

(Z)-1-(2-Chlorophenyl)-3-methyl-4-[2-(4-nitrophenyl)hydrazin-1-ylidene]-1H-pyrazol-5(4H)-one

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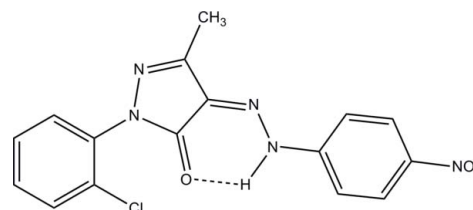
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Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.039; wR factor = 0.097; data-to-parameter ratio = 14.0.

There are two independent molecules, *A* and *B*, in the asymmetric unit of the title compound, $\text{C}_{16}\text{H}_{12}\text{ClN}_5\text{O}_3$. The relative orientations of the chlorophenyl ring with respect to the pyrazole ring in the two crystallographically independent molecules are different, and their corresponding dihedral angles are -53.3 (2) and 114.09 (18) $^\circ$ in molecules *A* and *B*, respectively. There are two strong intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, and two weak intramolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen bonds. The crystal packing is constructed by weak $\text{C}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ interactions, and two $\pi-\pi$ stacking interactions [centroid-centroid distances = 3.7894 (9) and 3.5719 (10) Å], forming a molecular ladder along the *a* axis.

Related literature

For synthesis and related literature, see: Abdel-Aziz *et al.* (2009); Bustos *et al.* (2006, 2007, 2009, 2012). For the biological activity of this class of compounds, see: Castagnolo *et al.* (2009); Chauhan *et al.* (1993); El-Hawash *et al.* (2006); Gunasekaran *et al.* (2011); Himly *et al.* (2003); Jolly *et al.* (1991); Kalluraya *et al.* (2007); Kawai *et al.* (1997); Moreau *et al.* (2008); Pasha *et al.* (2009); Radi *et al.* (2009); Singh (1991); Wu *et al.* (2002).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{12}\text{ClN}_5\text{O}_3$	$\gamma = 79.774$ (1) $^\circ$
$M_r = 357.76$	$V = 1612.7$ (2) Å ³
Triclinic, $P\bar{1}$	$Z = 4$
$a = 7.2715$ (6) Å	Mo $K\alpha$ radiation
$b = 14.7757$ (12) Å	$\mu = 0.26$ mm ⁻¹
$c = 15.7609$ (12) Å	$T = 150$ K
$\alpha = 75.408$ (1) $^\circ$	$0.24 \times 0.22 \times 0.10$ mm
$\beta = 86.943$ (1) $^\circ$	

Data collection

Bruker D8 Discover diffractometer with SMART CCD area-detector	6469 independent reflections
13022 measured reflections	4430 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.097$	$\Delta\rho_{\text{max}} = 0.44$ e Å ⁻³
$S = 0.91$	$\Delta\rho_{\text{min}} = -0.50$ e Å ⁻³
6469 reflections	
461 parameters	

Table 1

Hydrogen-bond geometry (Å, $^\circ$).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N4}-\text{H7}\cdots\text{O1}$	0.910 (19)	2.084 (18)	2.777 (2)	132.1 (14)
$\text{N9}-\text{H8}\cdots\text{O4}$	0.863 (19)	2.222 (18)	2.8647 (19)	131.2 (16)
$\text{C28}-\text{H28}\cdots\text{O2}$	0.95	2.57	3.265 (2)	130
$\text{C15}-\text{H15}\cdots\text{Cl2}$	0.95	2.93	3.4770 (18)	118
$\text{N9}-\text{H8}\cdots\text{O2}$	0.86 (2)	2.654 (18)	3.266 (2)	129.0 (13)
$\text{N9}-\text{H8}\cdots\text{O1}^{\text{i}}$	0.863 (19)	2.478 (17)	3.094 (2)	128.9 (14)
$\text{C4}-\text{H4}\cdots\text{O5}^{\text{ii}}$	0.95	2.37	3.202 (3)	146
$\text{C10}-\text{H10B}\cdots\text{O3}^{\text{iii}}$	0.98	2.52	3.392 (3)	148
$\text{C10}-\text{H10C}\cdots\text{O6}^{\text{iv}}$	0.98	2.43	3.175 (3)	133
$\text{C32}-\text{H32}\cdots\text{Cl2}^{\text{v}}$	0.95	2.80	3.6898 (18)	157

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP in SHELXTL-PC* (Sheldrick, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2200).

References

- Abdel-Aziz, M., Abuo-Rahma, G. E.-D. A. & Hassan, A. A. (2009). *Eur. J. Med. Chem.* **44**, 3480–3487.
- Bruker (2000). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2001). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bustos, C., Pérez-Cerda, M., Alvarez-Thon, L., Barrales-Salcedo, E. & Garland, M. T. (2012). *Acta Cryst.* **E68**, o353–o354.
- Bustos, C., Sánchez, C., Schott, E., Alvarez-Thon, L. & Fuentealba, M. (2007). *Acta Cryst.* **E63**, o1138–o1139.
- Bustos, C., Schott, E., Mac-Leod-Carey, D. A., Ibañez, A. & Alvarez-Thon, L. (2006). *Acta Cryst.* **E62**, o2499–o2501.
- Bustos, C., Schott, E., Rios, M., Sanchez, C. & Carcamo, J. G. (2009). *J. Chil. Chem. Soc.* **54**, 267–268.
- Castagnolo, D., Manetti, F., Radi, M., Bechi, B., Pagano, M., De Logu, A., Meleddu, R., Saggi, M. & Botta, M. (2009). *Bioorg. Med. Chem.* **17**, 5716–5721.
- Chauhan, P. M. S., Singh, S. & Chatterjee, R. K. (1993). *Indian J. Chem.* **32**, 858–861.
- El-Hawash, S. A. M., Badawey, E. A. M. & El-Ashmawey, I. M. (2006). *Eur. J. Med. Chem.* **41**, 155–165.
- Gunasekaran, P., Perumal, S., Yogeeswari, P. & Sriram, D. (2011). *Eur. J. Med. Chem.* **46**, 4530–4536.
- Himly, M., Jahn-Schmid, B., Pittertschatscher, K., Bohle, B., Grubmayr, K., Ferreira, F., Ebner, H. & Ebner, C. (2003). *J. Allergy Clin. Immunol.* **111**, 882–888.
- Jolly, V. S., Dalvi, M. Y. & Shrivastava, A. K. (1991). *J. Indian Chem. Soc.* **68**, 513–514.
- Kalluraya, B., Lingappa, B. & Rai, N. S. (2007). *Indian J. Heterocycl. Chem.* **17**, 67–70.
- Kawai, H., Nakai, H., Suga, M., Yuki, S., Watanabe, T. & Saito, K. I. (1997). *J. Pharmacol. Exp. Ther.* **281**, 921–927.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Moreau, F., Desroy, N., Genevard, J. M., Vongsouthi, V., Gerusz, V., Le Fralliec, G., Oliveira, C., Floquet, S., Denis, A., Escaich, S., Wolf, K., Busemann, M. & Aschenbrenner, A. (2008). *Bioorg. Med. Chem. Lett.* **18**, 4022–4026.
- Pasha, F. A., Muddassar, M., Neaz, M. M. & Cho, S. J. (2009). *J. Mol. Graph. Model.* **28**, 54–61.
- Radi, M., Bernardo, V., Bechi, B., Castagnolo, D., Pagano, M. & Botta, M. (2009). *Tetrahedron Lett.* **50**, 6572–6575.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Singh, D. (1991). *J. Indian Chem. Soc.* **68**, 165–167.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Wu, T. W., Zeng, L. H., Wu, J. & Fung, K. P. (2002). *Life Sci.* **71**, 2249–2255.

supporting information

Acta Cryst. (2012). E68, o2351–o2352 [https://doi.org/10.1107/S1600536812029790]

(Z)-1-(2-Chlorophenyl)-3-methyl-4-[2-(4-nitrophenyl)hydrazin-1-ylidene]-1H-pyrazol-5(4H)-one

Carlos Bustos, Andrés Escobar-Fuentealba, Luis Alvarez-Thon, Rodrigo Faundez-Gutierrez and Maria Teresa Garland

S1. Comment

In previous papers, we have informed preliminary results about the synthesis of a large library of pyrazoles by reaction of β -diketohydrazone with substituted arylhydrazines (Bustos *et al.*, 2009) and three molecular structures of this type of compounds have been reported (Bustos *et al.*, 2006, 2007, 2012). As a consequence of these studies we have found that α -hydrazo- β -ketoesters yield pyrazolones by reaction with substituted arylhydrazines. Now, we present the synthesis and the molecular structure of the title compound, prepared by reaction of (*E*)-ethyl 2-(2-(4-nitrophenyl)hydrazinylidene)-3-oxobutanoate with (2-chlorophenyl)hydrazine.

The title compound shown in Fig. 1, crystallizes with two independent molecules in the asymmetric unit, where the chlorophenyl ring display a different orientation with respect to the pyrazole ring in molecules A and B. The corresponding dihedral angles between the chlorophenyl and pyrazole rings, in molecule A and molecule B are $-53.3(2)^\circ$ and $114.09(18)^\circ$ respectively. In the crystal, two strong intramolecular hydrogen bonds (N4—H7 \cdots O1 and N9—H8 \cdots O4) are observed (Fig. 1, Table 1). As shown in Fig. 1 and Table 1, there are three additional weak intramolecular contacts that link molecules A and B (N9—H8 \cdots O2, C15—H15 \cdots C12 and C28—H28 \cdots O2). The atoms in the C1—C6 ring are slightly disordered due to thermal motion.

The partial packing of (I), shows that the two molecules in the asymmetric unit form inversion dimers *via* a pair of weak C32—H32 \cdots C12^v bonds (Fig. 2, Table 1). There is another pair of weak C10—H10B \cdots O3ⁱⁱⁱ bonds that form a dimer involving molecule A. In addition, there are two π - π stacking interactions, one with $Cg\cdots Cg^j$ distance of 3.6112(9) Å and the other with $Cg\cdots Cg^{iii}$ distance of 3.7894(9) Å, where Cg is the centroid of the C11—C16 ring. These interactions form a molecular ladder that runs parallel to the *a* axis (Fig. 3, Table 1). Finally the crystal packing is completed with three weak contacts: C4—H4 \cdots O5ⁱⁱ, N9—H8 \cdots O1ⁱ and C10—H10C \cdots O6^{iv} (Table 1). [Symmetry codes: (i) $-x, -y + 1, -z + 1$; (ii) $x - 1, y + 1, z + 1$; (iii) $-x + 1, -y + 1, -z + 1$; (iv) $x, y, z + 1$; (v) $-x + 1, -y, -z + 1$].

S2. Experimental

In a 100 ml round-bottomed flask were added 2.34 g (8.38 mmole) of (*E*)-ethyl 2-(2-(4-nitrophenyl)hydrazinylidene)-3-oxobutanoate, 1.60 g (8.94 mmole) 2-(2-chlorophenyl)hydrazine hydrochloride, 5 ml of glacial acetic acid and 50 ml of ethanol. The reaction mixture was magnetically stirred and heated under reflux during 36 h. Then, after cooling at room temperature, the precipitate was filtrated by suction and dried in a vacuum oven at 45°C during 24 h. Yield 78% of crude product. Single crystals suitable for X-ray studies were obtained by recrystallization from tetrahydrofuran. Melting point: 182–183 °C.

S3. Refinement

All H atoms were found in difference Fourier maps. The H atoms attached to the N9 and N4 atoms were refined freely against the diffraction data, but all other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with aromatic C—H = 0.95 Å, methyl C—H = 0.98 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{aromatic C})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl C})$.

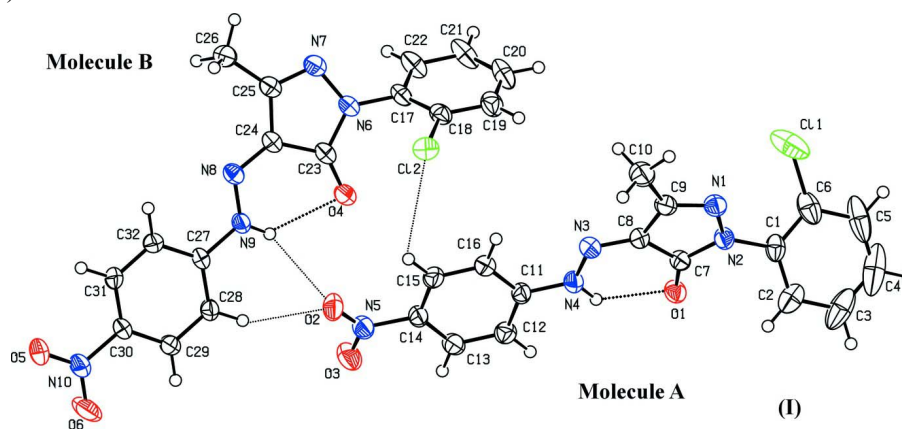


Figure 1

View of the title compound with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. The strong intramolecular hydrogen bonds (N9—H8...O4 and N4—H7...O1) are depicted with dashed lines. The weak intramolecular contacts (N9—H8...O2, C15—H15...Cl2 and C28—H28...O2) are depicted with dotted lines.

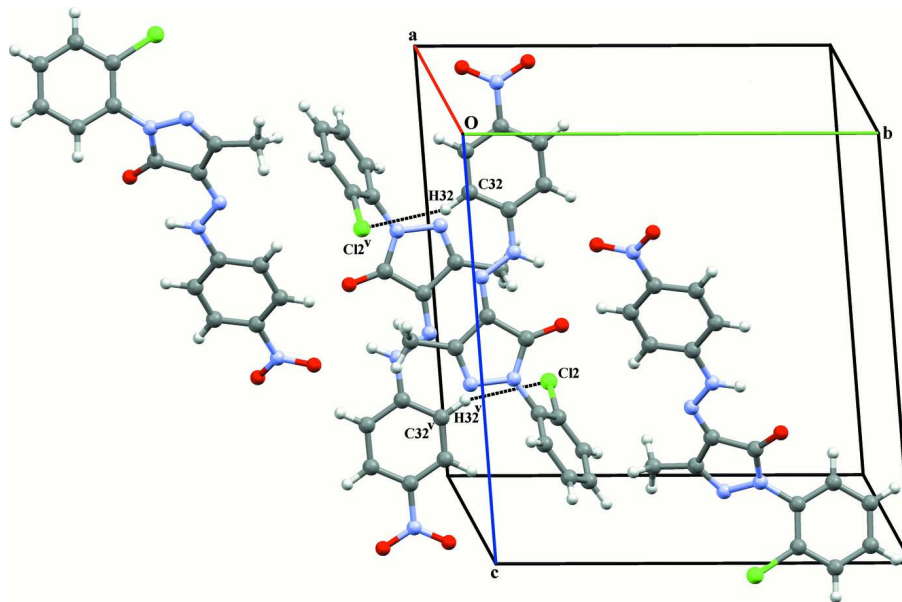


Figure 2

Part of the crystal packing showing the formation of an inversion-related dimer *via* C12—H32...Cl2^v weak contacts (dashed lines). [Symmetry code: (v) $-x + 1, -y, -z + 1$].

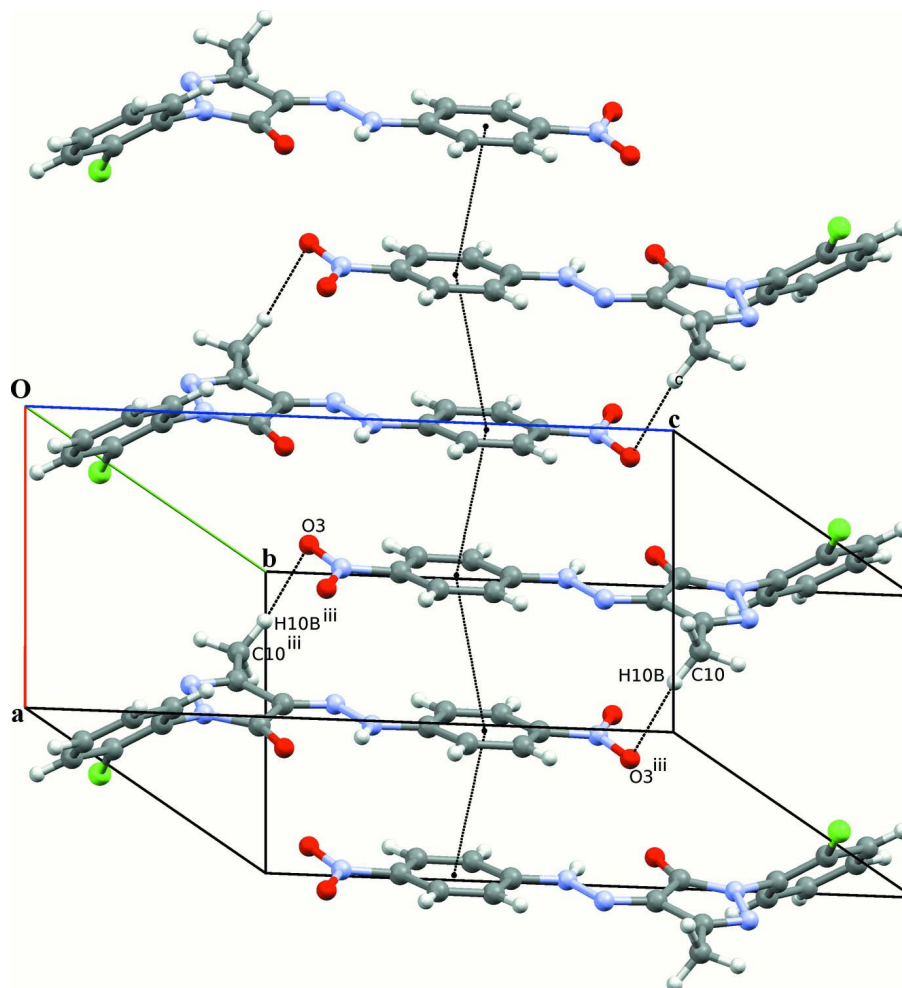


Figure 3

Part of the crystal packing showing the formation of a dimer *via* C10—H10B...O3ⁱⁱⁱ weak contacts (dashed lines). In addition, there are two π - π stacking interactions ($Cg \cdots Cg^i$ and $Cg \cdots Cg^{iii}$) that form a molecular ladder along the *a* direction, where *Cg* is the centroid of the C11—C16 ring. [Symmetry codes: (i) $-x, -y + 1, -z + 1$; (iii) $-x + 1, -y + 1, -z + 1$].

(*Z*)-1-(2-Chlorophenyl)-3-methyl-4-[2-(4-nitrophenyl)hydrazin-1-ylidene]-1*H*-pyrazol-5(4*H*)-one

Crystal data

$C_{16}H_{12}ClN_5O_3$

$M_r = 357.76$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 7.2715$ (6) Å

$b = 14.7757$ (12) Å

$c = 15.7609$ (12) Å

$\alpha = 75.408$ (1)°

$\beta = 86.943$ (1)°

$\gamma = 79.774$ (1)°

$V = 1612.7$ (2) Å³

$Z = 4$

$F(000) = 736$

$D_x = 1.474$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 999 reflections

$\theta = 1.7$ – 26.3 °

$\mu = 0.26$ mm⁻¹

$T = 150$ K

Polyhedron, orange

$0.24 \times 0.22 \times 0.10$ mm

Data collection

Bruker D8 Discover
 diffractometer with SMART CCD area-detector
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 13022 measured reflections
 6469 independent reflections

4430 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$
 $\theta_{\text{max}} = 26.3^\circ$, $\theta_{\text{min}} = 1.7^\circ$
 $h = -9 \rightarrow 9$
 $k = -18 \rightarrow 18$
 $l = -19 \rightarrow 18$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.097$
 $S = 0.91$
 6469 reflections
 461 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0531P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.44 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.50 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R -factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating $-R$ -factor-obs *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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	-0.01333 (8)	0.62030 (5)	1.02576 (4)	0.0783 (2)
O1	0.04690 (16)	0.71458 (8)	0.70638 (7)	0.0356 (4)
O2	0.39096 (19)	0.34058 (10)	0.33864 (9)	0.0506 (5)
O3	0.1869 (2)	0.45770 (10)	0.26804 (9)	0.0556 (5)
N1	0.3064 (2)	0.59306 (10)	0.89571 (9)	0.0348 (5)
N2	0.17363 (19)	0.66873 (10)	0.84708 (9)	0.0325 (5)
N3	0.2942 (2)	0.52887 (10)	0.69715 (9)	0.0337 (5)
N4	0.2057 (2)	0.57477 (11)	0.62260 (9)	0.0325 (5)
N5	0.2826 (2)	0.41611 (12)	0.33324 (11)	0.0427 (6)
C1	0.0966 (2)	0.74606 (13)	0.88317 (12)	0.0360 (6)
C2	0.1016 (3)	0.83741 (13)	0.83373 (15)	0.0472 (7)
C3	0.0258 (3)	0.91360 (16)	0.8665 (2)	0.0746 (10)
C4	-0.0534 (4)	0.8998 (2)	0.9486 (3)	0.0919 (14)
C5	-0.0606 (3)	0.8097 (2)	0.99852 (17)	0.0776 (10)
C6	0.0128 (3)	0.73186 (16)	0.96534 (13)	0.0509 (7)
C7	0.1476 (2)	0.65996 (12)	0.76493 (11)	0.0303 (6)

C8	0.2673 (2)	0.56924 (11)	0.76232 (11)	0.0303 (6)
C9	0.3580 (2)	0.53480 (12)	0.84638 (11)	0.0334 (6)
C10	0.4958 (3)	0.44607 (13)	0.87643 (13)	0.0461 (7)
C11	0.2284 (2)	0.53490 (12)	0.55094 (11)	0.0304 (6)
C12	0.1493 (2)	0.58808 (12)	0.47130 (11)	0.0345 (6)
C13	0.1677 (2)	0.54937 (13)	0.39973 (12)	0.0364 (6)
C14	0.2656 (2)	0.45854 (12)	0.40817 (11)	0.0337 (6)
C15	0.3460 (2)	0.40554 (12)	0.48640 (11)	0.0347 (6)
C16	0.3273 (2)	0.44349 (12)	0.55815 (11)	0.0328 (6)
Cl2	0.38569 (6)	0.19954 (3)	0.65477 (3)	0.0401 (2)
O4	0.14560 (16)	0.21654 (9)	0.47521 (7)	0.0372 (4)
O5	0.6349 (2)	0.07315 (9)	-0.02481 (8)	0.0530 (5)
O6	0.5293 (3)	0.22216 (11)	-0.05986 (9)	0.0832 (7)
N6	0.1295 (2)	0.08970 (10)	0.59660 (9)	0.0339 (5)
N7	0.16950 (19)	-0.01115 (10)	0.61679 (9)	0.0338 (5)
N8	0.31059 (18)	0.04899 (10)	0.39600 (9)	0.0291 (5)
N9	0.3055 (2)	0.13105 (11)	0.33806 (9)	0.0293 (5)
N10	0.5588 (2)	0.14595 (11)	-0.00578 (10)	0.0417 (6)
C17	0.0555 (2)	0.13690 (12)	0.66200 (11)	0.0338 (6)
C18	0.1585 (2)	0.19249 (12)	0.69232 (11)	0.0332 (6)
C19	0.0843 (3)	0.23988 (13)	0.75496 (12)	0.0417 (6)
C20	-0.0926 (3)	0.22971 (15)	0.78852 (12)	0.0495 (7)
C21	-0.1929 (3)	0.17270 (16)	0.76076 (12)	0.0503 (7)
C22	-0.1210 (3)	0.12697 (15)	0.69684 (12)	0.0443 (7)
C23	0.1699 (2)	0.13106 (13)	0.51121 (11)	0.0310 (6)
C24	0.2456 (2)	0.04932 (12)	0.47505 (10)	0.0284 (5)
C25	0.2384 (2)	-0.03413 (12)	0.54576 (11)	0.0303 (5)
C26	0.2990 (3)	-0.13494 (12)	0.54268 (12)	0.0379 (6)
C27	0.3760 (2)	0.13371 (12)	0.25320 (10)	0.0263 (5)
C28	0.3623 (2)	0.22144 (12)	0.19267 (11)	0.0306 (5)
C29	0.4246 (2)	0.22550 (12)	0.10800 (11)	0.0324 (6)
C30	0.4999 (2)	0.14203 (12)	0.08492 (11)	0.0306 (6)
C31	0.5177 (2)	0.05511 (12)	0.14479 (11)	0.0316 (6)
C32	0.4578 (2)	0.05063 (12)	0.22955 (11)	0.0302 (6)
H2	0.15770	0.84740	0.77700	0.0570*
H3	0.02820	0.97620	0.83220	0.0900*
H4	-0.10380	0.95290	0.97120	0.1100*
H5	-0.11540	0.80060	1.05550	0.0930*
H7	0.135 (2)	0.6333 (13)	0.6166 (11)	0.040 (5)*
H10A	0.53790	0.44200	0.93550	0.0690*
H10B	0.60330	0.44670	0.83620	0.0690*
H10C	0.43690	0.39110	0.87740	0.0690*
H12	0.08310	0.65070	0.46640	0.0410*
H13	0.11360	0.58480	0.34520	0.0440*
H15	0.41360	0.34340	0.49060	0.0420*
H16	0.38160	0.40760	0.61240	0.0390*
H8	0.252 (2)	0.1836 (13)	0.3497 (12)	0.040 (5)*
H19	0.15420	0.27900	0.77470	0.0500*

H20	-0.14490	0.26260	0.83110	0.0590*
H21	-0.31260	0.16450	0.78560	0.0600*
H22	-0.19250	0.08870	0.67670	0.0530*
H26A	0.27280	-0.17660	0.59930	0.0570*
H26B	0.43350	-0.14620	0.53010	0.0570*
H26C	0.23080	-0.14830	0.49650	0.0570*
H28	0.31030	0.27800	0.20970	0.0370*
H29	0.41610	0.28480	0.06590	0.0390*
H31	0.57090	-0.00110	0.12750	0.0380*
H32	0.47180	-0.00860	0.27180	0.0360*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0652 (4)	0.1022 (5)	0.0431 (3)	0.0082 (3)	0.0134 (3)	0.0083 (3)
O1	0.0404 (7)	0.0362 (7)	0.0283 (7)	-0.0009 (6)	-0.0055 (6)	-0.0072 (6)
O2	0.0640 (10)	0.0469 (9)	0.0502 (9)	-0.0167 (7)	0.0170 (7)	-0.0278 (7)
O3	0.0732 (10)	0.0675 (10)	0.0328 (8)	-0.0217 (8)	-0.0005 (7)	-0.0177 (7)
N1	0.0385 (9)	0.0318 (8)	0.0303 (8)	0.0003 (7)	-0.0039 (7)	-0.0042 (7)
N2	0.0385 (8)	0.0304 (8)	0.0274 (8)	0.0003 (6)	-0.0035 (7)	-0.0087 (6)
N3	0.0362 (8)	0.0349 (8)	0.0302 (8)	-0.0073 (7)	0.0034 (7)	-0.0083 (7)
N4	0.0374 (9)	0.0304 (9)	0.0294 (8)	-0.0020 (7)	0.0016 (7)	-0.0101 (7)
N5	0.0531 (11)	0.0469 (10)	0.0357 (10)	-0.0223 (9)	0.0117 (8)	-0.0168 (8)
C1	0.0342 (10)	0.0395 (11)	0.0364 (11)	0.0042 (8)	-0.0118 (8)	-0.0180 (9)
C2	0.0425 (12)	0.0357 (11)	0.0646 (14)	0.0002 (9)	-0.0204 (10)	-0.0152 (10)
C3	0.0645 (17)	0.0444 (14)	0.122 (2)	0.0115 (12)	-0.0504 (17)	-0.0375 (16)
C4	0.0711 (19)	0.093 (2)	0.129 (3)	0.0405 (16)	-0.0581 (19)	-0.085 (2)
C5	0.0541 (15)	0.123 (2)	0.0606 (16)	0.0365 (16)	-0.0269 (12)	-0.0621 (18)
C6	0.0436 (12)	0.0706 (15)	0.0355 (11)	0.0138 (10)	-0.0139 (9)	-0.0212 (11)
C7	0.0329 (10)	0.0316 (10)	0.0269 (9)	-0.0078 (8)	0.0020 (8)	-0.0071 (8)
C8	0.0337 (10)	0.0292 (9)	0.0286 (10)	-0.0057 (8)	0.0036 (7)	-0.0087 (8)
C9	0.0360 (10)	0.0316 (10)	0.0300 (10)	-0.0042 (8)	0.0028 (8)	-0.0046 (8)
C10	0.0519 (12)	0.0399 (11)	0.0392 (11)	0.0053 (9)	-0.0015 (9)	-0.0055 (9)
C11	0.0322 (10)	0.0320 (10)	0.0304 (10)	-0.0096 (8)	0.0061 (7)	-0.0122 (8)
C12	0.0350 (10)	0.0322 (10)	0.0359 (10)	-0.0047 (8)	0.0009 (8)	-0.0084 (8)
C13	0.0402 (11)	0.0401 (11)	0.0293 (10)	-0.0107 (9)	0.0024 (8)	-0.0072 (8)
C14	0.0390 (10)	0.0369 (10)	0.0307 (10)	-0.0154 (8)	0.0086 (8)	-0.0143 (8)
C15	0.0382 (10)	0.0291 (10)	0.0376 (11)	-0.0070 (8)	0.0085 (8)	-0.0107 (8)
C16	0.0364 (10)	0.0314 (10)	0.0303 (10)	-0.0061 (8)	0.0026 (8)	-0.0072 (8)
Cl2	0.0379 (3)	0.0390 (3)	0.0408 (3)	-0.0072 (2)	-0.0009 (2)	-0.0045 (2)
O4	0.0464 (8)	0.0364 (7)	0.0264 (7)	-0.0047 (6)	0.0047 (6)	-0.0062 (6)
O5	0.0758 (10)	0.0461 (8)	0.0401 (8)	-0.0067 (7)	0.0197 (7)	-0.0230 (7)
O6	0.1420 (16)	0.0506 (10)	0.0322 (9)	0.0191 (10)	0.0312 (9)	0.0043 (8)
N6	0.0407 (9)	0.0369 (9)	0.0240 (8)	-0.0084 (7)	0.0068 (7)	-0.0078 (7)
N7	0.0346 (8)	0.0387 (9)	0.0279 (8)	-0.0092 (7)	0.0018 (6)	-0.0063 (7)
N8	0.0271 (8)	0.0363 (8)	0.0240 (8)	-0.0066 (6)	-0.0010 (6)	-0.0068 (7)
N9	0.0341 (8)	0.0292 (8)	0.0238 (8)	-0.0014 (7)	0.0032 (6)	-0.0084 (7)
N10	0.0516 (10)	0.0410 (10)	0.0314 (9)	-0.0039 (8)	0.0125 (8)	-0.0125 (8)

C17	0.0376 (10)	0.0413 (11)	0.0204 (9)	-0.0029 (8)	0.0022 (7)	-0.0067 (8)
C18	0.0359 (10)	0.0345 (10)	0.0241 (9)	0.0012 (8)	-0.0012 (8)	-0.0028 (8)
C19	0.0504 (12)	0.0427 (11)	0.0298 (10)	0.0011 (9)	-0.0070 (9)	-0.0099 (9)
C20	0.0506 (13)	0.0653 (14)	0.0271 (11)	0.0139 (11)	-0.0031 (9)	-0.0173 (10)
C21	0.0360 (11)	0.0856 (16)	0.0260 (10)	-0.0011 (11)	0.0032 (8)	-0.0151 (11)
C22	0.0363 (11)	0.0712 (14)	0.0270 (10)	-0.0124 (10)	0.0019 (8)	-0.0134 (10)
C23	0.0306 (9)	0.0399 (11)	0.0225 (9)	-0.0065 (8)	0.0011 (7)	-0.0078 (8)
C24	0.0256 (9)	0.0367 (10)	0.0234 (9)	-0.0060 (7)	0.0007 (7)	-0.0083 (8)
C25	0.0267 (9)	0.0401 (10)	0.0253 (9)	-0.0106 (8)	-0.0007 (7)	-0.0067 (8)
C26	0.0420 (11)	0.0385 (11)	0.0329 (10)	-0.0102 (9)	0.0015 (8)	-0.0063 (9)
C27	0.0256 (9)	0.0338 (9)	0.0213 (9)	-0.0044 (7)	0.0009 (7)	-0.0107 (7)
C28	0.0361 (10)	0.0284 (9)	0.0273 (9)	-0.0013 (8)	0.0018 (8)	-0.0104 (8)
C29	0.0388 (10)	0.0298 (10)	0.0259 (9)	-0.0026 (8)	0.0034 (8)	-0.0050 (8)
C30	0.0347 (10)	0.0344 (10)	0.0234 (9)	-0.0057 (8)	0.0051 (7)	-0.0098 (8)
C31	0.0362 (10)	0.0293 (10)	0.0312 (10)	-0.0043 (8)	0.0060 (8)	-0.0133 (8)
C32	0.0344 (10)	0.0265 (9)	0.0286 (10)	-0.0044 (7)	0.0020 (7)	-0.0057 (8)

Geometric parameters (Å, °)

C11—C6	1.721 (2)	C14—C15	1.380 (2)
C12—C18	1.7367 (16)	C15—C16	1.374 (2)
O1—C7	1.235 (2)	C2—H2	0.9500
O2—N5	1.233 (2)	C3—H3	0.9500
O3—N5	1.235 (2)	C4—H4	0.9500
O4—C23	1.232 (2)	C5—H5	0.9500
O5—N10	1.219 (2)	C10—H10A	0.9800
O6—N10	1.221 (2)	C10—H10B	0.9800
N1—C9	1.295 (2)	C10—H10C	0.9800
N1—N2	1.426 (2)	C12—H12	0.9500
N2—C1	1.417 (2)	C13—H13	0.9500
N2—C7	1.360 (2)	C15—H15	0.9500
N3—C8	1.302 (2)	C16—H16	0.9500
N3—N4	1.334 (2)	C17—C22	1.386 (3)
N4—C11	1.390 (2)	C17—C18	1.385 (2)
N5—C14	1.461 (2)	C18—C19	1.383 (3)
N4—H7	0.910 (19)	C19—C20	1.383 (3)
N6—C23	1.371 (2)	C20—C21	1.368 (3)
N6—N7	1.423 (2)	C21—C22	1.381 (3)
N6—C17	1.417 (2)	C23—C24	1.471 (3)
N7—C25	1.300 (2)	C24—C25	1.446 (2)
N8—N9	1.317 (2)	C25—C26	1.489 (3)
N8—C24	1.310 (2)	C27—C32	1.396 (2)
N9—C27	1.400 (2)	C27—C28	1.393 (2)
N10—C30	1.460 (2)	C28—C29	1.376 (2)
N9—H8	0.863 (19)	C29—C30	1.385 (3)
C1—C6	1.385 (3)	C30—C31	1.378 (2)
C1—C2	1.384 (3)	C31—C32	1.372 (2)
C2—C3	1.371 (3)	C19—H19	0.9500

C3—C4	1.370 (5)	C20—H20	0.9500
C4—C5	1.374 (5)	C21—H21	0.9500
C5—C6	1.392 (4)	C22—H22	0.9500
C7—C8	1.472 (2)	C26—H26A	0.9800
C8—C9	1.443 (2)	C26—H26B	0.9800
C9—C10	1.490 (3)	C26—H26C	0.9800
C11—C16	1.393 (2)	C28—H28	0.9500
C11—C12	1.394 (2)	C29—H29	0.9500
C12—C13	1.378 (3)	C31—H31	0.9500
C13—C14	1.381 (3)	C32—H32	0.9500
C11…N1	3.0568 (16)	C12…C14 ⁱⁱⁱ	3.557 (2)
C11…N2	3.0352 (15)	C13…C12 ⁱⁱⁱ	3.557 (2)
C12…O4	3.3366 (12)	C13…C11 ⁱⁱⁱ	3.316 (2)
C12…N6	2.9917 (16)	C14…C12 ⁱⁱⁱ	3.557 (2)
C12…C15	3.4770 (18)	C15…O4	3.417 (2)
C12…C16	3.5002 (19)	C15…C12	3.4770 (18)
C12…C21 ⁱ	3.481 (2)	C15…C11 ^{iv}	3.348 (2)
C12…C22 ⁱ	3.604 (2)	C15…C16 ^{iv}	3.468 (2)
C12…C23	3.2713 (18)	C16…C16 ^{iv}	3.451 (2)
C12…H15	2.9300	C16…C12	3.5002 (19)
C12…H16	2.9700	C16…C15 ^{iv}	3.468 (2)
C12…H21 ⁱ	2.9800	C18…O4	3.355 (2)
C12…H32 ⁱⁱ	2.8000	C20…C5 ^{xiv}	3.468 (3)
O1…C2	3.108 (2)	C21…C12 ^{xvi}	3.481 (2)
O1…O3 ⁱⁱⁱ	3.2371 (19)	C22…C12 ^{xvi}	3.604 (2)
O1…N3	3.0341 (19)	C23…C26 ^{xii}	3.545 (3)
O1…N4	2.777 (2)	C23…C12	3.2713 (18)
O1…C28 ⁱⁱⁱ	3.396 (2)	C25…N8 ⁱⁱ	3.407 (2)
O1…O4 ⁱⁱⁱ	3.1049 (16)	C26…C23 ^{xii}	3.545 (3)
O1…N9 ⁱⁱⁱ	3.094 (2)	C28…O1 ⁱⁱⁱ	3.396 (2)
O2…N9	3.266 (2)	C28…C1 ⁱⁱⁱ	3.516 (2)
O2…C8 ^{iv}	3.189 (2)	C28…O2	3.265 (2)
O2…C28	3.265 (2)	C29…C1 ^{iv}	3.594 (2)
O2…O4	3.1526 (18)	C29…C2 ^{iv}	3.514 (3)
O2…N3 ^{iv}	3.188 (2)	C29…C6 ⁱⁱⁱ	3.341 (3)
O3…C10 ^{iv}	3.392 (3)	C29…C5 ⁱⁱⁱ	3.391 (3)
O3…O1 ⁱⁱⁱ	3.2371 (19)	C30…C3 ^{iv}	3.481 (3)
O3…C7 ⁱⁱⁱ	3.362 (2)	C30…C2 ^{iv}	3.322 (3)
O4…C15	3.417 (2)	C30…C5 ⁱⁱⁱ	3.407 (3)
O4…O1 ⁱⁱⁱ	3.1049 (16)	C30…C4 ⁱⁱⁱ	3.502 (3)
O4…C12	3.3366 (12)	C31…O5 ^v	3.343 (2)
O4…N8	3.077 (2)	C31…C3 ^{iv}	3.421 (3)
O4…O2	3.1526 (18)	C31…C2 ^{iv}	3.497 (3)
O4…C18	3.355 (2)	C5…H20 ^{xiv}	3.0000
O4…N9	2.8647 (19)	C7…H7	2.476 (17)
O5…C31 ^v	3.343 (2)	C7…H2	2.8400
O5…C4 ^{vi}	3.202 (3)	C19…H13 ⁱⁱⁱ	2.8500

O5...O5 ^v	3.102 (2)	C19...H5 ^{xiv}	2.9100
O6...C10 ^{vii}	3.175 (3)	C20...H13 ⁱⁱⁱ	2.9900
O1...H7	2.084 (18)	C20...H5 ^{xiv}	2.8500
O1...H28 ⁱⁱⁱ	2.8500	C21...H3 ^{xi}	3.0300
O1...H26A ^{viii}	2.7200	C22...H3 ^{xi}	2.7700
O1...H8 ⁱⁱⁱ	2.478 (17)	C23...H8	2.539 (18)
O1...H2	2.7400	C23...H26C ^{xii}	2.8900
O2...H15	2.4200	C23...H26B ⁱⁱ	2.9600
O2...H8	2.654 (18)	C24...H26B ⁱⁱ	2.9300
O2...H28	2.5700	H2...O1	2.7400
O3...H10B ^{iv}	2.5200	H2...N7 ^{viii}	2.8500
O3...H13	2.4500	H2...C7	2.8400
O4...H12 ⁱⁱⁱ	2.6500	H3...C21 ^{viii}	3.0300
O4...H8	2.222 (18)	H3...C22 ^{viii}	2.7700
O4...H7 ⁱⁱⁱ	2.876 (17)	H4...O5 ^{xiii}	2.3700
O5...H4 ^{vi}	2.3700	H4...H4 ^{xvii}	2.5600
O5...H31	2.4300	H5...C19 ^{xiv}	2.9100
O5...H31 ^v	2.7700	H5...C20 ^{xiv}	2.8500
O6...H10C ^{vii}	2.4300	H5...H20 ^{xiv}	2.5600
O6...H20 ^{ix}	2.9100	H7...O1	2.084 (18)
O6...H21 ^{ix}	2.8900	H7...O4 ⁱⁱⁱ	2.876 (17)
O6...H29	2.4400	H7...C7	2.476 (17)
N1...C11	3.0568 (16)	H7...H12	2.3600
N2...C11	3.0352 (15)	H8...O4	2.222 (18)
N3...O2 ^{iv}	3.188 (2)	H8...O1 ⁱⁱⁱ	2.478 (17)
N3...O1	3.0341 (19)	H8...C23	2.539 (18)
N4...O1	2.777 (2)	H8...H28	2.3600
N6...C12	2.9917 (16)	H8...O2	2.654 (18)
N8...C25 ⁱⁱ	3.407 (2)	H10A...N1 ^x	2.8400
N8...O4	3.077 (2)	H10B...O3 ^{iv}	2.5200
N9...O1 ⁱⁱⁱ	3.094 (2)	H10C...O6 ^{xv}	2.4300
N9...O2	3.266 (2)	H12...H7	2.3600
N9...O4	2.8647 (19)	H12...O4 ⁱⁱⁱ	2.6500
N1...H10A ^x	2.8400	H13...O3	2.4500
N3...H16	2.4700	H13...C19 ⁱⁱⁱ	2.8500
N7...H2 ^{xi}	2.8500	H13...C20 ⁱⁱⁱ	2.9900
N8...H22 ^{xii}	2.8400	H15...C12	2.9300
N8...H32	2.4800	H15...O2	2.4200
C1...C28 ⁱⁱⁱ	3.516 (2)	H16...C12	2.9700
C1...C29 ^{iv}	3.594 (2)	H16...N3	2.4700
C2...O1	3.108 (2)	H20...O6 ^{xviii}	2.9100
C2...C29 ^{iv}	3.514 (3)	H20...C5 ^{xiv}	3.0000
C2...C30 ^{iv}	3.322 (3)	H20...H5 ^{xiv}	2.5600
C2...C31 ^{iv}	3.497 (3)	H21...C12 ^{xvi}	2.9800
C3...C31 ^{iv}	3.421 (3)	H21...O6 ^{xviii}	2.8900
C3...C30 ^{iv}	3.481 (3)	H22...N8 ^{xii}	2.8400
C4...O5 ^{xiii}	3.202 (3)	H22...H32 ^{xii}	2.5300
C4...C30 ⁱⁱⁱ	3.502 (3)	H26A...O1 ^{xi}	2.7200

C5...C29 ⁱⁱⁱ	3.391 (3)	H26B...C23 ⁱⁱ	2.9600
C5...C20 ^{xiv}	3.468 (3)	H26B...C24 ⁱⁱ	2.9300
C5...C30 ⁱⁱⁱ	3.407 (3)	H26C...C23 ^{xii}	2.8900
C6...C29 ⁱⁱⁱ	3.341 (3)	H28...O2	2.5700
C7...O3 ⁱⁱⁱ	3.362 (2)	H28...H8	2.3600
C8...O2 ^{iv}	3.189 (2)	H28...O1 ⁱⁱⁱ	2.8500
C10...O6 ^{xv}	3.175 (3)	H29...O6	2.4400
C10...O3 ^{iv}	3.392 (3)	H31...O5	2.4300
C11...C15 ^{iv}	3.348 (2)	H31...O5 ^v	2.7700
C11...C13 ⁱⁱⁱ	3.316 (2)	H32...N8	2.4800
C12...C13 ⁱⁱⁱ	3.557 (2)	H32...C12 ⁱⁱ	2.8000
C12...C12 ⁱⁱⁱ	3.594 (2)	H32...H22 ^{xii}	2.5300
N2—N1—C9	106.83 (13)	H10A—C10—H10B	109.00
N1—N2—C1	119.93 (13)	H10A—C10—H10C	109.00
N1—N2—C7	112.75 (14)	C9—C10—H10A	110.00
C1—N2—C7	127.10 (15)	C11—C12—H12	120.00
N4—N3—C8	117.15 (15)	C13—C12—H12	120.00
N3—N4—C11	119.70 (15)	C14—C13—H13	120.00
O2—N5—O3	123.58 (17)	C12—C13—H13	120.00
O2—N5—C14	118.30 (15)	C14—C15—H15	120.00
O3—N5—C14	118.12 (16)	C16—C15—H15	120.00
C11—N4—H7	118.9 (11)	C11—C16—H16	120.00
N3—N4—H7	121.4 (11)	C15—C16—H16	120.00
C17—N6—C23	126.83 (15)	C18—C17—C22	119.34 (16)
N7—N6—C23	112.99 (14)	N6—C17—C22	119.98 (16)
N7—N6—C17	120.19 (13)	N6—C17—C18	120.68 (14)
N6—N7—C25	106.64 (13)	C12—C18—C17	120.02 (13)
N9—N8—C24	118.23 (15)	C12—C18—C19	119.47 (14)
N8—N9—C27	119.93 (15)	C17—C18—C19	120.47 (16)
O5—N10—O6	122.44 (16)	C18—C19—C20	119.38 (18)
O5—N10—C30	118.93 (15)	C19—C20—C21	120.46 (19)
O6—N10—C30	118.63 (16)	C20—C21—C22	120.3 (2)
N8—N9—H8	121.6 (12)	C17—C22—C21	120.0 (2)
C27—N9—H8	118.3 (12)	O4—C23—N6	127.09 (17)
C2—C1—C6	119.81 (19)	O4—C23—C24	129.70 (15)
N2—C1—C2	118.81 (16)	N6—C23—C24	103.21 (15)
N2—C1—C6	121.35 (18)	N8—C24—C23	128.65 (16)
C1—C2—C3	120.1 (2)	C23—C24—C25	106.06 (14)
C2—C3—C4	120.3 (3)	N8—C24—C25	125.28 (16)
C3—C4—C5	120.5 (3)	N7—C25—C26	121.74 (16)
C4—C5—C6	119.7 (3)	C24—C25—C26	127.17 (15)
C1—C6—C5	119.6 (2)	N7—C25—C24	111.09 (15)
C11—C6—C5	118.78 (17)	C28—C27—C32	120.65 (15)
C11—C6—C1	121.59 (17)	N9—C27—C28	118.40 (16)
O1—C7—C8	128.04 (16)	N9—C27—C32	120.95 (15)
O1—C7—N2	128.49 (16)	C27—C28—C29	119.44 (16)
N2—C7—C8	103.47 (14)	C28—C29—C30	119.11 (16)

C7—C8—C9	105.96 (14)	N10—C30—C31	119.01 (16)
N3—C8—C7	127.95 (15)	N10—C30—C29	119.11 (15)
N3—C8—C9	126.04 (15)	C29—C30—C31	121.87 (16)
N1—C9—C10	121.84 (15)	C30—C31—C32	119.33 (16)
C8—C9—C10	127.23 (16)	C27—C32—C31	119.53 (16)
N1—C9—C8	110.92 (15)	C18—C19—H19	120.00
N4—C11—C16	120.98 (15)	C20—C19—H19	120.00
C12—C11—C16	120.35 (16)	C19—C20—H20	120.00
N4—C11—C12	118.67 (16)	C21—C20—H20	120.00
C11—C12—C13	119.63 (16)	C20—C21—H21	120.00
C12—C13—C14	119.22 (16)	C22—C21—H21	120.00
N5—C14—C15	118.64 (16)	C17—C22—H22	120.00
C13—C14—C15	121.69 (16)	C21—C22—H22	120.00
N5—C14—C13	119.66 (15)	C25—C26—H26A	109.00
C14—C15—C16	119.39 (16)	C25—C26—H26B	109.00
C11—C16—C15	119.71 (16)	C25—C26—H26C	109.00
C1—C2—H2	120.00	H26A—C26—H26B	109.00
C3—C2—H2	120.00	H26A—C26—H26C	110.00
C4—C3—H3	120.00	H26B—C26—H26C	109.00
C2—C3—H3	120.00	C27—C28—H28	120.00
C5—C4—H4	120.00	C29—C28—H28	120.00
C3—C4—H4	120.00	C28—C29—H29	120.00
C4—C5—H5	120.00	C30—C29—H29	120.00
C6—C5—H5	120.00	C30—C31—H31	120.00
C9—C10—H10C	109.00	C32—C31—H31	120.00
C9—C10—H10B	109.00	C27—C32—H32	120.00
H10B—C10—H10C	110.00	C31—C32—H32	120.00
C9—N1—N2—C1	-177.60 (14)	C3—C4—C5—C6	-0.1 (4)
C9—N1—N2—C7	-2.68 (18)	C4—C5—C6—C11	-175.3 (2)
N2—N1—C9—C10	-179.42 (15)	C4—C5—C6—C1	1.6 (3)
N2—N1—C9—C8	1.49 (18)	N2—C7—C8—C9	-1.58 (16)
C7—N2—C1—C2	-45.7 (2)	N2—C7—C8—N3	-178.96 (16)
N1—N2—C1—C6	-53.3 (2)	O1—C7—C8—N3	1.4 (3)
C7—N2—C1—C6	132.57 (19)	O1—C7—C8—C9	178.79 (16)
C1—N2—C7—C8	177.05 (15)	N3—C8—C9—N1	177.47 (16)
N1—N2—C7—O1	-177.80 (16)	N3—C8—C9—C10	-1.6 (3)
C1—N2—C7—O1	-3.3 (3)	C7—C8—C9—C10	-179.01 (16)
N1—N2—C1—C2	128.42 (18)	C7—C8—C9—N1	0.03 (19)
N1—N2—C7—C8	2.58 (17)	N4—C11—C12—C13	-179.02 (15)
C8—N3—N4—C11	-179.70 (15)	N4—C11—C16—C15	179.38 (15)
N4—N3—C8—C7	1.0 (2)	C12—C11—C16—C15	-0.3 (2)
N4—N3—C8—C9	-175.90 (15)	C16—C11—C12—C13	0.7 (2)
N3—N4—C11—C16	6.4 (2)	C11—C12—C13—C14	-0.4 (2)
N3—N4—C11—C12	-173.89 (15)	C12—C13—C14—N5	178.73 (14)
O2—N5—C14—C15	-10.7 (2)	C12—C13—C14—C15	-0.2 (2)
O3—N5—C14—C13	-10.0 (2)	C13—C14—C15—C16	0.5 (2)
O2—N5—C14—C13	170.37 (15)	N5—C14—C15—C16	-178.37 (14)

O3—N5—C14—C15	168.98 (15)	C14—C15—C16—C11	-0.3 (2)
N7—N6—C17—C22	-65.3 (2)	C22—C17—C18—C12	175.76 (14)
C23—N6—C17—C18	-66.0 (2)	N6—C17—C18—C12	-3.7 (2)
C23—N6—C17—C22	114.6 (2)	C22—C17—C18—C19	-1.8 (3)
C17—N6—C23—C24	178.77 (15)	N6—C17—C18—C19	178.76 (16)
N7—N6—C17—C18	114.09 (18)	C18—C17—C22—C21	0.4 (3)
N7—N6—C23—O4	178.25 (15)	N6—C17—C22—C21	179.79 (18)
C17—N6—C23—O4	-1.7 (3)	C17—C18—C19—C20	1.3 (3)
N7—N6—C23—C24	-1.28 (17)	C12—C18—C19—C20	-176.25 (15)
C23—N6—N7—C25	1.31 (18)	C18—C19—C20—C21	0.6 (3)
C17—N6—N7—C25	-178.74 (14)	C19—C20—C21—C22	-2.1 (3)
N6—N7—C25—C24	-0.71 (17)	C20—C21—C22—C17	1.6 (3)
N6—N7—C25—C26	179.30 (15)	O4—C23—C24—N8	2.4 (3)
C24—N8—N9—C27	179.13 (14)	O4—C23—C24—C25	-178.71 (16)
N9—N8—C24—C25	179.09 (14)	N6—C23—C24—N8	-178.13 (16)
N9—N8—C24—C23	-2.2 (2)	N6—C23—C24—C25	0.80 (16)
N8—N9—C27—C28	177.73 (14)	N8—C24—C25—N7	178.93 (15)
N8—N9—C27—C32	-2.2 (2)	N8—C24—C25—C26	-1.1 (3)
O5—N10—C30—C31	-5.4 (2)	C23—C24—C25—N7	-0.05 (18)
O5—N10—C30—C29	175.80 (15)	C23—C24—C25—C26	179.94 (16)
O6—N10—C30—C29	-4.4 (2)	N9—C27—C28—C29	-177.71 (14)
O6—N10—C30—C31	174.38 (17)	C32—C27—C28—C29	2.2 (2)
N2—C1—C6—C5	179.79 (18)	N9—C27—C32—C31	176.99 (14)
C2—C1—C6—C11	174.84 (16)	C28—C27—C32—C31	-2.9 (2)
C6—C1—C2—C3	0.8 (3)	C27—C28—C29—C30	-0.1 (2)
C2—C1—C6—C5	-2.0 (3)	C28—C29—C30—N10	177.47 (14)
N2—C1—C6—C11	-3.4 (3)	C28—C29—C30—C31	-1.3 (2)
N2—C1—C2—C3	179.09 (19)	N10—C30—C31—C32	-178.19 (14)
C1—C2—C3—C4	0.7 (4)	C29—C30—C31—C32	0.6 (2)
C2—C3—C4—C5	-1.1 (4)	C30—C31—C32—C27	1.5 (2)

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y, -z+1$; (iii) $-x, -y+1, -z+1$; (iv) $-x+1, -y+1, -z+1$; (v) $-x+1, -y, -z$; (vi) $x+1, y-1, z-1$; (vii) $x, y, z-1$; (viii) $x, y+1, z$; (ix) $x+1, y, z-1$; (x) $-x+1, -y+1, -z+2$; (xi) $x, y-1, z$; (xii) $-x, -y, -z+1$; (xiii) $x-1, y+1, z+1$; (xiv) $-x, -y+1, -z+2$; (xv) $x, y, z+1$; (xvi) $x-1, y, z$; (xvii) $-x, -y+2, -z+2$; (xviii) $x-1, y, z+1$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N4—H7 \cdots O1	0.910 (19)	2.084 (18)	2.777 (2)	132.1 (14)
N9—H8 \cdots O4	0.863 (19)	2.222 (18)	2.8647 (19)	131.2 (16)
C28—H28 \cdots O2	0.95	2.57	3.265 (2)	130
C15—H15 \cdots C12	0.95	2.93	3.4770 (18)	118
N9—H8 \cdots O2	0.86 (2)	2.654 (18)	3.266 (2)	129.0 (13)
N9—H8 \cdots O1 ⁱⁱⁱ	0.863 (19)	2.478 (17)	3.094 (2)	128.9 (14)
C4—H4 \cdots O5 ^{xiii}	0.95	2.37	3.202 (3)	146
C10—H10B \cdots O3 ^{iv}	0.98	2.52	3.392 (3)	148
C10—H10C \cdots O6 ^{xv}	0.98	2.43	3.175 (3)	133
C32—H32 \cdots C12 ⁱⁱ	0.95	2.80	3.6898 (18)	157

Symmetry codes: (ii) $-x+1, -y, -z+1$; (iii) $-x, -y+1, -z+1$; (iv) $-x+1, -y+1, -z+1$; (xiii) $x-1, y+1, z+1$; (xv) $x, y, z+1$.