Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 1-Chloroacetyl-2,6-bis(2-chlorophenyl)-3,5-dimethylpiperidin-4-one

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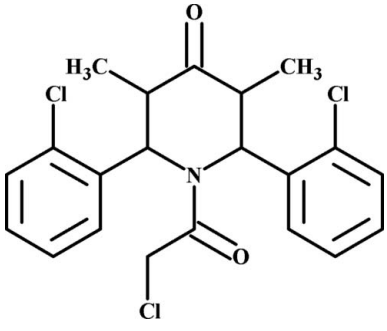
Received 15 July 2010; accepted 4 August 2010

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.045; $wR$ factor = 0.141; data-to-parameter ratio = 28.1.

In the title compound, $C_{21}H_{20}Cl_3NO_2$, the piperidin-4-one ring adopts a boat conformation. The two 2-chlorophenyl groups are approximately perpendicular to each other, making a dihedral angle of 74.07 (8)°.

## Related literature

For the biological activity of related structures, see: Parthiban *et al.* (2009); Aridoss *et al.* (2007). For spectroscopic studies of piperidin-4-ones, see: Ravindran *et al.* (1991); Krishnakumar *et al.* (1996). For ring conformational analysis, see: Cremer & Pople (1975); Nardelli (1983). For the synthesis of the title compound, see: Ramachandran *et al.* (2008); Aridoss *et al.* (2010).




## Experimental

### Crystal data

$C_{21}H_{20}Cl_3NO_2$ $M_r$ = 424.73
Monoclinic, $P2_1/n$
$a$ = 11.6295 (4) Å
$b$ = 9.6955 (3) Å
$c$ = 17.4743 (5) Å
$\beta$ = 90.481 (1)°
$V$ = 1970.22 (11) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.48 mm$^{-1}$
$T$ = 293 K
0.22 × 0.16 × 0.16 mm

### Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (Blessing, 1995)
$T_{min}$ = 0.901, $T_{max}$ = 0.927
27536 measured reflections
6864 independent reflections
4998 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.023

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.141
$S$ = 1.01
6864 reflections
244 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 e Å$^{-3}$

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

RR and YTJ are grateful for the support provided by the Industrial Technology Development Program–Ministry of Knowledge Economy of the Korean Government and 2010 Post-Doc. Research Program funded by Pukyong National University. The authors are thankful to the SAIF, Indian Institute of Technology, Madras, for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2226).

## References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
Aridoss, G., Balasubramanian, S., Parthiban, P., Ramachandran, R. & Kabilan, S. (2007). *Med. Chem. Res.* **16**, 188–204.
Aridoss, G., Sundaramoorthy, S., Velmurugan, D., Park, K. S. & Jeong, Y. T. (2010). *Acta Cryst.* E**66**, o1479.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Bruker (2004). *APEX2*, *SAINT* and *XPREP*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Krishnakumar, R. & Krishnapillay, M. (1996). *Indian J. Chem. Sect. B*, **35**, 418–425.
Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.
Parthiban, P., Aridoss, G., Rathika, P., Ramkumar, V. & Kabilan, S. (2009). *Bioorg. Med. Chem. Lett.* **19**, 2981–2985.
Ramachandran, R., Aridoss, G., Velmurugan, D., Kabilan, S. & Jeong, Y. T. (2008). *Acta Cryst.* E**64**, o2009–o2010.
Ravindran, T., Jeyaraman, R., Murray, R. W. & Singh, M. J. (1991). *J. Org. Chem.* **56**, 4833–4840.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supplementary materials

*Acta Cryst.* (2010). E**66**, o2284 [ **doi:10.1107/S1600536810031247** ]

# 1-Chloroacetyl-2,6-bis(2-chlorophenyl)-3,5-dimethylpiperidin-4-one

**R. Ramachandran, P. Parthiban, M. Rani, S. Kabilan and Y. T. Jeong**

## Comment

2,6-Disubstituted piperidones and their N-substituted compounds are of great interest due to their significant pharmacological properties (Parthiban *et al.*, 2009; Aridoss *et al.*, 2007). The introduction of electron withdrawing groups such as –CHO, $COCH_3$, COPh, NO, *etc*., at the ring nitrogen cause a major change in ring conformation (Ravindran *et al.*, 1991; Krishnakumar *et al.*, 1996). Hence, we introduced the chloroacetyl ($COCH_2Cl$) group into the piperidine ring in order to analyse the ring conformation through a single-crystal X-ray diffraction study.

In the molecular structure ($C_{21}H_{20}Cl_2NO_2$), the piperidine ring adopts a boat conformation with puckering parameters (Cremer & Pople, 1975) as follows: Total puckering amplitude, $Q_T$=0.6960 (15)Å and phase angle θ=85.52 (12)°. The smallest displacement asymmetry parameters (Nardelli, 1983) $q_1$ and $q_2$ are 0.6939 (15) Å and 0.0544 (15) Å, respectively. The dihedral angle between the two *o*-chlorophenyl rings is 74.07 (8) °.

## Experimental

The title compound was obtained by adopting an earlier method (Ramachandran *et al.* (2008); Aridoss *et al.*, 2010). To a well stirred solution of 3,5-dimethyl-2,6-bis(*o*-chloroyphenyl)piperidin-4-one (2 g, 4.71 mmol) and triethylamine (1.42 g, 14.13 mmol) in freshly distilled benzene (50 ml), chloroacetyl chloride (0.79 g, 7.06 mmol) in benzene (10 ml) was added drop-wise through the addition funnel over about half an hour. Stirring was continued until the completion of reaction. The reaction mixture was then poured into water and extracted with DCM. The solvent was removed under reduced pressure. The crude sample was purified by column chromatography. Upon recrystallization from absolute ethanol this afforded fine white crystals suitable for X-ray diffraction analysis.

## Refinement

H-atoms were positioned and refined using a riding model, with aromatic C—H = 0.93 Å, methine C—H = 0.98 Å, methylene C—H = 0.97 Å and methyl C—H = 0.96 Å. The displacement parameters were set for phenyl, methylene and aliphatic H atoms at $U_{iso}$(H)=1.2$U_{eq}$(C) or 1.5$_{eq}$(methyl C).

## Figures

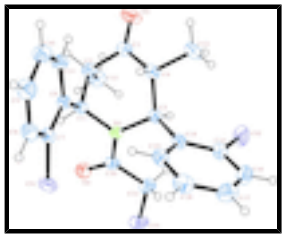


Fig. 1. The molecular structure of title compound, showing 50% probability displacement ellipsoids.

### 1-Chloroacetyl-2,6-bis(2-chlorophenyl)-3,5-dimethylpiperidin-4-one

*Crystal data*

| | |
|---|---|
| $C_{21}H_{20}Cl_3NO_2$ | $F(000) = 880$ |
| $M_r = 424.73$ | $D_x = 1.432$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2yn | Cell parameters from 5467 reflections |
| $a = 11.6295$ (4) Å | $\theta = 2.1$–$25.0°$ |
| $b = 9.6955$ (3) Å | $\mu = 0.48$ mm$^{-1}$ |
| $c = 17.4743$ (5) Å | $T = 293$ K |
| $\beta = 90.481$ (1)° | Prism, colourless |
| $V = 1970.22$ (11) Å$^3$ | 0.22 × 0.16 × 0.16 mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Bruker Kappa APEXII CCD diffractometer | 6864 independent reflections |
| Radiation source: fine-focus sealed tube | 4998 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.023$ |
| ω and φ scan | $\theta_{max} = 32.1°$, $\theta_{min} = 2.1°$ |
| Absorption correction: multi-scan (Blessing, 1995) | $h = -17\rightarrow17$ |
| $T_{min} = 0.901$, $T_{max} = 0.927$ | $k = -14\rightarrow14$ |
| 27536 measured reflections | $l = -26\rightarrow26$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.045$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.141$ | H-atom parameters constrained |
| $S = 1.01$ | $w = 1/[\sigma^2(F_o^2) + (0.0727P)^2 + 0.5226P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 6864 reflections | $(\Delta/\sigma)_{max} = 0.001$ |
| 244 parameters | $\Delta\rho_{max} = 0.40$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.31$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.85514 (12) | 0.37161 (13) | −0.00740 (8) | 0.0341 (3) |
| H1A | 0.8746 | 0.4692 | −0.0004 | 0.041* |
| C2 | 0.96025 (12) | 0.30363 (15) | −0.04343 (8) | 0.0385 (3) |
| H2A | 1.0237 | 0.3032 | −0.0062 | 0.046* |
| C3 | 0.93368 (13) | 0.15720 (15) | −0.06701 (9) | 0.0415 (3) |
| C4 | 0.80789 (13) | 0.11575 (13) | −0.07058 (8) | 0.0368 (3) |
| H4A | 0.7839 | 0.0981 | −0.0178 | 0.044* |
| C5 | 0.73061 (12) | 0.23387 (13) | −0.10157 (7) | 0.0340 (2) |
| H5A | 0.7456 | 0.2437 | −0.1564 | 0.041* |
| C6 | 0.82292 (12) | 0.31664 (14) | 0.07135 (7) | 0.0346 (3) |
| C7 | 0.73896 (13) | 0.37879 (15) | 0.11624 (8) | 0.0393 (3) |
| C8 | 0.70953 (16) | 0.32934 (19) | 0.18766 (9) | 0.0505 (4) |
| H8A | 0.6519 | 0.3724 | 0.2155 | 0.061* |
| C9 | 0.76589 (18) | 0.2157 (2) | 0.21762 (9) | 0.0562 (4) |
| H9A | 0.7457 | 0.1810 | 0.2653 | 0.067* |
| C10 | 0.85181 (17) | 0.15487 (18) | 0.17638 (10) | 0.0529 (4) |
| H10A | 0.8911 | 0.0794 | 0.1965 | 0.063* |
| C11 | 0.88047 (14) | 0.20513 (16) | 0.10491 (9) | 0.0432 (3) |
| H11A | 0.9400 | 0.1633 | 0.0783 | 0.052* |
| C12 | 0.99879 (15) | 0.37953 (19) | −0.11617 (10) | 0.0494 (4) |
| H12A | 1.0647 | 0.3339 | −0.1371 | 0.074* |
| H12B | 1.0184 | 0.4731 | −0.1036 | 0.074* |
| H12C | 0.9373 | 0.3790 | −0.1532 | 0.074* |
| C13 | 0.79149 (17) | −0.01856 (16) | −0.11449 (11) | 0.0516 (4) |
| H13A | 0.8409 | −0.0882 | −0.0931 | 0.077* |
| H13B | 0.8104 | −0.0044 | −0.1673 | 0.077* |
| H13C | 0.7129 | −0.0478 | −0.1108 | 0.077* |
| C14 | 0.60451 (13) | 0.19672 (14) | −0.09241 (8) | 0.0380 (3) |
| C15 | 0.53718 (15) | 0.14574 (17) | −0.15177 (10) | 0.0491 (4) |
| C16 | 0.42350 (17) | 0.1070 (2) | −0.14119 (13) | 0.0619 (5) |
| H16A | 0.3809 | 0.0702 | −0.1816 | 0.074* |
| C17 | 0.37424 (16) | 0.12352 (19) | −0.07036 (14) | 0.0614 (5) |
| H17A | 0.2978 | 0.0989 | −0.0630 | 0.074* |
| C18 | 0.43777 (16) | 0.17623 (19) | −0.01064 (12) | 0.0548 (4) |
| H18A | 0.4041 | 0.1889 | 0.0369 | 0.066* |
| C19 | 0.55201 (14) | 0.21042 (16) | −0.02132 (9) | 0.0432 (3) |
| H19A | 0.5949 | 0.2434 | 0.0199 | 0.052* |
| C20 | 0.73202 (12) | 0.49115 (14) | −0.09783 (8) | 0.0379 (3) |

| | | | | |
|---|---|---|---|---|
| C21 | 0.63849 (16) | 0.48474 (18) | −0.15890 (10) | 0.0514 (4) |
| H21A | 0.5701 | 0.4434 | −0.1374 | 0.062* |
| H21B | 0.6640 | 0.4265 | −0.2006 | 0.062* |
| O1 | 1.01004 (12) | 0.07846 (14) | −0.08337 (10) | 0.0692 (4) |
| O2 | 0.77569 (11) | 0.59995 (11) | −0.07971 (7) | 0.0528 (3) |
| N1 | 0.75946 (10) | 0.36719 (11) | −0.06397 (6) | 0.0326 (2) |
| Cl1 | 0.66924 (4) | 0.52622 (4) | 0.08431 (3) | 0.05574 (13) |
| Cl2 | 0.59251 (5) | 0.13268 (7) | −0.24418 (3) | 0.07505 (18) |
| Cl3 | 0.60408 (4) | 0.64954 (5) | −0.19502 (3) | 0.06205 (14) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0384 (6) | 0.0286 (6) | 0.0353 (6) | 0.0014 (5) | −0.0026 (5) | 0.0020 (4) |
| C2 | 0.0353 (6) | 0.0382 (7) | 0.0421 (7) | 0.0015 (5) | 0.0007 (5) | 0.0047 (5) |
| C3 | 0.0434 (7) | 0.0362 (7) | 0.0452 (7) | 0.0071 (6) | 0.0061 (6) | 0.0042 (5) |
| C4 | 0.0440 (7) | 0.0285 (6) | 0.0379 (6) | 0.0028 (5) | 0.0056 (5) | −0.0003 (5) |
| C5 | 0.0386 (6) | 0.0313 (6) | 0.0320 (6) | 0.0013 (5) | 0.0025 (5) | −0.0023 (4) |
| C6 | 0.0407 (6) | 0.0305 (6) | 0.0325 (6) | 0.0025 (5) | −0.0022 (5) | −0.0009 (5) |
| C7 | 0.0441 (7) | 0.0364 (6) | 0.0373 (6) | 0.0048 (5) | −0.0030 (5) | −0.0051 (5) |
| C8 | 0.0543 (9) | 0.0583 (9) | 0.0390 (7) | 0.0055 (7) | 0.0056 (7) | −0.0077 (7) |
| C9 | 0.0723 (11) | 0.0619 (10) | 0.0345 (7) | 0.0008 (9) | 0.0044 (7) | 0.0058 (7) |
| C10 | 0.0677 (11) | 0.0490 (9) | 0.0419 (8) | 0.0094 (8) | −0.0027 (7) | 0.0106 (6) |
| C11 | 0.0509 (8) | 0.0402 (7) | 0.0386 (7) | 0.0104 (6) | −0.0005 (6) | 0.0041 (6) |
| C12 | 0.0462 (8) | 0.0539 (9) | 0.0482 (8) | −0.0054 (7) | 0.0069 (7) | 0.0089 (7) |
| C13 | 0.0646 (10) | 0.0333 (7) | 0.0571 (9) | 0.0017 (7) | 0.0077 (8) | −0.0078 (6) |
| C14 | 0.0404 (7) | 0.0328 (6) | 0.0410 (7) | −0.0012 (5) | 0.0011 (5) | −0.0040 (5) |
| C15 | 0.0493 (8) | 0.0474 (8) | 0.0504 (8) | −0.0004 (7) | −0.0060 (7) | −0.0089 (7) |
| C16 | 0.0525 (10) | 0.0523 (10) | 0.0804 (13) | −0.0082 (8) | −0.0200 (9) | −0.0034 (9) |
| C17 | 0.0431 (9) | 0.0483 (9) | 0.0929 (15) | −0.0075 (7) | 0.0031 (9) | 0.0139 (9) |
| C18 | 0.0503 (9) | 0.0458 (8) | 0.0686 (11) | −0.0028 (7) | 0.0156 (8) | 0.0093 (8) |
| C19 | 0.0464 (8) | 0.0377 (7) | 0.0455 (7) | −0.0033 (6) | 0.0074 (6) | −0.0003 (6) |
| C20 | 0.0412 (7) | 0.0346 (6) | 0.0379 (6) | 0.0049 (5) | 0.0015 (5) | 0.0057 (5) |
| C21 | 0.0559 (9) | 0.0466 (8) | 0.0516 (9) | 0.0061 (7) | −0.0126 (7) | 0.0126 (7) |
| O1 | 0.0535 (7) | 0.0504 (7) | 0.1039 (12) | 0.0162 (6) | 0.0167 (7) | −0.0077 (7) |
| O2 | 0.0664 (8) | 0.0322 (5) | 0.0596 (7) | −0.0005 (5) | −0.0111 (6) | 0.0084 (5) |
| N1 | 0.0375 (5) | 0.0277 (5) | 0.0325 (5) | 0.0015 (4) | −0.0017 (4) | 0.0018 (4) |
| Cl1 | 0.0653 (3) | 0.0465 (2) | 0.0555 (2) | 0.02331 (18) | 0.00334 (19) | −0.00359 (17) |
| Cl2 | 0.0761 (3) | 0.1018 (4) | 0.0471 (2) | 0.0034 (3) | −0.0109 (2) | −0.0279 (2) |
| Cl3 | 0.0654 (3) | 0.0601 (3) | 0.0607 (3) | 0.0222 (2) | −0.0028 (2) | 0.0202 (2) |

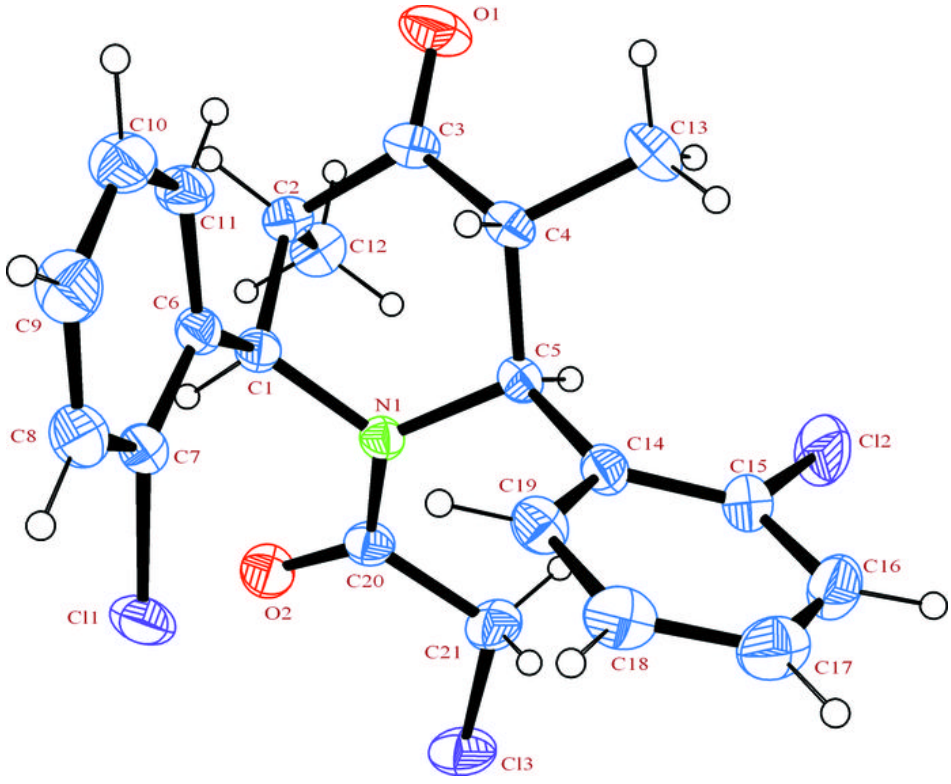
*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—N1 | 1.4828 (17) | C11—H11A | 0.9300 |
| C1—C6 | 1.5253 (18) | C12—H12A | 0.9600 |
| C1—C2 | 1.5291 (19) | C12—H12B | 0.9600 |
| C1—H1A | 0.9800 | C12—H12C | 0.9600 |
| C2—C3 | 1.510 (2) | C13—H13A | 0.9600 |
| C2—C12 | 1.539 (2) | C13—H13B | 0.9600 |

| | | | |
|---|---|---|---|
| C2—H2A | 0.9800 | C13—H13C | 0.9600 |
| C3—O1 | 1.2072 (18) | C14—C15 | 1.386 (2) |
| C3—C4 | 1.518 (2) | C14—C19 | 1.395 (2) |
| C4—C13 | 1.523 (2) | C15—C16 | 1.388 (3) |
| C4—C5 | 1.5506 (19) | C15—Cl2 | 1.7480 (19) |
| C4—H4A | 0.9800 | C16—C17 | 1.378 (3) |
| C5—N1 | 1.4871 (17) | C16—H16A | 0.9300 |
| C5—C14 | 1.520 (2) | C17—C18 | 1.372 (3) |
| C5—H5A | 0.9800 | C17—H17A | 0.9300 |
| C6—C7 | 1.3946 (19) | C18—C19 | 1.383 (2) |
| C6—C11 | 1.3979 (19) | C18—H18A | 0.9300 |
| C7—C8 | 1.383 (2) | C19—H19A | 0.9300 |
| C7—Cl1 | 1.7333 (15) | C20—O2 | 1.2117 (19) |
| C8—C9 | 1.382 (3) | C20—N1 | 1.3760 (16) |
| C8—H8A | 0.9300 | C20—C21 | 1.519 (2) |
| C9—C10 | 1.370 (3) | C21—Cl3 | 1.7628 (16) |
| C9—H9A | 0.9300 | C21—H21A | 0.9700 |
| C10—C11 | 1.384 (2) | C21—H21B | 0.9700 |
| C10—H10A | 0.9300 | | |
| N1—C1—C6 | 113.75 (11) | C6—C11—H11A | 118.9 |
| N1—C1—C2 | 108.12 (11) | C2—C12—H12A | 109.5 |
| C6—C1—C2 | 115.10 (11) | C2—C12—H12B | 109.5 |
| N1—C1—H1A | 106.4 | H12A—C12—H12B | 109.5 |
| C6—C1—H1A | 106.4 | C2—C12—H12C | 109.5 |
| C2—C1—H1A | 106.4 | H12A—C12—H12C | 109.5 |
| C3—C2—C1 | 110.81 (12) | H12B—C12—H12C | 109.5 |
| C3—C2—C12 | 106.54 (13) | C4—C13—H13A | 109.5 |
| C1—C2—C12 | 111.93 (12) | C4—C13—H13B | 109.5 |
| C3—C2—H2A | 109.2 | H13A—C13—H13B | 109.5 |
| C1—C2—H2A | 109.2 | C4—C13—H13C | 109.5 |
| C12—C2—H2A | 109.2 | H13A—C13—H13C | 109.5 |
| O1—C3—C2 | 120.67 (15) | H13B—C13—H13C | 109.5 |
| O1—C3—C4 | 122.25 (15) | C15—C14—C19 | 116.86 (14) |
| C2—C3—C4 | 117.08 (12) | C15—C14—C5 | 123.05 (13) |
| C3—C4—C13 | 111.32 (13) | C19—C14—C5 | 120.08 (13) |
| C3—C4—C5 | 111.98 (11) | C14—C15—C16 | 121.98 (17) |
| C13—C4—C5 | 112.70 (13) | C14—C15—Cl2 | 120.50 (13) |
| C3—C4—H4A | 106.8 | C16—C15—Cl2 | 117.50 (14) |
| C13—C4—H4A | 106.8 | C17—C16—C15 | 119.46 (18) |
| C5—C4—H4A | 106.8 | C17—C16—H16A | 120.3 |
| N1—C5—C14 | 111.94 (10) | C15—C16—H16A | 120.3 |
| N1—C5—C4 | 111.08 (11) | C18—C17—C16 | 120.09 (17) |
| C14—C5—C4 | 110.19 (11) | C18—C17—H17A | 120.0 |
| N1—C5—H5A | 107.8 | C16—C17—H17A | 120.0 |
| C14—C5—H5A | 107.8 | C17—C18—C19 | 119.88 (18) |
| C4—C5—H5A | 107.8 | C17—C18—H18A | 120.1 |
| C7—C6—C11 | 115.67 (13) | C19—C18—H18A | 120.1 |
| C7—C6—C1 | 122.34 (12) | C18—C19—C14 | 121.68 (16) |
| C11—C6—C1 | 121.90 (12) | C18—C19—H19A | 119.2 |

| | | | |
|---|---|---|---|
| C8—C7—C6 | 122.53 (14) | C14—C19—H19A | 119.2 |
| C8—C7—Cl1 | 117.25 (12) | O2—C20—N1 | 123.57 (13) |
| C6—C7—Cl1 | 120.20 (11) | O2—C20—C21 | 121.02 (13) |
| C9—C8—C7 | 119.87 (15) | N1—C20—C21 | 115.37 (13) |
| C9—C8—H8A | 120.1 | C20—C21—Cl3 | 111.94 (12) |
| C7—C8—H8A | 120.1 | C20—C21—H21A | 109.2 |
| C10—C9—C8 | 119.31 (15) | Cl3—C21—H21A | 109.2 |
| C10—C9—H9A | 120.3 | C20—C21—H21B | 109.2 |
| C8—C9—H9A | 120.3 | Cl3—C21—H21B | 109.2 |
| C9—C10—C11 | 120.33 (16) | H21A—C21—H21B | 107.9 |
| C9—C10—H10A | 119.8 | C20—N1—C1 | 115.56 (11) |
| C11—C10—H10A | 119.8 | C20—N1—C5 | 121.24 (11) |
| C10—C11—C6 | 122.20 (15) | C1—N1—C5 | 119.01 (10) |
| C10—C11—H11A | 118.9 | | |
| N1—C1—C2—C3 | −58.03 (14) | C1—C6—C11—C10 | 179.89 (15) |
| C6—C1—C2—C3 | 70.37 (15) | N1—C5—C14—C15 | 136.19 (14) |
| N1—C1—C2—C12 | 60.74 (15) | C4—C5—C14—C15 | −99.67 (16) |
| C6—C1—C2—C12 | −170.85 (12) | N1—C5—C14—C19 | −45.29 (17) |
| C1—C2—C3—O1 | −166.27 (15) | C4—C5—C14—C19 | 78.86 (16) |
| C12—C2—C3—O1 | 71.75 (19) | C19—C14—C15—C16 | −1.4 (2) |
| C1—C2—C3—C4 | 14.92 (17) | C5—C14—C15—C16 | 177.20 (16) |
| C12—C2—C3—C4 | −107.06 (14) | C19—C14—C15—Cl2 | 176.79 (12) |
| O1—C3—C4—C13 | −13.9 (2) | C5—C14—C15—Cl2 | −4.6 (2) |
| C2—C3—C4—C13 | 164.91 (13) | C14—C15—C16—C17 | 2.2 (3) |
| O1—C3—C4—C5 | −141.05 (16) | Cl2—C15—C16—C17 | −176.06 (15) |
| C2—C3—C4—C5 | 37.74 (17) | C15—C16—C17—C18 | −0.8 (3) |
| C3—C4—C5—N1 | −46.40 (15) | C16—C17—C18—C19 | −1.2 (3) |
| C13—C4—C5—N1 | −172.84 (12) | C17—C18—C19—C14 | 2.0 (3) |
| C3—C4—C5—C14 | −171.04 (11) | C15—C14—C19—C18 | −0.7 (2) |
| C13—C4—C5—C14 | 62.52 (15) | C5—C14—C19—C18 | −179.31 (14) |
| N1—C1—C6—C7 | −62.84 (17) | O2—C20—C21—Cl3 | −1.2 (2) |
| C2—C1—C6—C7 | 171.62 (13) | N1—C20—C21—Cl3 | 176.76 (11) |
| N1—C1—C6—C11 | 120.85 (14) | O2—C20—N1—C1 | −5.4 (2) |
| C2—C1—C6—C11 | −4.69 (19) | C21—C20—N1—C1 | 176.75 (13) |
| C11—C6—C7—C8 | −3.4 (2) | O2—C20—N1—C5 | −162.10 (14) |
| C1—C6—C7—C8 | −179.97 (14) | C21—C20—N1—C5 | 20.01 (19) |
| C11—C6—C7—Cl1 | 174.97 (12) | C6—C1—N1—C20 | 123.99 (12) |
| C1—C6—C7—Cl1 | −1.55 (19) | C2—C1—N1—C20 | −106.85 (13) |
| C6—C7—C8—C9 | 1.4 (3) | C6—C1—N1—C5 | −78.72 (14) |
| Cl1—C7—C8—C9 | −177.11 (14) | C2—C1—N1—C5 | 50.45 (14) |
| C7—C8—C9—C10 | 1.0 (3) | C14—C5—N1—C20 | −78.22 (15) |
| C8—C9—C10—C11 | −1.1 (3) | C4—C5—N1—C20 | 158.14 (12) |
| C9—C10—C11—C6 | −1.2 (3) | C14—C5—N1—C1 | 125.81 (12) |
| C7—C6—C11—C10 | 3.4 (2) | C4—C5—N1—C1 | 2.17 (16) |

**Fig. 1**


O1
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C18
C17
Cl3