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# *N*-*tert*-Butyl-2-{2-[2-(4-chlorophenyl)-4-hydroxy-1-(5-methylisoxazol-3-yl)-5-oxo-2,5-dihydro-1*H*-pyrrol-3-yl]-*N*-(4-methoxyphenyl)acetamido}-2-(4-methoxyphenyl)acetamide methanol monosolvate: single-crystal X-ray diffraction study and Hirshfeld surface analysis

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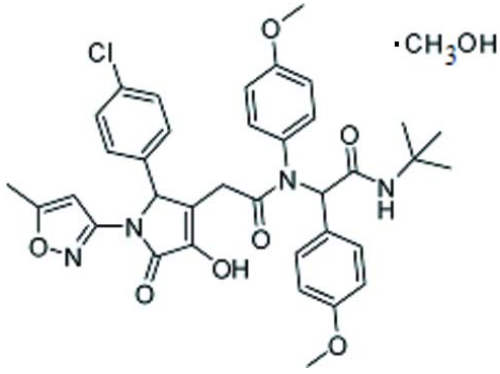
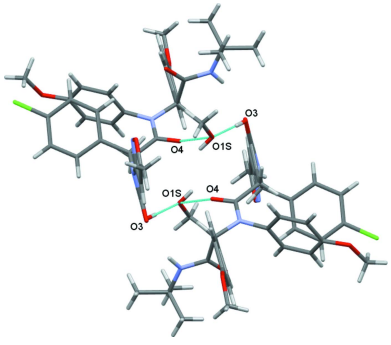
The title compound, $C_{36}H_{37}ClN_4O_7\cdot CH_3OH$, which crystallizes as a methanol solvate, may possess biological activity, which is inherent for a natural peptide or protein. In the crystal, molecules of the title compound form hydrogen-bonded tetramers with the solvate molecules acting as bridges as a result of the O—H···O and N—H···O intermolecular hydrogen bonds. Hirshfeld surface analysis was used to study the different types of intermolecular interactions whose contributions are: H···H = 53.8%, O···H/H···O = 19.0%, C···H/H···C = 14.8%, Cl···H/H···Cl = 5.3%, N···H/H···N = 3.2%.

## 1. Chemical context

The combined application of Doebner and Ugi-type multicomponent reactions, with the participation of the azoloazine type of carboxylic acid as an acid component in the Ugi reaction to increase the molecular diversity of the target heterocyclic compounds, was reported in our previous publication (Murlykina *et al.*, 2019).




In the current work, the final product was synthesized *via* the four-component Ugi reaction of 2-[2-(4-chlorophenyl)-4-hydroxy-1-(5-methylisoxazol-3-yl)-5-oxo-2,5-dihydro-1*H*-pyrrol-3-yl]acetic acid, 4-methoxyaniline, 4-methoxybenzaldehyde and *tert*-butylisocyanide. The target product contains a heterocyclic core bound to peptidomimetics, compounds that mimic a natural peptide or protein and which may have high biological activity. The pyrrolone fragment is also a privileged



motif because of its biological activities, namely antibacterial (Murlykina *et al.*, 2013), antiviral (Murlykina *et al.*, 2015; Rashid *et al.*, 2012; Pace *et al.*, 2008), antitumor (Mori *et al.*, 2013; Koz'minykh *et al.*, 2002) and antimicrobial (Khalaf *et al.*, 2004; Gein *et al.*, 2006).

## 2. Structural commentary

The title compound crystallizes as a methanol solvate (Fig. 1). The methanol molecule is disordered over two positions (*A* and *B*) with the populations of *A*:*B* in a 0.303 (10):0.697 (10) ratio. All atoms of the partially saturated five-membered heterocycle are in the same plane with an accuracy of 0.008 Å. The N2—C4 bond length of 1.380 (3) Å and the C8—N2—C4—N1 torsion angle of 2.2 (4)° indicate conjugation between the $\pi$-systems of the partially saturated and oxazole cycles. The *para*-chlorophenyl substituent is located in the pseudo-equatorial position and is turned in relation to the C7—C8 endocyclic bond [C6—C7—C8—C9 = −120.9 (3)° and C7—C8—C9—C10 = 60.9 (3)°]. The C16(═O4)—N3 carbamide group is located in the *-ac* position in relation to the C6—C7 endocyclic bond [C6—C7—C15—C16 = −107.2 (3)°], and the C16═O4 carbonyl group is slightly non-coplanar to the C7—C15 bond [C7—C15—C16—O4 = 22.5 (4)°]. The *para*-methoxyphenyl substituent at the nitrogen atom is turned almost orthogonally to the plane of the carbamide group [C16—N3—C17—C22 = −99.5 (3)°]. The *para*-methoxyphenyl substituent at the carbon atom is located in a position intermediate between *sp* and *−sc* and is also rotated almost orthogonally to the plane of the carbamide group [the C17—N3—C24—C25 and N3—C24—C25—C26 torsion angles are −33.4 (3) and −83.4 (3)°, respectively]. In both *para*-methoxyphenyl substituents, the methoxy group is coplanar with the plane of the aromatic ring [the C19—C20—O5—C23 and C29—C28—O7—C31 torsion angles are −3.3 (5) and 3.6 (4)°, respectively] despite the steric repulsion between the methyl group and the aromatic ring atoms (the shortened contacts are: C23···H19 = 2.52, H23*B*···C19 = 2.73, H23*A*···C19 = 2.76 and H29···C31 = 2.50, H31*C*···C29 = 2.77 and H31*B*···C29 = 2.70 Å as compared with the C···H van der Waals radii sum of 2.87 Å). The substituent at the C24 atom is located in the *−sc* position relative to the N3—C16 bond [C32—C24—N3—C16 = −78.1 (3)°] and the C32—O6 carbonyl group is slightly non-coplanar to the N3—C24 bond [O6—C32—C24—N3 = −27.8 (3)°]. The *tert*-butyl substituent is located in anti-perplanar position to the C32—C24 bond [C33—N4—C32—C24 = 172.9 (2)°].

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O8*A* | 0.75 (4) | 1.88 (5) | 2.61 (3) | 162 (4) |
| O3—H3···O8*B* | 0.75 (4) | 1.93 (4) | 2.680 (10) | 175 (4) |
| N4—H4···O2$^{i}$ | 0.87 (3) | 2.33 (3) | 3.193 (3) | 170 (3) |
| O8*A*—H8*A*···O4$^{i}$ | 0.82 | 1.88 | 2.60 (3) | 145 |
| O8*B*—H8*B*···O4$^{i}$ | 0.82 | 2.50 | 2.843 (8) | 107 |

Symmetry code: (i) $-x+1, -y+2, -z+1$.

## 3. Supramolecular features

In the crystal, the molecules of the title compound are linked by bridging methanol molecules due to the formation of the O3—H8*A*···O8*A*, O3—H8*B*···O8*B*, O8*A*—H8*A*···O4 and O8*B*–H8*B*···O4 intermolecular hydrogen bonds (Table 1). Additionally, two main molecules are bound by N4—H4···O2 hydrogen bonds (Table 1) within this dimer. As a result, a hydrogen-bonded tetramer may be recognized as a structural motif of the crystal packing (Fig. 2).

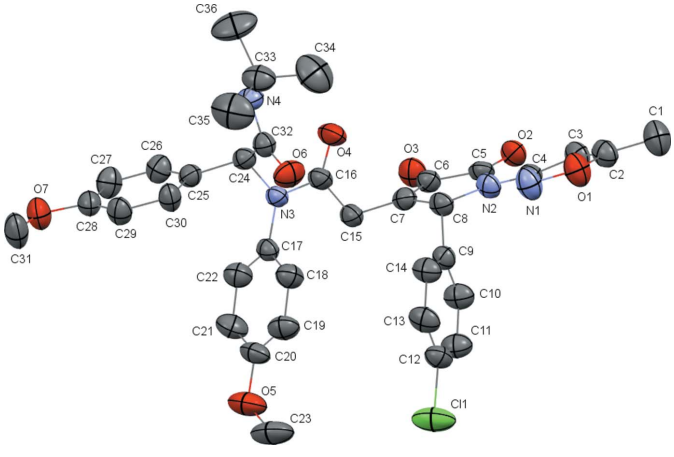



**Figure 1**
Molecular structure of the title compound (solvent molecule and hydrogen atoms are omitted for clarity). Displacement ellipsoids are shown at the 50% probability level.

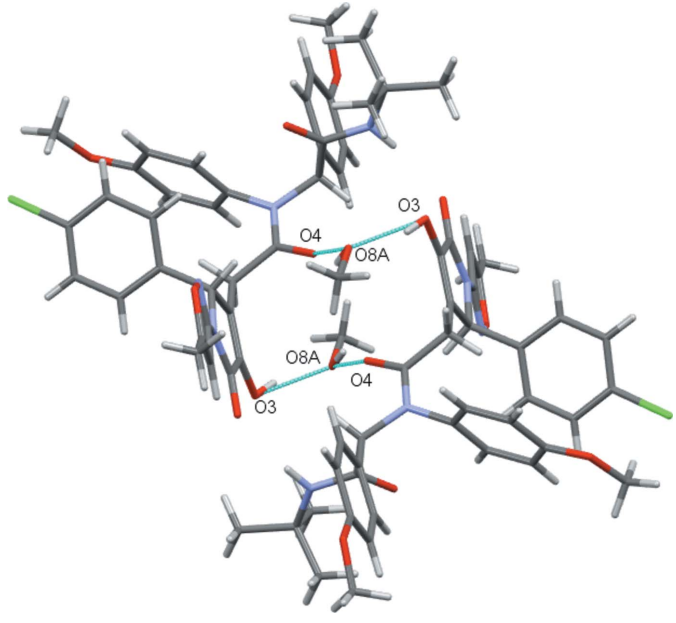



**Figure 2**
Tetramer of hydrogen-bonded title molecules linked through methanol solvent molecules.

## 4. Hirshfeld surface analysis

Different types of intra- and intermolecular interactions in a crystal structure can be identified and visualized with Hirshfeld surface analysis (Turner *et al.*, 2017). The molecular Hirshfeld surface of the major compound was generated using a high surface resolution with three-dimensional $d_{norm}$ surfaces. The areas that are coloured red on the $d_{norm}$ surfaces correspond to contacts that are shorter than the van der Waals radii sum of the closest atoms (Fig. 3). These red spots indicate atoms participating in hydrogen bonding or short contacts. The brightest red spots are observed at the hydroxyl groups of both the main and the methanol molecules, indicating a strong O—H···O intermolecular hydrogen bond. In addition, bright-red spots are observed at the carboxyl group and at the hydrogen atom of the amino group, indicating short contacts. It should be mentioned that smaller red areas are found at the nitrogen atom of the partially saturated five-membered heterocycle, at the C31H$_3$ methyl group and at the C32═O6 carboxyl group, indicating short contacts.

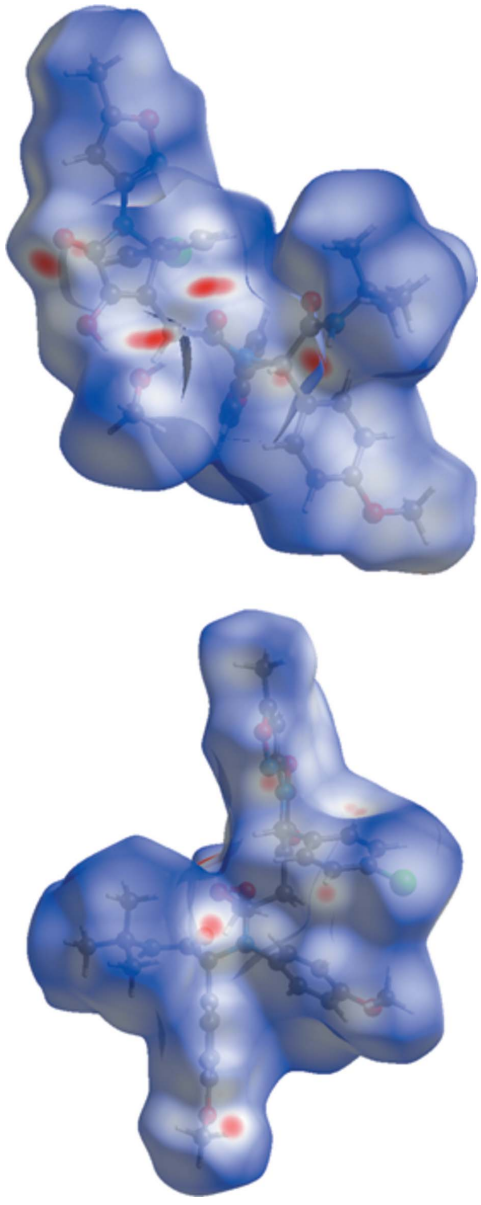


**Figure 3**
Two views of the Hirshfeld surface of the title molecule mapped over $d_{norm}$ in the range −0.295 to 1.590 a.u.

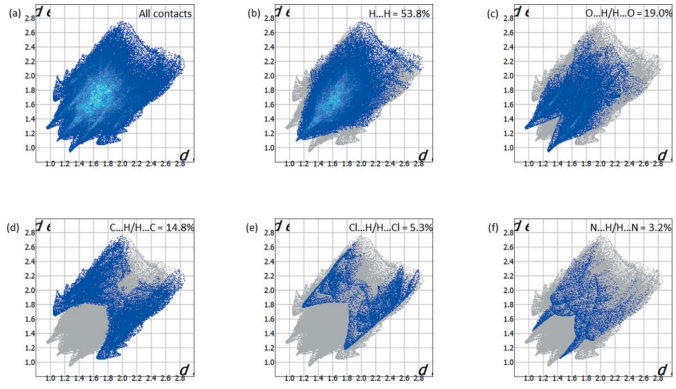



**Figure 4**
Two-dimensional fingerprint plot for the title compound showing (*a*) all interactions, and delineated into (*b*) H···H, (*c*) O···H/H···O, (*d*) C···H/H···C, (*e*) Cl···H/H···Cl. (*f*) N···H/H···N contacts.

In the two-dimensional fingerprint plots the pair of sharp spikes indicate strong hydrogen bonds and short contacts in the crystal structure (Fig. 4*a*). The highest contribution is from H···H contacts (53.8%), while these made by the O···H/H···O (19.0%) and C···H/H···C (14.8%) interactions are similar (Fig. 4*c*, 4*d*). The contributions of Cl···H/H···Cl (5.3%) and N···H/H···N (3.2%) interactions (Fig. 4*e*, 4*f*) are very small.

## 5. Database survey

A search of the Cambridge Structural Database (CSD Version 5.42, update of November 2020; Groom *et al.*, 2016) for the 3-hydroxy-1,5-dihydro-pyrrol-2-one fragment revealed 79 hits. Only 27 of these hits contain a fragment with the same structure as that of the title compound [refcodes: BOQXEN (del Corte *et al.*, 2019), CIKPAQ (Sarkar *et al.*, 2018), EVIYUD (Aliev *et al.*, 2003*b*), GEJZAY (Mashevskaya *et al.*, 2011), GIMGEQ (Sarkar *et al.*, 2013), GITCAQ, GITDEV (Saha *et al.*, 2017), IRUBUS (Aliev *et al.*, 2003*a*), LIFBEJ, LIFBOT (Sun *et al.*, 2011), NUXPIG (Wiedemann *et al.*, 2009), PADHUA (Zonouz *et al.*, 2015), PASTOT (Nicolaou *et al.*, 2005), QIPNAH (Bhajammanavar *et al.*, 2019), ROHNAG (Hosseinzadeh *et al.*, 2019), TOMPER (Sakhno *et al.*, 2008), UJEXOY (Ahankar *et al.*, 2016), VILQEP (Gein *et al.*, 2018), VIPNAJ (Mylari *et al.*, 1991), VIQDOP (Guseinov *et al.*, 2006), VOWGAP (Kazakov *et al.*, 1990), WAPMIM (Dubovtsev *et al.*, 2016), XINHIL (Aliev *et al.*, 2001), XOKRAT, XOKRAT01 (Ramazani *et al.*, 2019), YAJMOM (Wei *et al.*, 2004), YIYFAP (Denislamova *et al.*, 2014)]. All these structures and the title compound have the same electron density distribution within the 3-hydroxy-1,5-dihydro-pyrrol-2-one fragment.

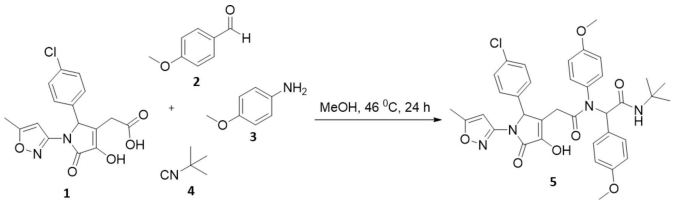



**Figure 5**
Reaction scheme.

## 6. Synthesis and crystallization

4-Methoxyaniline (**3**) (0.5 mmol) and 4-methoxybenzaldehyde (**2**) (0.5 mmol) were dissolved in methanol (4 mL) and stirred for 0.5 h. Then, 2-[2-(4-chlorophenyl)-4-hydroxy-1-(5-methyl-isoxazol-3-yl)-5-oxo-2,5-dihydro-1*H*-pyrrol-3-yl]acetic acid (**1**) (0.5 mmol) and *tert*-butylisocyanide (**4**) (0.5 mmol) were added consistently and the reaction mixture was stirred for 24 h at 319 K. The mixture was allowed to stand overnight. The crystal precipitate was filtered off and dried. The reaction scheme is shown in Fig. 5.

## 7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms were located in difference-Fourier maps. They were included in calculated positions and treated as riding with C—H = 0.96 Å and O—H = 0.82 Å, $U_{iso}$(H) = 1.5$U_{eq}$(C,O) for methyl and hydroxyl groups and with Car—H = 0.93 Å, C$sp^3$—H = 0.97 Å, N—H = 0.89 Å $U_{iso}$(H) = 1.2$U_{eq}$(C,N) for all other hydrogen atoms. The solvent molecule is disordered over two positions (*A* and *B*) in a 0.303 (10):0.697 (10) occupancy ratio. C$sp^3$—O bonds were refined with fixed length of 1.413 Å.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{36}H_{37}ClN_4O_7{\cdot}CH_4O$ |
| $M_r$ | 705.19 |
| Crystal system, space group | Triclinic, $P\overline{1}$ |
| Temperature (K) | 293 |
| *a*, *b*, *c* (Å) | 10.3211 (7), 13.9316 (11), 15.0269 (10) |
| $\alpha$, $\beta$, $\gamma$ (°) | 107.179 (6), 100.769 (6), 109.649 (7) |
| *V* (Å$^3$) | 1843.2 (2) |
| *Z* | 2 |
| Radiation type | Mo *K*$\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.16 |
| Crystal size (mm) | 0.12 × 0.08 × 0.04 |
| | |
| Data collection | |
| Diffractometer | Rigaku Xcalibur, Sapphire3 |
| Absorption correction | Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018) |
| $T_{min}$, $T_{max}$ | 0.718, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 12120, 6484, 3993 |
| $R_{int}$ | 0.062 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.595 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S* | 0.063, 0.180, 0.98 |
| No. of reflections | 6484 |
| No. of parameters | 488 |
| No. of restraints | 14 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.37, −0.37 |

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXT2014/5* (Sheldrick, 2015*a*), *SHELXL2017/1* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020) and *OLEX2* (Dolomanov *et al.*, 2009).


## Funding information

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# supporting information

*Acta Cryst.* (2021). E**77**, 1208-1212 [https://doi.org/10.1107/S2056989021011312]

# *N*-*tert*-Butyl-2-{2-[2-(4-chlorophenyl)-4-hydroxy-1-(5-methylisoxazol-3-yl)-5-oxo-2,5-dihydro-1*H*-pyrrol-3-yl]-*N*-(4-methoxyphenyl)acetamido}-2-(4-methoxyphenyl)acetamide methanol monosolvate: single-crystal X-ray diffraction study and Hirshfeld surface analysis

**Mariia O. Shyshkina, Yana I. Sakhno, Oleksandr V. Radchenko, Svitlana V. Shishkina, Sergey M. Desenko and Valentyn A. Chebanov**

## Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2018); cell refinement: *CrysAlis PRO* (Rigaku OD, 2018); data reduction: *CrysAlis PRO* (Rigaku OD, 2018); program(s) used to solve structure: *SHELXT2014/5* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2017/1* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

**_N_-*tert*-Butyl-2-{2-[2-(4-chlorophenyl)-4-hydroxy-1-(5-methylisoxazol-3-yl)-5-oxo-2,5-dihydro-1*H*-pyrrol-3-yl]-*N*-(4-methoxyphenyl)acetamido}-2-(4-methoxyphenyl)acetamide methanol monosolvate**

*Crystal data*

$C_{36}H_{37}ClN_4O_7 \cdot CH_4O$
$M_r = 705.19$
Triclinic, $P\overline{1}$
$a = 10.3211$ (7) Å
$b = 13.9316$ (11) Å
$c = 15.0269$ (10) Å
$\alpha = 107.179$ (6)°
$\beta = 100.769$ (6)°
$\gamma = 109.649$ (7)°
$V = 1843.2$ (2) Å$^3$
$Z = 2$
$F(000) = 744$
$D_x = 1.271$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2243 reflections
$\theta = 3.6$–$24.8°$
$\mu = 0.16$ mm$^{-1}$
$T = 293$ K
Plate, colourless
0.12 × 0.08 × 0.04 mm

*Data collection*

Rigaku Xcalibur, Sapphire3 diffractometer
Radiation source: fine-focus sealed X-ray tube
Detector resolution: 16.1827 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2018)
$T_{min} = 0.718$, $T_{max} = 1.000$
12120 measured reflections
6484 independent reflections
3993 reflections with $I > 2\sigma(I)$
$R_{int} = 0.062$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.0°$
$h = -12 \rightarrow 12$
$k = -16 \rightarrow 16$
$l = -17 \rightarrow 17$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.180$
$S = 0.98$
6484 reflections
488 parameters
14 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0804P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å$^{-3}$
$\Delta\rho_{min} = -0.37$ 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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Cl1 | 0.18482 (10) | 0.27201 (7) | 0.45318 (8) | 0.0879 (4) | |
| O1 | 0.1363 (2) | 0.78835 (18) | 0.78187 (15) | 0.0656 (6) | |
| O2 | 0.6392 (2) | 0.97400 (15) | 0.79614 (13) | 0.0527 (5) | |
| O3 | 0.7446 (2) | 0.94517 (18) | 0.63187 (18) | 0.0595 (6) | |
| H3 | 0.741 (4) | 0.957 (3) | 0.586 (3) | 0.083 (15)* | |
| O4 | 0.3890 (3) | 0.87873 (18) | 0.40675 (14) | 0.0708 (7) | |
| O5 | 0.3359 (3) | 0.30481 (19) | 0.15424 (18) | 0.0802 (8) | |
| O6 | 0.0559 (2) | 0.67660 (18) | 0.23246 (16) | 0.0630 (6) | |
| O7 | 0.3038 (2) | 0.61623 (18) | −0.17618 (14) | 0.0609 (6) | |
| N1 | 0.1951 (3) | 0.7675 (2) | 0.70394 (18) | 0.0612 (7) | |
| N2 | 0.4227 (2) | 0.83021 (18) | 0.68609 (15) | 0.0460 (6) | |
| N3 | 0.3530 (2) | 0.72925 (16) | 0.27770 (14) | 0.0392 (5) | |
| N4 | 0.0835 (3) | 0.8240 (2) | 0.19070 (17) | 0.0490 (6) | |
| H4 | 0.152 (4) | 0.884 (3) | 0.195 (2) | 0.074 (11)* | |
| C1 | 0.2010 (4) | 0.8940 (3) | 0.9544 (2) | 0.0736 (10) | |
| H1A | 0.128840 | 0.922066 | 0.943675 | 0.110* | |
| H1B | 0.284465 | 0.949601 | 1.008583 | 0.110* | |
| H1C | 0.161991 | 0.829562 | 0.968871 | 0.110* | |
| C2 | 0.2440 (3) | 0.8644 (3) | 0.8645 (2) | 0.0546 (8) | |
| C3 | 0.3694 (3) | 0.8946 (2) | 0.8449 (2) | 0.0531 (8) | |
| H3A | 0.460502 | 0.945953 | 0.888417 | 0.064* | |
| C4 | 0.3337 (3) | 0.8316 (2) | 0.74402 (19) | 0.0434 (7) | |
| C5 | 0.5651 (3) | 0.9051 (2) | 0.71337 (19) | 0.0436 (7) | |
| C6 | 0.6065 (3) | 0.8850 (2) | 0.62320 (19) | 0.0438 (7) | |
| C7 | 0.4959 (3) | 0.8067 (2) | 0.54664 (19) | 0.0432 (7) | |
| C8 | 0.3665 (3) | 0.7620 (2) | 0.57990 (18) | 0.0442 (7) | |
| H8 | 0.286697 | 0.776140 | 0.548349 | 0.053* | |
| C9 | 0.3169 (3) | 0.6397 (2) | 0.55535 (18) | 0.0416 (7) | |
| C10 | 0.4071 (3) | 0.5977 (2) | 0.5935 (2) | 0.0537 (8) | |
| H10 | 0.496737 | 0.646313 | 0.640469 | 0.064* | |

| | | | | |
|---|---|---|---|---|
| C11 | 0.3678 (3) | 0.4858 (2) | 0.5639 (2) | 0.0560 (8) |
| H11 | 0.429826 | 0.458959 | 0.590555 | 0.067* |
| C12 | 0.2347 (3) | 0.4139 (2) | 0.4940 (2) | 0.0496 (7) |
| C13 | 0.1416 (3) | 0.4526 (2) | 0.4563 (2) | 0.0562 (8) |
| H13 | 0.051140 | 0.403604 | 0.410486 | 0.067* |
| C14 | 0.1827 (3) | 0.5653 (2) | 0.4868 (2) | 0.0513 (7) |
| H14 | 0.119336 | 0.591657 | 0.460966 | 0.062* |
| C15 | 0.4877 (3) | 0.7567 (2) | 0.44215 (18) | 0.0467 (7) |
| H15A | 0.441428 | 0.676778 | 0.419640 | 0.056* |
| H15B | 0.585368 | 0.777049 | 0.438053 | 0.056* |
| C16 | 0.4042 (3) | 0.7932 (2) | 0.37513 (19) | 0.0450 (7) |
| C17 | 0.3430 (3) | 0.6180 (2) | 0.24142 (18) | 0.0396 (6) |
| C18 | 0.2315 (3) | 0.5338 (2) | 0.2456 (2) | 0.0530 (8) |
| H18 | 0.159408 | 0.548169 | 0.268223 | 0.064* |
| C19 | 0.2238 (3) | 0.4270 (2) | 0.2165 (2) | 0.0587 (8) |
| H19 | 0.147233 | 0.370399 | 0.219647 | 0.070* |
| C20 | 0.3309 (3) | 0.4061 (2) | 0.1831 (2) | 0.0555 (8) |
| C21 | 0.4416 (4) | 0.4902 (3) | 0.1765 (2) | 0.0644 (9) |
| H21 | 0.512658 | 0.475722 | 0.152511 | 0.077* |
| C22 | 0.4478 (3) | 0.5952 (2) | 0.2053 (2) | 0.0545 (8) |
| H22 | 0.522867 | 0.651377 | 0.200380 | 0.065* |
| C23 | 0.2286 (4) | 0.2176 (3) | 0.1641 (3) | 0.0916 (13) |
| H23A | 0.135048 | 0.201790 | 0.122460 | 0.137* |
| H23B | 0.229750 | 0.238635 | 0.231160 | 0.137* |
| H23C | 0.247922 | 0.152986 | 0.145215 | 0.137* |
| C24 | 0.2850 (3) | 0.7726 (2) | 0.21251 (18) | 0.0404 (6) |
| H24 | 0.342636 | 0.852694 | 0.238280 | 0.048* |
| C25 | 0.2869 (3) | 0.7253 (2) | 0.10845 (19) | 0.0421 (6) |
| C26 | 0.4104 (3) | 0.7724 (2) | 0.0842 (2) | 0.0512 (8) |
| H26 | 0.491865 | 0.831257 | 0.132629 | 0.061* |
| C27 | 0.4136 (3) | 0.7330 (3) | −0.0107 (2) | 0.0564 (8) |
| H27 | 0.497132 | 0.764969 | −0.025541 | 0.068* |
| C28 | 0.2927 (3) | 0.6460 (2) | −0.0838 (2) | 0.0475 (7) |
| C29 | 0.1706 (3) | 0.5974 (2) | −0.0612 (2) | 0.0533 (8) |
| H29 | 0.089435 | 0.538224 | −0.109724 | 0.064* |
| C30 | 0.1692 (3) | 0.6372 (2) | 0.03492 (19) | 0.0494 (7) |
| H30 | 0.086484 | 0.603566 | 0.049952 | 0.059* |
| C31 | 0.1776 (4) | 0.5334 (3) | −0.2546 (2) | 0.0748 (11) |
| H31A | 0.196560 | 0.524816 | −0.315984 | 0.112* |
| H31B | 0.098515 | 0.554956 | −0.254578 | 0.112* |
| H31C | 0.152642 | 0.464518 | −0.246083 | 0.112* |
| C32 | 0.1300 (3) | 0.7536 (2) | 0.21472 (19) | 0.0434 (7) |
| C33 | −0.0649 (3) | 0.8177 (3) | 0.1762 (3) | 0.0637 (9) |
| C34 | −0.1035 (4) | 0.8284 (4) | 0.2694 (3) | 0.0987 (14) |
| H34A | −0.104076 | 0.767117 | 0.286450 | 0.148* |
| H34B | −0.197859 | 0.828863 | 0.259680 | 0.148* |
| H34C | −0.033068 | 0.896180 | 0.321512 | 0.148* |
| C35 | −0.1715 (4) | 0.7095 (3) | 0.0922 (3) | 0.1000 (14) |

| | | | | | |
|---|---|---|---|---|---|
| H35A | −0.140232 | 0.702183 | 0.035187 | 0.150* | |
| H35B | −0.266155 | 0.709336 | 0.077334 | 0.150* | |
| H35C | −0.175204 | 0.648757 | 0.111037 | 0.150* | |
| C36 | −0.0624 (5) | 0.9159 (4) | 0.1482 (4) | 0.1060 (15) | |
| H36A | 0.007832 | 0.983643 | 0.200456 | 0.159* | |
| H36B | −0.156610 | 0.916650 | 0.137701 | 0.159* | |
| H36C | −0.036727 | 0.908644 | 0.089156 | 0.159* | |
| O8A | 0.754 (3) | 1.031 (2) | 0.4990 (12) | 0.127 (8) | 0.303 (10) |
| H8A | 0.682895 | 1.041989 | 0.506038 | 0.191* | 0.303 (10) |
| C37A | 0.744 (3) | 0.9975 (16) | 0.3987 (13) | 0.117 (6) | 0.303 (10) |
| H37A | 0.802378 | 1.059842 | 0.387193 | 0.175* | 0.303 (10) |
| H37B | 0.644924 | 0.969545 | 0.359214 | 0.175* | 0.303 (10) |
| H37C | 0.778623 | 0.940725 | 0.381619 | 0.175* | 0.303 (10) |
| O8B | 0.7271 (8) | 0.9964 (6) | 0.4729 (6) | 0.084 (2) | 0.697 (10) |
| H8B | 0.655470 | 1.005555 | 0.450322 | 0.127* | 0.697 (10) |
| C37B | 0.8511 (8) | 1.0727 (6) | 0.4653 (7) | 0.101 (3) | 0.697 (10) |
| H37D | 0.926332 | 1.046763 | 0.469376 | 0.151* | 0.697 (10) |
| H37E | 0.884329 | 1.143282 | 0.517811 | 0.151* | 0.697 (10) |
| H37F | 0.826852 | 1.079852 | 0.403396 | 0.151* | 0.697 (10) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0789 (6) | 0.0378 (5) | 0.1184 (8) | 0.0190 (4) | 0.0063 (6) | 0.0136 (5) |
| O1 | 0.0552 (13) | 0.0732 (16) | 0.0524 (13) | 0.0196 (12) | 0.0169 (11) | 0.0114 (11) |
| O2 | 0.0585 (12) | 0.0386 (11) | 0.0372 (11) | 0.0093 (10) | 0.0026 (10) | 0.0033 (9) |
| O3 | 0.0551 (14) | 0.0508 (14) | 0.0535 (14) | 0.0101 (11) | 0.0117 (12) | 0.0120 (11) |
| O4 | 0.1146 (19) | 0.0506 (14) | 0.0430 (12) | 0.0494 (14) | 0.0060 (12) | 0.0071 (10) |
| O5 | 0.0854 (17) | 0.0464 (15) | 0.0958 (18) | 0.0347 (13) | 0.0134 (14) | 0.0115 (13) |
| O6 | 0.0582 (13) | 0.0572 (14) | 0.0817 (16) | 0.0205 (11) | 0.0289 (12) | 0.0376 (12) |
| O7 | 0.0605 (13) | 0.0701 (15) | 0.0466 (12) | 0.0214 (11) | 0.0230 (11) | 0.0184 (10) |
| N1 | 0.0548 (16) | 0.0588 (17) | 0.0458 (14) | 0.0111 (13) | 0.0117 (13) | 0.0052 (12) |
| N2 | 0.0468 (13) | 0.0391 (14) | 0.0361 (12) | 0.0111 (11) | 0.0070 (11) | 0.0050 (10) |
| N3 | 0.0475 (13) | 0.0298 (12) | 0.0326 (11) | 0.0155 (10) | 0.0055 (10) | 0.0070 (9) |
| N4 | 0.0488 (15) | 0.0428 (15) | 0.0511 (15) | 0.0198 (13) | 0.0066 (12) | 0.0172 (12) |
| C1 | 0.073 (2) | 0.087 (3) | 0.054 (2) | 0.032 (2) | 0.0218 (18) | 0.0199 (18) |
| C2 | 0.0558 (19) | 0.053 (2) | 0.0407 (16) | 0.0199 (16) | 0.0050 (15) | 0.0088 (14) |
| C3 | 0.0506 (18) | 0.0526 (19) | 0.0384 (16) | 0.0162 (15) | 0.0043 (14) | 0.0063 (13) |
| C4 | 0.0489 (17) | 0.0353 (16) | 0.0370 (14) | 0.0144 (13) | 0.0064 (13) | 0.0099 (12) |
| C5 | 0.0529 (17) | 0.0263 (14) | 0.0381 (15) | 0.0125 (13) | 0.0017 (14) | 0.0070 (12) |
| C6 | 0.0486 (17) | 0.0333 (15) | 0.0397 (15) | 0.0123 (13) | 0.0084 (13) | 0.0102 (12) |
| C7 | 0.0485 (16) | 0.0356 (15) | 0.0371 (14) | 0.0159 (13) | 0.0032 (13) | 0.0113 (12) |
| C8 | 0.0476 (16) | 0.0343 (15) | 0.0335 (14) | 0.0125 (13) | −0.0001 (13) | 0.0036 (11) |
| C9 | 0.0414 (15) | 0.0370 (16) | 0.0358 (14) | 0.0112 (13) | 0.0064 (12) | 0.0093 (12) |
| C10 | 0.0450 (17) | 0.0417 (18) | 0.0537 (17) | 0.0112 (14) | −0.0023 (14) | 0.0102 (14) |
| C11 | 0.0502 (18) | 0.047 (2) | 0.065 (2) | 0.0211 (15) | 0.0082 (16) | 0.0205 (15) |
| C12 | 0.0516 (17) | 0.0326 (16) | 0.0550 (17) | 0.0135 (14) | 0.0154 (15) | 0.0097 (13) |
| C13 | 0.0469 (17) | 0.0413 (18) | 0.0533 (18) | 0.0102 (14) | −0.0001 (14) | 0.0015 (14) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C14 | 0.0445 (16) | 0.0437 (18) | 0.0499 (17) | 0.0165 (14) | −0.0004 (14) | 0.0089 (13) |
| C15 | 0.0612 (18) | 0.0397 (16) | 0.0347 (14) | 0.0241 (14) | 0.0075 (14) | 0.0094 (12) |
| C16 | 0.0551 (17) | 0.0311 (15) | 0.0381 (15) | 0.0162 (13) | 0.0063 (13) | 0.0065 (12) |
| C17 | 0.0427 (15) | 0.0375 (16) | 0.0327 (14) | 0.0177 (13) | 0.0058 (12) | 0.0084 (11) |
| C18 | 0.0536 (18) | 0.0432 (18) | 0.0590 (19) | 0.0194 (15) | 0.0188 (15) | 0.0159 (14) |
| C19 | 0.0585 (19) | 0.0346 (18) | 0.068 (2) | 0.0119 (15) | 0.0101 (17) | 0.0143 (15) |
| C20 | 0.062 (2) | 0.0386 (18) | 0.0523 (18) | 0.0274 (16) | 0.0026 (16) | 0.0020 (14) |
| C21 | 0.067 (2) | 0.056 (2) | 0.075 (2) | 0.0359 (18) | 0.0266 (19) | 0.0170 (17) |
| C22 | 0.0540 (18) | 0.0471 (19) | 0.0589 (19) | 0.0210 (15) | 0.0161 (16) | 0.0174 (14) |
| C23 | 0.097 (3) | 0.043 (2) | 0.113 (3) | 0.028 (2) | 0.004 (3) | 0.020 (2) |
| C24 | 0.0437 (15) | 0.0331 (15) | 0.0366 (14) | 0.0128 (12) | 0.0044 (12) | 0.0121 (11) |
| C25 | 0.0468 (16) | 0.0378 (16) | 0.0384 (14) | 0.0150 (13) | 0.0099 (13) | 0.0154 (12) |
| C26 | 0.0442 (17) | 0.0541 (19) | 0.0468 (17) | 0.0149 (14) | 0.0071 (14) | 0.0189 (14) |
| C27 | 0.0449 (17) | 0.068 (2) | 0.0549 (19) | 0.0178 (16) | 0.0179 (15) | 0.0269 (16) |
| C28 | 0.0540 (18) | 0.0513 (18) | 0.0412 (16) | 0.0246 (15) | 0.0173 (14) | 0.0188 (14) |
| C29 | 0.0538 (18) | 0.0444 (18) | 0.0424 (16) | 0.0069 (14) | 0.0121 (14) | 0.0089 (13) |
| C30 | 0.0496 (17) | 0.0475 (18) | 0.0383 (15) | 0.0087 (14) | 0.0160 (14) | 0.0116 (13) |
| C31 | 0.087 (3) | 0.071 (2) | 0.0449 (18) | 0.013 (2) | 0.0268 (19) | 0.0129 (17) |
| C32 | 0.0462 (16) | 0.0418 (17) | 0.0360 (14) | 0.0166 (14) | 0.0091 (13) | 0.0111 (12) |
| C33 | 0.0533 (19) | 0.059 (2) | 0.071 (2) | 0.0274 (17) | 0.0067 (17) | 0.0194 (17) |
| C34 | 0.074 (3) | 0.119 (4) | 0.106 (3) | 0.051 (3) | 0.038 (2) | 0.030 (3) |
| C35 | 0.066 (2) | 0.088 (3) | 0.099 (3) | 0.023 (2) | −0.017 (2) | 0.011 (2) |
| C36 | 0.092 (3) | 0.101 (3) | 0.153 (4) | 0.061 (3) | 0.020 (3) | 0.074 (3) |
| O8A | 0.140 (12) | 0.122 (14) | 0.101 (11) | 0.012 (11) | 0.040 (9) | 0.065 (10) |
| C37A | 0.178 (15) | 0.106 (12) | 0.098 (11) | 0.062 (11) | 0.086 (10) | 0.050 (9) |
| O8B | 0.088 (4) | 0.073 (4) | 0.096 (6) | 0.033 (3) | 0.032 (3) | 0.036 (3) |
| C37B | 0.107 (6) | 0.111 (6) | 0.137 (7) | 0.060 (5) | 0.068 (5) | 0.081 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C12 | 1.741 (3) | C18—H18 | 0.9300 |
| O1—N1 | 1.417 (3) | C18—C19 | 1.391 (4) |
| O1—C2 | 1.348 (3) | C19—H19 | 0.9300 |
| O2—C5 | 1.224 (3) | C19—C20 | 1.377 (4) |
| O3—H3 | 0.75 (4) | C20—C21 | 1.377 (5) |
| O3—C6 | 1.346 (3) | C21—H21 | 0.9300 |
| O4—C16 | 1.221 (3) | C21—C22 | 1.373 (4) |
| O5—C20 | 1.371 (3) | C22—H22 | 0.9300 |
| O5—C23 | 1.409 (4) | C23—H23A | 0.9600 |
| O6—C32 | 1.225 (3) | C23—H23B | 0.9600 |
| O7—C28 | 1.365 (3) | C23—H23C | 0.9600 |
| O7—C31 | 1.425 (4) | C24—H24 | 0.9800 |
| N1—C4 | 1.307 (3) | C24—C25 | 1.514 (4) |
| N2—C4 | 1.380 (3) | C24—C32 | 1.540 (4) |
| N2—C5 | 1.381 (3) | C25—C26 | 1.393 (4) |
| N2—C8 | 1.474 (3) | C25—C30 | 1.377 (4) |
| N3—C16 | 1.357 (3) | C26—H26 | 0.9300 |
| N3—C17 | 1.443 (3) | C26—C27 | 1.378 (4) |

| | | | |
|---|---|---|---|
| N3—C24 | 1.482 (3) | C27—H27 | 0.9300 |
| N4—H4 | 0.87 (3) | C27—C28 | 1.384 (4) |
| N4—C32 | 1.335 (4) | C28—C29 | 1.373 (4) |
| N4—C33 | 1.475 (4) | C29—H29 | 0.9300 |
| C1—H1A | 0.9600 | C29—C30 | 1.390 (4) |
| C1—H1B | 0.9600 | C30—H30 | 0.9300 |
| C1—H1C | 0.9600 | C31—H31A | 0.9600 |
| C1—C2 | 1.486 (4) | C31—H31B | 0.9600 |
| C2—C3 | 1.337 (4) | C31—H31C | 0.9600 |
| C3—H3A | 0.9300 | C33—C34 | 1.505 (5) |
| C3—C4 | 1.412 (4) | C33—C35 | 1.522 (5) |
| C5—C6 | 1.473 (4) | C33—C36 | 1.539 (5) |
| C6—C7 | 1.322 (4) | C34—H34A | 0.9600 |
| C7—C8 | 1.515 (4) | C34—H34B | 0.9600 |
| C7—C15 | 1.491 (4) | C34—H34C | 0.9600 |
| C8—H8 | 0.9800 | C35—H35A | 0.9600 |
| C8—C9 | 1.507 (4) | C35—H35B | 0.9600 |
| C9—C10 | 1.379 (4) | C35—H35C | 0.9600 |
| C9—C14 | 1.388 (4) | C36—H36A | 0.9600 |
| C10—H10 | 0.9300 | C36—H36B | 0.9600 |
| C10—C11 | 1.375 (4) | C36—H36C | 0.9600 |
| C11—H11 | 0.9300 | O8A—H8A | 0.8200 |
| C11—C12 | 1.381 (4) | O8A—C37A | 1.412 (5) |
| C12—C13 | 1.364 (4) | C37A—H37A | 0.9600 |
| C13—H13 | 0.9300 | C37A—H37B | 0.9600 |
| C13—C14 | 1.381 (4) | C37A—H37C | 0.9600 |
| C14—H14 | 0.9300 | O8B—H8B | 0.8200 |
| C15—H15A | 0.9700 | O8B—C37B | 1.412 (5) |
| C15—H15B | 0.9700 | C37B—H37D | 0.9600 |
| C15—C16 | 1.513 (4) | C37B—H37E | 0.9600 |
| C17—C18 | 1.366 (4) | C37B—H37F | 0.9600 |
| C17—C22 | 1.383 (4) | | |

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| C2—O1—N1 | 108.9 (2) | C20—C21—H21 | 119.7 |
| C6—O3—H3 | 106 (3) | C22—C21—C20 | 120.5 (3) |
| C20—O5—C23 | 117.9 (3) | C22—C21—H21 | 119.7 |
| C28—O7—C31 | 117.5 (2) | C17—C22—H22 | 119.8 |
| C4—N1—O1 | 104.6 (2) | C21—C22—C17 | 120.4 (3) |
| C4—N2—C5 | 125.3 (2) | C21—C22—H22 | 119.8 |
| C4—N2—C8 | 122.4 (2) | O5—C23—H23A | 109.5 |
| C5—N2—C8 | 111.2 (2) | O5—C23—H23B | 109.5 |
| C16—N3—C17 | 121.8 (2) | O5—C23—H23C | 109.5 |
| C16—N3—C24 | 115.9 (2) | H23A—C23—H23B | 109.5 |
| C17—N3—C24 | 121.48 (19) | H23A—C23—H23C | 109.5 |
| C32—N4—H4 | 114 (2) | H23B—C23—H23C | 109.5 |
| C32—N4—C33 | 125.7 (3) | N3—C24—H24 | 107.3 |
| C33—N4—H4 | 119 (2) | N3—C24—C25 | 112.2 (2) |
| H1A—C1—H1B | 109.5 | N3—C24—C32 | 111.1 (2) |

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| H1A—C1—H1C | 109.5 | C25—C24—H24 | 107.3 |
| H1B—C1—H1C | 109.5 | C25—C24—C32 | 111.3 (2) |
| C2—C1—H1A | 109.5 | C32—C24—H24 | 107.3 |
| C2—C1—H1B | 109.5 | C26—C25—C24 | 119.8 (2) |
| C2—C1—H1C | 109.5 | C30—C25—C24 | 122.5 (2) |
| O1—C2—C1 | 115.8 (3) | C30—C25—C26 | 117.8 (3) |
| C3—C2—O1 | 109.4 (3) | C25—C26—H26 | 119.5 |
| C3—C2—C1 | 134.7 (3) | C27—C26—C25 | 121.0 (3) |
| C2—C3—H3A | 127.4 | C27—C26—H26 | 119.5 |
| C2—C3—C4 | 105.1 (3) | C26—C27—H27 | 119.9 |
| C4—C3—H3A | 127.4 | C26—C27—C28 | 120.2 (3) |
| N1—C4—N2 | 118.8 (2) | C28—C27—H27 | 119.9 |
| N1—C4—C3 | 111.9 (3) | O7—C28—C27 | 115.9 (3) |
| N2—C4—C3 | 129.3 (3) | O7—C28—C29 | 124.3 (3) |
| O2—C5—N2 | 126.0 (3) | C29—C28—C27 | 119.8 (3) |
| O2—C5—C6 | 128.1 (3) | C28—C29—H29 | 120.3 |
| N2—C5—C6 | 106.0 (2) | C28—C29—C30 | 119.5 (3) |
| O3—C6—C5 | 117.3 (2) | C30—C29—H29 | 120.3 |
| C7—C6—O3 | 131.8 (3) | C25—C30—C29 | 121.8 (3) |
| C7—C6—C5 | 110.9 (2) | C25—C30—H30 | 119.1 |
| C6—C7—C8 | 109.5 (2) | C29—C30—H30 | 119.1 |
| C6—C7—C15 | 130.0 (3) | O7—C31—H31A | 109.5 |
| C15—C7—C8 | 120.3 (2) | O7—C31—H31B | 109.5 |
| N2—C8—C7 | 102.3 (2) | O7—C31—H31C | 109.5 |
| N2—C8—H8 | 109.6 | H31A—C31—H31B | 109.5 |
| N2—C8—C9 | 114.2 (2) | H31A—C31—H31C | 109.5 |
| C7—C8—H8 | 109.6 | H31B—C31—H31C | 109.5 |
| C9—C8—C7 | 111.2 (2) | O6—C32—N4 | 124.3 (3) |
| C9—C8—H8 | 109.6 | O6—C32—C24 | 121.5 (3) |
| C10—C9—C8 | 121.5 (2) | N4—C32—C24 | 114.0 (3) |
| C10—C9—C14 | 117.9 (3) | N4—C33—C34 | 110.8 (3) |
| C14—C9—C8 | 120.4 (2) | N4—C33—C35 | 109.5 (3) |
| C9—C10—H10 | 119.2 | N4—C33—C36 | 105.1 (3) |
| C11—C10—C9 | 121.7 (3) | C34—C33—C35 | 111.2 (3) |
| C11—C10—H10 | 119.2 | C34—C33—C36 | 110.1 (3) |
| C10—C11—H11 | 120.5 | C35—C33—C36 | 109.9 (3) |
| C10—C11—C12 | 119.0 (3) | C33—C34—H34A | 109.5 |
| C12—C11—H11 | 120.5 | C33—C34—H34B | 109.5 |
| C11—C12—Cl1 | 119.7 (2) | C33—C34—H34C | 109.5 |
| C13—C12—Cl1 | 119.4 (2) | H34A—C34—H34B | 109.5 |
| C13—C12—C11 | 120.9 (3) | H34A—C34—H34C | 109.5 |
| C12—C13—H13 | 120.3 | H34B—C34—H34C | 109.5 |
| C12—C13—C14 | 119.4 (3) | C33—C35—H35A | 109.5 |
| C14—C13—H13 | 120.3 | C33—C35—H35B | 109.5 |
| C9—C14—H14 | 119.4 | C33—C35—H35C | 109.5 |
| C13—C14—C9 | 121.1 (3) | H35A—C35—H35B | 109.5 |
| C13—C14—H14 | 119.4 | H35A—C35—H35C | 109.5 |
| C7—C15—H15A | 109.0 | H35B—C35—H35C | 109.5 |

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| C7—C15—H15B | 109.0 | C33—C36—H36A | 109.5 |
| C7—C15—C16 | 112.7 (2) | C33—C36—H36B | 109.5 |
| H15A—C15—H15B | 107.8 | C33—C36—H36C | 109.5 |
| C16—C15—H15A | 109.0 | H36A—C36—H36B | 109.5 |
| C16—C15—H15B | 109.0 | H36A—C36—H36C | 109.5 |
| O4—C16—N3 | 121.5 (2) | H36B—C36—H36C | 109.5 |
| O4—C16—C15 | 121.5 (2) | C37A—O8A—H8A | 109.5 |
| N3—C16—C15 | 116.9 (2) | O8A—C37A—H37A | 109.5 |
| C18—C17—N3 | 120.0 (2) | O8A—C37A—H37B | 109.5 |
| C18—C17—C22 | 118.9 (3) | O8A—C37A—H37C | 109.5 |
| C22—C17—N3 | 121.0 (3) | H37A—C37A—H37B | 109.5 |
| C17—C18—H18 | 119.4 | H37A—C37A—H37C | 109.5 |
| C17—C18—C19 | 121.3 (3) | H37B—C37A—H37C | 109.5 |
| C19—C18—H18 | 119.4 | C37B—O8B—H8B | 109.5 |
| C18—C19—H19 | 120.4 | O8B—C37B—H37D | 109.5 |
| C20—C19—C18 | 119.2 (3) | O8B—C37B—H37E | 109.5 |
| C20—C19—H19 | 120.4 | O8B—C37B—H37F | 109.5 |
| O5—C20—C19 | 124.3 (3) | H37D—C37B—H37E | 109.5 |
| O5—C20—C21 | 116.0 (3) | H37D—C37B—H37F | 109.5 |
| C19—C20—C21 | 119.7 (3) | H37E—C37B—H37F | 109.5 |

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| Cl1—C12—C13—C14 | −178.3 (2) | C9—C10—C11—C12 | 0.2 (5) |
| O1—N1—C4—N2 | 179.9 (2) | C10—C9—C14—C13 | −1.2 (4) |
| O1—N1—C4—C3 | −0.3 (3) | C10—C11—C12—Cl1 | 178.4 (2) |
| O1—C2—C3—C4 | −0.4 (3) | C10—C11—C12—C13 | −1.6 (5) |
| O2—C5—C6—O3 | 3.6 (4) | C11—C12—C13—C14 | 1.7 (5) |
| O2—C5—C6—C7 | −177.3 (3) | C12—C13—C14—C9 | −0.2 (5) |
| O3—C6—C7—C8 | 176.7 (3) | C14—C9—C10—C11 | 1.2 (4) |
| O3—C6—C7—C15 | 1.0 (5) | C15—C7—C8—N2 | 177.6 (2) |
| O5—C20—C21—C22 | −178.9 (3) | C15—C7—C8—C9 | 55.3 (3) |
| O7—C28—C29—C30 | −177.4 (3) | C16—N3—C17—C18 | 78.2 (3) |
| N1—O1—C2—C1 | −178.7 (3) | C16—N3—C17—C22 | −99.5 (3) |
| N1—O1—C2—C3 | 0.2 (3) | C16—N3—C24—C25 | 156.7 (2) |
| N2—C5—C6—O3 | −176.9 (2) | C16—N3—C24—C32 | −78.1 (3) |
| N2—C5—C6—C7 | 2.2 (3) | C17—N3—C16—O4 | −166.9 (3) |
| N2—C8—C9—C10 | −54.2 (4) | C17—N3—C16—C15 | 16.2 (4) |
| N2—C8—C9—C14 | 131.3 (3) | C17—N3—C24—C25 | −33.4 (3) |
| N3—C17—C18—C19 | −176.2 (2) | C17—N3—C24—C32 | 91.9 (3) |
| N3—C17—C22—C21 | 176.0 (3) | C17—C18—C19—C20 | 0.1 (5) |
| N3—C24—C25—C26 | −83.4 (3) | C18—C17—C22—C21 | −1.7 (4) |
| N3—C24—C25—C30 | 97.7 (3) | C18—C19—C20—O5 | 178.8 (3) |
| N3—C24—C32—O6 | −27.8 (3) | C18—C19—C20—C21 | −1.6 (5) |
| N3—C24—C32—N4 | 155.6 (2) | C19—C20—C21—C22 | 1.5 (5) |
| C1—C2—C3—C4 | 178.2 (4) | C20—C21—C22—C17 | 0.2 (5) |
| C2—O1—N1—C4 | 0.1 (3) | C22—C17—C18—C19 | 1.6 (4) |
| C2—C3—C4—N1 | 0.5 (4) | C23—O5—C20—C19 | −3.3 (5) |
| C2—C3—C4—N2 | −179.8 (3) | C23—O5—C20—C21 | 177.1 (3) |
| C4—N2—C5—O2 | 10.4 (5) | C24—N3—C16—O4 | 3.0 (4) |

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| C4—N2—C5—C6 | −169.1 (2) | C24—N3—C16—C15 | −173.9 (2) |
| C4—N2—C8—C7 | 168.3 (2) | C24—N3—C17—C18 | −91.1 (3) |
| C4—N2—C8—C9 | −71.4 (3) | C24—N3—C17—C22 | 91.1 (3) |
| C5—N2—C4—N1 | 168.8 (3) | C24—C25—C26—C27 | −178.1 (3) |
| C5—N2—C4—C3 | −10.9 (5) | C24—C25—C30—C29 | 177.5 (2) |
| C5—N2—C8—C7 | −0.1 (3) | C25—C24—C32—O6 | 97.9 (3) |
| C5—N2—C8—C9 | 120.2 (3) | C25—C24—C32—N4 | −78.7 (3) |
| C5—C6—C7—C8 | −2.2 (3) | C25—C26—C27—C28 | 0.5 (5) |
| C5—C6—C7—C15 | −177.9 (3) | C26—C25—C30—C29 | −1.5 (4) |
| C6—C7—C8—N2 | 1.4 (3) | C26—C27—C28—O7 | 177.0 (3) |
| C6—C7—C8—C9 | −120.9 (3) | C26—C27—C28—C29 | −1.4 (5) |
| C6—C7—C15—C16 | −107.2 (3) | C27—C28—C29—C30 | 0.9 (4) |
| C7—C8—C9—C10 | 60.9 (3) | C28—C29—C30—C25 | 0.6 (5) |
| C7—C8—C9—C14 | −113.6 (3) | C30—C25—C26—C27 | 0.9 (4) |
| C7—C15—C16—O4 | 22.5 (4) | C31—O7—C28—C27 | −174.8 (3) |
| C7—C15—C16—N3 | −160.6 (2) | C31—O7—C28—C29 | 3.6 (4) |
| C8—N2—C4—N1 | 2.2 (4) | C32—N4—C33—C34 | 61.9 (4) |
| C8—N2—C4—C3 | −177.5 (3) | C32—N4—C33—C35 | −61.1 (4) |
| C8—N2—C5—O2 | 178.3 (3) | C32—N4—C33—C36 | −179.1 (3) |
| C8—N2—C5—C6 | −1.2 (3) | C32—C24—C25—C26 | 151.4 (2) |
| C8—C7—C15—C16 | 77.5 (3) | C32—C24—C25—C30 | −27.5 (4) |
| C8—C9—C10—C11 | −173.5 (3) | C33—N4—C32—O6 | −3.6 (4) |
| C8—C9—C14—C13 | 173.5 (3) | C33—N4—C32—C24 | 172.9 (2) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O8*A* | 0.75 (4) | 1.88 (5) | 2.61 (3) | 162 (4) |
| O3—H3···O8*B* | 0.75 (4) | 1.93 (4) | 2.680 (10) | 175 (4) |
| N4—H4···O2$^{i}$ | 0.87 (3) | 2.33 (3) | 3.193 (3) | 170 (3) |
| O8*A*—H8*A*···O4$^{i}$ | 0.82 | 1.88 | 2.60 (3) | 145 |
| O8*B*—H8*B*···O4$^{i}$ | 0.82 | 2.50 | 2.843 (8) | 107 |

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+1.