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Crystal structure of ethyl 2-(4-chlorophenyl)-3-cyclopentyl-4-oxo-1-propylimidazolidine-5-carboxylate

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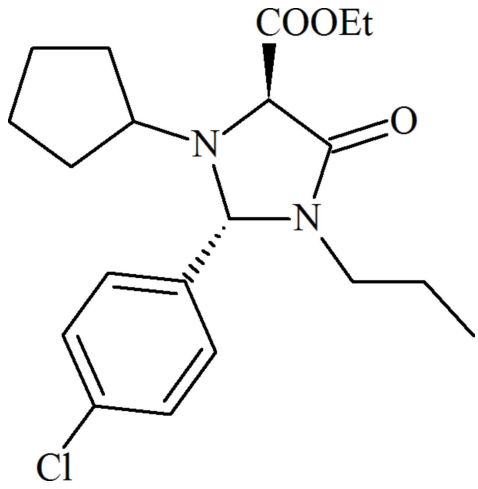
The title compound, $C_{20}H_{27}ClN_2O_3$, was obtained *via* an original synthesis method. The central heterocyclic ring adopts a shallow envelope conformation, with the N atom bearing the cyclopentane ring as the flap [deviation from the other atoms = 0.442 (2) Å]. The cyclopentane ring adopts a twisted conformation about one of the C_N—C bonds: the exocyclic C—N bond adopts an equatorial orientation. The dihedral angles between the central ring (all atoms) and the pendant five- and six-membered rings are 10.3 (2) and 87.76 (14)°, respectively. In the crystal, C—H···O interactions link the molecules into [011] chains. A weak C—H···Cl interaction links the chains into (100) sheets. A mechanism for the cyclization reaction is proposed.

Keywords: crystal structure; synthesis; aziridine rearrangement; C—H···O and C—H···O interactions.

CCDC reference: 1419261

1. Related literature

For background to the biological properties of imidazolidin-4-one rings, see: Chambel *et al.* (2006); Vale *et al.* (2008*a*,*b*,*c*); Gomes *et al.* (2004); Araujo *et al.* (2005); Qin *et al.* (2009). For imidazolidin-4-one rings in Diels–Alder reactions, see: Lin *et al.* (2013). For the synthesis and mechanistic studies, see: Gomes *et al.* (2006); Zhang *et al.* (2008).



2. Experimental

2.1. Crystal data

$C_{20}H_{27}ClN_2O_3$
M_r = 378.89
Triclinic, $P\overline{1}$
a = 9.083 (7) Å
b = 11.201 (6) Å
c = 11.846 (6) Å
α = 117.75 (4)°
β = 90.49 (5)°
γ = 104.08 (6)°
V = 1024.1 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 298 K
0.4 × 0.3 × 0.2 mm

2.2. Data collection

Enraf–Nonius CAD-4 diffractometer
6270 measured reflections
4439 independent reflections
2533 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
2 standard reflections every 120 reflections
intensity decay: 4%

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.180
S = 0.99
4439 reflections
295 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H2···O1^{i}	1.00 (2)	2.50 (3)	3.454 (4)	160 (2)
C3—H12···O3^{ii}	0.99 (4)	2.59 (4)	3.439 (5)	143 (3)
C16—H16*B*···Cl1^{iii}	0.97	2.80	3.662 (6)	148

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

Data collection: *CAD-4 EXPRESS* (Duisenberg, 1992; Macíček & Yordanov, 1992); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7486).

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supporting information

Acta Cryst. (2015). E**71**, o682–o683 [doi:10.1107/S2056989015015364]

Crystal structure of ethyl 2-(4-chlorophenyl)-3-cyclopentyl-4-oxo-1-propyl-imidazolidine-5-carboxylate

Mohamed Ali Tabarki, Youssef Ben Smida, Abderrahmen Guesmi and Rafâa Besbes

S1. comment

The imidazolidin-4-one rings are of major interest and constitute a very important class of heterocyclic compounds because of their presence in several biologically active synthetic products (Chambel *et al.* 2006; Vale *et al.* 2008a) and their use as high antimalarial drugs (Vale *et al.* 2008b&c). These products exhibit also antibacterial activity (Gomes *et al.* 2004; Araujo *et al.* 2005) and inhibit binding of VCAM-1 to VLA-4 (Qin *et al.* 2009). On the other hand, imidazolidnione was used as organocatalyst for Diels-Alder reactions (Lin *et al.*2013).

In the present work we have developed an efficient strategy for the synthesis of 1-cyclopenty-2-parachlorophenyl-3-propyl-5-ethoxycarbonylimidazolidin-4-one (I) (Fig.1) via ring expansion of aziridine-2-carboxylate upon reaction with propylisocyanate. It should be mentioned that in a similar protocol, Gomes *et al.* (2006) report that aziridines rearrange under the effect of heating or radiation and transform into azomethines. The latter reacts subsequently on various electrophiles systems.

A result similar to one described by Zhang *et al.*(2008), but the authors did not explain the formation of the compounds obtained. To explain the formation of the imidazolidin-4-one we based on work that was performed by Gomes *et al.* (2006) and in which the authors suggest that aziridines rearrange under the effect of heating or irradiation and become an azomethine. The latter reacts subsequently on various electrophile systems. In our case, the attack of the isocyanate by the carbanion of azomethine, formed upon the refluxing in toluene aziridine, adequately explains obtaining imidazolidin-4-one after cyclization of the intermediate formed.

S2. Experimental

S2.1. Synthesis and crystallization

To a solution of ethyl 3-(4-chlorophenyl)-1-cyclopentylaziridine-2-carboxylate (2.20 mmol) in toluene (10 ml) under nitrogen atmosphere, were added n-Propylisocyanate (2.64 mmol). The mixture was refluxed during 20 hours. After completeness of the reaction, the mixture was concentrated under reduced pressure and the residue was purified by silica gel column chromatography using a mixture of n-hexane / EtOAc (5:5) as eluent to afford colourless prisms of the studied compound.

S2.2. Refinement

Hydrogen atoms were treated by a mixture of independent and constrained refinement. In fact hydrogen atoms from H1 to H15 were located in the difference Fourier Map. The others H atoms were located geometrically and refined using a riding model.

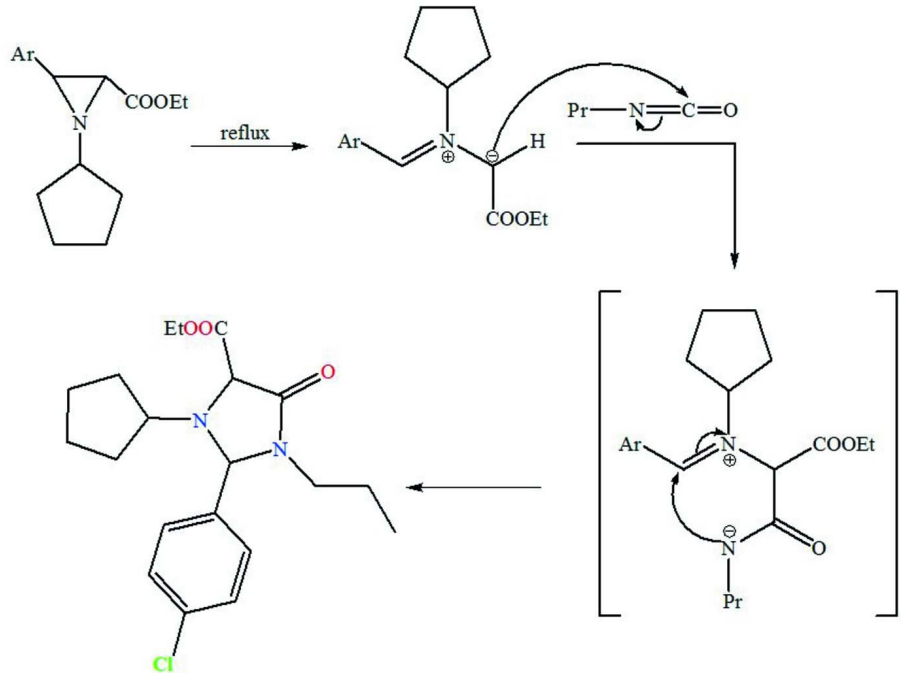

Figure 1

Synthesis protocol of $C_{20}H_{27}ClN_2O_3$.

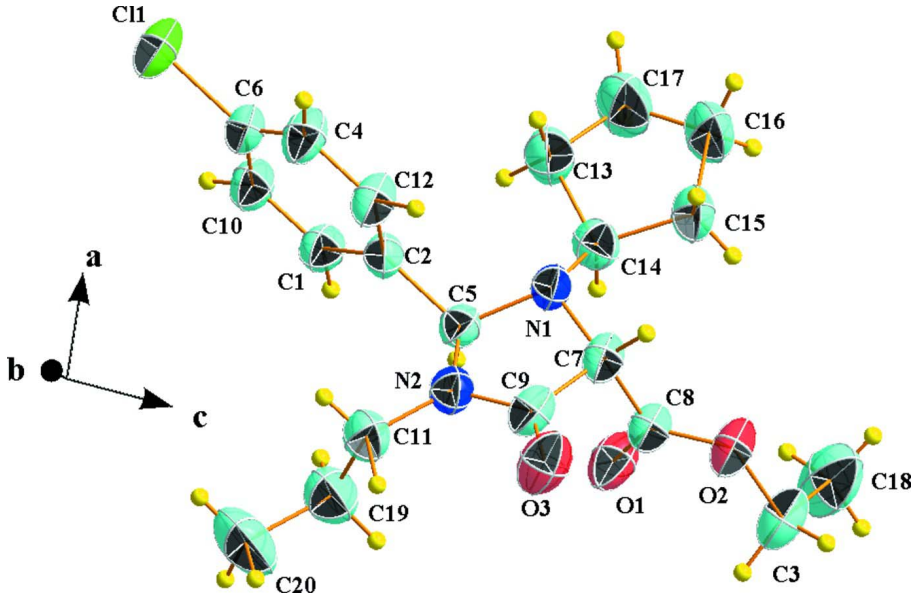

Figure 2

Perspective view of the title compound showing 50% displacement ellipsoids.

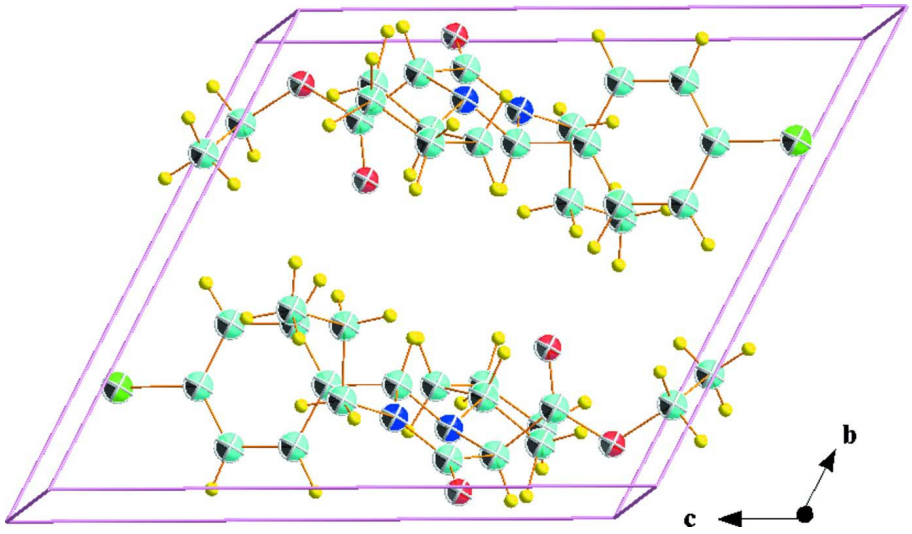

Figure 3

Unit cell projection of $C_{20}H_{27}ClN_2O_3$ showing two molecules per cell.

Ethyl 2-(4-chlorophenyl)-3-cyclopentyl-4-oxo-1-propylimidazolidine-5-carboxylate

Crystal data

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$M_r = 378.89$
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Hall symbol: -P 1
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$c = 11.846\ (6)$ Å
$\alpha = 117.75\ (4)°$
$\beta = 90.49\ (5)°$
$\gamma = 104.08\ (6)°$
$V = 1024.1\ (11)$ Å^3
$Z = 2$
$F(000) = 404$
$D_x = 1.229$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10–15°$
$\mu = 0.21$ mm^{-1}
$T = 298$ K
Prism, colorless
0.4 × 0.3 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
6270 measured reflections
4439 independent reflections
2533 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.1°$
$h = -11\rightarrow3$
$k = -14\rightarrow14$
$l = -15\rightarrow15$
2 standard reflections every 120 reflections
intensity decay: 4%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.180$
$S = 0.99$
4439 reflections
295 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.1064P)^2 + 0.0609P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.043$

$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	1.01028 (10)	0.71959 (8)	0.06769 (7)	0.0791 (3)
O1	0.5260 (2)	0.67479 (19)	0.72389 (18)	0.0698 (6)
O2	0.5879 (2)	0.87245 (18)	0.91481 (16)	0.0650 (5)
O3	0.4728 (3)	0.9785 (2)	0.69803 (19)	0.0759 (6)
N1	0.7774 (2)	0.82492 (19)	0.64046 (17)	0.0461 (5)
N2	0.5635 (2)	0.8158 (2)	0.53131 (18)	0.0502 (5)
C1	0.8327 (3)	0.6004 (3)	0.1915 (2)	0.0570 (7)
C2	0.7634 (3)	0.7335 (2)	0.4011 (2)	0.0441 (5)
C3	0.4957 (4)	0.7979 (4)	0.9770 (3)	0.0732 (9)
C4	0.9202 (4)	0.8525 (3)	0.3002 (2)	0.0606 (7)
C5	0.6794 (3)	0.7399 (2)	0.5138 (2)	0.0442 (5)
C6	0.9138 (3)	0.7244 (3)	0.1961 (2)	0.0538 (6)
C7	0.6788 (3)	0.8889 (2)	0.7346 (2)	0.0503 (6)
C8	0.5893 (3)	0.7974 (3)	0.7891 (2)	0.0502 (6)
C9	0.5592 (3)	0.9044 (2)	0.6558 (2)	0.0527 (6)
C10	0.7574 (3)	0.6052 (2)	0.2946 (2)	0.0507 (6)
C11	0.4526 (4)	0.7868 (3)	0.4255 (3)	0.0635 (7)
C12	0.8435 (3)	0.8565 (3)	0.4024 (2)	0.0559 (7)
C13	1.0021 (4)	0.7211 (4)	0.5907 (3)	0.0706 (8)
C14	0.8731 (3)	0.7538 (3)	0.6745 (2)	0.0555 (6)
C15	0.9604 (4)	0.8461 (4)	0.8118 (3)	0.0717 (8)
C16	1.1058 (4)	0.8014 (6)	0.8072 (4)	0.1159 (15)
H16A	1.1940	0.8836	0.8458	0.139*
H16B	1.1023	0.7511	0.8556	0.139*
C17	1.1201 (4)	0.7121 (5)	0.6750 (4)	0.1030 (12)
H17A	1.1022	0.6159	0.6581	0.124*
H17B	1.2224	0.7432	0.6574	0.124*
C18	0.5848 (5)	0.7287 (5)	1.0162 (4)	0.1136 (14)
H18A	0.5229	0.6808	1.0561	0.170*
H18B	0.6730	0.7974	1.0764	0.170*
H18C	0.6176	0.6619	0.9419	0.170*
C19	0.3480 (4)	0.6381 (3)	0.3616 (3)	0.0785 (9)

H19A	0.4093	0.5724	0.3275	0.094*
H19B	0.2937	0.6217	0.4255	0.094*
C20	0.2353 (5)	0.6120 (6)	0.2556 (4)	0.1300 (17)
H20A	0.1705	0.5173	0.2180	0.195*
H20B	0.2886	0.6256	0.1911	0.195*
H20C	0.1737	0.6762	0.2892	0.195*
H1	0.626 (2)	0.645 (2)	0.4965 (19)	0.032 (5)*
H2	0.697 (3)	0.519 (2)	0.297 (2)	0.041 (6)*
H3	0.734 (3)	0.979 (3)	0.802 (3)	0.067 (8)*
H4	0.392 (4)	0.738 (4)	0.923 (3)	0.096 (11)*
H5	0.826 (3)	0.513 (3)	0.115 (3)	0.073 (8)*
H6	0.972 (3)	0.937 (3)	0.310 (3)	0.073 (9)*
H7	0.806 (3)	0.668 (3)	0.667 (2)	0.051 (6)*
H8	0.497 (4)	0.796 (3)	0.365 (3)	0.082 (10)*
H9	0.952 (4)	0.624 (3)	0.516 (3)	0.091 (10)*
H10	0.842 (3)	0.951 (3)	0.476 (3)	0.075 (8)*
H11	0.908 (4)	0.827 (3)	0.868 (3)	0.090 (10)*
H12	0.470 (4)	0.875 (4)	1.053 (4)	0.098 (11)*
H13	0.375 (4)	0.834 (4)	0.460 (3)	0.107 (12)*
H14	1.051 (4)	0.812 (4)	0.585 (3)	0.100 (11)*
H15	0.982 (4)	0.953 (4)	0.834 (3)	0.091 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0968 (6)	0.0929 (6)	0.0563 (4)	0.0280 (5)	0.0331 (4)	0.0418 (4)
O1	0.0915 (14)	0.0523 (11)	0.0569 (11)	0.0042 (10)	0.0224 (10)	0.0269 (9)
O2	0.0894 (14)	0.0616 (11)	0.0461 (9)	0.0212 (10)	0.0243 (9)	0.0274 (8)
O3	0.0993 (16)	0.0773 (13)	0.0715 (13)	0.0525 (12)	0.0327 (11)	0.0386 (11)
N1	0.0511 (11)	0.0486 (11)	0.0392 (9)	0.0109 (9)	0.0068 (8)	0.0231 (8)
N2	0.0523 (12)	0.0537 (11)	0.0478 (11)	0.0174 (9)	0.0079 (9)	0.0259 (9)
C1	0.0669 (17)	0.0512 (14)	0.0442 (13)	0.0164 (13)	0.0117 (12)	0.0160 (12)
C2	0.0476 (13)	0.0434 (12)	0.0386 (11)	0.0092 (10)	0.0040 (10)	0.0195 (10)
C3	0.098 (3)	0.077 (2)	0.0580 (17)	0.034 (2)	0.0333 (18)	0.0390 (16)
C4	0.082 (2)	0.0509 (15)	0.0496 (14)	0.0093 (14)	0.0154 (13)	0.0292 (13)
C5	0.0466 (13)	0.0408 (12)	0.0431 (12)	0.0079 (10)	0.0092 (10)	0.0206 (10)
C6	0.0582 (15)	0.0662 (16)	0.0408 (12)	0.0165 (12)	0.0115 (11)	0.0291 (12)
C7	0.0661 (16)	0.0399 (12)	0.0402 (12)	0.0088 (11)	0.0116 (11)	0.0185 (10)
C8	0.0585 (15)	0.0484 (14)	0.0449 (12)	0.0150 (12)	0.0134 (11)	0.0232 (11)
C9	0.0648 (16)	0.0467 (13)	0.0522 (14)	0.0180 (12)	0.0199 (12)	0.0268 (11)
C10	0.0552 (15)	0.0440 (13)	0.0485 (13)	0.0104 (11)	0.0102 (11)	0.0205 (11)
C11	0.0627 (18)	0.0772 (19)	0.0590 (17)	0.0239 (16)	0.0066 (14)	0.0375 (16)
C12	0.0757 (18)	0.0411 (13)	0.0445 (13)	0.0083 (12)	0.0145 (12)	0.0192 (11)
C13	0.074 (2)	0.081 (2)	0.0568 (17)	0.0360 (18)	0.0077 (15)	0.0262 (17)
C14	0.0581 (16)	0.0553 (15)	0.0593 (15)	0.0118 (13)	0.0038 (13)	0.0346 (13)
C15	0.073 (2)	0.100 (3)	0.0519 (16)	0.0268 (18)	0.0068 (14)	0.0434 (17)
C16	0.080 (3)	0.208 (5)	0.073 (2)	0.058 (3)	0.0089 (19)	0.070 (3)
C17	0.087 (3)	0.151 (3)	0.083 (2)	0.061 (3)	0.008 (2)	0.053 (2)

C18	0.129 (3)	0.156 (4)	0.120 (3)	0.067 (3)	0.041 (3)	0.104 (3)
C19	0.072 (2)	0.084 (2)	0.0719 (19)	0.0244 (17)	0.0029 (16)	0.0309 (17)
C20	0.087 (3)	0.165 (4)	0.110 (3)	0.025 (3)	−0.030 (2)	0.050 (3)

Geometric parameters (Å, º)

Cl1—C6	1.747 (3)	C11—C19	1.515 (5)
O1—C8	1.197 (3)	C11—H8	0.86 (3)
O2—C8	1.328 (3)	C11—H13	0.96 (4)
O2—C3	1.478 (3)	C12—H10	1.01 (3)
O3—C9	1.222 (3)	C13—C17	1.511 (5)
N1—C7	1.464 (3)	C13—C14	1.543 (4)
N1—C5	1.478 (3)	C13—H9	1.01 (3)
N1—C14	1.478 (3)	C13—H14	1.03 (4)
N2—C9	1.348 (3)	C14—C15	1.535 (4)
N2—C11	1.460 (4)	C14—H7	0.97 (2)
N2—C5	1.466 (3)	C15—C16	1.515 (5)
C1—C6	1.380 (4)	C15—H11	0.90 (3)
C1—C10	1.391 (3)	C15—H15	1.07 (3)
C1—H5	0.96 (3)	C16—C17	1.441 (5)
C2—C12	1.385 (3)	C16—H16A	0.9700
C2—C10	1.390 (3)	C16—H16B	0.9700
C2—C5	1.525 (3)	C17—H17A	0.9700
C3—C18	1.452 (5)	C17—H17B	0.9701
C3—H4	1.01 (4)	C18—H18A	0.9600
C3—H12	0.99 (4)	C18—H18B	0.9600
C4—C6	1.376 (4)	C18—H18C	0.9600
C4—C12	1.390 (4)	C19—C20	1.483 (5)
C4—H6	0.90 (3)	C19—H19A	0.9700
C5—H1	0.97 (2)	C19—H19B	0.9700
C7—C9	1.518 (4)	C20—H20A	0.9600
C7—C8	1.533 (3)	C20—H20B	0.9599
C7—H3	0.95 (3)	C20—H20C	0.9600
C10—H2	1.00 (2)		
C8—O2—C3	116.5 (2)	C2—C12—C4	120.8 (2)
C7—N1—C5	106.60 (19)	C2—C12—H10	119.8 (16)
C7—N1—C14	116.08 (19)	C4—C12—H10	119.3 (16)
C5—N1—C14	115.95 (19)	C17—C13—C14	103.6 (3)
C9—N2—C11	123.2 (2)	C17—C13—H9	109.5 (18)
C9—N2—C5	113.4 (2)	C14—C13—H9	103.7 (19)
C11—N2—C5	123.1 (2)	C17—C13—H14	105 (2)
C6—C1—C10	119.3 (2)	C14—C13—H14	105.6 (19)
C6—C1—H5	118.8 (17)	H9—C13—H14	127 (3)
C10—C1—H5	121.8 (17)	N1—C14—C15	112.0 (2)
C12—C2—C10	119.2 (2)	N1—C14—C13	113.7 (2)
C12—C2—C5	120.1 (2)	C15—C14—C13	103.3 (2)
C10—C2—C5	120.7 (2)	N1—C14—H7	107.9 (14)

C18—C3—O2	110.8 (3)	C15—C14—H7	109.7 (14)
C18—C3—H4	117 (2)	C13—C14—H7	110.2 (14)
O2—C3—H4	111 (2)	C16—C15—C14	104.7 (3)
C18—C3—H12	111 (2)	C16—C15—H11	107 (2)
O2—C3—H12	103 (2)	C14—C15—H11	111 (2)
H4—C3—H12	103 (3)	C16—C15—H15	113.1 (18)
C6—C4—C12	119.1 (2)	C14—C15—H15	107.8 (18)
C6—C4—H6	125.9 (19)	H11—C15—H15	113 (3)
C12—C4—H6	115.0 (19)	C17—C16—C15	109.3 (3)
N2—C5—N1	101.47 (18)	C17—C16—H16A	109.8
N2—C5—C2	110.85 (19)	C15—C16—H16A	109.8
N1—C5—C2	113.85 (19)	C17—C16—H16B	109.8
N2—C5—H1	107.4 (12)	C15—C16—H16B	109.8
N1—C5—H1	113.1 (12)	H16A—C16—H16B	108.3
C2—C5—H1	109.7 (12)	C16—C17—C13	107.5 (3)
C4—C6—C1	121.2 (2)	C16—C17—H17A	110.2
C4—C6—Cl1	119.1 (2)	C13—C17—H17A	110.2
C1—C6—Cl1	119.7 (2)	C16—C17—H17B	110.2
N1—C7—C9	103.22 (19)	C13—C17—H17B	110.2
N1—C7—C8	115.3 (2)	H17A—C17—H17B	108.5
C9—C7—C8	105.8 (2)	C3—C18—H18A	109.5
N1—C7—H3	111.6 (16)	C3—C18—H18B	109.5
C9—C7—H3	109.3 (16)	H18A—C18—H18B	109.5
C8—C7—H3	111.0 (16)	C3—C18—H18C	109.5
O1—C8—O2	125.5 (2)	H18A—C18—H18C	109.5
O1—C8—C7	123.1 (2)	H18B—C18—H18C	109.5
O2—C8—C7	111.4 (2)	C20—C19—C11	111.8 (3)
O3—C9—N2	127.3 (3)	C20—C19—H19A	109.3
O3—C9—C7	126.4 (2)	C11—C19—H19A	109.3
N2—C9—C7	106.2 (2)	C20—C19—H19B	109.3
C2—C10—C1	120.4 (2)	C11—C19—H19B	109.3
C2—C10—H2	116.7 (12)	H19A—C19—H19B	107.9
C1—C10—H2	122.9 (12)	C19—C20—H20A	109.5
N2—C11—C19	112.9 (3)	C19—C20—H20B	109.5
N2—C11—H8	112 (2)	H20A—C20—H20B	109.5
C19—C11—H8	106 (2)	C19—C20—H20C	109.5
N2—C11—H13	109 (2)	H20A—C20—H20C	109.5
C19—C11—H13	97 (2)	H20B—C20—H20C	109.5
H8—C11—H13	119 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H2···O1^{i}	1.00 (2)	2.50 (3)	3.454 (4)	160 (2)
C3—H12···O3^{ii}	0.99 (4)	2.59 (4)	3.439 (5)	143 (3)
C16—H16*B*···Cl1^{iii}	0.97	2.80	3.662 (6)	148

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1, −*y*+2, −*z*+2; (iii) *x*, *y*, *z*+1.