# data reports

## Crystal structure of the adduct (4-chlorophenyl)(4-hydroxypiperidin-1-yl)methanone–(4-chlorophenyl)-(piperidin-1-yl)methanone (0.75/0.25)

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In the title compound, $0.75C_{12}H_{14}ClNO_2 \cdot 0.25C_{12}H_{14}ClNO$, which is an adduct comprising 0.75 4-hydroxypiperidin-1-yl or 0.25 4-piperidin-1-yl substituents on a common (4-chlorophenyl)methanone component; the dihedral angles between the benzene ring and the two piperidine rings are 51.6 (3) and 89.5 (7)°, respectively. The hydroxypiperidine ring is in a bisectional orientation (bi) with the phenyl ring. In the crystal, intermolecular O—H⋯O hydrogen bonds between the hydroxypiperidine group and the keto O atom lead to the formation of chains extending along the c- axis direction.

## 1. Related literature

For the synthesis, see: Revathi et al. (2015). For the biological activity of piperidine derivatives, see: Ramalingan et al. (2004); Sargent & May (1970); Rubiralta et al. (1991). For related structures, see: Revathi et al. (2015); Prathebha et al. (2015).
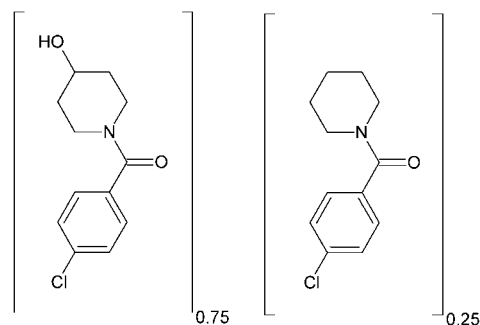


## 2. Experimental

### 2.1. Crystal data

| | |
|---|---|
| $0.75C_{12}H_{14}ClNO_2 \cdot 0.25C_{12}H_{14}ClNO$ | $V = 1193.5$ (3) $Å^3$ |
| $M_r = 235.69$ | $Z = 4$ |
| Orthorhombic, $Pca2_1$ | Mo $K\alpha$ radiation |
| $a = 24.312$ (4) Å | $\mu = 0.30$ mm$^{-1}$ |
| $b = 6.1628$ (10) Å | $T = 293$ K |
| $c = 7.9654$ (11) Å | $0.25 \times 0.20 \times 0.20$ mm |

### 2.2. Data collection

| | |
|---|---|
| Bruker Kappa APEXII CCD diffractometer | 17321 measured reflections |
| Absorption correction: multi-scan (SADABS; Bruker, 2004) | 2356 independent reflections |
| | 1539 reflections with $I > 2\sigma(I)$ |
| $T_{min} = 0.930$, $T_{max} = 0.941$ | $R_{int} = 0.031$ |

### 2.3. Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.048$ | $\Delta\rho_{min} = -0.22$ e Å$^{-3}$ |
| $wR(F^2) = 0.144$ | Absolute structure: Flack $x$ determined using 583 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al., 2013) |
| $S = 1.02$ | |
| 2356 reflections | |
| 200 parameters | |
| 121 restraints | Absolute structure parameter: 0.03 (3) |
| H-atom parameters constrained | |
| $\Delta\rho_{max} = 0.31$ e Å$^{-3}$ | |

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

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| O2—H2A⋯O1[i] | 0.82 | 2.05 | 2.693 (7) | 135 |

Symmetry code: (i) $x, y, z - 1$.

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: SHELXL2014 (Sheldrick, 2015); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012) and Mercury (Bruno et al., 2002); software used to prepare material for publication: SHELXL2014.

## Acknowledgements

the computing facility and SAIF, IIT, Madras, for the X-ray data collection facility.

## References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
Bruker (2004). *SAINT, APEX2, XPREP* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruno, V., Castaldo, A., Centore, R., Sirigu, A., Sarcinelli, F., Casalboni, M. & Pizzoferrato, R. (2002). *J. Polym. Sci. A Polym. Chem.* **40**, 1468–1475.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Prathebha, K., Reuben Jonathan, D., Revathi, B. K., Sathya, S. & Usha, G. (2015). *Acta Cryst.* E**71**, o39–o40.
Ramalingan, C., Balasubramanian, S., Kabilan, S. & Vasudevan, M. (2004). *Eur. J. Med. Chem.* **39**, 527–533.
Revathi, B. K., Reuben Jonathan, D., Sathya, S., Prathebha, K. & Usha, G. (2015). *Acta Cryst.* E**71**, o359–o360.
Rubiralta, M., Giralt, E. & Diez, A. (1991). *Piperidine: Structure, Preparation, Reactivity, and Synthetic Applications of Piperidine and its Derivatives*, pp. 225–312. Amsterdam: Elsevier.
Sargent, L. J. & May, E. L. (1970). *J. Med. Chem.* **13**, 1061–1063.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

# supporting information

## Crystal structure of the adduct (4-chlorophenyl)(4-hydroxypiperidin-1-yl)methanone–(4-chlorophenyl)(piperidin-1-yl)methanone (0.75/0.25)

### B. K. Revathi, D. Reuben Jonathan, K. Kalai Sevi, K. Dhanalakshmi and G. Usha

### S1. Comment

Piperidine is very important pharmacophore because of its presence in numerous alkaloids, pharmaceuticals, agrochemicals and as synthetic intermediates. Biologically active alkaloids with substituted piperidine ring systems have been targeted for their total or partial synthesis (Ramalingan *et al.*, 2004). Piperidines are known to have CNS depressant action at low dosage levels and stimulant activity with increased dosages. In addition, the nucleus also possesses analgesic, ganglionic blocking and anesthetic properties as well (Sargent & May, 1970; Rubiralta *et al.*, 1991).

   In the title compound, $0.75(C_{12}H_{14}NO_2Cl)$ . $0.25(C_{12}H_{14}NOCl)$, which is an adduct comprising 0.75(4-hydroxy-piperidin-1-yl) or 0.25(4-piperidin-1-yl) substituents on a common (4-chlorophenyl)methanone component, the dihedral angles between the benzene ring and the two piperidine rings defined by N1–C12 and N1′–C12′ are 51.6 (3) and 89.5 (7)°, respectively (Figs. 1, 2). The C—C distances in the hydroxypiperidine ring and the benzene ring are in the range [1.472 (9)–1.529 (8)Å and 1.346 (7)–1.390 (8)Å], respectively and are in good agreement with literature values (Allen *et al.*, 1987). The C—N distances are in the range [1.346 (7)Å - 1.465 (7)Å] and are in good agreement with values in a similar reported structure (Revathi *et al.*, 2015). The C—O distance [1.223 (6)Å] indicates double bond character and is comparable with the value reported previously (Prathebha *et al.*, 2015). The hydroxypiperdine ring is in a bisectional orientation (*bi*) with the phenyl ring. The sum of the bond angle around the N1 atom is [359.9 (5)°], showing $sp^2$ hybridization of the atoms. The torsion angle C8—N1—C7—O1 [12.3 (10)°], indicates that the keto group is in a +*syn-periplanar* (+*sp*) orientation with the hydroxy piperidine ring. The hydroxypiperidine ring adopts a chair conformation with puckering parameters of q2 = 0.029Å, phi2 = -173.57° q3 = -0.555Å, QT = 0.555Å and theta2 = 176.95°.

   In the crystal, molecules are linked by O2—H···O1$^i$ hydrogen bonds (Table 1), forming one-dimensional chains extending along *c* (Fig. 3). Present also are very weak inter-chain C12′—H···Cl1$^{ii}$ interactions [3.63 (2) Å]. For symmetry code (ii) -*x* + 3/2, -*y* + 1, *z* - 1.

### S2. Experimental

The title compound was synthesized by utilizing a reported procedure (Revathi *et al.*, 2015). In a 250 ml round-bottomed flask, 130 ml of ethylmethylketone was added to 4-hydroxypiperidiene (0.04 mol) and stirred well. Triethylamine (0.04 mol) was then added and the mixture was stirred for 10 min. 4-Chlorobenzoyl chloride (0.04 mol) was added and the reaction mixture was stirred at room temperature for about 2 hr. A white precipitate of triethylammonium chloride was produced, which was filtered and the filtrate was evaporated to obtain the crude product, crystallization from ethyl-methylketone gave colourless block-like crystals of the unexpected title adduct (yield: 88%).

## S3. Refinement

H atoms were positioned geometrically and treated as riding on their parent atoms and refined with, C—H distances of 0.93–0.98 Å, an O—H distance of 0.82 Å, with $U_{iso}(H) = 1.5 U_{eq}(C\text{-methyl})$, $U_{iso}(H) = 1.2U_{eq}(C,O)$ for other H atoms. The value of the absolute structure parameter (Parsons *et al.*, 2013), although of no relevance for the present structure was determined as 0.03 (3) using 583 quotients $[(I+)-(I-)]/[(I+)+(I-)]$.



**Figure 1**

The molecular structure of the major (73%) 4-hydroxypiperidin-1-yl substituted component of the title adduct, showing atom numbering, with displacement ellipsoids drawn at the 30% probability level.

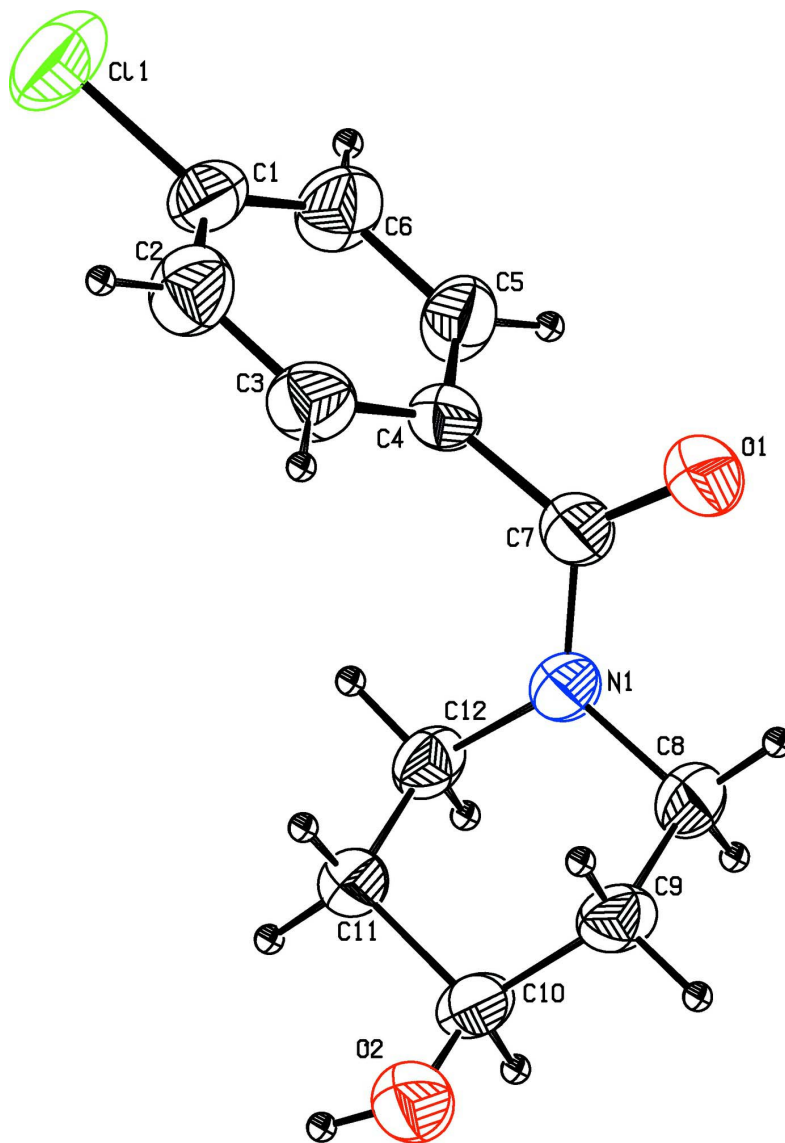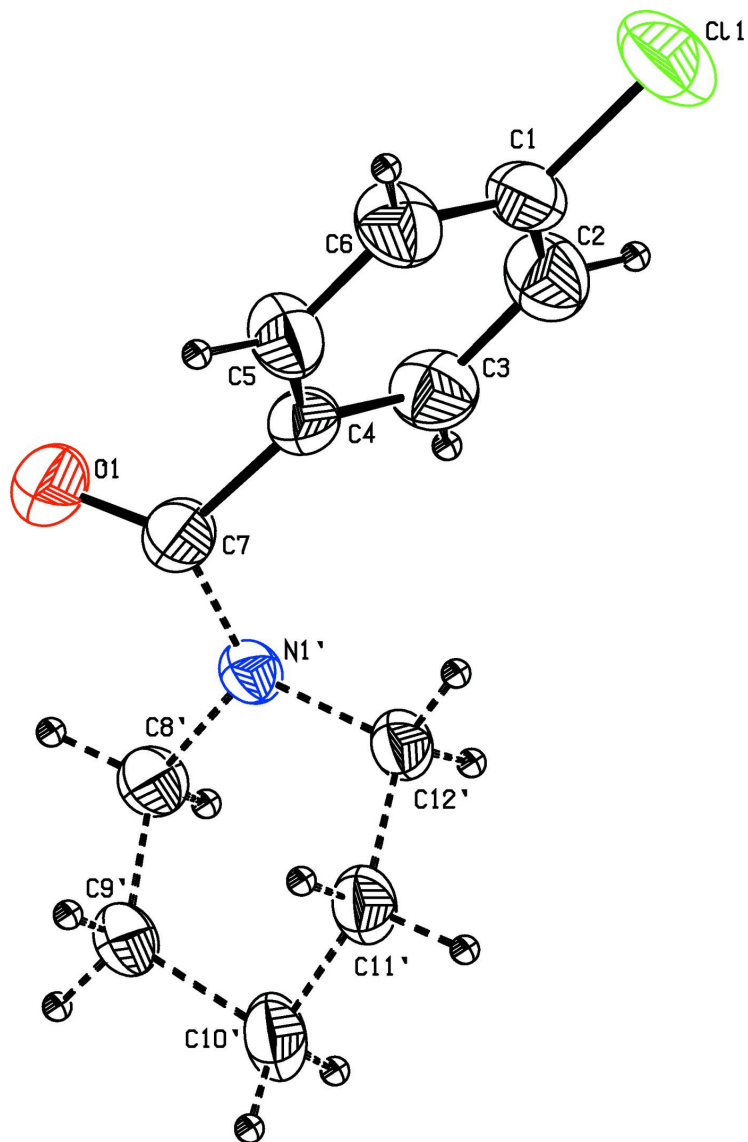**Figure 2**

The molecular structure of the minor (25%) piperidin-1-yl substituted component of the title adduct, showing atom numbering, with displacement ellipsoids drawn at the 30% probability level. The bonds for the minor-occupancy piperidinyl group are shown as dashed lines
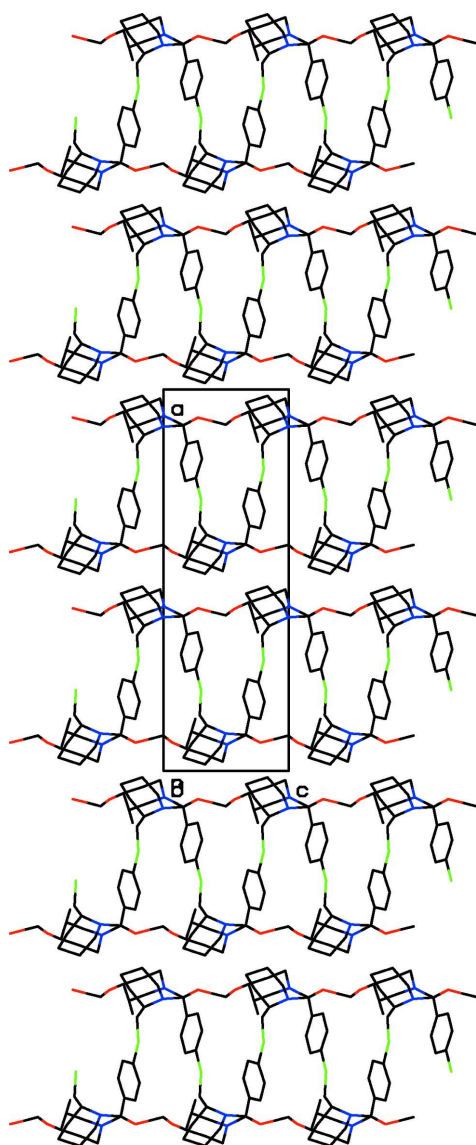
**Figure 3**

The packing of the molecules in the crystal structure. The dashed lines indicate the O—H⋯O hydrogen bonds and weak inter-chain C—H⋯Cl interactions.

**(4-Chlorophenyl)(4-hydroxypiperidin-1-yl)methanone–(4-chlorophenyl)(piperidin-1-yl)methanone (0.75/0.25)**

*Crystal data*

| | |
|---|---|
| $0.75C_{12}H_{14}ClNO_2 \cdot 0.25C_{12}H_{14}ClNO$ | $D_x = 1.312$ Mg m$^{-3}$ |
| $M_r = 235.69$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Orthorhombic, $Pca2_1$ | Cell parameters from 3452 reflections |
| $a = 24.312$ (4) Å | $\theta = 2.8$–23.2° |
| $b = 6.1628$ (10) Å | $\mu = 0.30$ mm$^{-1}$ |
| $c = 7.9654$ (11) Å | $T = 293$ K |
| $V = 1193.5$ (3) Å$^3$ | Block, colourless |
| $Z = 4$ | $0.25 \times 0.20 \times 0.20$ mm |
| $F(000) = 496$ | |

*Data collection*

| | |
|---|---|
| Bruker Kappa APEXII CCD diffractometer | 2356 independent reflections |
| Radiation source: Sealed tube | 1539 reflections with $I > 2\sigma(I)$ |
| $\omega$ and $\varphi$ scan | $R_{int} = 0.031$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2004) | $\theta_{max} = 26.1°$, $\theta_{min} = 3.1°$ |
| | $h = -30 \rightarrow 30$ |
| $T_{min} = 0.930$, $T_{max} = 0.941$ | $k = -7 \rightarrow 7$ |
| 17321 measured reflections | $l = -9 \rightarrow 9$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.048$ | $w = 1/[\sigma^2(F_o^2) + (0.0664P)^2 + 0.3379P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.144$ | |
| $S = 1.02$ | |
| 2356 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 200 parameters | $\Delta\rho_{max} = 0.31$ e Å$^{-3}$ |
| 121 restraints | $\Delta\rho_{min} = -0.22$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Absolute structure: Flack $x$ determined using 583 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013) |
| Secondary atom site location: difference Fourier map | Absolute structure parameter: 0.03 (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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.73230 (19) | 0.1884 (8) | 0.7580 (6) | 0.0743 (13) | |
| C2 | 0.7397 (2) | 0.3762 (10) | 0.6747 (8) | 0.0909 (16) | |
| H2 | 0.7748 | 0.4191 | 0.6416 | 0.109* | |
| C3 | 0.6943 (2) | 0.5050 (9) | 0.6390 (7) | 0.0832 (15) | |
| H3 | 0.6991 | 0.6353 | 0.5819 | 0.100* | |
| C4 | 0.64254 (19) | 0.4423 (8) | 0.6867 (5) | 0.0656 (12) | |
| C5 | 0.63637 (19) | 0.2510 (8) | 0.7729 (8) | 0.0811 (14) | |
| H5 | 0.6016 | 0.2076 | 0.8082 | 0.097* | |
| C6 | 0.6815 (2) | 0.1230 (8) | 0.8074 (8) | 0.0890 (16) | |
| H6 | 0.6771 | −0.0076 | 0.8643 | 0.107* | |
| C7 | 0.5944 (2) | 0.5838 (9) | 0.6575 (6) | 0.0773 (14) | |
| C8 | 0.5243 (3) | 0.7357 (12) | 0.4730 (9) | 0.0749 (18) | 0.75 |
| H8A | 0.5155 | 0.8165 | 0.5739 | 0.090* | 0.75 |
| H8B | 0.4918 | 0.6565 | 0.4381 | 0.090* | 0.75 |
| C9 | 0.5419 (3) | 0.8877 (11) | 0.3372 (9) | 0.0681 (17) | 0.75 |
| H9A | 0.5727 | 0.9741 | 0.3766 | 0.082* | 0.75 |
| H9B | 0.5119 | 0.9855 | 0.3109 | 0.082* | 0.75 |
| C10 | 0.5587 (3) | 0.7668 (13) | 0.1804 (9) | 0.0711 (19) | 0.75 |

| | | | | | |
|---|---|---|---|---|---|
| H10 | 0.5263 | 0.6897 | 0.1375 | 0.085* | 0.75 |
| C11 | 0.6028 (3) | 0.5988 (12) | 0.2233 (8) | 0.0690 (16) | 0.75 |
| H11A | 0.6102 | 0.5109 | 0.1249 | 0.083* | 0.75 |
| H11B | 0.6365 | 0.6732 | 0.2537 | 0.083* | 0.75 |
| C12 | 0.5859 (3) | 0.4567 (10) | 0.3627 (8) | 0.0640 (14) | 0.75 |
| H12A | 0.5554 | 0.3660 | 0.3269 | 0.077* | 0.75 |
| H12B | 0.6162 | 0.3627 | 0.3935 | 0.077* | 0.75 |
| N1 | 0.5694 (2) | 0.5841 (9) | 0.5067 (6) | 0.0659 (14) | 0.75 |
| O2 | 0.5746 (2) | 0.8993 (9) | 0.0685 (7) | 0.0870 (14) | 0.75 |
| H2A | 0.5924 | 0.8339 | −0.0030 | 0.105 (17)* | 0.75 |
| C8′ | 0.5628 (8) | 0.863 (3) | 0.454 (3) | 0.069 (4) | 0.25 |
| H8′1 | 0.5850 | 0.9758 | 0.4031 | 0.083* | 0.25 |
| H8′2 | 0.5466 | 0.9203 | 0.5556 | 0.083* | 0.25 |
| C9′ | 0.5169 (8) | 0.795 (4) | 0.331 (2) | 0.072 (5) | 0.25 |
| H9′1 | 0.4939 | 0.9210 | 0.3107 | 0.087* | 0.25 |
| H9′2 | 0.4942 | 0.6872 | 0.3865 | 0.087* | 0.25 |
| C10′ | 0.5350 (9) | 0.703 (4) | 0.160 (2) | 0.077 (6) | 0.25 |
| H10A | 0.5559 | 0.8073 | 0.0953 | 0.093* | 0.25 |
| H10B | 0.5040 | 0.6510 | 0.0946 | 0.093* | 0.25 |
| C11′ | 0.5714 (8) | 0.515 (3) | 0.226 (3) | 0.069 (5) | 0.25 |
| H11C | 0.5475 | 0.4128 | 0.2838 | 0.082* | 0.25 |
| H11D | 0.5864 | 0.4396 | 0.1295 | 0.082* | 0.25 |
| C12′ | 0.6196 (7) | 0.568 (3) | 0.345 (2) | 0.057 (4) | 0.25 |
| H12C | 0.6383 | 0.4350 | 0.3775 | 0.069* | 0.25 |
| H12D | 0.6458 | 0.6610 | 0.2887 | 0.069* | 0.25 |
| N1′ | 0.5977 (6) | 0.677 (2) | 0.4953 (15) | 0.053 (3) | 0.25 |
| O1 | 0.57385 (17) | 0.6828 (7) | 0.7749 (5) | 0.1119 (14) | |
| Cl1 | 0.78823 (7) | 0.0274 (3) | 0.7998 (3) | 0.1274 (8) | |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.067 (3) | 0.080 (3) | 0.076 (3) | 0.011 (2) | −0.016 (2) | −0.004 (3) |
| C2 | 0.070 (3) | 0.098 (4) | 0.104 (4) | −0.010 (3) | 0.001 (3) | 0.003 (4) |
| C3 | 0.091 (4) | 0.076 (3) | 0.083 (4) | −0.003 (3) | −0.002 (3) | 0.018 (3) |
| C4 | 0.073 (3) | 0.071 (3) | 0.053 (2) | 0.012 (2) | 0.002 (2) | 0.010 (2) |
| C5 | 0.066 (3) | 0.080 (3) | 0.097 (3) | 0.007 (3) | 0.011 (3) | 0.019 (3) |
| C6 | 0.092 (4) | 0.072 (3) | 0.103 (4) | 0.017 (3) | 0.001 (3) | 0.023 (3) |
| C7 | 0.090 (3) | 0.082 (3) | 0.061 (2) | 0.029 (3) | 0.005 (2) | 0.006 (2) |
| C8 | 0.074 (4) | 0.076 (4) | 0.075 (4) | 0.031 (3) | 0.005 (3) | 0.011 (3) |
| C9 | 0.080 (5) | 0.057 (3) | 0.068 (4) | 0.018 (3) | −0.012 (3) | 0.004 (3) |
| C10 | 0.067 (4) | 0.081 (4) | 0.066 (3) | 0.020 (3) | −0.018 (3) | −0.003 (3) |
| C11 | 0.067 (4) | 0.082 (4) | 0.058 (4) | 0.020 (3) | −0.004 (3) | −0.004 (3) |
| C12 | 0.063 (3) | 0.056 (3) | 0.074 (3) | 0.017 (3) | −0.010 (3) | −0.008 (3) |
| N1 | 0.072 (3) | 0.064 (3) | 0.062 (3) | 0.023 (3) | −0.001 (2) | 0.006 (2) |
| O2 | 0.097 (4) | 0.088 (3) | 0.076 (3) | 0.017 (3) | 0.004 (3) | 0.004 (3) |
| C8′ | 0.079 (9) | 0.064 (8) | 0.064 (9) | 0.032 (7) | −0.003 (8) | −0.001 (6) |
| C9′ | 0.067 (9) | 0.080 (12) | 0.071 (9) | 0.034 (8) | −0.001 (7) | −0.007 (9) |

| | | | | | |
|---|---|---|---|---|---|
| C10′ | 0.057 (11) | 0.085 (12) | 0.090 (9) | 0.012 (9) | 0.012 (7) | −0.020 (8) |
| C11′ | 0.054 (8) | 0.079 (10) | 0.073 (9) | 0.006 (7) | 0.002 (7) | −0.023 (7) |
| C12′ | 0.050 (7) | 0.059 (9) | 0.062 (7) | 0.009 (7) | 0.001 (6) | −0.008 (6) |
| N1′ | 0.051 (7) | 0.052 (6) | 0.055 (4) | 0.012 (5) | −0.004 (4) | −0.004 (4) |
| O1 | 0.121 (3) | 0.147 (3) | 0.067 (2) | 0.057 (3) | 0.008 (2) | −0.004 (3) |
| Cl1 | 0.0952 (10) | 0.1290 (13) | 0.1579 (17) | 0.0462 (9) | −0.0304 (12) | −0.0134 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.346 (7) | C10—H10 | 0.9800 |
| C1—C6 | 1.358 (7) | C11—C12 | 1.472 (9) |
| C1—Cl1 | 1.716 (5) | C11—H11A | 0.9700 |
| C2—C3 | 1.390 (8) | C11—H11B | 0.9700 |
| C2—H2 | 0.9300 | C12—N1 | 1.447 (8) |
| C3—C4 | 1.370 (7) | C12—H12A | 0.9700 |
| C3—H3 | 0.9300 | C12—H12B | 0.9700 |
| C4—C5 | 1.373 (7) | O2—H2A | 0.8200 |
| C4—C7 | 1.478 (7) | C8′—N1′ | 1.460 (16) |
| C5—C6 | 1.378 (6) | C8′—C9′ | 1.54 (2) |
| C5—H5 | 0.9300 | C8′—H8′1 | 0.9700 |
| C6—H6 | 0.9300 | C8′—H8′2 | 0.9700 |
| C7—O1 | 1.223 (6) | C9′—C10′ | 1.540 (12) |
| C7—N1 | 1.346 (7) | C9′—H9′1 | 0.9700 |
| C7—N1′ | 1.417 (13) | C9′—H9′2 | 0.9700 |
| C8—N1 | 1.465 (7) | C10′—C11′ | 1.552 (12) |
| C8—C9 | 1.494 (9) | C10′—H10A | 0.9700 |
| C8—H8A | 0.9700 | C10′—H10B | 0.9700 |
| C8—H8B | 0.9700 | C11′—C12′ | 1.542 (19) |
| C9—C10 | 1.510 (9) | C11′—H11C | 0.9700 |
| C9—H9A | 0.9700 | C11′—H11D | 0.9700 |
| C9—H9B | 0.9700 | C12′—N1′ | 1.474 (16) |
| C10—O2 | 1.269 (9) | C12′—H12C | 0.9700 |
| C10—C11 | 1.529 (8) | C12′—H12D | 0.9700 |
| | | | |
| C2—C1—C6 | 121.4 (5) | C10—C11—H11B | 109.2 |
| C2—C1—Cl1 | 119.1 (4) | H11A—C11—H11B | 107.9 |
| C6—C1—Cl1 | 119.5 (4) | N1—C12—C11 | 110.6 (5) |
| C1—C2—C3 | 119.0 (5) | N1—C12—H12A | 109.5 |
| C1—C2—H2 | 120.5 | C11—C12—H12A | 109.5 |
| C3—C2—H2 | 120.5 | N1—C12—H12B | 109.5 |
| C4—C3—C2 | 120.8 (5) | C11—C12—H12B | 109.5 |
| C4—C3—H3 | 119.6 | H12A—C12—H12B | 108.1 |
| C2—C3—H3 | 119.6 | C7—N1—C12 | 125.6 (5) |
| C3—C4—C5 | 118.8 (4) | C7—N1—C8 | 120.2 (5) |
| C3—C4—C7 | 121.1 (4) | C12—N1—C8 | 114.1 (5) |
| C5—C4—C7 | 119.9 (4) | C10—O2—H2A | 109.5 |
| C4—C5—C6 | 120.3 (5) | N1′—C8′—C9′ | 110.8 (15) |
| C4—C5—H5 | 119.9 | N1′—C8′—H8′1 | 109.5 |

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| C6—C5—H5 | 119.9 | C9′—C8′—H8′1 | 109.5 |
| C1—C6—C5 | 119.7 (5) | N1′—C8′—H8′2 | 109.5 |
| C1—C6—H6 | 120.1 | C9′—C8′—H8′2 | 109.5 |
| C5—C6—H6 | 120.1 | H8′1—C8′—H8′2 | 108.1 |
| O1—C7—N1 | 119.8 (5) | C8′—C9′—C10′ | 116.9 (17) |
| O1—C7—N1′ | 121.2 (6) | C8′—C9′—H9′1 | 108.1 |
| O1—C7—C4 | 119.8 (4) | C10′—C9′—H9′1 | 108.1 |
| N1—C7—C4 | 119.9 (5) | C8′—C9′—H9′2 | 108.1 |
| N1′—C7—C4 | 109.8 (6) | C10′—C9′—H9′2 | 108.1 |
| N1—C8—C9 | 108.5 (6) | H9′1—C9′—H9′2 | 107.3 |
| N1—C8—H8A | 110.0 | C9′—C10′—C11′ | 98.0 (15) |
| C9—C8—H8A | 110.0 | C9′—C10′—H10A | 112.2 |
| N1—C8—H8B | 110.0 | C11′—C10′—H10A | 112.2 |
| C9—C8—H8B | 110.0 | C9′—C10′—H10B | 112.2 |
| H8A—C8—H8B | 108.4 | C11′—C10′—H10B | 112.2 |
| C8—C9—C10 | 111.5 (6) | H10A—C10′—H10B | 109.8 |
| C8—C9—H9A | 109.3 | C12′—C11′—C10′ | 118.9 (17) |
| C10—C9—H9A | 109.3 | C12′—C11′—H11C | 107.6 |
| C8—C9—H9B | 109.3 | C10′—C11′—H11C | 107.6 |
| C10—C9—H9B | 109.3 | C12′—C11′—H11D | 107.6 |
| H9A—C9—H9B | 108.0 | C10′—C11′—H11D | 107.6 |
| O2—C10—C9 | 110.2 (6) | H11C—C11′—H11D | 107.0 |
| O2—C10—C11 | 112.2 (6) | N1′—C12′—C11′ | 108.7 (13) |
| C9—C10—C11 | 109.8 (5) | N1′—C12′—H12C | 109.9 |
| O2—C10—H10 | 108.2 | C11′—C12′—H12C | 109.9 |
| C9—C10—H10 | 108.2 | N1′—C12′—H12D | 109.9 |
| C11—C10—H10 | 108.2 | C11′—C12′—H12D | 109.9 |
| C12—C11—C10 | 112.0 (6) | H12C—C12′—H12D | 108.3 |
| C12—C11—H11A | 109.2 | C7—N1′—C8′ | 119.5 (12) |
| C10—C11—H11A | 109.2 | C7—N1′—C12′ | 125.0 (12) |
| C12—C11—H11B | 109.2 | C8′—N1′—C12′ | 112.7 (13) |
| | | | |
| C6—C1—C2—C3 | 0.0 (8) | C10—C11—C12—N1 | −53.4 (9) |
| Cl1—C1—C2—C3 | 179.1 (5) | O1—C7—N1—C12 | −173.2 (6) |
| C1—C2—C3—C4 | −0.3 (8) | C4—C7—N1—C12 | −1.0 (10) |
| C2—C3—C4—C5 | 0.9 (8) | O1—C7—N1—C8 | 12.3 (10) |
| C2—C3—C4—C7 | 176.6 (5) | C4—C7—N1—C8 | −175.4 (6) |
| C3—C4—C5—C6 | −1.3 (8) | C11—C12—N1—C7 | −116.7 (7) |
| C7—C4—C5—C6 | −177.0 (5) | C11—C12—N1—C8 | 58.0 (9) |
| C2—C1—C6—C5 | −0.3 (9) | C9—C8—N1—C7 | 115.9 (7) |
| Cl1—C1—C6—C5 | −179.5 (5) | C9—C8—N1—C12 | −59.2 (9) |
| C4—C5—C6—C1 | 1.0 (9) | N1′—C8′—C9′—C10′ | −60 (3) |
| C3—C4—C7—O1 | −104.8 (7) | C8′—C9′—C10′—C11′ | 56 (2) |
| C5—C4—C7—O1 | 70.8 (7) | C9′—C10′—C11′—C12′ | −57 (2) |
| C3—C4—C7—N1 | 82.9 (7) | C10′—C11′—C12′—N1′ | 59 (2) |
| C5—C4—C7—N1 | −101.4 (6) | O1—C7—N1′—C8′ | −18.1 (18) |
| C3—C4—C7—N1′ | 42.4 (9) | C4—C7—N1′—C8′ | −164.8 (13) |
| C5—C4—C7—N1′ | −142.0 (8) | O1—C7—N1′—C12′ | −177.3 (12) |

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| N1—C8—C9—C10 | 56.9 (9) | C4—C7—N1′—C12′ | 36.0 (17) |
| C8—C9—C10—O2 | −178.5 (7) | C9′—C8′—N1′—C7 | −108.1 (19) |
| C8—C9—C10—C11 | −54.4 (9) | C9′—C8′—N1′—C12′ | 54 (2) |
| O2—C10—C11—C12 | 175.3 (7) | C11′—C12′—N1′—C7 | 108.5 (19) |
| C9—C10—C11—C12 | 52.4 (9) | C11′—C12′—N1′—C8′ | −52 (2) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H⋯*A* | *D*—H | H⋯*A* | *D*⋯*A* | *D*—H⋯*A* |
|---|---|---|---|---|
| O2—H2*A*⋯O1[i] | 0.82 | 2.05 | 2.693 (7) | 135 |
| C12′—H12*D*⋯Cl1[ii] | 0.97 | 2.77 | 3.63 (2) | 148 |

Symmetry codes: (i) *x*, *y*, *z*−1; (ii) −*x*+3/2, *y*+1, *z*−1/2.