

Crystal structure of 1-butyl-3-[2-[(indan-5-yl)-amino]-2-oxoethyl]-1*H*-imidazol-3-ium chloride

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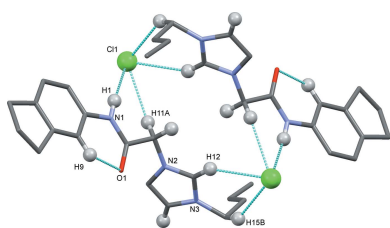
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In the cation of the title molecular salt, C₁₈H₂₄N₃O⁺·Cl[−], an intramolecular C—H···O hydrogen bond stabilizes the almost coplanar orientation of the aromatic ring of the indane unit and the amide plane. In the crystal, the packing is dominated by intermolecular C—H···Cl hydrogen-bonding interactions that result in the formation of slab-like structures propagating along [010]. The slabs are linked by weak C—H···O interactions, forming layers lying parallel to (100). The methylene carbon atom of the indanyl substituent is disordered over two positions with a refined occupancy ratio of 0.84 (2):0.16 (2). The crystal studied was refined as a twin with matrix [1 0 0.9, 0 $\bar{1}$ 0, 0 0 $\bar{1}$]; the resulting BASF value is 0.30.

1. Chemical context

N-Heterocyclic carbenes (NHCs) are neutral compounds in which a 6e[−]-containing divalent carbon atom is placed between two hetero atoms. They are typically derived from their parent imidazolium salts by deprotonation of the carbon atom located in between the two nitrogen atoms (Bhatia *et al.*, 2013). The high reactivity in the case of carbenes can be attributed to the presence of an incomplete octet resulting in a strong electron-donating ability (Hopkinson *et al.*, 2014). Arduengo was the first to successfully isolate a free carbene and characterize it by obtaining a single crystal X-ray structure for the same. This study opened a new era in organic chemistry allowing the investigation of the so-called NHCs as ligands (Arduengo *et al.*, 1991). To date, a tremendous amount of research on NHCs has enhanced the popularity of these carbene compounds in organic synthesis, organometallic chemistry, organocatalysis, medicinal and pharmaceutical applications and essentially every discipline of modern day science. Over the past two decades, *N*-heterocyclic carbene (NHC) ligands have been among the most exploited in organic synthesis. They can be considered superior to phosphine ligands as their electronic and steric properties can be easily fine-tuned by simple variations in their structures (Díez-González *et al.*, 2009; Hermann, 2002; Froese *et al.*, 2017). Attempts have been made to tune or modify the electronic and steric properties of NHCs by changing the substituent at one or both nitrogen centres. These changes in electronics and steric properties may further provide subtle information about the mechanism of catalytic transformations (Huynh, 2018; Peris, 2018). Although the term hemilability for a coordinated ligand was first introduced in 1979 (Jeffrey & Rauchfuss,



1979), the first hemilabile NHC ligand was developed some twenty years later (McGuinness & Cavell, 2000). The presence of hemilabile coordination sites in a ligand system plays a crucial role in catalysis as well as in biological sciences (cytotoxicity). The modular electronic and steric properties of the hemilabile ligand systems provide extra stability to transition metal complexes (Peris, 2018; Normand & Cavell, 2008). Herein we present the synthesis and crystal structure of the chloride salt of the potentially hemilabile amido-functionalized NHC ligand precursor, 1-butyl-3-{2-[(indan-5-yl)amino]-2-oxoethyl}-1*H*-imidazol-3-ium.



2. Structural commentary

The title compound, consists of a chloride anion and an *N*-substituted imidazolium cation, combining the NHC precursor moiety with an amide ($-\text{NH}-\text{C}(\text{O})-\text{CH}_2-$) moiety. The amide group is linked to one nitrogen of the imidazolium ring, N2, by a methylene group, and bears on its opposite side an indanyl substituent bound to the amide nitrogen atom N1. The other (non-amidic) substituent on the second nitrogen atom, N3, of the imidazolium ring is an extended *n*-butyl chain, whose mean plane (C15–C18) is inclined to the plane of the imidazolium ring (N2/N3/C12–C14) by $73.2(6)^\circ$. The central $-\text{CH}_2-\text{C}$ atom, C1, of the indanyl substituent is disordered over two positions with a refined occupancy ratio of C1:C1' = 0.84(2):0.16(2). Atom C1 resides $0.393(12)$ Å below the plane (on the opposite side of the imidazolium moiety) of the four planar C atoms of the pentene ring (C2–C5), while atom C1' is $0.40(4)$ Å above this plane (*i.e.* on the same side as the imidazolium moiety).

Crystallographic data of NHC precursor cations substituted by $\text{CH}_2-\text{C}(\text{O})-\text{NH}$ functional groups (amides) are relatively scarce. A search of the Cambridge Structural Database (CSD, version 3.59, August 2018; Groom *et al.*, 2016) yielded only 16 hits. Compared to published values of imidazolium salts with amide substituents, the geometrical parameters of the title compound are decidedly unexceptional, falling within the reported ranges. Only the C–C bond between methylene atom C11 and the carbonyl carbon C10 is relatively short [C10–C11 = $1.506(6)$ Å] and thereby close to the shortest such bond reported to date, *i.e.* 1.502 Å for a related compound with no substituent on the amide and a dodecyl chain on the other side of the imidazolium cation (Lee *et al.*, 2003*a*). In general, all bond lengths of the two moieties and the methylene linker are in rather close ranges with the largest differences observed being those which lead to further substituents. These are the N–C bond of the amide to its substituent on N ranging from *ca.* 1.409 Å for a phenyl (Samantaray *et al.*, 2007) to 1.482 Å for a *t*-butyl (Ray *et al.*, 2007), and the N–C bond of the imidazolium ring to the

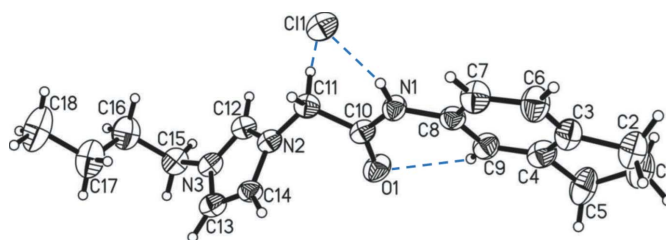


Figure 1

Molecular structure of the title molecular salt, with the atom labelling and displacement ellipsoids drawn at the 50% probability level. In this and subsequent figures, only the major component of the disordered atom C1 is shown. The hydrogen bonds (Table 1) are shown as blue dashed lines.

second substituent ranging from *ca.* 1.422 Å for a pyrimidyl (Lee *et al.*, 2009) to 1.483 Å for a rather bulky 3,5-di-*tert*-butyl-2-hydroxybenzyl (Wan & Zhang, 2016). Given the variety of the substituents on both sides in published structures, this observation is not surprising as the potential extension of the π system beyond the imidazolium and amide moieties would be expected to have a considerable influence on these bond lengths. Strictly within the imidazolium and amide moieties, the strongest deviation is found for the amide C(O)–N bond [here C10–N1 = $1.339(6)$ Å] ranging from *ca.* 1.301 Å for an unsubstituted amide, *i.e.* $-\text{C}(\text{O})-\text{NH}_2$, (Lee *et al.*, 2003*b*) to 1.355 Å for a phenyl-substituted amide (Lee & Zeng, 2012). A shorter C10–N1 bond is indicative of a strong tautomeric effect, *i.e.* C=O double-bond delocalization towards a C=N double bond. In the title compound, the nitrogen atom of the amide is bound to an indanyl group and the C10–N1 bond length of $1.339(6)$ Å comprises a rather average value for a $-\text{C}(\text{O})-\text{NH}-$ bond. A value that often varies in such compounds is the angle at which the plane of the amide moiety [$-\text{CH}_2-\text{C}(\text{O})-\text{NH}$, calculated without H-atom positions] is arranged with respect to the imidazolium ring plane (C₃N₂). Here the dihedral angles range from *ca.* 42.64° (Lee *et al.*, 2003*b*) to 85.95° (Lee *et al.*, 2012). In the title compound, this dihedral angle is $71.9(3)^\circ$. At this angle, resonance between the two moieties (amide and imidazolium) can clearly be excluded. In contrast, the angle between the amide moiety and the aromatic ring of the indanyl substituent is only $18.1(2)^\circ$, suggesting together with the N1–C8 bond length of only $1.427(6)$ Å, that the resonance of the aromatic ring extends to the amide moiety and/or *vice versa*. This relative orientation of the two aromatic systems is probably supported by a weak intramolecular C–H \cdots O hydrogen bond, between the amide oxygen atom (O1) and the aromatic carbon atom C9 (Table 1 and Fig. 1).

3. Supramolecular features

The comparably large unit cell of the crystal structure with $Z = 8$ is rather thin with a short *b* axis of $5.3986(11)$ Å, and the eight imidazolium cations are arranged in a single layer within the cell. The chloride anions and imidazolium cations form symmetric pairs, two-by-two, supported by C–H \cdots Cl hydrogen bonds involving hydrogen atoms of an *n*-butyl

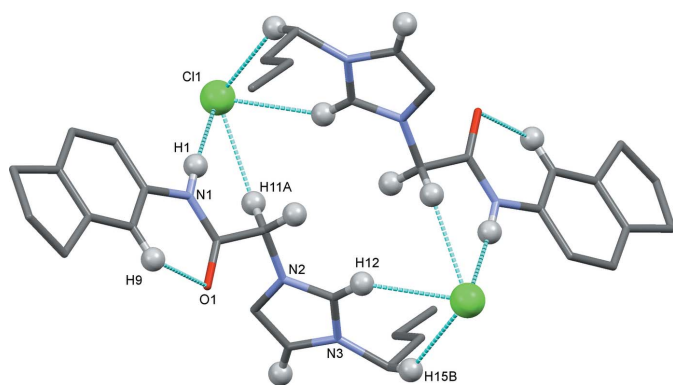


Figure 2

A view of the two-by-two hydrogen-bonded unit (dashed lines; see Table 1 for details). Only the H atoms (grey balls) involved in the intra- and intermolecular interactions have been included. The unlabelled atoms are related to the labelled atoms by the symmetry operation $-x + \frac{1}{2}, -y + \frac{3}{2}, -z$.

methylene C atom (C15–H15B), an imidazolium C atom (C12–H12), the amide N atom (N1–H1) and a C atom of the methylene linker (C11–H11A), and the chloride anion Cl1 (Table 1 and Fig. 2). In the crystal, these two-by-two units are linked by C11–H11B...Cl1ⁱⁱⁱ hydrogen bonds, forming slab-like structures propagating along the *b*-axis direction (Table 1 and Fig. 3). Weak C13–H13...O1ⁱⁱⁱ interactions link the slabs to form layers lying parallel to the *bc* plane (Table 1 and Fig. 3)

4. Synthesis and crystallization

The title compound, was synthesized by the simple reaction of *n*-butyl imidazole with 2-chloro-*N*-(indan-5-yl)acetamide in dry acetonitrile as solvent. All reagents and solvents required for the synthesis were purchased commercially and used without any further purification.

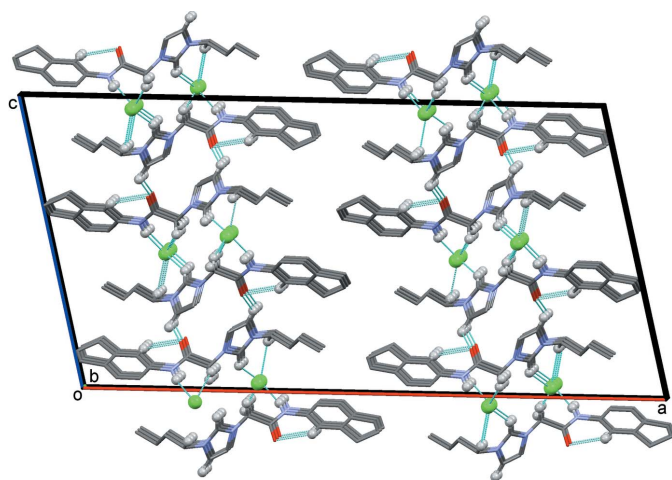


Figure 3

Crystal packing of the title molecular salt, viewed along the *b* axis, showing the various hydrogen bonds as dashed lines (see Table 1 for details). Only the H atoms (grey balls) involved in these interactions have been included.

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>
C9–H9...O1	0.95	2.35	2.915 (6)	118
N1–H1...CH	0.87 (5)	2.32 (5)	3.177 (4)	167 (4)
C11–H11A...CH ⁱ	0.99	2.75	3.541 (4)	137
C12–H12...CH ⁱ	0.95	2.73	3.530 (4)	143
C15–H15B...CH ⁱ	0.99	2.71	3.493 (5)	136
C11–H11B...CH ⁱⁱ	0.99	2.54	3.432 (5)	150
C13–H13...O1 ⁱⁱⁱ	0.95	2.54	3.066 (5)	115

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

Synthesis of 1-butyl-3-[2-[(indan-5-yl)amino]-2-oxoethyl]-1*H*-imidazol-3-ium chloride:

The synthesis of the imidazolium salt was carried out under a nitrogen atmosphere. An oven-dried Schlenk tube was charged with a stirring bar, 1.00 mmol of 2-chloro-*N*-(indan-5-yl)acetamide, 1.5 mmol of *n*-butyl imidazole, and 2 ml of dry acetonitrile. The reaction mixture was stirred for 12 h at 353 K. After the reaction mixture was allowed to cool to r.t., diethyl ether was added to the reaction mixture upon which the product precipitated leading already to sufficient separation. The precipitate was isolated by carefully decanting off the solvent, then washed with acetone (2 × 5 ml) and hexane (2 × 5 ml), and dried under vacuum. The product was obtained as a colourless (white) solid; yield: 94%. Colourless prismatic crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a solution in ethanol.

¹H NMR (400 MHz, DMSO-*d*₆): δ 10.99 (*s*, 1H), 9.26 (*s*, 1H), 7.79 (*d*, *J* = 10.5 Hz, 2H), 7.50 (*s*, 1H), 7.34 (*d*, *J* = 9.1 Hz, 1H), 7.11 (*d*, *J* = 8.1 Hz, 1H), 5.24 (*s*, 2H), 4.20 (*t*, *J* = 7.1 Hz, 2H), 2.77 (*q*, *J* = 7.7 Hz, 4H), 1.95 (*qui*, *J* = 7.4 Hz, 2H), 1.79–1.70 (*m*, 2H), 1.28–1.18 (*m*, 2H), 0.87 (*t*, *J* = 7.4 Hz, 3H). ¹³C NMR (101 MHz, DMSO-*d*₆): δ 163.8, 144.6, 139.3, 137.8, 137.1, 124.7, 124.4, 122.1, 117.6, 115.7, 51.7, 49.0, 32.9, 32.1, 31.7, 25.5, 19.1, 13.7. Analysis calculated for C₁₇H₂₄ClN₃O: C, 64.76; H, 7.25; N, 12.59. Found: C, 64.59; H, 7.12; N, 12.68. IR: C=O Stretching 1700.23 cm⁻¹.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The N-bound hydrogen atom (H1) was located in a difference-Fourier map and freely refined. The C-bound H atoms were placed in calculated positions and treated as riding: C–H = 0.95–0.99 Å with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C-methyl})$ and $1.2U_{\text{eq}}(\text{C})$ for other H atoms.

The methylene carbon atom C1 of the indanyl substituent is disordered over two positions with a refined occupancy ratio of 0.84 (2):0.16 (2). This disorder was modelled with constraints (SADI for all C–C bonds involving C1, SIMU and DELU). The crystal studied was refined as a twin with matrix [1 0 0.9, 0 1 0, 0 0 1]; the resulting BASF value is 0.30.

Acknowledgements

ARK and CS acknowledge ‘The Alexander von Humboldt Foundation’ for the research cooperation programme, which is

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₁₈ H ₂₄ N ₃ O ⁺ ·Cl ⁻
<i>M_r</i>	333.85
Crystal system, space group	Monoclinic, C2/c
Temperature (K)	170
<i>a</i> , <i>b</i> , <i>c</i> (Å)	36.270 (7), 5.3986 (11), 18.620 (4)
β (°)	103.34 (3)
<i>V</i> (Å ³)	3547.6 (13)
<i>Z</i>	8
Radiation type	Mo Kα
μ (mm ⁻¹)	0.22
Crystal size (mm)	0.35 × 0.27 × 0.13
Data collection	
Diffractometer	Stoe IPDS2T
Absorption correction	Numerical (<i>X-RED32</i> and <i>X-SHAPE</i> ; Stoe & Cie, 2010)
<i>T_{min}</i> , <i>T_{max}</i>	0.561, 0.968
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	12392, 3134, 1953
<i>R_{int}</i>	0.119
(sin θ/λ) _{max} (Å ⁻¹)	0.595
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.066, 0.199, 1.05
No. of reflections	3134
No. of parameters	224
No. of restraints	36
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.44, -0.40

Computer programs: *X-AREA* (Stoe & Cie, 2010), *X-RED32* (Stoe & Cie, 2010), *SHELXT2018* (Sheldrick, 2015a), *XP* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008), *SHELXL2018* (Sheldrick, 2015b), *WinGX* (Farrugia, 2012), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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Crystal structure of 1-butyl-3-{2-[(indan-5-yl)amino]-2-oxoethyl}-1*H*-imidazol-3-ium chloride

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Computing details

Data collection: *X-AREA* (Stoe & Cie, 2010); cell refinement: *X-AREA* (Stoe & Cie, 2010); data reduction: *X-RED32* (Stoe & Cie, 2010); program(s) used to solve structure: *SHELXT2018* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b); molecular graphics: *XP* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2018* (Sheldrick, 2015b), *WinGX* (Farrugia, 2012), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

1-Butyl-3-{2-[(indan-5-yl)amino]-2-oxoethyl}-1*H*-imidazol-3-ium chloride

Crystal data

$C_{18}H_{24}N_3O^+Cl^-$
 $M_r = 333.85$
 Monoclinic, *C2/c*
 $a = 36.270$ (7) Å
 $b = 5.3986$ (11) Å
 $c = 18.620$ (4) Å
 $\beta = 103.34$ (3)°
 $V = 3547.6$ (13) Å³
 $Z = 8$

$F(000) = 1424$
 $D_x = 1.250$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 19866 reflections
 $\theta = 6.6$ – 59.3 °
 $\mu = 0.22$ mm⁻¹
 $T = 170$ K
 Prism, colourless
 $0.35 \times 0.27 \times 0.13$ mm

Data collection

Stoe IPDS2T
 diffractometer
 Radiation source: fine-focus sealed tube
 Detector resolution: 6.67 pixels mm⁻¹
 ω scans
 Absorption correction: numerical
 (X-Red32 and X-Shape; Stoe & Cie, 2010)
 $T_{\min} = 0.561$, $T_{\max} = 0.968$

12392 measured reflections
 3134 independent reflections
 1953 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.119$
 $\theta_{\max} = 25.0$ °, $\theta_{\min} = 3.5$ °
 $h = -37 \rightarrow 42$
 $k = -6 \rightarrow 6$
 $l = -21 \rightarrow 22$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.066$
 $wR(F^2) = 0.199$
 $S = 1.05$
 3134 reflections
 224 parameters

36 restraints
 Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map
 Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.1126P)^2 + 0.3489P]$
where $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.44 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.40 \text{ e } \text{\AA}^{-3}$$

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. Refined as a 2-component twin.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
N1	0.16282 (10)	0.4778 (7)	0.0721 (2)	0.0366 (9)	
N2	0.25866 (10)	0.7006 (6)	0.13822 (19)	0.0336 (8)	
N3	0.31102 (11)	0.8875 (6)	0.1857 (2)	0.0358 (8)	
O1	0.18710 (10)	0.7509 (6)	0.16419 (18)	0.0477 (8)	
C1	0.0069 (2)	0.6310 (17)	0.1297 (7)	0.065 (3)	0.84 (2)
H1A	-0.005777	0.746242	0.090345	0.078*	0.84 (2)
H1B	-0.008010	0.623539	0.167931	0.078*	0.84 (2)
C1'	0.0140 (11)	0.538 (7)	0.163 (2)	0.056 (9)	0.16 (2)
H1'1	0.019558	0.439312	0.209509	0.067*	0.16 (2)
H1'2	-0.009825	0.630514	0.160560	0.067*	0.16 (2)
C2	0.01033 (15)	0.3703 (9)	0.0973 (4)	0.0588 (15)	
H2A	0.007424	0.238929	0.132728	0.071*	
H2B	-0.008830	0.345562	0.050560	0.071*	
C3	0.05004 (14)	0.3727 (9)	0.0845 (3)	0.0479 (12)	
C4	0.07030 (15)	0.5732 (8)	0.1194 (3)	0.0464 (12)	
C5	0.04634 (15)	0.7158 (10)	0.1626 (4)	0.0629 (16)	
H5A	0.053825	0.675188	0.215787	0.075*	
H5B	0.048800	0.896697	0.156268	0.075*	
C6	0.06731 (15)	0.2103 (10)	0.0450 (3)	0.0539 (13)	
H6	0.053560	0.074806	0.019341	0.065*	
C7	0.10502 (14)	0.2478 (8)	0.0434 (3)	0.0489 (12)	
H7	0.117384	0.133484	0.018010	0.059*	
C8	0.12457 (12)	0.4493 (8)	0.0782 (2)	0.0361 (10)	
C9	0.10734 (13)	0.6165 (8)	0.1167 (3)	0.0404 (10)	
H9	0.120702	0.756550	0.140374	0.048*	
C10	0.19013 (13)	0.6220 (7)	0.1116 (2)	0.0351 (10)	
C11	0.22575 (12)	0.6170 (8)	0.0831 (2)	0.0366 (10)	
H11A	0.230239	0.446019	0.067916	0.044*	
H11B	0.222334	0.724566	0.038996	0.044*	
C12	0.28154 (12)	0.8823 (7)	0.1286 (2)	0.0337 (10)	
H12	0.277478	0.991130	0.087397	0.040*	
C13	0.30716 (14)	0.7024 (8)	0.2339 (3)	0.0404 (11)	
H13	0.324273	0.664753	0.279459	0.048*	
C14	0.27447 (12)	0.5842 (7)	0.2044 (2)	0.0346 (10)	

H14	0.264207	0.446855	0.225054	0.042*
C15	0.34367 (14)	1.0541 (8)	0.1924 (3)	0.0448 (11)
H15A	0.355727	1.079649	0.245245	0.054*
H15B	0.334866	1.217060	0.170671	0.054*
C16	0.37271 (15)	0.9480 (10)	0.1533 (4)	0.0598 (15)
H16A	0.359918	0.915740	0.101139	0.072*
H16B	0.392405	1.075129	0.153460	0.072*
C17	0.39176 (17)	0.7154 (11)	0.1855 (4)	0.0678 (17)
H17A	0.372720	0.581383	0.180888	0.081*
H17B	0.403047	0.741053	0.238706	0.081*
C18	0.42267 (19)	0.6376 (13)	0.1468 (5)	0.089 (2)
H18A	0.411994	0.626255	0.093572	0.133*
H18B	0.432805	0.475814	0.165602	0.133*
H18C	0.443086	0.760623	0.156499	0.133*
H1	0.1675 (12)	0.378 (8)	0.039 (3)	0.029 (11)*
Cl1	0.19326 (3)	0.10403 (18)	-0.03275 (7)	0.0429 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.036 (2)	0.037 (2)	0.038 (2)	0.0015 (16)	0.0122 (17)	-0.0060 (18)
N2	0.042 (2)	0.0261 (17)	0.035 (2)	0.0016 (15)	0.0149 (17)	-0.0008 (15)
N3	0.044 (2)	0.0271 (17)	0.040 (2)	-0.0053 (16)	0.0163 (17)	-0.0018 (15)
O1	0.052 (2)	0.0487 (18)	0.047 (2)	-0.0017 (15)	0.0207 (16)	-0.0168 (16)
C1	0.052 (4)	0.058 (5)	0.091 (7)	0.015 (3)	0.027 (4)	-0.003 (5)
C1'	0.043 (15)	0.046 (17)	0.091 (19)	0.016 (14)	0.042 (14)	0.001 (15)
C2	0.049 (3)	0.054 (3)	0.079 (4)	0.001 (2)	0.026 (3)	0.003 (3)
C3	0.044 (3)	0.046 (3)	0.056 (3)	0.002 (2)	0.016 (2)	0.003 (2)
C4	0.055 (3)	0.041 (2)	0.046 (3)	0.009 (2)	0.018 (2)	0.002 (2)
C5	0.055 (3)	0.053 (3)	0.091 (4)	0.010 (3)	0.037 (3)	-0.002 (3)
C6	0.047 (3)	0.048 (3)	0.069 (4)	-0.004 (2)	0.017 (3)	-0.009 (3)
C7	0.051 (3)	0.040 (3)	0.057 (3)	0.000 (2)	0.015 (3)	-0.010 (2)
C8	0.040 (2)	0.036 (2)	0.033 (2)	0.0031 (18)	0.0089 (19)	0.0037 (18)
C9	0.044 (3)	0.037 (2)	0.042 (3)	0.004 (2)	0.014 (2)	-0.001 (2)
C10	0.044 (3)	0.028 (2)	0.036 (2)	0.0026 (19)	0.015 (2)	-0.0012 (19)
C11	0.042 (2)	0.032 (2)	0.036 (2)	-0.0015 (19)	0.0079 (19)	-0.0058 (19)
C12	0.045 (3)	0.0210 (19)	0.037 (2)	-0.0004 (18)	0.012 (2)	-0.0011 (17)
C13	0.054 (3)	0.031 (2)	0.040 (3)	0.003 (2)	0.018 (2)	0.0033 (19)
C14	0.043 (2)	0.031 (2)	0.031 (2)	0.0051 (19)	0.0115 (19)	0.0088 (18)
C15	0.050 (3)	0.033 (2)	0.053 (3)	-0.008 (2)	0.014 (2)	-0.003 (2)
C16	0.052 (3)	0.051 (3)	0.079 (4)	-0.014 (2)	0.019 (3)	-0.003 (3)
C17	0.060 (4)	0.051 (3)	0.097 (5)	-0.005 (3)	0.027 (3)	-0.014 (3)
C18	0.071 (4)	0.074 (4)	0.136 (7)	-0.005 (3)	0.055 (5)	-0.026 (4)
Cl1	0.0575 (7)	0.0305 (5)	0.0449 (7)	0.0024 (5)	0.0200 (5)	-0.0028 (5)

Geometric parameters (Å, °)

N1—C10	1.339 (6)	C2—C3	1.514 (7)
N1—C8	1.427 (6)	C3—C4	1.383 (7)
N2—C12	1.324 (5)	C3—C6	1.384 (7)
N2—C14	1.384 (5)	C4—C9	1.376 (7)
N2—C11	1.455 (6)	C4—C5	1.522 (7)
N3—C12	1.323 (6)	C6—C7	1.390 (7)
N3—C13	1.372 (5)	C7—C8	1.376 (6)
N3—C15	1.469 (6)	C8—C9	1.388 (6)
O1—C10	1.227 (5)	C10—C11	1.506 (6)
C1—C5	1.491 (8)	C13—C14	1.347 (7)
C1—C2	1.547 (8)	C15—C16	1.524 (7)
C1'—C2	1.508 (16)	C16—C17	1.491 (8)
C1'—C5	1.518 (16)	C17—C18	1.525 (8)
C10—N1—C8	129.0 (4)	C1'—C5—C4	102.8 (11)
C12—N2—C14	108.3 (4)	C3—C6—C7	119.2 (5)
C12—N2—C11	124.9 (4)	C8—C7—C6	120.5 (4)
C14—N2—C11	126.2 (3)	C7—C8—C9	120.9 (4)
C12—N3—C13	109.0 (4)	C7—C8—N1	116.9 (4)
C12—N3—C15	124.5 (4)	C9—C8—N1	122.2 (4)
C13—N3—C15	126.3 (4)	C4—C9—C8	117.9 (4)
C5—C1—C2	106.5 (5)	O1—C10—N1	125.3 (4)
C2—C1'—C5	107.1 (13)	O1—C10—C11	122.2 (4)
C1'—C2—C3	102.4 (11)	N1—C10—C11	112.5 (4)
C3—C2—C1	102.6 (5)	N2—C11—C10	112.1 (4)
C4—C3—C6	119.3 (5)	N3—C12—N2	108.8 (4)
C4—C3—C2	110.8 (4)	C14—C13—N3	106.9 (4)
C6—C3—C2	130.0 (5)	C13—C14—N2	107.1 (4)
C9—C4—C3	122.2 (4)	N3—C15—C16	111.2 (4)
C9—C4—C5	128.0 (5)	C17—C16—C15	115.5 (5)
C3—C4—C5	109.7 (4)	C16—C17—C18	111.4 (6)
C1—C5—C4	103.8 (5)		

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>
C9—H9...O1	0.95	2.35	2.915 (6)	118
N1—H1...C11	0.87 (5)	2.32 (5)	3.177 (4)	167 (4)
C11—H11 <i>A</i> ...C11	0.99	2.75	3.541 (4)	137
C12—H12...C11 ⁱ	0.95	2.73	3.530 (4)	143
C15—H15 <i>B</i> ...C11 ⁱ	0.99	2.71	3.493 (5)	136
C11—H11 <i>B</i> ...C11 ⁱⁱ	0.99	2.54	3.432 (5)	150
C13—H13...O1 ⁱⁱⁱ	0.95	2.54	3.066 (5)	115

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