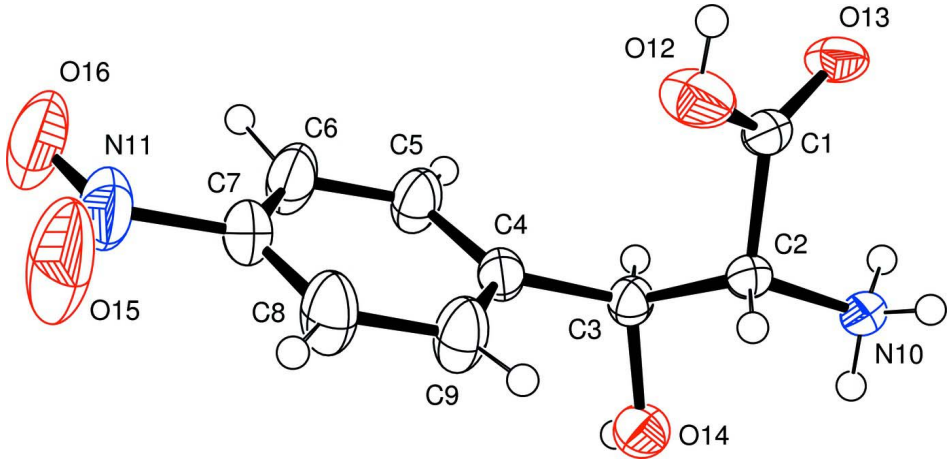

Figure 2

The molecular structure of the title compound (V), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

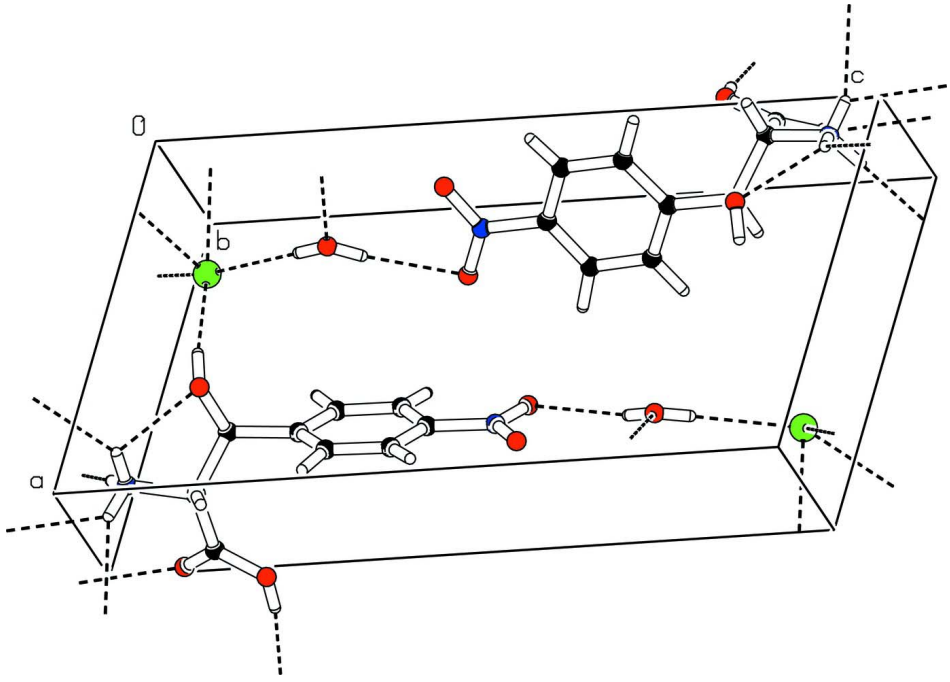

Figure 3

Crystal packing of the title compound. Intra and intermolecular hydrogen bonds are shown as dashed lines. H atoms not involved in the interactions have been omitted.

(2*R*,3*S*)-2-Ammonio-3-hydroxy-3-(4-nitrophenyl)propanoic acid chloride monohydrate

Crystal data

$C_9H_{11}N_2O_5^+\cdot Cl^-\cdot H_2O$
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Hall symbol: P 2yb
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$b = 5.056\ (3)$ Å
$c = 15.848\ (3)$ Å
$\beta = 104.626\ (17)°$
$V = 630.2\ (4)$ Å^3
$Z = 2$
$F(000) = 292$
$D_x = 1.479$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8–18°$
$\mu = 0.33$ mm^{-1}
$T = 293$ K
Elongated prism, colourless
0.49 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North et al., 1968)
$T_{min} = 0.872$, $T_{max} = 0.931$
6026 measured reflections
5517 independent reflections
5058 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 35.0°$, $\theta_{min} = 1.3°$
$h = -13\rightarrow13$
$k = -8\rightarrow8$
$l = -25\rightarrow25$
3 standard reflections every 63 reflections
intensity decay: 3%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.115$
$S = 1.09$
5517 reflections
216 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0825P)^2 + 0.0202P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.60$ e Å^{-3}
$\Delta\rho_{min} = -0.63$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.089 (10)
Absolute structure: Flack (1983), 2752 Friedel pairs
Absolute structure parameter: 0.00 (4)

Special details

Experimental. North A.C.T., Phillips D.C. & Mathews F.S. (1968) Acta. Cryst. A24, 351. Number of psi-scan sets used was 4. Theta correction was applied. Weighted transmission curves were used. No Fourier smoothing was applied.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl	0.71520 (3)	0.93514 (6)	0.922526 (17)	0.03591 (8)
C1	−0.00336 (13)	1.2561 (2)	0.83505 (7)	0.02674 (16)
C2	0.09722 (12)	1.00092 (18)	0.85613 (6)	0.02405 (15)
C3	0.27337 (13)	1.0067 (2)	0.83713 (6)	0.02743 (16)
C4	0.26848 (14)	1.0458 (2)	0.74189 (7)	0.02986 (18)
C5	0.3717 (2)	1.2362 (3)	0.71889 (9)	0.0427 (3)
C6	0.3767 (3)	1.2672 (4)	0.63235 (10)	0.0499 (4)
C7	0.2774 (2)	1.1044 (3)	0.57084 (8)	0.0415 (3)
C8	0.1743 (2)	0.9107 (4)	0.59137 (9)	0.0506 (4)
C9	0.1720 (2)	0.8796 (3)	0.67820 (9)	0.0459 (3)
N10	0.12271 (11)	0.9477 (2)	0.95052 (5)	0.02702 (14)
N11	0.2786 (2)	1.1354 (4)	0.47874 (9)	0.0569 (4)
O12	−0.08726 (19)	1.2705 (3)	0.75327 (6)	0.0491 (3)
O13	−0.00519 (12)	1.42165 (18)	0.88974 (5)	0.03491 (16)
O14	0.34476 (13)	0.7557 (2)	0.86696 (7)	0.03708 (19)
O15	0.1882 (3)	0.9890 (7)	0.42545 (9)	0.0966 (9)
O16	0.3691 (3)	1.3062 (5)	0.45945 (10)	0.0847 (6)
H2	0.034 (3)	0.872 (5)	0.8304 (13)	0.037 (5)*
H3	0.343 (3)	1.127 (5)	0.8692 (13)	0.032 (5)*
H5	0.445 (3)	1.340 (6)	0.7648 (17)	0.052 (6)*
H6	0.453 (4)	1.424 (10)	0.620 (2)	0.091 (10)*
H8	0.112 (5)	0.807 (10)	0.548 (2)	0.087 (10)*
H9	0.090 (4)	0.766 (8)	0.6899 (19)	0.073 (9)*
H101	0.032 (2)	0.927 (4)	0.9615 (10)	0.025 (3)*
H102	0.186 (2)	0.816 (4)	0.9639 (12)	0.030 (4)*
H103	0.174 (4)	1.087 (7)	0.9905 (16)	0.058 (7)*
H12	−0.147 (5)	1.395 (10)	0.743 (2)	0.087 (10)*

H14	0.448 (3)	0.778 (6)	0.8856 (15)	0.049 (6)*
O17	0.29100 (18)	0.1787 (3)	0.26402 (8)	0.0491 (3)
H171	0.307 (4)	0.250 (8)	0.309 (2)	0.073 (9)*
H172	0.290 (3)	0.237 (6)	0.2194 (17)	0.053 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.03069 (11)	0.03544 (12)	0.03887 (13)	0.00116 (10)	0.00372 (8)	−0.00215 (10)
C1	0.0319 (4)	0.0221 (3)	0.0267 (4)	0.0005 (3)	0.0084 (3)	0.0022 (3)
C2	0.0281 (3)	0.0200 (3)	0.0249 (3)	−0.0009 (3)	0.0083 (3)	0.0000 (2)
C3	0.0288 (4)	0.0274 (4)	0.0276 (4)	−0.0040 (3)	0.0099 (3)	−0.0011 (3)
C4	0.0339 (4)	0.0297 (4)	0.0287 (4)	−0.0028 (3)	0.0130 (3)	−0.0025 (3)
C5	0.0576 (7)	0.0393 (6)	0.0359 (5)	−0.0182 (6)	0.0206 (5)	−0.0051 (4)
C6	0.0705 (10)	0.0481 (7)	0.0377 (6)	−0.0171 (7)	0.0260 (6)	−0.0004 (5)
C7	0.0531 (7)	0.0457 (7)	0.0295 (5)	0.0025 (5)	0.0176 (5)	0.0005 (4)
C8	0.0595 (8)	0.0634 (10)	0.0304 (5)	−0.0190 (8)	0.0141 (5)	−0.0110 (6)
C9	0.0582 (8)	0.0504 (8)	0.0323 (5)	−0.0227 (6)	0.0172 (5)	−0.0090 (5)
N10	0.0337 (3)	0.0226 (3)	0.0266 (3)	0.0022 (3)	0.0110 (2)	0.0036 (3)
N11	0.0739 (10)	0.0692 (11)	0.0324 (5)	−0.0018 (8)	0.0221 (6)	0.0009 (6)
O12	0.0684 (7)	0.0451 (5)	0.0274 (4)	0.0240 (5)	0.0005 (4)	0.0003 (4)
O13	0.0471 (4)	0.0216 (3)	0.0339 (3)	0.0039 (3)	0.0062 (3)	−0.0026 (3)
O14	0.0310 (4)	0.0371 (4)	0.0441 (5)	0.0061 (3)	0.0112 (3)	0.0058 (4)
O15	0.1309 (16)	0.127 (2)	0.0336 (5)	−0.0504 (17)	0.0250 (8)	−0.0151 (8)
O16	0.1330 (17)	0.0846 (12)	0.0465 (7)	−0.0305 (13)	0.0414 (9)	0.0055 (8)
O17	0.0652 (7)	0.0431 (5)	0.0357 (5)	−0.0145 (5)	0.0070 (4)	0.0004 (4)

Geometric parameters (Å, º)

C1—O13	1.2078 (14)	C7—C8	1.380 (2)
C1—O12	1.3053 (14)	C7—N11	1.4704 (17)
C1—C2	1.5188 (15)	C8—C9	1.3898 (18)
C2—N10	1.4821 (12)	C8—H8	0.91 (4)
C2—C3	1.5360 (14)	C9—H9	0.94 (4)
C2—H2	0.87 (2)	N10—H101	0.810 (17)
C3—O14	1.4253 (16)	N10—H102	0.83 (2)
C3—C4	1.5128 (14)	N10—H103	0.97 (3)
C3—H3	0.90 (2)	N11—O15	1.219 (3)
C4—C5	1.3851 (17)	N11—O16	1.222 (3)
C4—C9	1.3931 (18)	O12—H12	0.78 (5)
C5—C6	1.3913 (19)	O14—H14	0.82 (3)
C5—H5	0.97 (3)	O17—H171	0.78 (3)
C6—C7	1.372 (2)	O17—H172	0.76 (3)
C6—H6	1.05 (4)		
O13—C1—O12	125.15 (11)	C5—C6—H6	115.3 (17)
O13—C1—C2	122.31 (10)	C6—C7—C8	122.79 (12)
O12—C1—C2	112.53 (9)	C6—C7—N11	119.42 (14)

N10—C2—C1	107.85 (8)	C8—C7—N11	117.79 (14)
N10—C2—C3	107.55 (8)	C7—C8—C9	118.33 (14)
C1—C2—C3	114.68 (8)	C7—C8—H8	119 (3)
N10—C2—H2	104.6 (14)	C9—C8—H8	123 (3)
C1—C2—H2	108.4 (15)	C8—C9—C4	120.22 (13)
C3—C2—H2	113.1 (14)	C8—C9—H9	117.0 (19)
O14—C3—C4	110.69 (9)	C4—C9—H9	121.9 (19)
O14—C3—C2	103.87 (8)	C2—N10—H101	109.8 (11)
C4—C3—C2	114.02 (9)	C2—N10—H102	108.8 (13)
O14—C3—H3	105.5 (14)	H101—N10—H102	112 (2)
C4—C3—H3	109.3 (13)	C2—N10—H103	117.1 (17)
C2—C3—H3	113.1 (13)	H101—N10—H103	102 (2)
C5—C4—C9	119.76 (11)	H102—N10—H103	106 (2)
C5—C4—C3	119.24 (10)	O15—N11—O16	123.43 (16)
C9—C4—C3	120.81 (10)	O15—N11—C7	117.97 (18)
C4—C5—C6	120.54 (13)	O16—N11—C7	118.60 (17)
C4—C5—H5	118.4 (17)	C1—O12—H12	113 (2)
C6—C5—H5	120.9 (17)	C3—O14—H14	107 (2)
C7—C6—C5	118.32 (13)	H171—O17—H172	129 (4)
C7—C6—H6	126.2 (17)		

O15—N11—C7—C6	−179.5 (2)	O14—C3—C4—C9	62.57 (15)
O16—N11—C7—C6	0.2 (3)	O14—C3—C4—C5	−112.37 (13)
O15—N11—C7—C8	0.2 (3)	C2—C3—C4—C9	−54.08 (14)
O16—N11—C7—C8	179.9 (2)	C5—C4—C9—C8	−2.4 (2)
O12—C1—C2—C3	84.78 (12)	C3—C4—C9—C8	−177.27 (13)
O12—C1—C2—N10	−155.44 (11)	C9—C4—C5—C6	1.6 (2)
O13—C1—C2—N10	23.39 (14)	C3—C4—C5—C6	176.57 (15)
O13—C1—C2—C3	−96.39 (12)	C4—C5—C6—C7	−0.2 (3)
C1—C2—C3—O14	176.50 (9)	C5—C6—C7—N11	179.19 (18)
C1—C2—C3—C4	−62.95 (11)	C5—C6—C7—C8	−0.5 (3)
N10—C2—C3—C4	177.11 (8)	C6—C7—C8—C9	−0.2 (3)
N10—C2—C3—O14	56.56 (10)	N11—C7—C8—C9	−179.96 (17)
C2—C3—C4—C5	130.98 (12)	C7—C8—C9—C4	1.7 (2)

Hydrogen-bond geometry (Å, º)

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N10—H101···O13^{i}	0.810 (17)	2.420 (16)	2.9259 (13)	121.5 (14)
N10—H101···Cl^{ii}	0.810 (17)	2.489 (16)	3.2298 (11)	152.7 (15)
N10—H102···O14	0.83 (2)	2.265 (19)	2.6759 (14)	110.8 (16)
N10—H102···Cl^{iii}	0.83 (2)	2.62 (2)	3.3409 (14)	145.5 (16)
N10—H103···Cl^{iv}	0.97 (3)	2.28 (3)	3.2435 (14)	176 (3)
O12—H12···O17^{v}	0.78 (5)	1.84 (5)	2.6168 (19)	171 (4)
O14—H14···Cl	0.82 (3)	2.25 (3)	3.0539 (13)	166 (3)

O17—H171···O16^{vi}	0.78 (3)	2.33 (3)	3.069 (2)	159 (4)
O17—H172···Cl^{vii}	0.76 (3)	2.45 (3)	3.2170 (13)	178 (3)

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