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1-Ethoxy-3-[4-(ethoxycarbonyl)phenyl]-3-hydroxy-1-oxopropan-2-aminium chloride

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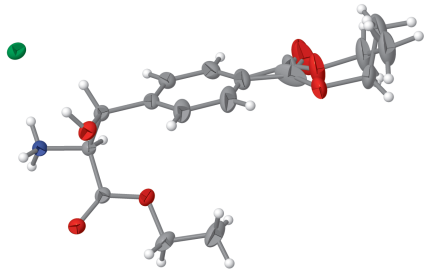
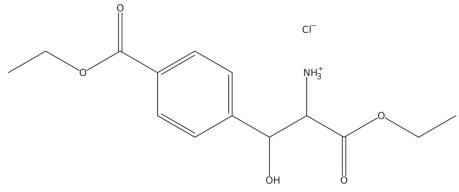
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{14}H_{20}NO_5^+\cdot Cl^-$, was prepared as a racemate of *R*,*R*- and *S*,*S*-enantiomers by reduction of the corresponding hydroxyiminoketone. In the crystal, layers are formed *via* hydrogen bridges of four ammonium groups to chloride ions; these lamellae are connected *via* interdigitated benzoic ester groups.

3D view



Chemical scheme



Structure description

The title compound, $[C_{14}H_{20}NO_5]^+\cdot Cl^-$ (Fig. 1), was prepared by nitrosation of the corresponding keto-ester followed by catalytic reduction of the resulting hydroxyiminoketone. As was observed with a similar oxime (Ebel & Deuschel, 1956), the hydrogenation occurred on both functional groups, the oxime and the ketone, generating two vicinal chiral centers. The crystalline compound obtained from the hydrogenation is a racemic mixture of the (*S*,*S*)- and (*R*,*R*)-enantiomers. The compound crystallizes with a disordered benzoic ester function, the ratio of the two conformers being 0.745/0.255 (11). The minor occupied conformer exhibits a dihedral angle between the plane of the benzene ring and the ester function (C16*A*—O17*A*—O18*A*—C19*A*—C20*A*) of 29.8 (9)°, whereas in the major occupied conformer, the planes of the ring and the ethyl ester (C16—O17—O18—C19—C20) subtend an angle of 14.0 (3)°, with a maximum deviation of −0.148 (7) Å from the ester mean plane at C19. The other ester moiety (C9—C10—O11—O12—C13—C14) is also nearly planar [maximum deviation from mean plane on C14: 0.0733 (19) Å]. This plane and the benzene ring subtend a dihedral angle of 49.16 (8)°. The hydroxy and amino groups are nearly staggered, the torsion angle O8—C7—C9—N15 being 71.81 (13)°. All the ammonium hydrogen atoms are connected with symmetry-related chloride ions *via* N—H···Cl hydrogen bonds (Fig. 2, Table 1); furthermore, H15*A* forms an intermolecular bond to the carbonyl oxygen O1, therefore, four molecules are connected *via* hydrogen bonds to one chloride ion and a carbonyl O atom. The packing in the crystal is dominated by the hydrogen bridges, resulting in

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···Cl1	1.00	2.89	3.4784 (13)	119
C7—H7···Cl1^{i}	1.00	2.96	3.6365 (14)	126
O8—H8···Cl1	0.84 (2)	2.24 (2)	3.0625 (11)	165.1 (19)
N15—H15A···Cl1^{i}	0.87 (2)	2.53 (2)	3.2470 (13)	140.7 (16)
N15—H15A···O11^{ii}	0.87 (2)	2.279 (19)	2.8484 (15)	123.0 (16)
N15—H15B···Cl1^{iii}	0.94 (2)	2.31 (2)	3.2011 (12)	159.3 (15)
N15—H15C···Cl1^{iv}	0.94 (2)	2.19 (2)	3.1155 (13)	168.4 (16)
C20—H20A···O17^{v}	0.98	2.64	3.520 (6)	149
C20A—H20D···O18A^{vi}	0.98	2.03	2.75 (2)	129

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

lamellae formation in the *bc* plane. These layers are connected in the *ac* plane *via* interdigitated benzoic ester groups, connected *via* van der Waals interactions.

Synthesis and crystallization

Ethyl 4-(3-ethoxy-3-oxopropanoyl)benzoate (Korsager *et al.*, 2013) (1.95 g, 7.4 mmol) was added to glacial acetic acid (1.8 ml) at 283 K. While stirring, a solution of 0.76 g $NaNO_2$ (11 mmol) in a minimal amount of water was added dropwise. The mixture was allowed to reach room temperature and after stirring for 1 h, the product was extracted with ethyl acetate, pooled extracts were deacidified with sodium bicarbonate, dried, and the product, ethyl 4-(3-ethoxy-2-hydroxyimino-3-oxopropanoyl)benzoate, was purified by column chromatography (SiO_2, petroleum ether/ethyl acetate 20/1, then toluene /ethyl acetate 9/1) colorless oil, yield: 1.43 g, 66%. 200 mg (0.7 mmol) of this compound were dissolved in ethanol (1 ml) and Pd/C (5%, 0.24 g) and hydrochloric acid (36%, 0.05 ml) were added, stirring for 48 h in a hydrogen atmosphere. The mixture was filtered through silica, the silica washed with ethanol and the solvent was slowly evaporated to obtain 63 mg (0.2 mmol, 32%) as a colorless solid with m.p.= 445-447 K. ^{1}H-NMR (400 MHz, D_2O): 7.97 (*m*, 2 H), 7.43 (*d*, J = 8.3 Hz, 2 H), 5.39 (*d*, 1 H), 4.44 (*q*, 1 H), 4.29 (*m*, J = 7.1 Hz, 2 H), 4.04 (*qd*, J = 7.2 Hz, 2 H), 1.28 (*t*, J = 7.1 Hz, 3 H), 0.97 (*t*, J = 7.1 Hz, 3 H). C-NMR (100 MHz, D_2O): 168.52, 167.16, 143.05, 130.05, 129.68, 126.07, 70.57, 63.29, 62,34, 58.16, 13.34, 12.89. IR: 2985, 2356, 1718, 1506, 1370, 1277, 1107, 1017, 960, 868, 862, 821, 805 cm^{-1}. MS (ESI) 282.13.

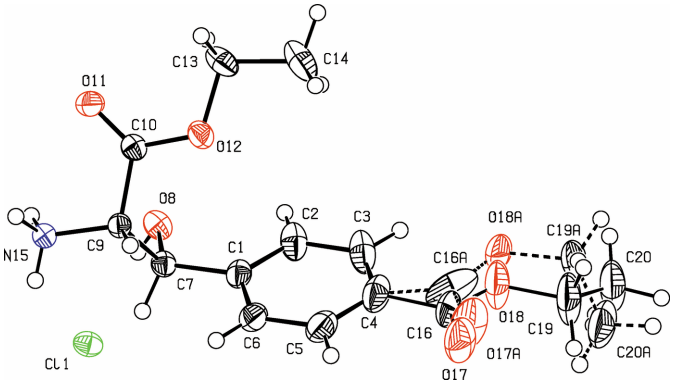


Figure 1
View of the title compound. Displacement ellipsoids are drawn at the 50% probability level. The bonds involving the minor occupied sites are drawn as broken bonds.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{20}NO_5^+\cdot Cl^-$
M_r	317.76
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	120
a, b, c (Å)	20.5205 (9), 8.1626 (3), 9.4732 (4)
β (°)	90.788 (4)
V (Å^3)	1586.62 (11)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.26
Crystal size (mm)	0.45 × 0.26 × 0.07
Data collection	
Diffractometer	Stoe *IPDS* 2T
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13552, 3856, 3370
R_{int}	0.025
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.663
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.100, 1.07
No. of reflections	3856
No. of parameters	253
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.37, −0.24

Computer programs: *X-AREA WinXpose*, *Recipe* and *Integrate* (Stoe & Cie, 2020), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2019/2* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2020).

Refinement

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

References

Ebel, F. & Deuschel, W. (1956). *Chem. Ber.* **89**, 2799–2807.
Korsager, S., Nielsen, D. U., Taaning, R. H. & Skrydstrup, T. (2013). *Angew. Chem. Int. Ed.* **52**, 9763–9766.

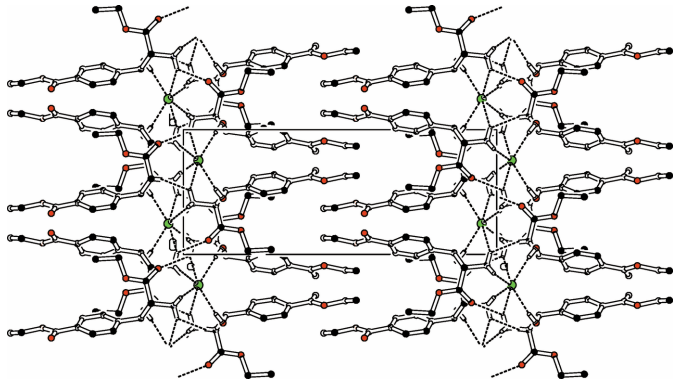


Figure 2
Part of the packing diagram. View along the *c* axis.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2020). *Acta Cryst.* E**76**, 1–11.
Stoe & Cie (2020). *X-RED* and *X-AREA*. Stoe & Cie, Darmstadt,

full crystallographic data

IUCrData (2024). **9**, x241032 [https://doi.org/10.1107/S2414314624010320]

1-Ethoxy-3-[4-(ethoxycarbonyl)phenyl]-3-hydroxy-1-oxopropan-2-aminium chloride

Dieter Schollmeyer, Igor Proz and Heiner Detert

1-Ethoxy-3-[4-(ethoxycarbonyl)phenyl]-3-hydroxy-1-oxopropan-2-aminium chloride

Crystal data

$C_{14}H_{20}NO_5^+ \cdot Cl^-$
M_r = 317.76
Monoclinic, $P2_1/c$
a = 20.5205 (9) Å
b = 8.1626 (3) Å
c = 9.4732 (4) Å
β = 90.788 (4)°
V = 1586.62 (11) Å^3
Z = 4
$F(000)$ = 672
D_x = 1.330 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 20325 reflections
θ = 2.7–28.6°
μ = 0.26 mm^{-1}
T = 120 K
Plate, colorless
0.45 × 0.26 × 0.07 mm

Data collection

Stoe IPDS 2T diffractometer
Radiation source: sealed X-ray tube, 12x0.4mm long-fine focus
Detector resolution: 6.67 pixels mm^{-1}
rotation method, ω scans
13552 measured reflections
3856 independent reflections
3370 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 28.1°, θ_{min} = 2.7°
h = −26→27
k = −10→10
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.100
S = 1.07
3856 reflections
253 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.8357P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Hydrogen atoms attached to carbons were placed at calculated positions and were refined in the riding-model approximation with $C_{aromatic}$–H = 0.95 Å, C_{methyl}–H = 0.98 Å, $C_{methylene}$–H = 0.99 Å, $C_{tertiary}$–H = 1.00 Å, and with U_{iso}(H) = 1.5 U_{eq}(C_{methyl}) or with U_{iso}(H) = 1.2 U_{eq}(C). The methyl groups were allowed to rotate but not to tip. The H atom bonded to O was freely refined. The coordinates of the H atoms bonded to N were freely refined, using the same displacement parameter for all of them.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl1	0.04788 (2)	0.74584 (4)	0.53772 (3)	0.02717 (10)	
C1	0.20763 (7)	0.53804 (17)	0.26788 (16)	0.0290 (3)	
C2	0.25645 (7)	0.4881 (2)	0.36094 (18)	0.0396 (4)	
H2	0.245492	0.436695	0.447396	0.047*	
C3	0.32166 (8)	0.5135 (3)	0.3275 (2)	0.0489 (5)	
H3	0.354962	0.480069	0.391941	0.059*	
C4	0.33832 (8)	0.5868 (2)	0.2013 (2)	0.0459 (4)	
C5	0.28952 (8)	0.6384 (2)	0.1090 (2)	0.0432 (4)	
H5	0.300619	0.689710	0.022632	0.052*	
C6	0.22461 (8)	0.61534 (19)	0.14216 (18)	0.0356 (3)	
H6	0.191439	0.652400	0.078923	0.043*	
C7	0.13615 (6)	0.51234 (17)	0.29996 (14)	0.0247 (3)	
H7	0.113579	0.620639	0.292571	0.030*	
O8	0.12629 (5)	0.44954 (13)	0.43765 (10)	0.0290 (2)	
H8	0.1037 (10)	0.520 (3)	0.479 (2)	0.044 (5)*	
C9	0.10290 (6)	0.39315 (16)	0.19502 (14)	0.0224 (3)	
H9	0.116469	0.419779	0.096663	0.027*	
C10	0.11986 (6)	0.21598 (16)	0.22877 (14)	0.0242 (3)	
O11	0.08032 (5)	0.11362 (12)	0.25813 (12)	0.0318 (2)	
O12	0.18364 (5)	0.19266 (13)	0.22279 (11)	0.0302 (2)	
C13	0.20667 (8)	0.0287 (2)	0.25851 (19)	0.0387 (4)	
H13A	0.192695	−0.050854	0.185213	0.046*	
H13B	0.188989	−0.006596	0.350352	0.046*	
C14	0.27998 (9)	0.0386 (3)	0.2664 (2)	0.0529 (5)	
H14A	0.297970	−0.070463	0.285645	0.079*	
H14B	0.293063	0.113733	0.342381	0.079*	
H14C	0.296576	0.079071	0.176389	0.079*	
N15	0.03125 (5)	0.41088 (15)	0.20662 (13)	0.0244 (2)	
H15A	0.0204 (9)	0.513 (3)	0.193 (2)	0.037 (3)*	
H15B	0.0158 (9)	0.381 (2)	0.296 (2)	0.037 (3)*	
H15C	0.0093 (9)	0.347 (2)	0.139 (2)	0.037 (3)*	
C16	0.40734 (16)	0.6211 (5)	0.1514 (7)	0.0389 (10)	0.745 (11)
O17	0.42199 (18)	0.6711 (5)	0.0352 (5)	0.0582 (9)	0.745 (11)
O18	0.44929 (13)	0.5912 (7)	0.2542 (4)	0.0615 (10)	0.745 (11)
C19	0.51701 (16)	0.6271 (9)	0.2243 (5)	0.0714 (15)	0.745 (11)
H19A	0.520857	0.734003	0.175759	0.086*	0.745 (11)
H19B	0.535769	0.541044	0.163486	0.086*	0.745 (11)
C20	0.55162 (16)	0.6316 (9)	0.3649 (5)	0.0711 (13)	0.745 (11)
H20A	0.530360	0.711548	0.426259	0.085*	0.745 (11)

H20B	0.597186	0.663354	0.351674	0.085*	0.745 (11)
H20C	0.549900	0.522924	0.408636	0.085*	0.745 (11)
C16A	0.4092 (10)	0.583 (2)	0.2027 (17)	0.062 (5)	0.255 (11)
O17A	0.4281 (7)	0.6462 (18)	0.0916 (14)	0.070 (3)	0.255 (11)
O18A	0.4507 (3)	0.5225 (12)	0.2963 (14)	0.049 (2)	0.255 (11)
C19A	0.5220 (4)	0.5443 (15)	0.2706 (19)	0.061 (4)	0.255 (11)
H19C	0.547117	0.463037	0.326574	0.073*	0.255 (11)
H19D	0.531053	0.525759	0.169490	0.073*	0.255 (11)
C20A	0.5421 (5)	0.710 (2)	0.311 (2)	0.069 (4)	0.255 (11)
H20D	0.521517	0.789492	0.246725	0.103*	0.255 (11)
H20E	0.589582	0.719229	0.304779	0.103*	0.255 (11)
H20F	0.528635	0.732273	0.407778	0.103*	0.255 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.03246 (18)	0.02436 (17)	0.02476 (16)	0.00383 (12)	0.00350 (12)	−0.00027 (12)
C1	0.0241 (6)	0.0261 (7)	0.0367 (7)	−0.0015 (5)	0.0013 (5)	−0.0113 (6)
C2	0.0272 (7)	0.0524 (10)	0.0391 (8)	0.0013 (7)	−0.0027 (6)	−0.0125 (7)
C3	0.0260 (8)	0.0657 (12)	0.0549 (11)	0.0042 (8)	−0.0059 (7)	−0.0210 (9)
C4	0.0282 (8)	0.0449 (9)	0.0649 (12)	−0.0056 (7)	0.0105 (7)	−0.0211 (9)
C5	0.0369 (8)	0.0345 (8)	0.0585 (11)	−0.0070 (7)	0.0125 (7)	−0.0056 (8)
C6	0.0305 (7)	0.0286 (7)	0.0479 (9)	−0.0028 (6)	0.0046 (6)	−0.0040 (6)
C7	0.0235 (6)	0.0248 (6)	0.0257 (6)	0.0012 (5)	0.0003 (5)	−0.0037 (5)
O8	0.0306 (5)	0.0323 (5)	0.0242 (5)	0.0066 (4)	0.0009 (4)	−0.0040 (4)
C9	0.0206 (6)	0.0227 (6)	0.0239 (6)	0.0000 (5)	0.0005 (5)	−0.0008 (5)
C10	0.0250 (6)	0.0252 (6)	0.0223 (6)	0.0026 (5)	0.0007 (5)	−0.0035 (5)
O11	0.0303 (5)	0.0238 (5)	0.0415 (6)	0.0008 (4)	0.0059 (4)	0.0021 (4)
O12	0.0250 (5)	0.0282 (5)	0.0372 (5)	0.0050 (4)	−0.0006 (4)	−0.0044 (4)
C13	0.0395 (8)	0.0339 (8)	0.0425 (9)	0.0154 (7)	−0.0041 (7)	−0.0026 (7)
C14	0.0408 (9)	0.0609 (12)	0.0566 (11)	0.0239 (9)	−0.0127 (8)	−0.0142 (9)
N15	0.0221 (5)	0.0233 (5)	0.0277 (6)	0.0006 (4)	−0.0010 (4)	0.0017 (5)
C16	0.0231 (14)	0.0503 (19)	0.044 (3)	−0.0071 (12)	0.0116 (14)	−0.0005 (17)
O17	0.0346 (11)	0.0832 (19)	0.057 (2)	−0.0068 (11)	0.0143 (15)	0.0125 (17)
O18	0.0223 (10)	0.098 (3)	0.0647 (17)	−0.0070 (14)	0.0010 (10)	0.0146 (17)
C19	0.0209 (13)	0.112 (4)	0.081 (2)	−0.0046 (19)	0.0032 (13)	0.014 (3)
C20	0.0244 (13)	0.106 (4)	0.083 (3)	−0.0029 (18)	−0.0030 (14)	0.012 (2)
C16A	0.101 (12)	0.054 (7)	0.033 (7)	−0.031 (6)	0.041 (7)	−0.003 (5)
O17A	0.054 (6)	0.113 (8)	0.044 (6)	−0.022 (5)	0.025 (5)	0.021 (5)
O18A	0.024 (3)	0.046 (4)	0.076 (6)	−0.005 (3)	0.014 (3)	0.001 (3)
C19A	0.021 (4)	0.054 (6)	0.108 (11)	0.007 (4)	0.011 (5)	−0.006 (6)
C20A	0.029 (5)	0.079 (9)	0.098 (11)	−0.013 (5)	0.015 (5)	−0.016 (7)

Geometric parameters (Å, º)

C1—C2	1.387 (2)	C13—H13B	0.9900
C1—C6	1.396 (2)	C14—H14A	0.9800
C1—C7	1.5165 (18)	C14—H14B	0.9800

C2—C3	1.395 (2)	C14—H14C	0.9800
C2—H2	0.9500	N15—H15A	0.87 (2)
C3—C4	1.384 (3)	N15—H15B	0.94 (2)
C3—H3	0.9500	N15—H15C	0.94 (2)
C4—C5	1.386 (3)	C16—O17	1.216 (5)
C4—C16A	1.45 (2)	C16—O18	1.314 (5)
C4—C16	1.525 (5)	O18—C19	1.452 (4)
C5—C6	1.385 (2)	C19—C20	1.501 (7)
C5—H5	0.9500	C19—H19A	0.9900
C6—H6	0.9500	C19—H19B	0.9900
C7—O8	1.4186 (16)	C20—H20A	0.9800
C7—C9	1.5433 (18)	C20—H20B	0.9800
C7—H7	1.0000	C20—H20C	0.9800
O8—H8	0.84 (2)	C16A—O17A	1.238 (10)
C9—N15	1.4831 (16)	C16A—O18A	1.32 (2)
C9—C10	1.5204 (18)	O18A—C19A	1.496 (11)
C9—H9	1.0000	C19A—C20A	1.463 (18)
C10—O11	1.2000 (17)	C19A—H19C	0.9900
C10—O12	1.3245 (16)	C19A—H19D	0.9900
O12—C13	1.4579 (18)	C20A—H20D	0.9800
C13—C14	1.508 (2)	C20A—H20E	0.9800
C13—H13A	0.9900	C20A—H20F	0.9800

C2—C1—C6	119.24 (14)	C13—C14—H14B	109.5
C2—C1—C7	121.62 (14)	H14A—C14—H14B	109.5
C6—C1—C7	119.14 (13)	C13—C14—H14C	109.5
C1—C2—C3	119.93 (17)	H14A—C14—H14C	109.5
C1—C2—H2	120.0	H14B—C14—H14C	109.5
C3—C2—H2	120.0	C9—N15—H15A	109.5 (12)
C4—C3—C2	120.63 (17)	C9—N15—H15B	112.9 (11)
C4—C3—H3	119.7	H15A—N15—H15B	107.4 (17)
C2—C3—H3	119.7	C9—N15—H15C	111.3 (11)
C3—C4—C5	119.42 (15)	H15A—N15—H15C	108.0 (17)
C3—C4—C16A	104.1 (7)	H15B—N15—H15C	107.7 (16)
C5—C4—C16A	136.5 (7)	O17—C16—O18	124.5 (3)
C3—C4—C16	126.1 (3)	O17—C16—C4	125.8 (3)
C5—C4—C16	114.5 (3)	O18—C16—C4	109.8 (4)
C6—C5—C4	120.30 (17)	C16—O18—C19	115.9 (3)
C6—C5—H5	119.8	O18—C19—C20	105.9 (3)
C4—C5—H5	119.8	O18—C19—H19A	110.6
C5—C6—C1	120.44 (16)	C20—C19—H19A	110.6
C5—C6—H6	119.8	O18—C19—H19B	110.6
C1—C6—H6	119.8	C20—C19—H19B	110.6
O8—C7—C1	112.63 (11)	H19A—C19—H19B	108.7
O8—C7—C9	107.28 (11)	C19—C20—H20A	109.5
C1—C7—C9	112.23 (11)	C19—C20—H20B	109.5
O8—C7—H7	108.2	H20A—C20—H20B	109.5
C1—C7—H7	108.2	C19—C20—H20C	109.5

C9—C7—H7	108.2	H20A—C20—H20C	109.5
C7—O8—H8	105.4 (14)	H20B—C20—H20C	109.5
N15—C9—C10	107.55 (10)	O17A—C16A—O18A	121.4 (15)
N15—C9—C7	108.70 (10)	O17A—C16A—C4	108.0 (16)
C10—C9—C7	111.49 (11)	O18A—C16A—C4	130.6 (9)
N15—C9—H9	109.7	C16A—O18A—C19A	118.1 (9)
C10—C9—H9	109.7	C20A—C19A—O18A	109.9 (8)
C7—C9—H9	109.7	C20A—C19A—H19C	109.7
O11—C10—O12	125.56 (13)	O18A—C19A—H19C	109.7
O11—C10—C9	123.88 (12)	C20A—C19A—H19D	109.7
O12—C10—C9	110.56 (11)	O18A—C19A—H19D	109.7
C10—O12—C13	116.06 (12)	H19C—C19A—H19D	108.2
O12—C13—C14	106.39 (15)	C19A—C20A—H20D	109.5
O12—C13—H13A	110.5	C19A—C20A—H20E	109.5
C14—C13—H13A	110.5	H20D—C20A—H20E	109.5
O12—C13—H13B	110.5	C19A—C20A—H20F	109.5
C14—C13—H13B	110.5	H20D—C20A—H20F	109.5
H13A—C13—H13B	108.6	H20E—C20A—H20F	109.5
C13—C14—H14A	109.5		

C6—C1—C2—C3	−0.8 (2)	C7—C9—C10—O11	119.45 (14)
C7—C1—C2—C3	179.81 (15)	N15—C9—C10—O12	−178.83 (11)
C1—C2—C3—C4	−0.6 (3)	C7—C9—C10—O12	−59.76 (14)
C2—C3—C4—C5	1.3 (3)	O11—C10—O12—C13	−1.5 (2)
C2—C3—C4—C16A	−177.1 (6)	C9—C10—O12—C13	177.71 (11)
C2—C3—C4—C16	−179.7 (2)	C10—O12—C13—C14	−171.43 (13)
C3—C4—C5—C6	−0.6 (3)	C3—C4—C16—O17	172.6 (4)
C16A—C4—C5—C6	177.3 (8)	C5—C4—C16—O17	−8.4 (5)
C16—C4—C5—C6	−179.65 (19)	C3—C4—C16—O18	−8.6 (4)
C4—C5—C6—C1	−0.9 (2)	C5—C4—C16—O18	170.4 (3)
C2—C1—C6—C5	1.6 (2)	O17—C16—O18—C19	2.1 (6)
C7—C1—C6—C5	−179.05 (14)	C4—C16—O18—C19	−176.8 (3)
C2—C1—C7—O8	6.05 (19)	C16—O18—C19—C20	163.8 (7)
C6—C1—C7—O8	−173.30 (12)	C3—C4—C16A—O17A	179.4 (10)
C2—C1—C7—C9	−115.16 (15)	C5—C4—C16A—O17A	1.4 (17)
C6—C1—C7—C9	65.49 (17)	C3—C4—C16A—O18A	1.8 (14)
O8—C7—C9—N15	71.81 (13)	C5—C4—C16A—O18A	−176.2 (8)
C1—C7—C9—N15	−163.95 (11)	O17A—C16A—O18A—C19A	5.2 (19)
O8—C7—C9—C10	−46.57 (14)	C4—C16A—O18A—C19A	−177.5 (10)
C1—C7—C9—C10	77.66 (14)	C16A—O18A—C19A—C20A	79.2 (19)
N15—C9—C10—O11	0.38 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···Cl1	1.00	2.89	3.4784 (13)	119
C7—H7···Cl1^{i}	1.00	2.96	3.6365 (14)	126
O8—H8···Cl1	0.84 (2)	2.24 (2)	3.0625 (11)	165.1 (19)

N15—H15*A*···Cl1^{i}	0.87 (2)	2.53 (2)	3.2470 (13)	140.7 (16)
N15—H15*A*···O11^{ii}	0.87 (2)	2.279 (19)	2.8484 (15)	123.0 (16)
N15—H15*B*···Cl1^{iii}	0.94 (2)	2.31 (2)	3.2011 (12)	159.3 (15)
N15—H15*C*···Cl1^{iv}	0.94 (2)	2.19 (2)	3.1155 (13)	168.4 (16)
C20—H20*A*···O17^{v}	0.98	2.64	3.520 (6)	149
C20*A*—H20*D*···O18*A*^{vi}	0.98	2.03	2.75 (2)	129

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