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Crystal structure and Hirshfeld surface analysis of 1-(2-chloroacetyl)-3-methyl-2,6-bis(4-methylphenyl)piperidin-4-one

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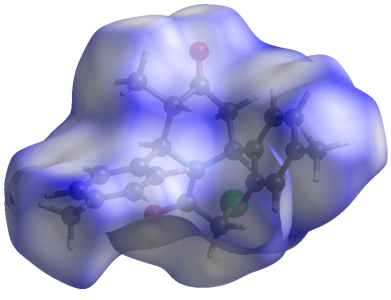
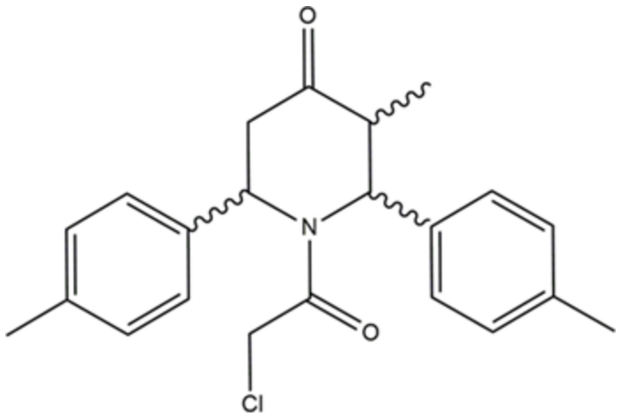
In the crystal structure of the title compound, $C_{22}H_{24}ClNO_2$, the piperidine ring adopts a boat conformation. Intra- and intermolecular C—H···Cl hydrogen bonds are observed. The intermolecular interactions were quantified and analysed using Hirshfeld surface analysis, revealing that H···H interactions contribute most to the crystal packing (56.1%).

1. Chemical context

Piperidin-4-one derivatives represent a significant class of heterocyclic compounds widely documented for their versatility in the field of medicinal chemistry. The piperidin-4-one scaffold serves as a valuable synthetic intermediate and as a promising pharmacophore showing diverse biological activities (Sahu *et al.*, 2013). Among piperidin-4-one derivatives, 3-alkyl-2,6-diarylpiperidin-4-one derivatives have been extensively investigated, particularly with respect to their synthesis, stereochemistry, and diverse biological activities. 3-Alkyl-2,6-diarylpiperidone derivatives predominantly adopt a chair conformation with an equatorial orientation of the alkyl and phenyl substituents (Pandiarajan *et al.*, 1991). The introduction of groups such as –NO, –CHO, $–COCH_3$, and $N—COCH_2Cl$ onto the ring nitrogen atom of a 2,6-diarylpiperidin-4-one moiety significantly alter the ring conformation and the orientation of its substituents. Delocalization of the nitrogen lone pair into the –CO*R* π-system imparts partial double-bond character to the —N—CO linkage, thereby restricting its rotation. The steric interaction between the N—CO group and the neighbouring equatorial substituent causes molecular strain, which is relieved by adopting a chair form with an axial orientation of the phenyl substituents or a boat form with one phenyl substituent in the flagpole position. The effects of such substitutions on the geometry of the piperidin-4-one nucleus have been extensively reported. Structural variations such as *N*-benzoyl (Krishnapillay *et al.*, 2000), *N*-nitroso (Ravindran *et al.*, 1991), *N*-formyl (Pandiarajan *et al.*, 1997), *N*-chloroacetyl (Aridoss *et al.*, 2007*a*,*b*; Divyabharathi *et al.*, 2024) and *N*-thiocyanatoacetyl (Karthiga *et al.*, 2024, 2025) derivatives have all been studied. Furthermore, investigations into the DNA-binding properties of *N*-acetyl analogues (Mohanraj & Ponnuswamy, 2018) and their antibacterial activities (Aridoss *et al.*, 2008) have also been reported. In the present work, crystal structure and Hirshfeld



surface analysis of 1-(2-chloroacetyl)-3-methyl-2,6-bis(4-methylphenyl)piperidin-4-one, are reported.



2. Structural commentary

The molecular structure is presented in Fig. 1. The compound is chiral due to the presence of stereogenic centres. Although the molecular structure depicted in Fig. 1 shows the 2*R*,3*S*,6*S* enantiomer, the crystal contains a racemic mixture of enantiomers. The O1—C3 [1.207 (2) Å] and O2—C6 [1.221 (2) Å] bond lengths confirm the double-bond character. The sum of the angles around atom N1 (357.1°) indicates that nitrogen adopts an almost trigonal–planar geometry. Conjugation between the carbonyl group and the adjacent C—C bond, combined with steric hindrance from the chloromethyl substituent, restricts free rotation about the C6—C7 bond. This limited rotational freedom results in distinct preferred conformations, which is reflected in the observed torsion angles O2—C6—C7—Cl1 [100.5 (2)°] and N1—C6—C7—Cl1 [−80.4 (2)°]. The piperidine ring adopts a boat conformation; the puckering parameters (Cremer & Pople, 1975) are: q_2 = 0.677 (2) Å, q_3 = −0.060 (2) Å, Q_T = 0.680 (2) Å and φ = 107.4 (2)°. Atoms C2 and C5 in the piperidine ring (N1/C1–C5) deviate by −0.528 (2) and −0.604 (2) Å, respectively, from the least-squares plane through the remaining four atoms. The methylphenyl rings C8–C13 and C15–C21 are planar, with their attached methyl atoms C14 and C22 deviate by −0.024 (3) and 0.003 (3) Å, respectively, from their ring planes. These methylphenyl rings are oriented with a dihedral angle of 51.7 (1)° with respect to each other. A weak intramolecular contact (Table 1) between a methine H atom and the Cl atom attached to the 2-chloroacetaldehyde moiety (C6/O2/C7/Cl1) leads to the stabilization of the molecular conformation. This C5—H5···Cl1 interaction forms an *S*(6) ring motif (Bernstein *et al.*, 1995), as shown in Fig. 1.

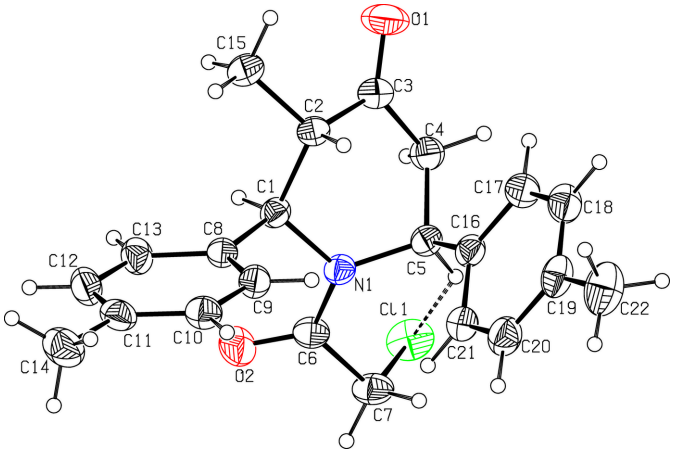


Figure 1
Molecular structure showing the atom-labelling scheme and the intramolecular hydrogen bond (dashed line). Ellipsoids are drawn at the 30% probability level.

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

Cg is the centroid of the C8–C13 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···Cl1	0.98	2.61	3.342 (2)	132
C1—H1···Cl1^{i}	0.98	2.79	3.674 (2)	151
C7—H7*A*···*Cg*^{ii}	0.97	2.85	3.575 (2)	133

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

3. Supramolecular features

In the crystal, molecules associate pairwise via C1—H1···Cl1^{i} hydrogen bonds (Table 1) into inversion dimers with an $R_2^2(12)$ graph-set motif (Etter *et al.*, 1990), as shown in Fig. 2. Moreover, molecules are further linked into an $R_2^2(14)$ graph-set motif by C—H···π interactions, C7—H7*A*···*Cg*, where *Cg* is the centroid of the symmetry-related C8–C13 benzene ring at $(2-x, 2-y, 1-z)$ (Table 1).

4. Hirshfeld surface analysis

The intermolecular interactions were quantified by a Hirshfeld surface (HS) analysis (Spackman & Jayatilaka, 2009) using *CrystalExplorer* (Spackman *et al.*, 2021). The HS mapped over d_{norm} is illustrated in Fig. 3. where no red spot occurs. This represents the non-availability of potential hydrogen bonds in

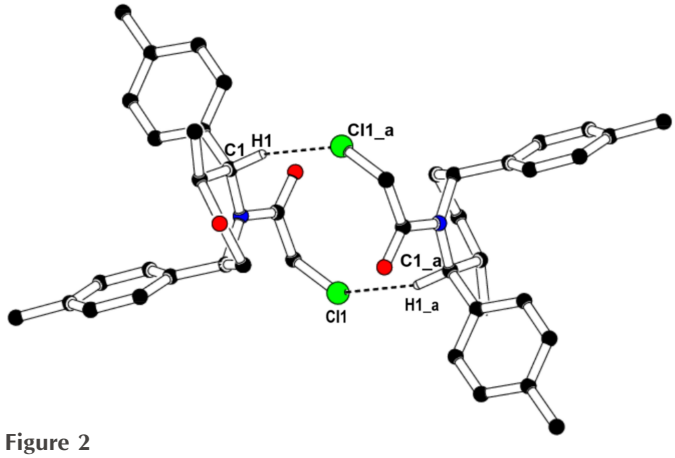


Figure 2
Centrosymmetric dimer through C—H···Cl hydrogen bonds [Symmetry code: (*a*) $-x+1, -y, -z+1$].

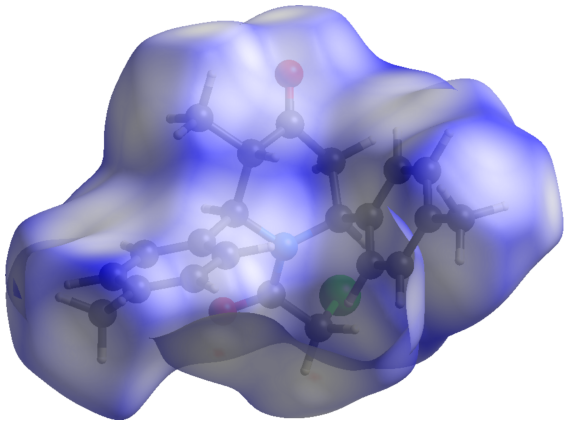


Figure 3
Hirshfeld surface mapped over d_{norm}.

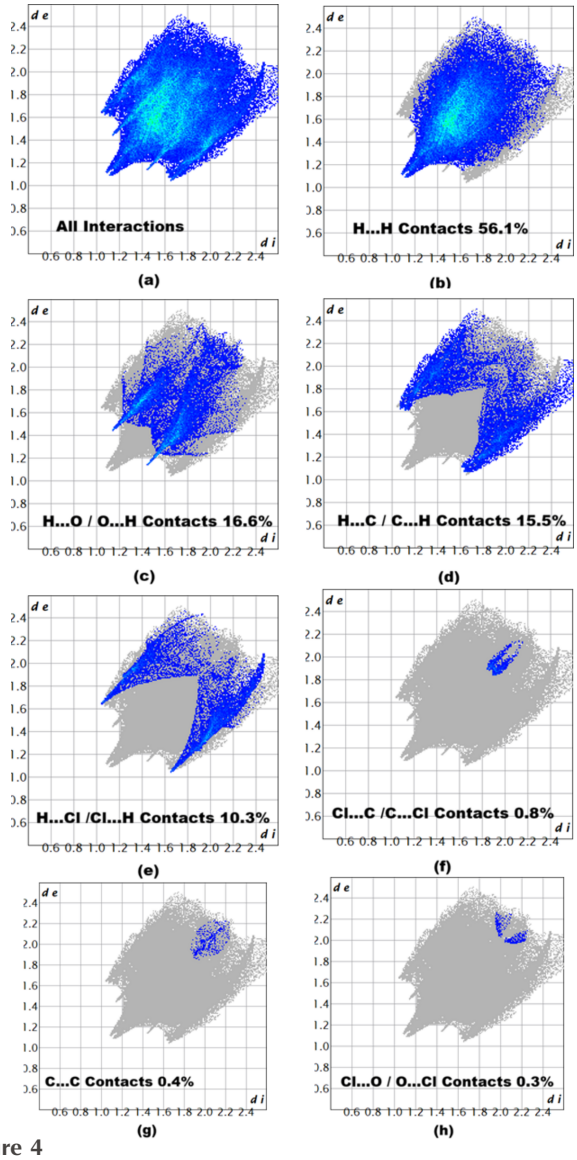


Figure 4
Two-dimensional fingerprint plots showing (*a*) all interactions, and delineated into (*b*) H···H, (*c*) H···O/O···H, (*d*) H···C/C···H, (*e*)H···Cl/Cl···H, (*f*) Cl···C/C···Cl, (*g*) C···C and (*h*) Cl···O/O···Cl interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{24}ClNO_2$
M_r	369.87
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	298
a, b, c (Å)	8.7654 (5), 11.3919 (6), 11.6090 (7)
α, β, γ (°)	110.594 (2), 102.709 (2), 107.595 (2)
V (Å^3)	962.71 (10)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.21
Crystal size (mm)	0.35 × 0.23 × 0.19
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.711, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	24328, 5355, 3183
R_{int}	0.043
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.706
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.050, 0.135, 1.03
No. of reflections	5355
No. of parameters	238
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.27

Computer programs: *APEX3* and *SAINT* (Bruker, 2017), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2019/2* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2020).

this crystal. The associated two-dimensional fingerprint plots (McKinnon *et al.*, 2007) provide quantitative information about the non-covalent interactions in the crystal packing in terms of the percentage contribution of the interatomic contacts (Spackman & McKinnon, 2002). The overall two-dimensional fingerprint plot is shown in Fig. 4*a* (top left). The HS analysis reveals that H···H and H···O/O···H contacts are the main contributors to the crystal packing followed by H···C/C···H, H···Cl/Cl···H, Cl···C/C···Cl, C···C and Cl···O/O···Cl contacts; see Fig. 4*b*–*h*.

5. Synthesis and crystallization

The compound has been previously reported, and all characterization data are consistent with those described by Aridoss *et al.*, 2007*a*,*b*. The compound was synthesized by mixing 3-methyl-2,6-di-*p*-tolylpiperidin-4-one (0.75 g, 2.5 mmol) and chloroacetyl chloride (1.0 mL, 10 mmol). The mixture was stirred in anhydrous benzene (50 mL) at room temperature. Then, triethylamine (1.4 mL, 10 mmol) was added as a base to initiate the reaction. The reaction mixture was maintained at room temperature for 6 h. Upon completion, the precipitated triethylammonium chloride salt was removed by filtration. The resulting organic layer was washed thoroughly with water then dried over anhydrous Na_2SO_4. The solvent was removed and the crude product was recrystallized from a mixture of petroleum ether and ethyl acetate (9:1, *v*/*v*) to afford the product as colourless crystals.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were placed in idealized positions and allowed to ride on their parent atoms: C—H = 0.93–0.98 Å with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and U_{iso}(H) = 1.2U_{eq}(C) for other H atoms.

Acknowledgements

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Acta Cryst. (2026). E**82**, 152-155 [https://doi.org/10.1107/S2056989026000083]

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Computing details

1-(2-Chloroacetyl)-3-methyl-2,6-bis(4-methylphenyl)piperidin-4-one

Crystal data

$C_{22}H_{24}ClNO_2$
$M_r = 369.87$
Triclinic, $P\overline{1}$
$a = 8.7654$ (5) Å
$b = 11.3919$ (6) Å
$c = 11.6090$ (7) Å
$\alpha = 110.594$ (2)°
$\beta = 102.709$ (2)°
$\gamma = 107.595$ (2)°
$V = 962.71$ (10) Å^3
$Z = 2$
$F(000) = 392$
$D_x = 1.276$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6744 reflections
$\theta = 2.6$–$28.8°$
$\mu = 0.21$ mm^{-1}
$T = 298$ K
Block, colourless
0.35 × 0.23 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: i-mu-s microfocus source
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
$T_{min} = 0.711$, $T_{max} = 0.746$
24328 measured reflections
5355 independent reflections
3183 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 30.1°$, $\theta_{min} = 2.1°$
$h = -11 \rightarrow 12$
$k = -15 \rightarrow 16$
$l = -15 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.135$
$S = 1.03$
5355 reflections
238 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 0.2977P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.25775 (8)	−0.21845 (5)	0.47830 (6)	0.0760 (2)
O1	0.85771 (18)	0.29415 (17)	0.90454 (19)	0.0905 (6)
O2	0.20241 (19)	0.06629 (14)	0.46708 (13)	0.0676 (4)
N1	0.34459 (17)	0.11084 (13)	0.67584 (13)	0.0436 (3)
C1	0.4496 (2)	0.25412 (16)	0.70160 (16)	0.0430 (4)
H1	0.493746	0.246576	0.629971	0.052*
C2	0.6072 (2)	0.33108 (17)	0.83254 (17)	0.0467 (4)
H2	0.563298	0.350761	0.904614	0.056*
C3	0.7039 (2)	0.2449 (2)	0.84836 (19)	0.0544 (4)
C4	0.5966 (2)	0.09311 (18)	0.79297 (19)	0.0523 (4)
H4A	0.639400	0.060054	0.854009	0.063*
H4B	0.608122	0.044809	0.710194	0.063*
C5	0.4065 (2)	0.05969 (17)	0.76895 (16)	0.0459 (4)
H5	0.345337	−0.041045	0.722924	0.055*
C6	0.2292 (2)	0.02538 (18)	0.55034 (17)	0.0498 (4)
C7	0.1282 (2)	−0.12542 (18)	0.5127 (2)	0.0589 (5)
H7A	0.025083	−0.164551	0.435285	0.071*
H7B	0.093917	−0.133089	0.584595	0.071*
C8	0.3460 (2)	0.33884 (16)	0.69974 (16)	0.0436 (4)
C9	0.2647 (2)	0.36976 (18)	0.78849 (18)	0.0501 (4)
H9	0.266432	0.332410	0.848017	0.060*
C10	0.1809 (2)	0.45532 (19)	0.79026 (19)	0.0551 (5)
H10	0.125999	0.473644	0.850156	0.066*
C11	0.1777 (2)	0.51415 (19)	0.70398 (19)	0.0577 (5)
C12	0.2585 (3)	0.4826 (2)	0.6152 (2)	0.0632 (5)
H12	0.257599	0.520543	0.556190	0.076*
C13	0.3407 (3)	0.39610 (19)	0.61200 (17)	0.0552 (5)
H13	0.392961	0.375967	0.550488	0.066*
C14	0.0891 (3)	0.6093 (2)	0.7089 (3)	0.0801 (7)
H14A	0.090380	0.653225	0.796661	0.120*
H14B	−0.027693	0.557154	0.647625	0.120*
H14C	0.148117	0.678073	0.685548	0.120*
C15	0.7241 (3)	0.4697 (2)	0.8473 (2)	0.0629 (5)
H15A	0.665406	0.529360	0.857365	0.094*
H15B	0.753294	0.455989	0.769991	0.094*
H15C	0.827119	0.511073	0.923769	0.094*
C16	0.3612 (2)	0.10762 (17)	0.89173 (17)	0.0459 (4)
C17	0.4756 (3)	0.15250 (19)	1.01738 (18)	0.0566 (5)
H17	0.586631	0.158729	1.028921	0.068*

C18	0.4274 (3)	0.1882 (2)	1.12598 (19)	0.0623 (5)
H18	0.507013	0.218030	1.209176	0.075*
C19	0.2642 (3)	0.18084 (19)	1.1142 (2)	0.0605 (5)
C20	0.1502 (3)	0.1367 (2)	0.9891 (2)	0.0628 (5)
H20	0.039547	0.131290	0.977987	0.075*
C21	0.1972 (2)	0.1003 (2)	0.87960 (19)	0.0546 (5)
H21	0.117321	0.070404	0.796475	0.066*
C22	0.2150 (4)	0.2203 (3)	1.2342 (2)	0.0888 (8)
H22A	0.095320	0.202382	1.207207	0.133*
H22B	0.282704	0.316607	1.292575	0.133*
H22C	0.235683	0.167058	1.279197	0.133*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0787 (4)	0.0539 (3)	0.0795 (4)	0.0288 (3)	0.0323 (3)	0.0103 (3)
O1	0.0434 (8)	0.0746 (10)	0.1260 (15)	0.0156 (8)	0.0014 (9)	0.0413 (10)
O2	0.0794 (10)	0.0584 (8)	0.0419 (7)	0.0190 (7)	0.0066 (7)	0.0156 (6)
N1	0.0441 (8)	0.0359 (7)	0.0393 (7)	0.0099 (6)	0.0108 (6)	0.0130 (6)
C1	0.0439 (9)	0.0388 (8)	0.0402 (9)	0.0113 (7)	0.0144 (7)	0.0168 (7)
C2	0.0419 (9)	0.0395 (9)	0.0453 (9)	0.0094 (7)	0.0092 (7)	0.0154 (7)
C3	0.0427 (10)	0.0543 (11)	0.0571 (11)	0.0145 (9)	0.0129 (8)	0.0234 (9)
C4	0.0499 (10)	0.0485 (10)	0.0582 (11)	0.0215 (8)	0.0182 (9)	0.0237 (9)
C5	0.0464 (9)	0.0366 (8)	0.0468 (9)	0.0126 (7)	0.0129 (8)	0.0170 (7)
C6	0.0468 (10)	0.0432 (9)	0.0425 (10)	0.0138 (8)	0.0104 (8)	0.0092 (8)
C7	0.0478 (10)	0.0428 (10)	0.0591 (11)	0.0111 (8)	0.0088 (9)	0.0073 (9)
C8	0.0431 (9)	0.0375 (8)	0.0372 (8)	0.0094 (7)	0.0073 (7)	0.0138 (7)
C9	0.0524 (10)	0.0475 (10)	0.0484 (10)	0.0181 (8)	0.0160 (8)	0.0231 (8)
C10	0.0506 (10)	0.0515 (10)	0.0549 (11)	0.0194 (9)	0.0157 (9)	0.0189 (9)
C11	0.0486 (10)	0.0469 (10)	0.0573 (12)	0.0149 (9)	0.0006 (9)	0.0175 (9)
C12	0.0713 (13)	0.0591 (12)	0.0531 (11)	0.0229 (11)	0.0084 (10)	0.0308 (10)
C13	0.0650 (12)	0.0543 (11)	0.0415 (10)	0.0214 (10)	0.0146 (9)	0.0224 (8)
C14	0.0723 (15)	0.0693 (14)	0.0925 (17)	0.0371 (12)	0.0105 (13)	0.0352 (13)
C15	0.0533 (11)	0.0474 (10)	0.0654 (13)	0.0044 (9)	0.0095 (9)	0.0230 (10)
C16	0.0494 (10)	0.0372 (8)	0.0466 (10)	0.0124 (7)	0.0155 (8)	0.0199 (7)
C17	0.0559 (11)	0.0534 (11)	0.0502 (11)	0.0165 (9)	0.0132 (9)	0.0215 (9)
C18	0.0768 (14)	0.0496 (11)	0.0446 (10)	0.0157 (10)	0.0128 (10)	0.0187 (9)
C19	0.0852 (15)	0.0444 (10)	0.0581 (12)	0.0258 (10)	0.0336 (11)	0.0264 (9)
C20	0.0664 (13)	0.0701 (13)	0.0721 (14)	0.0326 (11)	0.0370 (11)	0.0427 (11)
C21	0.0539 (11)	0.0593 (11)	0.0542 (11)	0.0202 (9)	0.0202 (9)	0.0319 (9)
C22	0.133 (2)	0.0797 (16)	0.0738 (16)	0.0489 (17)	0.0600 (16)	0.0382 (13)

Geometric parameters (Å, º)

Cl1—C7	1.789 (2)	C10—H10	0.9300
O1—C3	1.207 (2)	C11—C12	1.381 (3)
O2—C6	1.221 (2)	C11—C14	1.505 (3)
N1—C6	1.361 (2)	C12—C13	1.380 (3)

N1—C5	1.476 (2)	C12—H12	0.9300
N1—C1	1.493 (2)	C13—H13	0.9300
C1—C8	1.514 (2)	C14—H14A	0.9600
C1—C2	1.547 (2)	C14—H14B	0.9600
C1—H1	0.9800	C14—H14C	0.9600
C2—C3	1.509 (3)	C15—H15A	0.9600
C2—C15	1.526 (2)	C15—H15B	0.9600
C2—H2	0.9800	C15—H15C	0.9600
C3—C4	1.502 (3)	C16—C17	1.384 (2)
C4—C5	1.528 (2)	C16—C21	1.387 (3)
C4—H4A	0.9700	C17—C18	1.382 (3)
C4—H4B	0.9700	C17—H17	0.9300
C5—C16	1.523 (2)	C18—C19	1.380 (3)
C5—H5	0.9800	C18—H18	0.9300
C6—C7	1.519 (3)	C19—C20	1.381 (3)
C7—H7A	0.9700	C19—C22	1.506 (3)
C7—H7B	0.9700	C20—C21	1.385 (3)
C8—C9	1.383 (2)	C20—H20	0.9300
C8—C13	1.389 (2)	C21—H21	0.9300
C9—C10	1.384 (3)	C22—H22A	0.9600
C9—H9	0.9300	C22—H22B	0.9600
C10—C11	1.387 (3)	C22—H22C	0.9600

C6—N1—C5	122.82 (14)	C11—C10—H10	119.5
C6—N1—C1	115.84 (14)	C12—C11—C10	117.69 (18)
C5—N1—C1	118.41 (13)	C12—C11—C14	122.0 (2)
N1—C1—C8	113.14 (13)	C10—C11—C14	120.4 (2)
N1—C1—C2	112.07 (13)	C13—C12—C11	121.62 (18)
C8—C1—C2	110.05 (13)	C13—C12—H12	119.2
N1—C1—H1	107.1	C11—C12—H12	119.2
C8—C1—H1	107.1	C12—C13—C8	120.64 (18)
C2—C1—H1	107.1	C12—C13—H13	119.7
C3—C2—C15	112.14 (15)	C8—C13—H13	119.7
C3—C2—C1	112.78 (14)	C11—C14—H14A	109.5
C15—C2—C1	110.77 (14)	C11—C14—H14B	109.5
C3—C2—H2	106.9	H14A—C14—H14B	109.5
C15—C2—H2	106.9	C11—C14—H14C	109.5
C1—C2—H2	106.9	H14A—C14—H14C	109.5
O1—C3—C4	121.39 (18)	H14B—C14—H14C	109.5
O1—C3—C2	122.60 (18)	C2—C15—H15A	109.5
C4—C3—C2	115.99 (15)	C2—C15—H15B	109.5
C3—C4—C5	112.45 (15)	H15A—C15—H15B	109.5
C3—C4—H4A	109.1	C2—C15—H15C	109.5
C5—C4—H4A	109.1	H15A—C15—H15C	109.5
C3—C4—H4B	109.1	H15B—C15—H15C	109.5
C5—C4—H4B	109.1	C17—C16—C21	117.32 (17)
H4A—C4—H4B	107.8	C17—C16—C5	122.49 (16)
N1—C5—C16	112.38 (14)	C21—C16—C5	120.10 (15)

N1—C5—C4	108.01 (14)	C18—C17—C16	121.07 (19)
C16—C5—C4	116.21 (14)	C18—C17—H17	119.5
N1—C5—H5	106.5	C16—C17—H17	119.5
C16—C5—H5	106.5	C19—C18—C17	121.76 (19)
C4—C5—H5	106.5	C19—C18—H18	119.1
O2—C6—N1	122.11 (16)	C17—C18—H18	119.1
O2—C6—C7	118.71 (16)	C18—C19—C20	117.24 (18)
N1—C6—C7	119.17 (17)	C18—C19—C22	120.7 (2)
C6—C7—Cl1	109.87 (13)	C20—C19—C22	122.1 (2)
C6—C7—H7A	109.7	C19—C20—C21	121.4 (2)
Cl1—C7—H7A	109.7	C19—C20—H20	119.3
C6—C7—H7B	109.7	C21—C20—H20	119.3
Cl1—C7—H7B	109.7	C20—C21—C16	121.20 (18)
H7A—C7—H7B	108.2	C20—C21—H21	119.4
C9—C8—C13	117.95 (16)	C16—C21—H21	119.4
C9—C8—C1	122.47 (15)	C19—C22—H22A	109.5
C13—C8—C1	119.44 (16)	C19—C22—H22B	109.5
C8—C9—C10	121.14 (17)	H22A—C22—H22B	109.5
C8—C9—H9	119.4	C19—C22—H22C	109.5
C10—C9—H9	119.4	H22A—C22—H22C	109.5
C9—C10—C11	120.95 (19)	H22B—C22—H22C	109.5
C9—C10—H10	119.5		

C6—N1—C1—C8	70.04 (18)	C2—C1—C8—C9	−63.9 (2)
C5—N1—C1—C8	−128.78 (15)	N1—C1—C8—C13	−122.15 (16)
C6—N1—C1—C2	−164.83 (15)	C2—C1—C8—C13	111.63 (17)
C5—N1—C1—C2	−3.6 (2)	C13—C8—C9—C10	−0.1 (3)
N1—C1—C2—C3	46.1 (2)	C1—C8—C9—C10	175.48 (16)
C8—C1—C2—C3	172.88 (14)	C8—C9—C10—C11	−0.8 (3)
N1—C1—C2—C15	172.67 (15)	C9—C10—C11—C12	1.0 (3)
C8—C1—C2—C15	−60.51 (19)	C9—C10—C11—C14	−178.77 (18)
C15—C2—C3—O1	21.0 (3)	C10—C11—C12—C13	−0.3 (3)
C1—C2—C3—O1	146.9 (2)	C14—C11—C12—C13	179.49 (19)
C15—C2—C3—C4	−160.61 (16)	C11—C12—C13—C8	−0.7 (3)
C1—C2—C3—C4	−34.7 (2)	C9—C8—C13—C12	0.9 (3)
O1—C3—C4—C5	160.7 (2)	C1—C8—C13—C12	−174.90 (17)
C2—C3—C4—C5	−17.7 (2)	N1—C5—C16—C17	−138.29 (17)
C6—N1—C5—C16	−118.60 (17)	C4—C5—C16—C17	−13.2 (2)
C1—N1—C5—C16	81.61 (17)	N1—C5—C16—C21	45.3 (2)
C6—N1—C5—C4	111.89 (17)	C4—C5—C16—C21	170.40 (16)
C1—N1—C5—C4	−47.90 (18)	C21—C16—C17—C18	0.1 (3)
C3—C4—C5—N1	58.85 (19)	C5—C16—C17—C18	−176.44 (16)
C3—C4—C5—C16	−68.5 (2)	C16—C17—C18—C19	0.0 (3)
C5—N1—C6—O2	−166.44 (17)	C17—C18—C19—C20	−0.3 (3)
C1—N1—C6—O2	−6.2 (3)	C17—C18—C19—C22	179.97 (19)
C5—N1—C6—C7	14.5 (2)	C18—C19—C20—C21	0.5 (3)
C1—N1—C6—C7	174.76 (15)	C22—C19—C20—C21	−179.78 (19)
O2—C6—C7—Cl1	100.48 (19)	C19—C20—C21—C16	−0.4 (3)

N1—C6—C7—Cl1	−80.42 (19)	C17—C16—C21—C20	0.1 (3)
N1—C1—C8—C9	62.3 (2)	C5—C16—C21—C20	176.72 (16)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C8–C13 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···Cl1	0.98	2.61	3.342 (2)	132
C1—H1···Cl1^{i}	0.98	2.79	3.674 (2)	151
C7—H7*A*···*Cg*^{ii}	0.97	2.85	3.575 (2)	133

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