# 1-Chloroacetyl-3,3-dimethyl-2,6-diphenylpiperidin-4-one

**T. Kavitha,ᵃ S. Ponnuswamy,ᵇ M. Jamesh,ᵇ J. Umamaheshwariᵇ and M. N. Ponnuswamyᵃ***

ᵃCentre of Advanced Study in Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, and ᵇDepartment of Chemistry, Government Arts College (Autonomous), Coimbatore 641 018, India
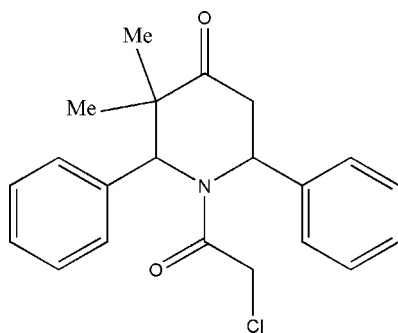Correspondence e-mail: mnpsy2004@yahoo.com

In the molecule of the title compound, $C_{21}H_{22}ClNO_2$, the piperidine ring adopts a distorted boat conformation. The two phenyl rings are nearly orthogonal to each other with a dihedral angle of 87.1 (1)°. In the crystal structure, the molecules are linked into a three-dimensional network by C—H···O and C—H···$\pi$ interactions.

## Related literature

For general background, see: Dimmock *et al.* (2001); Perumal *et al.* (2001). For ring conformational analysis, see: Cremer & Pople (1975); Nardelli (1983).

## Experimental

### Crystal data

| | |
|---|---|
| $C_{21}H_{22}ClNO_2$ | $V$ = 1858.08 (7) Å³ |
| $M_r$ = 355.85 | $Z$ = 4 |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation |
| $a$ = 13.7005 (3) Å | $\mu$ = 0.22 mm⁻¹ |
| $b$ = 9.8735 (2) Å | $T$ = 293 (2) K |
| $c$ = 14.8960 (3) Å | 0.32 × 0.26 × 0.20 mm |
| $\beta$ = 112.762 (1)° | |

### Data collection

| | |
|---|---|
| Bruker Kappa APEXII area-detector diffractometer | 22063 measured reflections |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001) | 4674 independent reflections |
| | 3424 reflections with $I > 2\sigma(I)$ |
| $T_{min}$ = 0.854, $T_{max}$ = 0.958 | $R_{int}$ = 0.025 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.050 | 226 parameters |
| $wR(F^2)$ = 0.154 | H-atom parameters constrained |
| $S$ = 1.01 | $\Delta\rho_{max}$ = 0.68 e Å⁻³ |
| 4674 reflections | $\Delta\rho_{min}$ = −0.66 e Å⁻³ |

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

*Cg*1 is the centroid of the C17–C22 ring.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C6—H6···O2ⁱ | 0.98 | 2.46 | 3.426 (2) | 168 |
| C8—H8B···O2ⁱ | 0.97 | 2.53 | 3.495 (3) | 172 |
| C21—H21···O1ⁱⁱ | 0.93 | 2.53 | 3.248 (3) | 134 |
| C11—H11···*Cg*1ⁱⁱⁱ | 0.93 | 2.73 | 3.568 (2) | 150 |

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

## References

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# supporting information

## 1-Chloroacetyl-3,3-dimethyl-2,6-diphenylpiperidin-4-one

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### S1. Comment

Piperidones are the important group of heterocyclic compounds in the field of medicinal chemistry due to their biological activities, including cytotoxic properties (Dimmock *et al.*, 2001). They were also reported to possess analgesic, anti-inflammatory, central nervous system (CNS), local anaesthetic, anticancer and antimicrobial activities (Perumal *et al.*, 2001).

The sum of bond angles around N1 atom (359.4°) indicates $sp^2$ hybridization. The N1—C7 [1.360 (2) Å] and C7—O2 [1.220 (2) Å] distances indicate electron delocalization. The piperidine ring adopts a distorted boat conformation, confirmed by puckering parameters $q_2$ = 0.612 (2) Å, $q_3$ = -0.122 (2) Å and $\varphi_2$ = 258.0 (2)° (Cremer & Pople, 1975) and the asymmetry parameter $\Delta_s$(C2) = $\Delta_s$(C5) = 19.2 (2)° (Nardelli, 1983). The two phenyl rings are nearly orthogonal to each other with a dihedral angle of 87.1 (1)°. The methyl substituents are oriented equatorially [N1—C2—C3—C16 = 175.3 (2)°] and axially [N1—C2—C3—C15 = 55.1 (2)°] at C3 position.

The crystal structure is stabilized by C—H⋯O and C—H⋯π intermolecular interactions. The glide-related molecules are linked into a chain along the *b* axis by C6—H6⋯O2[i] and C8—H8B⋯O2[i] hydrogen bonds, and the chains are cross-linked *via* C21—H21⋯O1[ii] hydrogen bonds; symmetry codes are given in Table 1. In addition, C—H⋯π interactions involving the C17–C22 ring (centroid *Cg*1) are observed.

### S2. Experimental

A mixture of 3,3-dimethyl-*cis*-2,6-diphenylpiperidin-4-one (1.4 g, 5 mmol), chloro acetylchloride (0.8 ml, 5 mmol) and triethylamine (2 ml, 14.4 mmol) in anhydrous benzene (20 ml) was stirred at room temperature for 7 h. The benzene solution was dried over anhydrous $Na_2SO_4$ and concentrated to obtain a pasty mass. It was purified by crystallization from benzene–petroleum ether (95:5, 60–80°C).

### S3. Refinement

H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and allowed to ride on their parent atoms, with $U_{iso}$(H) = 1.5$U_{eq}$(C) for methyl H atoms and 1.2$U_{eq}$(C) for other H atoms.
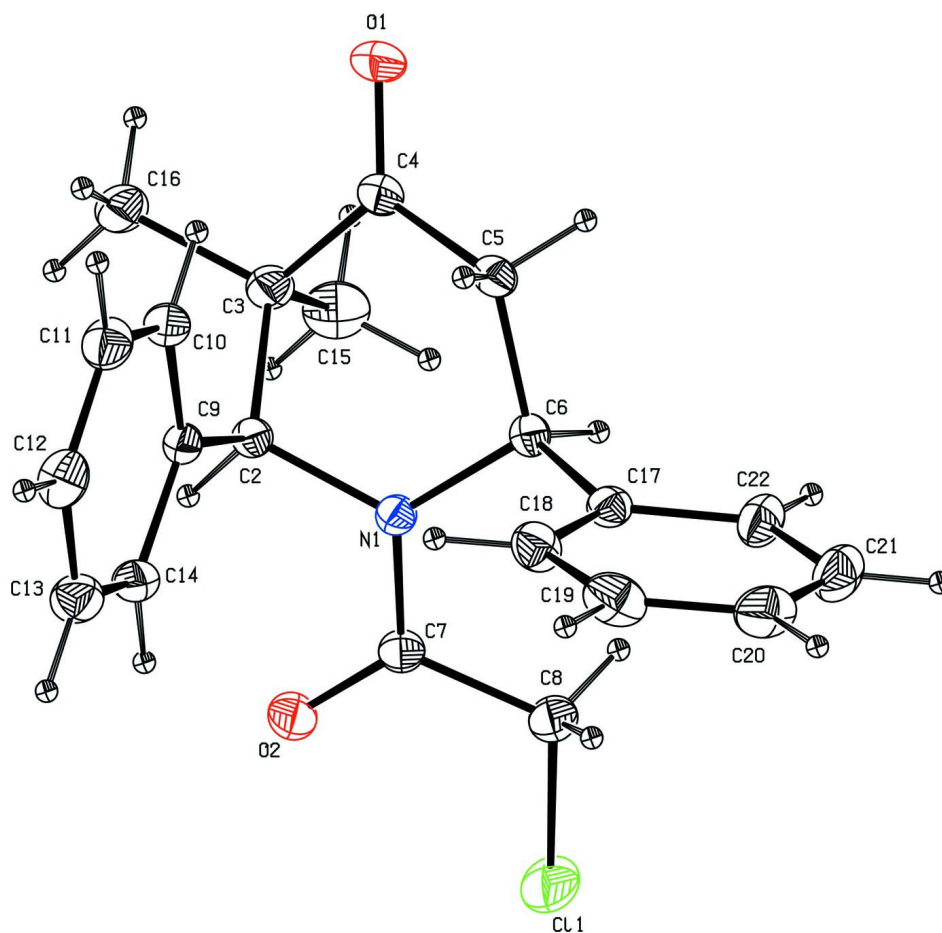
**Figure 1**

The molecular structure of the title compound, showing 20% probability displacement ellipsoids.
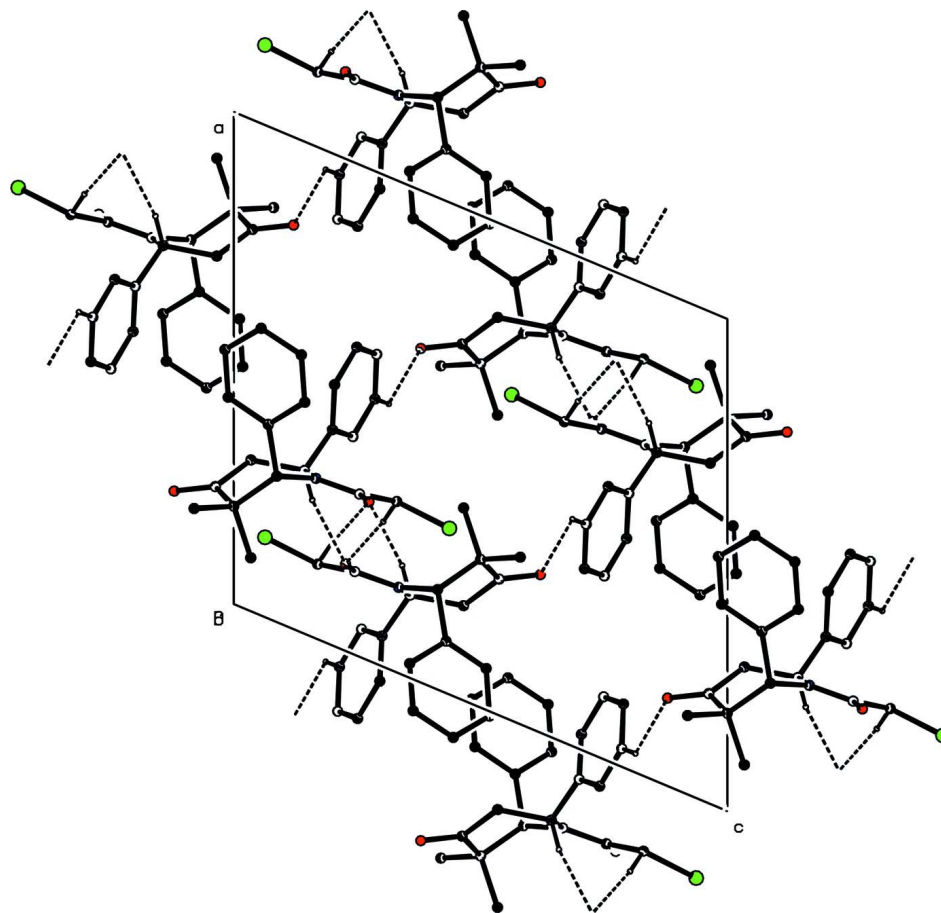
**Figure 2**
The crystal packing of the title compound, viewed along the *b* axis. Dashed lines indicate hydrogen bonds. H atoms not involed in hydrogen bonding have been omitted.

## 1-Chloroacetyl-3,3-dimethyl-2,6-diphenylpiperidin-4-one

*Crystal data*

| | |
|---|---|
| $C_{21}H_{22}ClNO_2$ | $F(000) = 752$ |
| $M_r = 355.85$ | $D_x = 1.272$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2yn | Cell parameters from 4674 reflections |
| $a = 13.7005$ (3) Å | $\theta = 2.5$–28.5° |
| $b = 9.8735$ (2) Å | $\mu = 0.22$ mm$^{-1}$ |
| $c = 14.8960$ (3) Å | $T = 293$ K |
| $\beta = 112.762$ (1)° | Block, colourless |
| $V = 1858.08$ (7) Å$^3$ | $0.32 \times 0.26 \times 0.20$ mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Bruker Kappa APEXII area-detector diffractometer | Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001) |
| Radiation source: fine-focus sealed tube | $T_{min} = 0.854$, $T_{max} = 0.958$ |
| Graphite monochromator | 22063 measured reflections |
| $\omega$ and $\varphi$ scans | 4674 independent reflections |

3424 reflections with $I > 2\sigma(I)$

$R_{int} = 0.025$

$\theta_{max} = 28.5°$, $\theta_{min} = 2.5°$

$h = -18 \rightarrow 18$

$k = -11 \rightarrow 13$

$l = -18 \rightarrow 19$

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.050$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.154$ | |
| $S = 1.01$ | H-atom parameters constrained |
| 4674 reflections | $w = 1/[\sigma^2(F_o^2) + (0.0741P)^2 + 0.7704P]$ |
| 226 parameters | where $P = (F_o^2 + 2F_c^2)/3$ |
| 0 restraints | $(\Delta/\sigma)_{max} = 0.001$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{max} = 0.68$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.66$ 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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C2 | 0.20338 (13) | 0.74492 (17) | 0.41291 (11) | 0.0364 (3) |
| H2 | 0.2263 | 0.8253 | 0.3878 | 0.044* |
| C3 | 0.30126 (14) | 0.6958 (2) | 0.50015 (13) | 0.0457 (4) |
| C4 | 0.27691 (14) | 0.56295 (19) | 0.53752 (13) | 0.0460 (4) |
| C5 | 0.19260 (15) | 0.47683 (18) | 0.46543 (13) | 0.0450 (4) |
| H5A | 0.1275 | 0.4912 | 0.4755 | 0.054* |
| H5B | 0.2125 | 0.3828 | 0.4808 | 0.054* |
| C6 | 0.16822 (13) | 0.49786 (17) | 0.35689 (12) | 0.0374 (3) |
| H6 | 0.2216 | 0.4486 | 0.3411 | 0.045* |
| C7 | 0.16925 (13) | 0.68513 (18) | 0.24518 (12) | 0.0392 (4) |
| C8 | 0.15274 (18) | 0.5752 (2) | 0.16986 (14) | 0.0536 (5) |
| H8A | 0.0834 | 0.5350 | 0.1537 | 0.064* |
| H8B | 0.2053 | 0.5048 | 0.1972 | 0.064* |
| C9 | 0.10539 (13) | 0.78759 (16) | 0.43001 (11) | 0.0372 (3) |
| C10 | 0.08432 (16) | 0.75494 (19) | 0.51136 (13) | 0.0462 (4) |
| H10 | 0.1339 | 0.7062 | 0.5620 | 0.055* |
| C11 | −0.00972 (18) | 0.7942 (2) | 0.51791 (15) | 0.0547 (5) |
| H11 | −0.0225 | 0.7717 | 0.5730 | 0.066* |
| C12 | −0.08371 (17) | 0.8654 (2) | 0.44463 (16) | 0.0569 (5) |
| H12 | −0.1470 | 0.8906 | 0.4493 | 0.068* |
| C13 | −0.06398 (16) | 0.8999 (2) | 0.36330 (15) | 0.0558 (5) |
| H13 | −0.1139 | 0.9490 | 0.3132 | 0.067* |

| C14 | 0.02930 (15) | 0.86157 (19) | 0.35629 (13) | 0.0451 (4) |
|---|---|---|---|---|
| H14 | 0.0418 | 0.8855 | 0.3013 | 0.054* |
| C15 | 0.39135 (16) | 0.6647 (3) | 0.46568 (19) | 0.0695 (7) |
| H15A | 0.4103 | 0.7459 | 0.4409 | 0.104* |
| H15B | 0.3681 | 0.5975 | 0.4152 | 0.104* |
| H15C | 0.4517 | 0.6313 | 0.5194 | 0.104* |
| C16 | 0.33953 (19) | 0.8035 (2) | 0.58046 (15) | 0.0635 (6) |
| H16A | 0.3542 | 0.8862 | 0.5541 | 0.095* |
| H16B | 0.4028 | 0.7724 | 0.6321 | 0.095* |
| H16C | 0.2856 | 0.8194 | 0.6054 | 0.095* |
| C17 | 0.06144 (13) | 0.43346 (18) | 0.30018 (12) | 0.0402 (4) |
| C18 | −0.03188 (15) | 0.4922 (2) | 0.29576 (15) | 0.0514 (5) |
| H18 | −0.0308 | 0.5772 | 0.3230 | 0.062* |
| C19 | −0.12687 (16) | 0.4244 (3) | 0.25075 (18) | 0.0653 (6) |
| H19 | −0.1896 | 0.4643 | 0.2477 | 0.078* |
| C20 | −0.12916 (18) | 0.2988 (3) | 0.21055 (17) | 0.0672 (6) |
| H20 | −0.1932 | 0.2534 | 0.1810 | 0.081* |
| C21 | −0.0373 (2) | 0.2404 (2) | 0.21401 (17) | 0.0645 (6) |
| H21 | −0.0389 | 0.1556 | 0.1862 | 0.077* |
| C22 | 0.05803 (17) | 0.3072 (2) | 0.25877 (14) | 0.0517 (5) |
| H22 | 0.1203 | 0.2669 | 0.2611 | 0.062* |
| Cl1 | 0.16240 (6) | 0.63835 (7) | 0.06314 (4) | 0.0778 (2) |
| N1 | 0.17457 (10) | 0.64277 (14) | 0.33383 (9) | 0.0350 (3) |
| O1 | 0.32116 (13) | 0.52655 (16) | 0.62105 (10) | 0.0706 (5) |
| O2 | 0.17704 (12) | 0.80385 (14) | 0.22634 (10) | 0.0518 (3) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C2 | 0.0408 (8) | 0.0334 (8) | 0.0325 (8) | −0.0040 (6) | 0.0113 (6) | 0.0006 (6) |
| C3 | 0.0407 (8) | 0.0456 (10) | 0.0409 (9) | −0.0039 (7) | 0.0049 (7) | 0.0007 (7) |
| C4 | 0.0477 (9) | 0.0416 (10) | 0.0396 (9) | 0.0071 (8) | 0.0070 (7) | 0.0041 (7) |
| C5 | 0.0533 (10) | 0.0358 (9) | 0.0399 (9) | −0.0014 (7) | 0.0114 (8) | 0.0063 (7) |
| C6 | 0.0398 (8) | 0.0331 (8) | 0.0384 (8) | −0.0020 (6) | 0.0143 (7) | 0.0000 (6) |
| C7 | 0.0394 (8) | 0.0433 (10) | 0.0375 (8) | −0.0005 (7) | 0.0177 (7) | 0.0022 (7) |
| C8 | 0.0761 (13) | 0.0507 (11) | 0.0404 (9) | 0.0006 (10) | 0.0295 (9) | 0.0007 (8) |
| C9 | 0.0452 (8) | 0.0321 (8) | 0.0328 (8) | −0.0034 (7) | 0.0136 (6) | −0.0017 (6) |
| C10 | 0.0599 (10) | 0.0423 (10) | 0.0382 (9) | −0.0002 (8) | 0.0210 (8) | 0.0036 (7) |
| C11 | 0.0692 (12) | 0.0547 (12) | 0.0506 (11) | −0.0083 (10) | 0.0346 (10) | −0.0026 (9) |
| C12 | 0.0509 (10) | 0.0616 (13) | 0.0633 (13) | −0.0042 (9) | 0.0278 (10) | −0.0119 (10) |
| C13 | 0.0501 (10) | 0.0628 (13) | 0.0487 (11) | 0.0097 (9) | 0.0127 (8) | −0.0009 (9) |
| C14 | 0.0521 (10) | 0.0471 (10) | 0.0356 (8) | 0.0046 (8) | 0.0164 (7) | 0.0025 (7) |
| C15 | 0.0394 (10) | 0.0911 (18) | 0.0705 (15) | 0.0000 (10) | 0.0129 (10) | 0.0047 (13) |
| C16 | 0.0668 (13) | 0.0553 (12) | 0.0468 (11) | −0.0150 (10) | −0.0016 (9) | −0.0027 (9) |
| C17 | 0.0453 (9) | 0.0390 (9) | 0.0351 (8) | −0.0074 (7) | 0.0144 (7) | 0.0031 (7) |
| C18 | 0.0458 (9) | 0.0456 (10) | 0.0566 (11) | −0.0039 (8) | 0.0129 (8) | 0.0041 (9) |
| C19 | 0.0424 (10) | 0.0691 (15) | 0.0728 (15) | −0.0048 (10) | 0.0096 (10) | 0.0161 (12) |
| C20 | 0.0586 (12) | 0.0710 (15) | 0.0568 (12) | −0.0287 (11) | 0.0057 (10) | 0.0037 (11) |

| | | | | | |
|---|---|---|---|---|---|
| C21 | 0.0760 (15) | 0.0581 (13) | 0.0567 (12) | −0.0258 (11) | 0.0225 (11) | −0.0140 (10) |
| C22 | 0.0605 (11) | 0.0497 (11) | 0.0489 (10) | −0.0143 (9) | 0.0256 (9) | −0.0101 (8) |
| Cl1 | 0.1234 (6) | 0.0747 (4) | 0.0530 (3) | −0.0077 (4) | 0.0534 (4) | −0.0031 (3) |
| N1 | 0.0387 (7) | 0.0337 (7) | 0.0327 (7) | −0.0025 (5) | 0.0139 (5) | 0.0002 (5) |
| O1 | 0.0861 (11) | 0.0565 (9) | 0.0427 (8) | 0.0064 (8) | −0.0041 (7) | 0.0112 (7) |
| O2 | 0.0714 (9) | 0.0441 (7) | 0.0471 (7) | −0.0043 (6) | 0.0308 (7) | 0.0057 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C2—N1 | 1.483 (2) | C11—C12 | 1.363 (3) |
| C2—C9 | 1.520 (2) | C11—H11 | 0.93 |
| C2—C3 | 1.541 (2) | C12—C13 | 1.382 (3) |
| C2—H2 | 0.98 | C12—H12 | 0.93 |
| C3—C4 | 1.512 (3) | C13—C14 | 1.375 (3) |
| C3—C16 | 1.534 (3) | C13—H13 | 0.93 |
| C3—C15 | 1.540 (3) | C14—H14 | 0.93 |
| C4—O1 | 1.209 (2) | C15—H15A | 0.96 |
| C4—C5 | 1.500 (3) | C15—H15B | 0.96 |
| C5—C6 | 1.533 (2) | C15—H15C | 0.96 |
| C5—H5A | 0.97 | C16—H16A | 0.96 |
| C5—H5B | 0.97 | C16—H16B | 0.96 |
| C6—N1 | 1.482 (2) | C16—H16C | 0.96 |
| C6—C17 | 1.517 (2) | C17—C18 | 1.382 (3) |
| C6—H6 | 0.98 | C17—C22 | 1.384 (3) |
| C7—O2 | 1.220 (2) | C18—C19 | 1.384 (3) |
| C7—N1 | 1.360 (2) | C18—H18 | 0.93 |
| C7—C8 | 1.514 (3) | C19—C20 | 1.372 (4) |
| C8—Cl1 | 1.7595 (19) | C19—H19 | 0.93 |
| C8—H8A | 0.97 | C20—C21 | 1.367 (4) |
| C8—H8B | 0.97 | C20—H20 | 0.93 |
| C9—C10 | 1.388 (2) | C21—C22 | 1.382 (3) |
| C9—C14 | 1.393 (2) | C21—H21 | 0.93 |
| C10—C11 | 1.385 (3) | C22—H22 | 0.93 |
| C10—H10 | 0.93 | | |
| | | | |
| N1—C2—C9 | 109.92 (12) | C10—C11—H11 | 119.6 |
| N1—C2—C3 | 109.45 (14) | C11—C12—C13 | 119.48 (19) |
| C9—C2—C3 | 118.87 (14) | C11—C12—H12 | 120.3 |
| N1—C2—H2 | 105.9 | C13—C12—H12 | 120.3 |
| C9—C2—H2 | 105.9 | C14—C13—C12 | 120.11 (19) |
| C3—C2—H2 | 105.9 | C14—C13—H13 | 119.9 |
| C4—C3—C16 | 111.85 (16) | C12—C13—H13 | 119.9 |
| C4—C3—C15 | 105.63 (18) | C13—C14—C9 | 121.22 (18) |
| C16—C3—C15 | 108.79 (18) | C13—C14—H14 | 119.4 |
| C4—C3—C2 | 109.86 (14) | C9—C14—H14 | 119.4 |
| C16—C3—C2 | 111.27 (16) | C3—C15—H15A | 109.5 |
| C15—C3—C2 | 109.26 (16) | C3—C15—H15B | 109.5 |
| O1—C4—C5 | 120.59 (18) | H15A—C15—H15B | 109.5 |

| | | | |
|---|---|---|---|
| O1—C4—C3 | 122.87 (17) | C3—C15—H15C | 109.5 |
| C5—C4—C3 | 116.54 (15) | H15A—C15—H15C | 109.5 |
| C4—C5—C6 | 118.12 (15) | H15B—C15—H15C | 109.5 |
| C4—C5—H5A | 107.8 | C3—C16—H16A | 109.5 |
| C6—C5—H5A | 107.8 | C3—C16—H16B | 109.5 |
| C4—C5—H5B | 107.8 | H16A—C16—H16B | 109.5 |
| C6—C5—H5B | 107.8 | C3—C16—H16C | 109.5 |
| H5A—C5—H5B | 107.1 | H16A—C16—H16C | 109.5 |
| N1—C6—C17 | 113.96 (13) | H16B—C16—H16C | 109.5 |
| N1—C6—C5 | 111.52 (14) | C18—C17—C22 | 119.07 (17) |
| C17—C6—C5 | 107.46 (13) | C18—C17—C6 | 121.70 (16) |
| N1—C6—H6 | 107.9 | C22—C17—C6 | 119.00 (16) |
| C17—C6—H6 | 107.9 | C17—C18—C19 | 119.9 (2) |
| C5—C6—H6 | 107.9 | C17—C18—H18 | 120.0 |
| O2—C7—N1 | 122.85 (16) | C19—C18—H18 | 120.0 |
| O2—C7—C8 | 121.32 (16) | C20—C19—C18 | 120.4 (2) |
| N1—C7—C8 | 115.83 (15) | C20—C19—H19 | 119.8 |
| C7—C8—Cl1 | 111.96 (14) | C18—C19—H19 | 119.8 |
| C7—C8—H8A | 109.2 | C21—C20—C19 | 120.0 (2) |
| Cl1—C8—H8A | 109.2 | C21—C20—H20 | 120.0 |
| C7—C8—H8B | 109.2 | C19—C20—H20 | 120.0 |
| Cl1—C8—H8B | 109.2 | C20—C21—C22 | 120.1 (2) |
| H8A—C8—H8B | 107.9 | C20—C21—H21 | 120.0 |
| C10—C9—C14 | 117.72 (16) | C22—C21—H21 | 120.0 |
| C10—C9—C2 | 125.29 (15) | C21—C22—C17 | 120.5 (2) |
| C14—C9—C2 | 116.96 (15) | C21—C22—H22 | 119.8 |
| C11—C10—C9 | 120.67 (18) | C17—C22—H22 | 119.8 |
| C11—C10—H10 | 119.7 | C7—N1—C6 | 122.42 (14) |
| C9—C10—H10 | 119.7 | C7—N1—C2 | 117.37 (13) |
| C12—C11—C10 | 120.80 (18) | C6—N1—C2 | 119.59 (13) |
| C12—C11—H11 | 119.6 | | |
| | | | |
| N1—C2—C3—C4 | −60.29 (18) | C12—C13—C14—C9 | 0.2 (3) |
| C9—C2—C3—C4 | 67.1 (2) | C10—C9—C14—C13 | −0.6 (3) |
| N1—C2—C3—C16 | 175.29 (15) | C2—C9—C14—C13 | 177.27 (17) |
| C9—C2—C3—C16 | −57.3 (2) | N1—C6—C17—C18 | −50.6 (2) |
| N1—C2—C3—C15 | 55.1 (2) | C5—C6—C17—C18 | 73.5 (2) |
| C9—C2—C3—C15 | −177.45 (17) | N1—C6—C17—C22 | 134.95 (16) |
| C16—C3—C4—O1 | −28.9 (3) | C5—C6—C17—C22 | −100.95 (19) |
| C15—C3—C4—O1 | 89.3 (2) | C22—C17—C18—C19 | 0.3 (3) |
| C2—C3—C4—O1 | −153.0 (2) | C6—C17—C18—C19 | −174.15 (18) |
| C16—C3—C4—C5 | 150.31 (18) | C17—C18—C19—C20 | 0.2 (3) |
| C15—C3—C4—C5 | −91.50 (19) | C18—C19—C20—C21 | −0.6 (4) |
| C2—C3—C4—C5 | 26.2 (2) | C19—C20—C21—C22 | 0.6 (4) |
| O1—C4—C5—C6 | −157.96 (19) | C20—C21—C22—C17 | −0.2 (3) |
| C3—C4—C5—C6 | 22.8 (2) | C18—C17—C22—C21 | −0.3 (3) |
| C4—C5—C6—N1 | −37.4 (2) | C6—C17—C22—C21 | 174.29 (18) |
| C4—C5—C6—C17 | −162.92 (16) | O2—C7—N1—C6 | −178.13 (16) |

| | | | |
|---|---|---|---|
| O2—C7—C8—Cl1 | 7.0 (2) | C8—C7—N1—C6 | 1.9 (2) |
| N1—C7—C8—Cl1 | −173.12 (13) | O2—C7—N1—C2 | −7.2 (2) |
| N1—C2—C9—C10 | 109.58 (18) | C8—C7—N1—C2 | 172.90 (15) |
| C3—C2—C9—C10 | −17.6 (2) | C17—C6—N1—C7 | −66.5 (2) |
| N1—C2—C9—C14 | −68.14 (18) | C5—C6—N1—C7 | 171.59 (15) |
| C3—C2—C9—C14 | 164.68 (16) | C17—C6—N1—C2 | 122.72 (15) |
| C14—C9—C10—C11 | 0.5 (3) | C5—C6—N1—C2 | 0.8 (2) |
| C2—C9—C10—C11 | −177.24 (17) | C9—C2—N1—C7 | 104.00 (16) |
| C9—C10—C11—C12 | 0.2 (3) | C3—C2—N1—C7 | −123.72 (15) |
| C10—C11—C12—C13 | −0.6 (3) | C9—C2—N1—C6 | −84.78 (17) |
| C11—C12—C13—C14 | 0.5 (3) | C3—C2—N1—C6 | 47.50 (18) |

*Hydrogen-bond geometry (Å, º)*

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C6—H6···O2[i] | 0.98 | 2.46 | 3.426 (2) | 168 |
| C8—H8*B*···O2[i] | 0.97 | 2.53 | 3.495 (3) | 172 |
| C21—H21···O1[ii] | 0.93 | 2.53 | 3.248 (3) | 134 |
| C11—H11···*Cg*1[iii] | 0.93 | 2.73 | 3.568 (2) | 150 |

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