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1-(3-Oxo-3-phenylpropyl)piperidinium chloride

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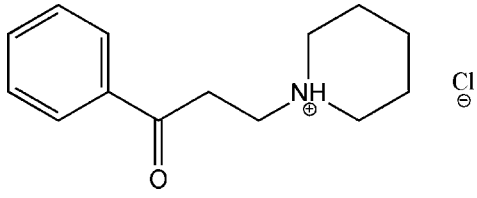
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.052; wR factor = 0.130; data-to-parameter ratio = 13.9.

In the title salt, $C_{14}H_{20}NO^{+}\cdot Cl^{-}$, the piperidine ring adopts a chair conformation. In the crystal, the cations and anions are linked by classical N—H···Cl hydrogen bond and weak C—H···Cl and C—H···O hydrogen bonds; the C—H···O hydrogen bonds exhibit $R_2^2(14)$ ring motifs while the C—H···Cl hydrogen bonds link the molecules into chains along the a-axis direction. π–π stacking is observed between parallel phenyl rings of adjacent cations, the centroid–centroid distance being 3.8164 (15) Å.

Related literature

For the synthesis and biological activity of piperidine derivatives, see: Vartanyan (1984). For standard bond lengths, see: Allen *et al.* (1987). For hydrogen-bond motifs see: Bernstein *et al.* (1995). For puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{14}H_{20}NO^{+}\cdot Cl^{-}$
M_r = 253.76
Monoclinic, $P2_1/c$
a = 11.2936 (13) Å
b = 12.0531 (15) Å
c = 10.9650 (13) Å
β = 112.971 (5)°
V = 1374.2 (3) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 2.33 mm^{-1}
T = 296 K
0.23 × 0.22 × 0.21 mm

Data collection

Bruker X8 Proteum diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.558, T_{max} = 0.614
7001 measured reflections
2217 independent reflections
1833 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.130
S = 1.09
2217 reflections
159 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H11···Cl1^{i}	0.95 (2)	2.15 (2)	3.0837 (18)	171 (2)
C4—H4···Cl1^{ii}	0.93	2.82	3.745 (3)	172
C14—H14B···O1^{iii}	0.97	2.45	3.249 (3)	139

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

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* and *Mercury*.

The authors are thankful to the IOE, University of Mysore, for providing the single-crystal X-ray diffraction facility. PN thanks the BET Academy of Higher Education for research facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5748).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2013). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Vartanyan, R. S. (1984). *Pharm. Chem. J.* **18**, 736–749.

supplementary materials

Acta Cryst. (2013). E**69**, o1748 [doi:10.1107/S1600536813029887]

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1. Comment

The piperidine hydrochloride is used as an intermediate for the synthesis of pharmaceuticals such as haloperidol (neuroleptic drug used to treat patients with psychotic illnesses, extreme agitation, or Tourette's syndrome) and loperamide which is a synthetic piperidine derivative, is an effective drug against diarrhea resulting from gastroenteritis or inflammatory bowel disease (Vartanyan *et al.*, 1984).

The piperidine ring (N1/C10—C14) of the title compound (Fig. 1) adopts chair conformation. The puckering parameters of the piperdine ring are Q = 0.571 (2) Å, θ = 180.0 (2)° and φ = 19 (10)° (Cremer & Pople, 1975). The bond lengths and angles are in normal ranges (Allen *et al.*, 1987).

The molecules are packed along *a* axis with inter molecular hydrogen bonds are shown in Figure 2. Bond lengths and angles of intermolecular hydrogen bonds (N1—H11···Cl1, C14—H14B···O1 and C4—H4···Cl1) are listed in table 1. Also, N···Cl intercontacts with a distance of 3.084 Å is observed. The C14—H14B···O1, exhibits $R^2_2(14)$ ring motifs (Bernstein *et al.*, 1995). The molecules are connected by infinite one dimensional chains along *a* axis by C4—H4···Cl1 hydrogen bonds. In addition, $\pi\cdots\pi$ interactions exists between phenyl rings *Cg*2···*Cg*2 with a distance of 3.8164 (15) Å, where *Cg*2 is C1/C2/C3/C4/C5/C6. Overall crystal structure of the title molecule exhibits three dimensional architecture.

2. Experimental

Single crystals (block) were obtained from slow evaporation of a solution of ethylacetate (m.p.:410–413 K).

3. Refinement

The H11 atom is located in a difference Fourier map and refined isotropically. Other H atoms were fixed geometrically (C—H= 0.93–0.96 Å) and allowed to ride on their parent atoms with U_{iso}(H) = 1.5U_{eq} for methyl H atom and 1.2U_{eq}(C) for other H atoms.

Computing details

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *Mercury* (Macrae *et al.*, 2008).

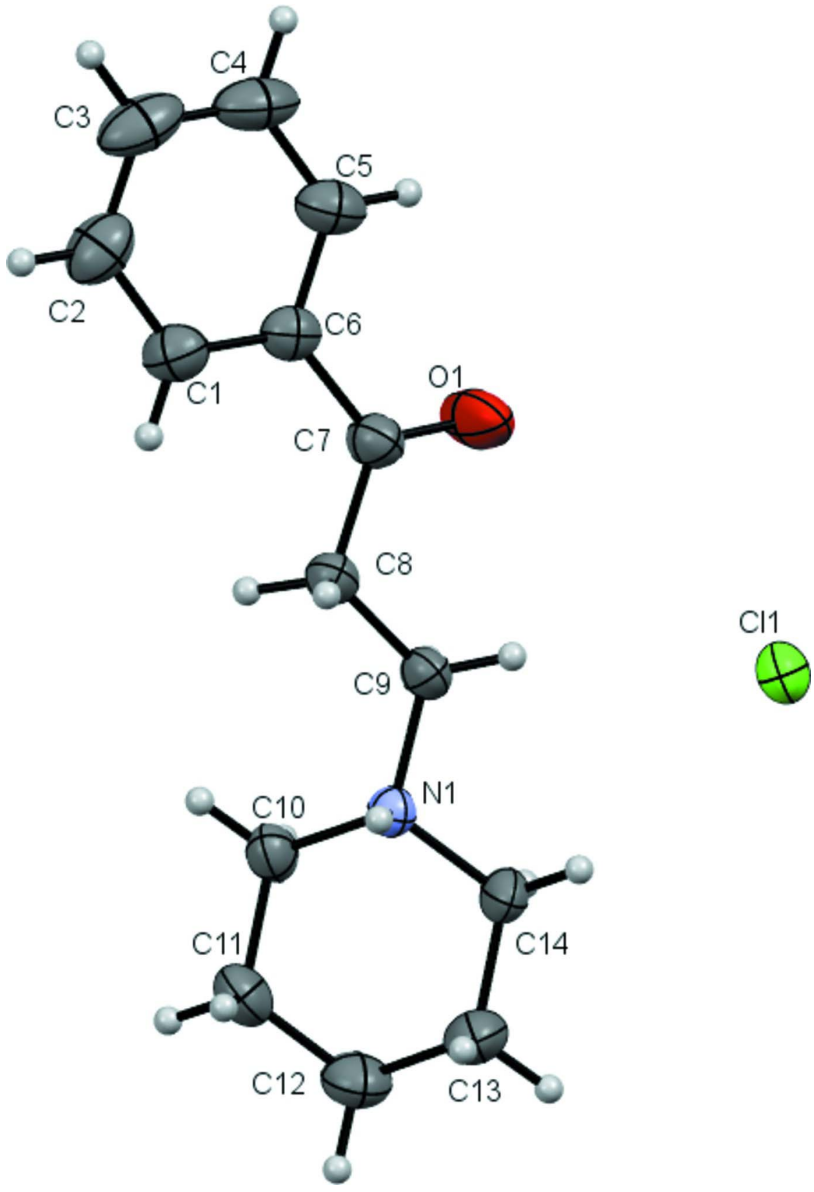

Figure 1

ORTEP diagram of the title compound with 50% probability ellipsoids.

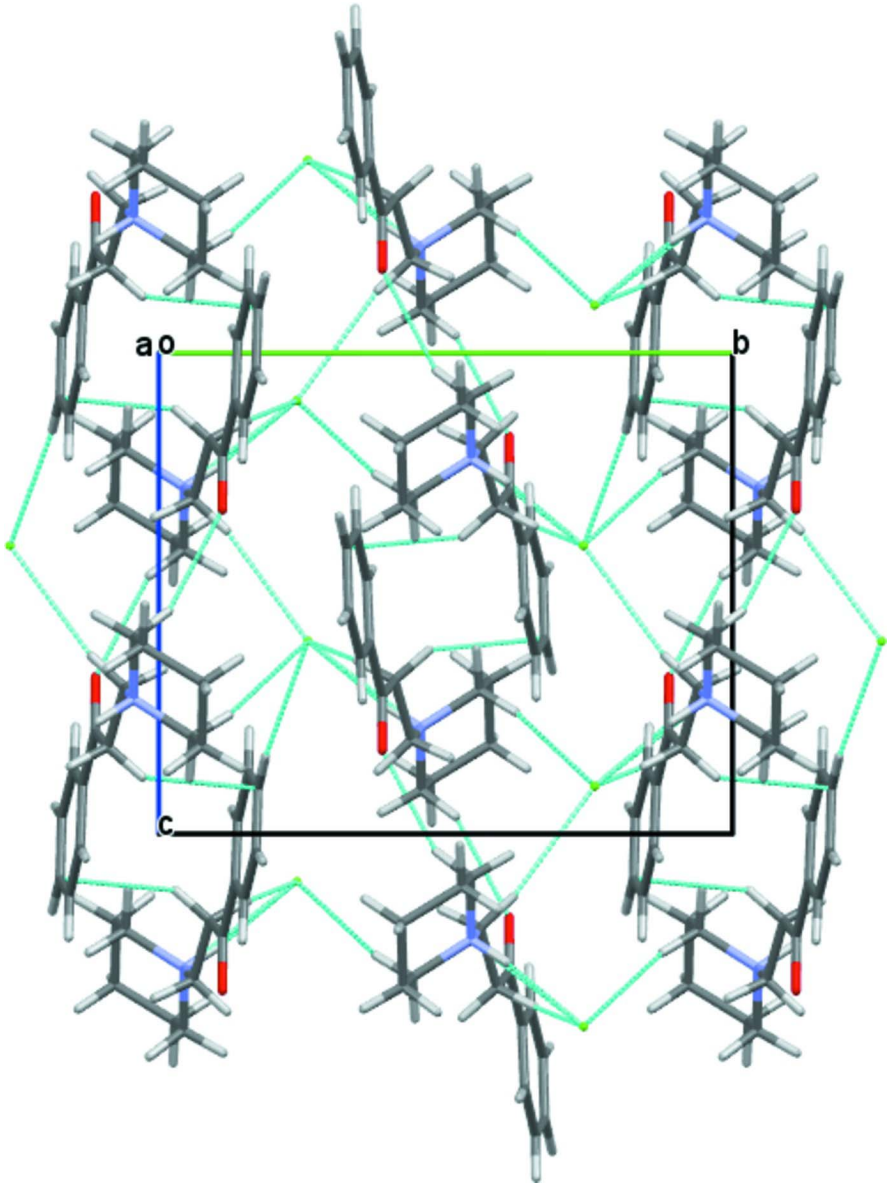

Figure 2

Packing diagram of molecule, viewed along the crystallographic *a* axis. Dotted lines indicate hydrogen bonds and short contacts involved.

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Crystal data

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Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.2936\ (13)$ Å
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$c = 10.9650\ (13)$ Å
$\beta = 112.971\ (5)°$
$V = 1374.2\ (3)$ Å^3
$Z = 4$
$F(000) = 544$
$D_x = 1.227$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 2217 reflections
$\theta = 4.3–64.4°$
$\mu = 2.33$ mm^{-1}

T = 296 K
Block, colourless

0.23 × 0.22 × 0.21 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Bruker MicroStar microfocus rotating anode
Helios multilayer optics monochromator
Detector resolution: 10.7 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)

T_{min} = 0.558, T_{max} = 0.614
7001 measured reflections
2217 independent reflections
1833 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
θ_{max} = 64.4°, θ_{min} = 4.3°
h = −13→12
k = −10→14
l = −10→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.130
S = 1.09
2217 reflections
159 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement
W = 1/[Σ^2(FO^2) + (0.0762P)^2 + 0.2853P] where P = (FO^2 + 2FC^2)/3
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), FC*=KFC[1+0.001XFC$^2\Lambda^3$/SIN(2Θ)]$^{-1/4}$
Extinction coefficient: 0.173 (7)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -R-factor-obs *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}
O1	−0.28618 (16)	0.3894 (2)	−0.17351 (15)	0.0736 (8)
N1	0.05732 (13)	0.45578 (15)	−0.22047 (13)	0.0286 (5)
C1	−0.43665 (19)	0.3624 (2)	−0.5286 (2)	0.0438 (7)
C2	−0.5574 (2)	0.3438 (2)	−0.6274 (2)	0.0533 (8)
C3	−0.6620 (2)	0.3275 (2)	−0.5943 (3)	0.0592 (9)
C4	−0.6478 (2)	0.3307 (2)	−0.4642 (3)	0.0586 (9)
C5	−0.5288 (2)	0.3513 (2)	−0.3656 (2)	0.0464 (8)
C6	−0.42153 (17)	0.36703 (19)	−0.39763 (18)	0.0353 (6)
C7	−0.29561 (18)	0.3890 (2)	−0.28717 (18)	0.0381 (7)
C8	−0.17901 (16)	0.41077 (19)	−0.31941 (17)	0.0353 (6)
C9	−0.06310 (17)	0.44018 (19)	−0.19508 (17)	0.0344 (6)
C10	0.05276 (19)	0.55864 (19)	−0.29863 (19)	0.0378 (6)
C11	0.1784 (2)	0.5746 (2)	−0.3175 (2)	0.0457 (8)
C12	0.2928 (2)	0.5773 (2)	−0.1862 (2)	0.0521 (8)

C13	0.29567 (18)	0.4721 (2)	−0.1097 (2)	0.0500 (8)
C14	0.17059 (17)	0.4572 (2)	−0.09057 (18)	0.0404 (7)
Cl1	0.05845 (4)	0.24029 (5)	0.09932 (4)	0.0406 (2)
H1	−0.36580	0.37180	−0.55080	0.0530*
H2	−0.56760	0.34230	−0.71580	0.0640*
H3	−0.74260	0.31430	−0.66040	0.0710*
H4	−0.71860	0.31900	−0.44240	0.0700*
H5	−0.51980	0.35480	−0.27770	0.0560*
H8A	−0.19730	0.47130	−0.38240	0.0420*
H8B	−0.16000	0.34530	−0.35990	0.0420*
H9A	−0.08110	0.50800	−0.15790	0.0410*
H9B	−0.04900	0.38170	−0.13020	0.0410*
H10A	0.03750	0.62240	−0.25280	0.0450*
H10B	−0.01790	0.55340	−0.38450	0.0450*
H11	0.066 (2)	0.393 (2)	−0.268 (2)	0.041 (6)*
H11A	0.18900	0.51460	−0.37120	0.0550*
H11B	0.17470	0.64360	−0.36440	0.0550*
H12A	0.37160	0.58350	−0.20140	0.0620*
H12B	0.28670	0.64120	−0.13530	0.0620*
H13A	0.36670	0.47550	−0.02390	0.0600*
H13B	0.30930	0.40890	−0.15740	0.0600*
H14A	0.17360	0.38800	−0.04420	0.0480*
H14B	0.16080	0.51720	−0.03640	0.0480*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0501 (9)	0.138 (2)	0.0387 (8)	−0.0265 (11)	0.0239 (7)	−0.0115 (10)
N1	0.0294 (8)	0.0315 (10)	0.0248 (7)	−0.0022 (6)	0.0104 (6)	−0.0011 (7)
C1	0.0371 (10)	0.0478 (15)	0.0450 (11)	−0.0014 (9)	0.0144 (8)	−0.0064 (10)
C2	0.0506 (13)	0.0500 (17)	0.0470 (11)	−0.0009 (11)	0.0057 (9)	−0.0092 (12)
C3	0.0373 (11)	0.0412 (16)	0.0810 (17)	−0.0043 (10)	0.0034 (11)	−0.0122 (13)
C4	0.0343 (11)	0.0499 (17)	0.0903 (18)	−0.0076 (11)	0.0230 (11)	−0.0031 (15)
C5	0.0403 (11)	0.0440 (15)	0.0596 (13)	−0.0058 (10)	0.0245 (9)	−0.0010 (11)
C6	0.0337 (10)	0.0307 (12)	0.0427 (11)	0.0001 (8)	0.0163 (8)	−0.0014 (9)
C7	0.0355 (10)	0.0450 (14)	0.0365 (10)	−0.0033 (9)	0.0171 (8)	−0.0022 (10)
C8	0.0311 (9)	0.0435 (14)	0.0329 (9)	0.0010 (8)	0.0144 (7)	0.0013 (9)
C9	0.0324 (9)	0.0444 (13)	0.0293 (9)	−0.0020 (8)	0.0153 (7)	0.0007 (9)
C10	0.0408 (10)	0.0381 (13)	0.0369 (10)	0.0035 (9)	0.0177 (8)	0.0087 (9)
C11	0.0490 (12)	0.0481 (16)	0.0450 (11)	−0.0047 (10)	0.0239 (9)	0.0090 (11)
C12	0.0430 (12)	0.0569 (18)	0.0565 (13)	−0.0150 (11)	0.0197 (10)	0.0001 (12)
C13	0.0321 (10)	0.0650 (18)	0.0467 (11)	−0.0061 (10)	0.0086 (8)	0.0066 (11)
C14	0.0349 (10)	0.0547 (15)	0.0263 (9)	−0.0078 (9)	0.0063 (7)	0.0054 (9)
Cl1	0.0444 (4)	0.0417 (4)	0.0369 (4)	−0.0004 (2)	0.0171 (2)	0.0073 (2)

Geometric parameters (Å, º)

O1—C7	1.209 (2)	C2—H2	0.9300
N1—C9	1.503 (3)	C3—H3	0.9300
N1—C10	1.497 (3)	C4—H4	0.9300

N1—C14	1.498 (2)	C5—H5	0.9300
N1—H11	0.95 (2)	C8—H8A	0.9700
C1—C6	1.380 (3)	C8—H8B	0.9700
C1—C2	1.389 (3)	C9—H9A	0.9700
C2—C3	1.379 (4)	C9—H9B	0.9700
C3—C4	1.373 (4)	C10—H10A	0.9700
C4—C5	1.379 (4)	C10—H10B	0.9700
C5—C6	1.400 (3)	C11—H11A	0.9700
C6—C7	1.488 (3)	C11—H11B	0.9700
C7—C8	1.514 (3)	C12—H12A	0.9700
C8—C9	1.518 (3)	C12—H12B	0.9700
C10—C11	1.524 (3)	C13—H13A	0.9700
C11—C12	1.514 (3)	C13—H13B	0.9700
C12—C13	1.514 (3)	C14—H14A	0.9700
C13—C14	1.518 (3)	C14—H14B	0.9700
C1—H1	0.9300		

C9—N1—C10	112.23 (16)	C7—C8—H8B	110.00
C9—N1—C14	108.91 (13)	C9—C8—H8A	109.00
C10—N1—C14	111.15 (16)	C9—C8—H8B	109.00
C9—N1—H11	107.3 (15)	H8A—C8—H8B	108.00
C10—N1—H11	109.6 (14)	N1—C9—H9A	109.00
C14—N1—H11	107.4 (13)	N1—C9—H9B	109.00
C2—C1—C6	120.2 (2)	C8—C9—H9A	109.00
C1—C2—C3	119.9 (2)	C8—C9—H9B	109.00
C2—C3—C4	120.3 (2)	H9A—C9—H9B	108.00
C3—C4—C5	120.2 (2)	N1—C10—H10A	109.00
C4—C5—C6	120.1 (2)	N1—C10—H10B	109.00
C5—C6—C7	117.75 (17)	C11—C10—H10A	109.00
C1—C6—C7	123.03 (19)	C11—C10—H10B	109.00
C1—C6—C5	119.22 (19)	H10A—C10—H10B	108.00
O1—C7—C6	120.8 (2)	C10—C11—H11A	109.00
O1—C7—C8	120.36 (19)	C10—C11—H11B	109.00
C6—C7—C8	118.87 (16)	C12—C11—H11A	109.00
C7—C8—C9	110.77 (15)	C12—C11—H11B	109.00
N1—C9—C8	112.86 (14)	H11A—C11—H11B	108.00
N1—C10—C11	110.89 (18)	C11—C12—H12A	110.00
C10—C11—C12	111.59 (17)	C11—C12—H12B	110.00
C11—C12—C13	109.56 (19)	C13—C12—H12A	110.00
C12—C13—C14	110.85 (19)	C13—C12—H12B	110.00
N1—C14—C13	111.44 (15)	H12A—C12—H12B	108.00
C2—C1—H1	120.00	C12—C13—H13A	109.00
C6—C1—H1	120.00	C12—C13—H13B	109.00
C1—C2—H2	120.00	C14—C13—H13A	109.00
C3—C2—H2	120.00	C14—C13—H13B	110.00
C2—C3—H3	120.00	H13A—C13—H13B	108.00
C4—C3—H3	120.00	N1—C14—H14A	109.00
C3—C4—H4	120.00	N1—C14—H14B	109.00
C5—C4—H4	120.00	C13—C14—H14A	109.00

C4—C5—H5	120.00	C13—C14—H14B	109.00
C6—C5—H5	120.00	H14A—C14—H14B	108.00
C7—C8—H8A	110.00		

C10—N1—C9—C8	69.9 (2)	C4—C5—C6—C7	180.0 (2)
C14—N1—C9—C8	−166.55 (18)	C1—C6—C7—O1	−178.0 (2)
C9—N1—C10—C11	177.56 (15)	C1—C6—C7—C8	1.9 (3)
C14—N1—C10—C11	55.3 (2)	C5—C6—C7—O1	2.4 (4)
C9—N1—C14—C13	179.67 (18)	C5—C6—C7—C8	−177.6 (2)
C10—N1—C14—C13	−56.2 (2)	O1—C7—C8—C9	−4.6 (3)
C6—C1—C2—C3	−1.4 (4)	C6—C7—C8—C9	175.5 (2)
C2—C1—C6—C5	0.9 (4)	C7—C8—C9—N1	176.49 (18)
C2—C1—C6—C7	−178.7 (2)	N1—C10—C11—C12	−56.2 (2)
C1—C2—C3—C4	0.7 (4)	C10—C11—C12—C13	56.4 (2)
C2—C3—C4—C5	0.6 (4)	C11—C12—C13—C14	−56.6 (2)
C3—C4—C5—C6	−1.2 (4)	C12—C13—C14—N1	57.1 (2)
C4—C5—C6—C1	0.4 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H11···Cl1^{i}	0.95 (2)	2.15 (2)	3.0837 (18)	171 (2)
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Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) *x*−1, −*y*+1/2, *z*−1/2; (iii) −*x*, −*y*+1, −*z*.