

Craig Williamson, John M. D.
Storey and William T. A.
Harrison*Department of Chemistry, University of
Aberdeen, Meston Walk, Aberdeen AB24
3UE, ScotlandCorrespondence e-mail:
w.harrison@abdn.ac.uk

Key indicators

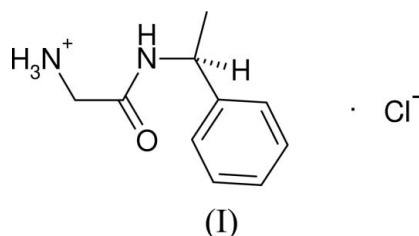
Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.042
 wR factor = 0.127
Data-to-parameter ratio = 13.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**{*N*-[(*S*)-1-Phenylethyl]carbamoyl}methylaminium
chloride**In the title compound, $\text{C}_{10}\text{H}_{15}\text{N}_2\text{O}^+\cdot\text{Cl}^-$, the crystal packing is
influenced by $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds,
resulting in a layered structure.

Received 24 August 2005

Accepted 26 August 2005

Online 7 September 2005

Comment

The known title compound, (I) (Fig. 1), was prepared as an
intermediate in the syntheses of new asymmetric catalysts,
following the literature procedure of Ho *et al.* (2001).

All the geometrical parameters for (I) lie within their expected ranges (Allen *et al.*, 1995). The absolute configuration of (I) is well defined and atom C7 has *S* configuration, as expected from the configuration of the equivalent C atom in the (*S*)-1-phenylethylamine starting material. The dihedral angle between the mean planes of the benzene ring (atoms C1–C6) and the C7/C9/C10/N1/O1 grouping is $66.14(13)^\circ$.

The crystal packing in (I) is influenced by hydrogen bonds (Table 1). An $\text{N}-\text{H}\cdots\text{O}$ bond arising from the N1 group links the cations into chains propagating in the *a* direction. The $-\text{NH}_3$ group participates in three $\text{N}-\text{H}\cdots\text{Cl}$ bonds [mean $\text{H}\cdots\text{Cl} = 2.32$ Å, mean $\text{N}\cdots\text{Cl} = 3.183(3)$ Å, mean $\text{N}-\text{H}\cdots\text{Cl} = 159^\circ$], which crosslink the [100] stacks in the *b* direction. The only intermolecular interactions in the *c*

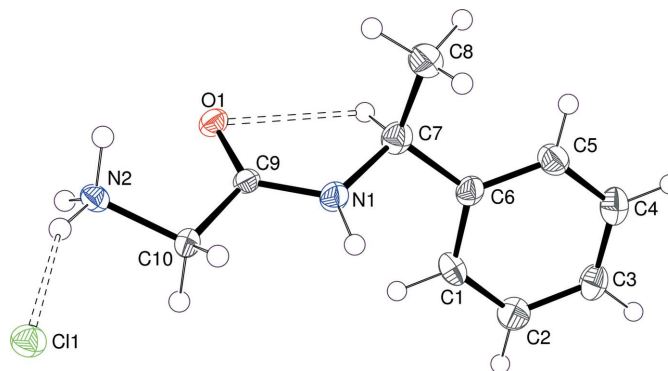


Figure 1

View of (I) (50% probability displacement ellipsoids; H atoms are drawn as small spheres of arbitrary radii). The $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bond and possible $\text{C}-\text{H}\cdots\text{O}$ interaction are indicated by dashed lines.

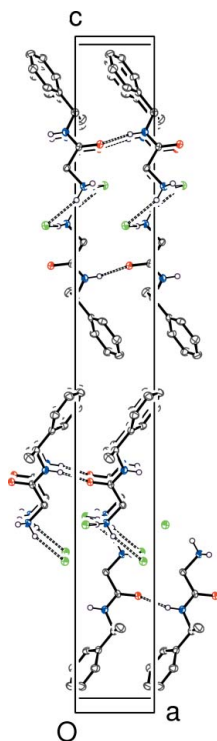


Figure 2
The packing of (I), viewed down [010], with all C-bound H atoms omitted for clarity and hydrogen bonds indicated by dashed lines.

direction are van der Waals forces (Fig. 2). A *PLATON* (Spek, 2003) analysis of (I) flagged a short intramolecular C—H...O distance (Fig. 1 and Table 1), although its structural significance – an attractive interaction or a repulsive steric contact – is not clear.

Experimental

N-Boc glycine (10 mmol, 1.75 g) was dissolved in dry THF (30 ml) in a dry flask under nitrogen. The solution was cooled to 195 K, and *N*-methyl morpholine (10 mmol, 1.01 g, 1.09 ml) was added with stirring. *t*Bu-chloroformate (10 mmol, 1.36 g, 1.30 ml) was added, and the solution stirred for 30 min. (*S*)-1-Phenylethylamine (10 mmol, 1.21 g, 1.29 ml) was added in one portion and the reaction mixture stirred at room temperature for 18 h. The solvent was removed *in vacuo*. The residue was taken up in EtOAc (30 ml), washed with 10% aqueous Na₂CO₃ (20 ml), 0.1 *M* aqueous HCl (20 ml) and saturated brine (20 ml), then dried over Na₂SO₄ and filtered, and the solvent was removed *in vacuo*. The resulting oil (1.37 g, 4.95 mmol) was dissolved in dry dichloromethane (DCM, 15 ml) and cooled to 273 K. Bubbling excess dry HCl through the reaction medium with stirring for 2 h allowed the collection of the desired product as a white precipitate, which was recrystallized from EtOH/Et₂O (1.09 g, 89%). Slow evaporation of a DCM solution of the purified material produced colourless needles of (I) suitable for diffraction; m.p. 446–449 K. [α]_D = –97.0°, *C* = 0.6 (MeOH); IR (KBr, cm^{–1}): ν_{\max} 3289 (C=O), 2967 (CH), 1660 (C=O), 1561 (C=O); ¹H NMR (250 MHz, CD₃OD): δ_{H} 9.2 (1H, *d*, *J* = 8.0 Hz, NH), 8.2 (3H, *s*, N⁺H₃), 7.3 (5H, *m*, Ph), 4.9 (1H, *q*, *J* = 7.0 Hz, CH), 3.6 (2H, *s*, CH₂), 1.3 (3H, *d*, *J* = 7.0 Hz, CH₃); ¹³C NMR (250 MHz, CD₃OD): δ_{C} 164.9 (C=O), 144.1, 128.3, 126.8, 126.1, 48.5 (CH), 40.1 (CH₂), 22.6 (CH₃); MS (ESI⁺):

calculated *m/z* 179.1179; found 179.1180 [M—Cl]⁺; (ESI[–]) 35.4 and 37.4 [Cl][–].

Crystal data

C₁₀H₁₅N₂O⁺·Cl[–]
M_r = 214.69
 Orthorhombic, *P*2₁2₁2₁
a = 4.6309 (3) Å
b = 5.8963 (4) Å
c = 39.939 (3) Å
V = 1090.54 (13) Å³
Z = 4
D_x = 1.308 Mg m^{–3}

Mo K α radiation
 Cell parameters from 856 reflections
 θ = 2.9–27.5°
 μ = 0.32 mm^{–1}
T = 120 (2) K
 Block cut from needle, colourless
 0.30 × 0.24 × 0.16 mm

Data collection

Nonius KappaCCD diffractometer
 ω and φ scans
 Absorption correction: multi-scan
 (*SADABS*; Bruker, 2003)
 T_{\min} = 0.910, T_{\max} = 0.951
 3682 measured reflections
 1768 independent reflections

1578 reflections with $I > 2\sigma(I)$
 R_{int} = 0.030
 θ_{\max} = 26.0°
 h = –5 → 5
 k = –6 → 7
 l = –48 → 48

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)]$ = 0.042
 $wR(F^2)$ = 0.127
 S = 1.14
 1768 reflections
 129 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0431P)^2 + 1.5926P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max}$ = 0.001
 $\Delta\rho_{\max}$ = 0.41 e Å^{–3}
 $\Delta\rho_{\min}$ = –0.39 e Å^{–3}
 Absolute structure: Flack (1983),
 439 Friedel pairs
 Flack parameter: 0.08 (13)

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1A...O1 ⁱ	0.88	2.01	2.839 (4)	156
N2—H2A...Cl1	0.91	2.32	3.181 (3)	157
N2—H2B...Cl1 ⁱⁱ	0.91	2.27	3.146 (3)	162
N2—H2C...Cl1 ⁱⁱⁱ	0.91	2.36	3.222 (3)	158
C7—H7...O1	1.00	2.45	2.809 (5)	101

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

All H atoms were placed in calculated positions (C—H = 0.95–0.99 Å and N—H = 0.88–0.91 Å) and refined as riding on their carrier atoms, allowing for rotation of the rigid terminal –XH₃ groups. The constraint $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl carrier})$ was applied as applicable.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor 1997), *SCALEPACK* and *SORTAV* (Blessing 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors thank the EPSRC National Mass Spectrometry Service (University of Swansea) and the EPSRC National Crystallography Service (University of Southampton) for data collections.

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1995). *International Tables for Crystallography*, Vol. C, edited by A. J. C. Wilson, pp. 685–706. Dordrecht: Kluwer.

- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Bruker (2003). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Ho, B., Crider, M. & Stables, J. P. (2001). *Eur. J. Med. Chem.* **36**, 265–286.
- Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

supporting information

Acta Cryst. (2005). E61, o3133–o3135 [doi:10.1107/S1600536805027182]

{*N*-[(*S*)-1-Phenylethyl]carbamoyl}methylaminium chloride**Craig Williamson, John M. D. Storey and William T. A. Harrison****S1. Comment**

The known title compound, (I) (Fig. 1), was prepared as an intermediate in the syntheses of new asymmetric catalysts, following the literature procedure of Ho *et al.* (2001).

All the geometrical parameters for (I) lie within their expected ranges (Allen *et al.*, 1995). The absolute structure of (I) is well defined and atom C7 has *S* conformation, as expected from the conformation of the equivalent C atom in the (*S*)-1-phenylethylamine starting material. The dihedral angle between the best planes of the benzene ring (atoms C1–C6) and the C7/C9/C10/N1/O1 grouping is 66.14 (13)°.

The crystal packing in (I) is influenced by hydrogen bonds (Table 1). An N—H···O bond arising from the N1 group links the molecules into chains propagating in the *a* direction. The —NH₃ group participates in three N—H···Cl bonds [mean H···Cl = 2.32 Å, mean N···Cl = 3.183 (3) Å, mean N—H···Cl = 159°], which crosslink the [100] stacks in the *b* direction. The only intermolecular interactions in the *c* direction are van der Waals forces (Fig. 2). A *PLATON* (Spek, 2003) analysis of (I) flagged a short intramolecular C—H···O distance (Fig. 1 and Table 1), although its structural significance – an attractive interaction or a repulsive steric contact – is not clear.

S2. Experimental

N-Boc glycine (10 mmol, 1.75 g) was dissolved in dry THF (30 ml) in a dry flask under nitrogen. The solution was cooled to 195 K, and *N*-methyl morpholine (10 mmol, 1.01 g, 1.09 ml) was added with stirring. *i*Bu-chloroformate (10 mmol, 1.36 g, 1.30 ml) was added, and the solution stirred for 30 min. (*S*)-1-Phenylethylamine (10 mmol, 1.21 g, 1.29 ml) was added in one portion and the reaction stirred at room temperature for 18 h. The solvent was removed *in vacuo*. The residue was taken up in EtOAc (30 ml), washed with 10% aqueous Na₂CO₃ (20 ml), 0.1 *M* aqueous HCl (20 ml) and saturated brine (20 ml), then dried over Na₂SO₄ and filtered, and the solvent was removed *in vacuo*. The resulting oil (1.37 g, 4.95 mmol) was dissolved in dry DCM (15 ml) and cooled to 273 K. Bubbling excess dry HCl through the reaction medium with stirring for 2 h allowed the collection of the desired product as a white precipitate, which was recrystallized from EtOH/Et₂O (1.09 g, 89%). Slow evaporation of a DCM solution of the purified material produced colourless needles of (I) suitable for diffraction; m.p. 446–449 K. [α]_D = –97.0°, *C* = 0.6 (MeOH); IR (KBr, cm^{–1}): ν_{\max} 3289 (C=O), 2967 (CH), 1660 (C=O), 1561 (C=O); ¹H NMR (250 MHz, CD₃OD): δ_{H} 9.2 (1H, *d*, *J* = 8.0 Hz, NH), 8.2 (3H, *s*, N⁺H₃), 7.3 (5H, *m*, Ph), 4.9 (1H, *q*, *J* = 7.0 Hz, CH), 3.6 (2H, *s*, CH₂), 1.3 (3H, *d*, *J* = 7.0 Hz, CH₃); ¹³C NMR (250 MHz, CD₃OD): δ_{C} 164.9 (C=O), 144.1, 128.3, 126.8, 126.1, 48.5 (CH), 40.1 (CH₂), 22.6 (CH₃); MS (ESI⁺): calculated *m/z* 179.1179; found 179.1180 [M—Cl⁺]; (ESI[–]) 35.4 and 37.4 [Cl[–]].

S3. Refinement

All H atoms were placed in calculated positions (C—H = 0.95–0.99 Å and N—H = 0.88–0.91 Å) and refined as riding on their carrier atoms, allowing for rotation of the rigid terminal —XH₃ groups. The constraint $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$ or

$U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl carrier})$ was applied as applicable.

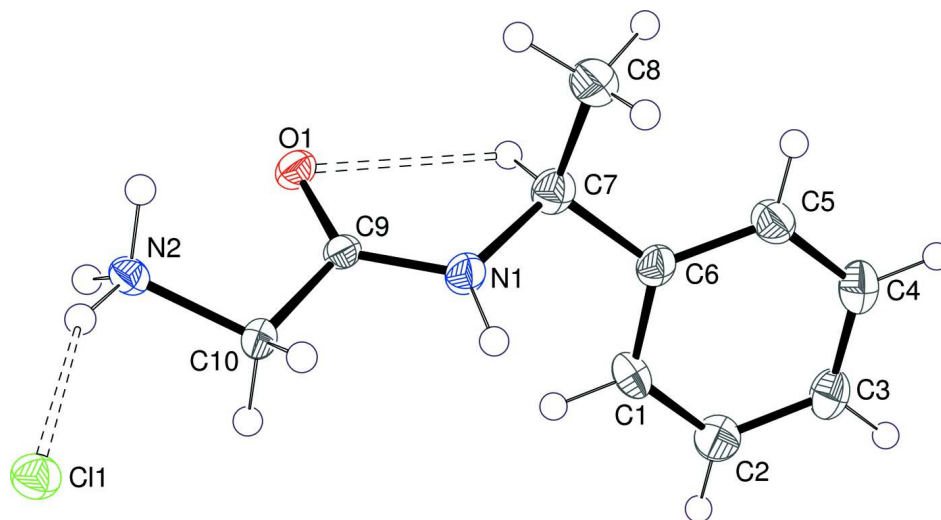


Figure 1

View of (I) (50% probability displacement ellipsoids; H atoms are drawn as small spheres of arbitrary radii). The N—H...Cl hydrogen bond and possible C—H...O interaction are indicated by dashed lines.

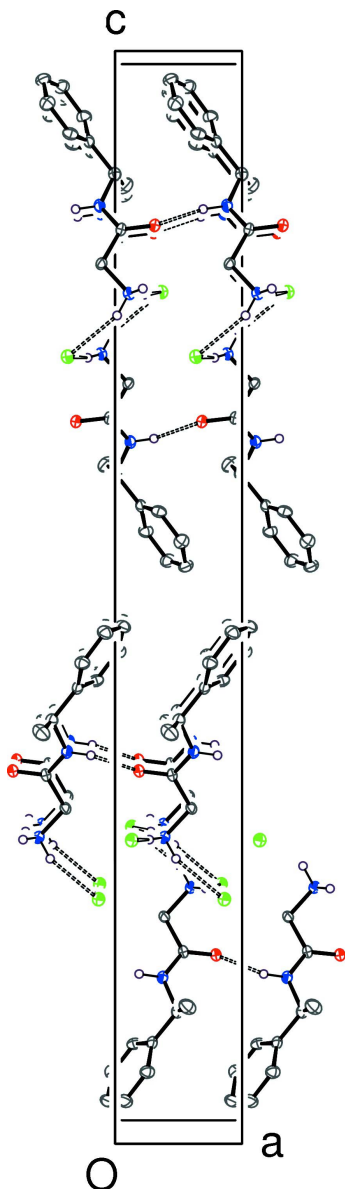


Figure 2

Unit-cell packing in (I), viewed down [010], with all C-bound H atoms omitted for clarity and hydrogen bonds indicated by dashed lines.

{N-[(S)-1-Phenylethyl]carbamoyl}methylaminium chloride

Crystal data

$C_{10}H_{15}N_2O^+ \cdot Cl^-$

$M_r = 214.69$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 4.6309 (3) \text{ \AA}$

$b = 5.8963 (4) \text{ \AA}$

$c = 39.939 (3) \text{ \AA}$

$V = 1090.54 (13) \text{ \AA}^3$

$Z = 4$

$F(000) = 456$

$D_x = 1.308 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 856 reflections

$\theta = 2.9\text{--}27.5^\circ$

$\mu = 0.32 \text{ mm}^{-1}$

$T = 120 \text{ K}$

Block cut from needle, colourless

$0.30 \times 0.24 \times 0.16 \text{ mm}$

Data collection

Nonius KappaCCD diffractometer	3682 measured reflections
Radiation source: fine-focus sealed tube	1768 independent reflections
Graphite monochromator	1578 reflections with $I > 2\sigma(I)$
ω and φ scans	$R_{\text{int}} = 0.030$
Absorption correction: multi-scan (SADABS; Bruker, 2003)	$\theta_{\text{max}} = 26.0^\circ$, $\theta_{\text{min}} = 4.0^\circ$
$T_{\text{min}} = 0.910$, $T_{\text{max}} = 0.951$	$h = -5 \rightarrow 5$
	$k = -6 \rightarrow 7$
	$l = -48 \rightarrow 48$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.042$	$w = 1/[\sigma^2(F_o^2) + (0.0431P)^2 + 1.5926P]$
$wR(F^2) = 0.127$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.14$	$(\Delta/\sigma)_{\text{max}} = 0.001$
1768 reflections	$\Delta\rho_{\text{max}} = 0.41 \text{ e } \text{\AA}^{-3}$
129 parameters	$\Delta\rho_{\text{min}} = -0.39 \text{ e } \text{\AA}^{-3}$
0 restraints	Absolute structure: Flack (1983), 439 Friedel pairs
Primary atom site location: structure-invariant direct methods	Absolute structure parameter: 0.08 (13)
Secondary atom site location: difference Fourier map	

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3476 (8)	0.2857 (6)	0.57786 (9)	0.0241 (8)
H1	0.3220	0.1673	0.5937	0.029*
C2	0.5263 (9)	0.2492 (7)	0.55044 (10)	0.0282 (9)
H2	0.6220	0.1079	0.5477	0.034*
C3	0.5636 (9)	0.4212 (7)	0.52715 (10)	0.0286 (9)
H3	0.6860	0.3984	0.5084	0.034*
C4	0.4228 (9)	0.6256 (7)	0.53132 (10)	0.0303 (10)
H4	0.4475	0.7432	0.5153	0.036*
C5	0.2449 (8)	0.6596 (7)	0.55885 (9)	0.0263 (9)
H5	0.1485	0.8007	0.5614	0.032*
C6	0.2054 (8)	0.4904 (7)	0.58274 (9)	0.0222 (8)
C7	0.0020 (9)	0.5204 (7)	0.61243 (10)	0.0247 (9)
H7	-0.1827	0.4417	0.6067	0.030*
C8	-0.0733 (10)	0.7653 (7)	0.62115 (11)	0.0368 (11)
H8A	-0.2023	0.7676	0.6406	0.055*

H8B	-0.1699	0.8368	0.6020	0.055*
H8C	0.1039	0.8489	0.6264	0.055*
C9	-0.0481 (7)	0.3063 (6)	0.66460 (8)	0.0167 (7)
C10	0.1047 (7)	0.2278 (6)	0.69611 (8)	0.0192 (7)
H10A	0.2041	0.0819	0.6919	0.023*
H10B	0.2515	0.3410	0.7028	0.023*
N1	0.1207 (7)	0.4076 (5)	0.64230 (7)	0.0219 (7)
H1A	0.3087	0.4073	0.6454	0.026*
N2	-0.1105 (6)	0.1991 (5)	0.72335 (7)	0.0174 (6)
H2A	-0.0173	0.1859	0.7433	0.021*
H2B	-0.2168	0.0720	0.7195	0.021*
H2C	-0.2293	0.3220	0.7239	0.021*
O1	-0.3109 (5)	0.2796 (5)	0.66108 (6)	0.0206 (6)
Cl1	0.36806 (18)	0.20605 (14)	0.78047 (2)	0.0210 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.032 (2)	0.0212 (17)	0.0196 (16)	-0.001 (2)	-0.0033 (16)	0.0040 (15)
C2	0.031 (2)	0.024 (2)	0.030 (2)	-0.0001 (17)	0.0023 (17)	-0.0045 (17)
C3	0.030 (2)	0.035 (2)	0.0206 (17)	-0.0040 (19)	0.0050 (17)	0.0005 (18)
C4	0.033 (2)	0.033 (2)	0.0248 (19)	-0.0061 (19)	0.0027 (18)	0.0046 (18)
C5	0.030 (2)	0.025 (2)	0.0239 (18)	0.0029 (17)	-0.0003 (16)	0.0030 (16)
C6	0.0155 (18)	0.028 (2)	0.0230 (17)	-0.0033 (16)	-0.0024 (15)	0.0032 (17)
C7	0.021 (2)	0.028 (2)	0.0247 (18)	-0.0027 (17)	-0.0014 (16)	0.0001 (18)
C8	0.040 (2)	0.036 (3)	0.035 (2)	0.012 (2)	0.010 (2)	0.011 (2)
C9	0.0138 (16)	0.0141 (16)	0.0220 (17)	0.0002 (15)	0.0023 (14)	-0.0001 (17)
C10	0.0163 (16)	0.0221 (18)	0.0193 (16)	-0.0005 (16)	0.0026 (14)	0.0041 (15)
N1	0.0154 (15)	0.0284 (16)	0.0219 (14)	-0.0011 (15)	-0.0013 (13)	0.0044 (13)
N2	0.0175 (13)	0.0143 (13)	0.0202 (13)	-0.0001 (15)	-0.0020 (12)	0.0020 (13)
O1	0.0152 (12)	0.0262 (13)	0.0204 (12)	0.0005 (11)	0.0010 (10)	0.0003 (12)
Cl1	0.0212 (4)	0.0177 (4)	0.0241 (4)	0.0013 (4)	-0.0005 (4)	0.0003 (4)

Geometric parameters (Å, °)

C1—C6	1.389 (5)	C7—H7	1.0000
C1—C2	1.390 (5)	C8—H8A	0.9800
C1—H1	0.9500	C8—H8B	0.9800
C2—C3	1.387 (6)	C8—H8C	0.9800
C2—H2	0.9500	C9—O1	1.235 (4)
C3—C4	1.380 (6)	C9—N1	1.327 (4)
C3—H3	0.9500	C9—C10	1.516 (5)
C4—C5	1.389 (5)	C10—N2	1.485 (4)
C4—H4	0.9500	C10—H10A	0.9900
C5—C6	1.393 (5)	C10—H10B	0.9900
C5—H5	0.9500	N1—H1A	0.8800
C6—C7	1.524 (5)	N2—H2A	0.9100
C7—N1	1.472 (5)	N2—H2B	0.9100

C7—C8	1.526 (6)	N2—H2C	0.9100
C6—C1—C2	121.9 (4)	C7—C8—H8A	109.5
C6—C1—H1	119.1	C7—C8—H8B	109.5
C2—C1—H1	119.1	H8A—C8—H8B	109.5
C3—C2—C1	119.3 (4)	C7—C8—H8C	109.5
C3—C2—H2	120.3	H8A—C8—H8C	109.5
C1—C2—H2	120.3	H8B—C8—H8C	109.5
C4—C3—C2	119.9 (4)	O1—C9—N1	124.1 (3)
C4—C3—H3	120.0	O1—C9—C10	121.0 (3)
C2—C3—H3	120.0	N1—C9—C10	114.8 (3)
C3—C4—C5	120.1 (4)	N2—C10—C9	109.2 (3)
C3—C4—H4	119.9	N2—C10—H10A	109.8
C5—C4—H4	119.9	C9—C10—H10A	109.8
C4—C5—C6	121.1 (4)	N2—C10—H10B	109.8
C4—C5—H5	119.4	C9—C10—H10B	109.8
C6—C5—H5	119.4	H10A—C10—H10B	108.3
C1—C6—C5	117.7 (3)	C9—N1—C7	121.8 (3)
C1—C6—C7	120.2 (4)	C9—N1—H1A	119.1
C5—C6—C7	122.1 (4)	C7—N1—H1A	119.1
N1—C7—C6	110.3 (3)	C10—N2—H2A	109.5
N1—C7—C8	109.1 (3)	C10—N2—H2B	109.5
C6—C7—C8	115.4 (3)	H2A—N2—H2B	109.5
N1—C7—H7	107.2	C10—N2—H2C	109.5
C6—C7—H7	107.2	H2A—N2—H2C	109.5
C8—C7—H7	107.2	H2B—N2—H2C	109.5

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots O1 ⁱ	0.88	2.01	2.839 (4)	156
N2—H2A \cdots C11	0.91	2.32	3.181 (3)	157
N2—H2B \cdots C11 ⁱⁱ	0.91	2.27	3.146 (3)	162
N2—H2C \cdots C11 ⁱⁱⁱ	0.91	2.36	3.222 (3)	158
C7—H7 \cdots O1	1.00	2.45	2.809 (5)	101

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