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ISSN 2414-3146

(1,4,8,11-Tetraazacyclotetradecane)palladium(II) diiodide monohydrate

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Received 18 July 2019
Accepted 19 July 2019

Edited by M. Weil, Vienna University of Technology, Austria

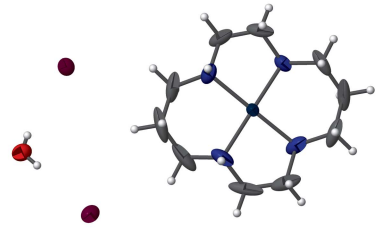
Keywords: crystal structure; palladium(II) complex; 1,4,8,11-tetraazacyclotetradecane; iodide anion.

CCDC reference: 1941633

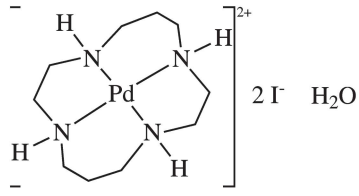
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $[Pd(C_{10}H_{24}N_4)]I_2\cdot H_2O$, the Pd^{II} ion is four-coordinated in a slightly distorted square-planar coordination environment defined by four N atoms from a 1,4,8,11-tetraazacyclotetradecane ligand. The cationic complex, two I^- anions and the solvent water molecule are linked through intermolecular hydrogen bonds into a three-dimensional network structure.

3D view



Chemical scheme



Structure description

With reference to the title compound, $[Pd(cyclam)]I_2\cdot H_2O$ (cyclam = 1,4,8,11-tetraazacyclotetradecane), the crystal structures of related cyclam-Pd^{II} complexes, *viz*. $[Pd(cyclam)]Cl_2\cdot 2CH_3OH$ (Hunter *et al.*, 2004) and $[Pd(cyclam)](CH_3CO_2)_2\cdot 2H_2O$ (Liang *et al.*, 2002), have been determined previously.

The title compound consists of a cationic $[Pd(cyclam)]^{2+}$ complex, two I^- counter-anions and a solvent water molecule. In the cationic complex, the central Pd^{II} ion is four-coordinated in a slightly distorted square-planar coordination environment defined by four N atoms from the tetradentate cyclam ligand (Fig. 1). The Pd—N bond lengths are almost equal with Pd—N = 2.0307 (19)-2.044 (2) Å. The six-membered chelate rings are in the stable chair conformations, and the torsion angles N1—C1—C2—N2 = 55.7 (3)° and N3—C6—C7—N4 = −57.1 (3)° reflect the *gauche* conformation for the group within the five-membered chelate rings. In the crystal, the complex molecules are stacked in columns parallel to the *b* axis. In the crystal structure, the complex cations, anions and solvent water molecule are linked through intermolecular O—H···I, N—H···O and N—H···I hydrogen bonds into a three-dimensional network structure (Table 1; Fig. 2).

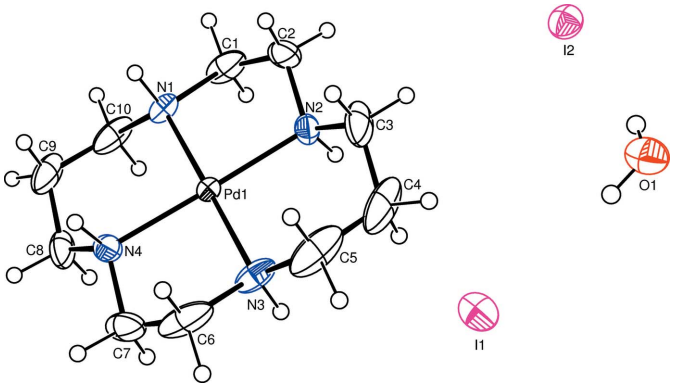


Figure 1
The molecular entities in the crystal structure of the title compound, showing the atom labelling and displacement ellipsoids drawn at the 40% probability level for non-H atoms.

Synthesis and crystallization

To a solution of $[PdI_2(pyridine)_2]$ (0.3322 g, 0.641 mmol) in acetone (30 ml) was added 1,4,8,11-tetraazacyclotetradecane (0.1296 g, 0.647 mmol) in MeOH (20 ml) and refluxed for 1 h. The formed dark-brown precipitate was removed by filtration. After evaporation of the solvent of the filtrate, the residue was washed with acetone, and dried at 323 K, to give a pale-yellow powder (0.3166 g). Yellow crystals suitable for X-ray analysis were obtained by slow evaporation from an MeOH/2-butanone solution at room temperature.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The highest peak (0.49 e Å^{-3}) and the deepest hole (−0.72 e Å^{-3}) in the difference Fourier map are located 0.79 and 0.77 Å, respectively, from the atoms I2 and I1.

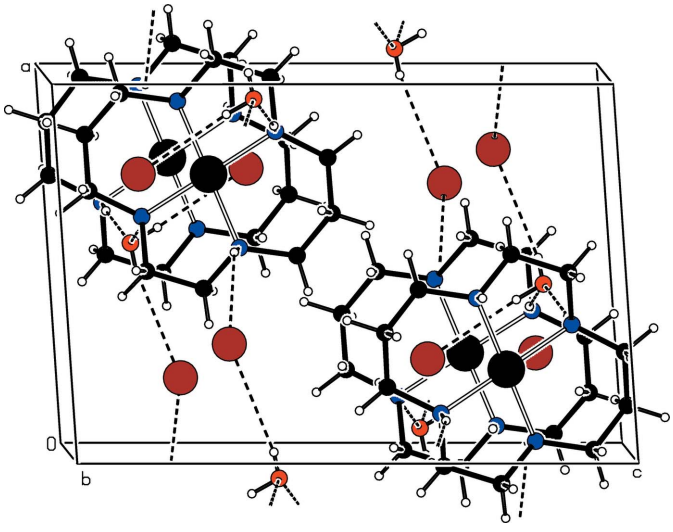


Figure 2
The packing in the crystal structure of the title compound, viewed approximately along the *b* axis. Hydrogen-bonding interactions are drawn as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H2*O*···I1	0.79 (4)	2.63 (5)	3.405 (3)	169 (4)
O1—H1*O*···I2	0.65 (4)	2.91 (4)	3.549 (3)	168 (5)
N2—H2···O1^{i}	0.78 (3)	2.22 (3)	2.993 (3)	173 (3)
N3—H3···I2^{ii}	0.82 (3)	3.02 (3)	3.656 (2)	137 (2)
N4—H4···O1^{iii}	0.78 (3)	2.29 (3)	3.035 (3)	161 (3)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Pd(C_{10}H_{24}N_4)]I_2·H_2O$
M_r	578.55
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	223
a, b, c (Å)	9.3993 (4), 13.7266 (5), 13.8797 (6)
β (°)	93.9017 (14)
V (Å^3)	1786.61 (13)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	4.49
Crystal size (mm)	0.23 × 0.18 × 0.11
Data collection	
Diffractometer	PHOTON 100 CMOS detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.595, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	48072, 3544, 3307
R_{int}	0.043
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.619
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.017, 0.041, 1.12
No. of reflections	3544
No. of parameters	188
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.49, −0.72

Computer programs: *APEX2* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014/7* (Sheldrick, 2015*b*) and *ORTEP-3 for Windows* (Farrugia, 2012).

Acknowledgements

The author thanks the KBSI, Seoul Center, for the X-ray data collection.

Funding information

This study was supported financially by Chonnam National University (grant No. 2018–3317).

References

Bruker (2016). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Hunter, T. M., Paisey, S. J., Park, H., Cleghorn, L., Parkin, A., Parsons, S. & Sadler, P. J. (2004). *J. Inorg. Biochem.* **98**, 713–719.

Liang, X., Parkinson, J. A., Weishäupl, M., Gould, R. O., Paisey, S. J., Park, H., Hunter, T. M., Blindauer, C. A., Parsons, S. & Sadler, P. J. (2002). *J. Am. Chem. Soc.* **124**, 9105–9112.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

full crystallographic data

IUCrData (2019). **4**, x191032 [https://doi.org/10.1107/S2414314619010320]

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Crystal data

$[Pd(C_{10}H_{24}N_4)]I_2·H_2O$
M_r = 578.55
Monoclinic, $P2_1/n$
a = 9.3993 (4) Å
b = 13.7266 (5) Å
c = 13.8797 (6) Å
β = 93.9017 (14)°
V = 1786.61 (13) Å^3
Z = 4
$F(000)$ = 1096
D_x = 2.151 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9932 reflections
θ = 2.5–28.3°
μ = 4.49 mm^{-1}
T = 223 K
Block, yellow
0.23 × 0.18 × 0.11 mm

Data collection

PHOTON 100 CMOS detector diffractometer
Radiation source: sealed tube
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.595, T_{max} = 0.745
48072 measured reflections
3544 independent reflections
3307 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 26.1°, θ_{min} = 2.1°
h = −11→11
k = −16→16
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.017
$wR(F^2)$ = 0.041
S = 1.12
3544 reflections
188 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0147P)^2 + 1.4653P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}
Extinction correction: SHELXL2014/7 (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00940 (16)

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.

Refinement. Hydrogen atoms on C atoms were positioned geometrically and allowed to ride on their respective parent atoms: C—H = 0.98 Å and U_{iso}(H) = 1.2U_{eq}(C). Hydrogen atoms bonded to N and O atoms were located from Fourier difference maps and refined isotropically.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Pd1	0.25793 (2)	0.38024 (2)	0.72122 (2)	0.02194 (6)
I1	0.74413 (2)	0.36724 (2)	0.35324 (2)	0.04099 (7)
I2	0.19189 (2)	0.37008 (2)	0.21133 (2)	0.03941 (6)
N1	0.0680 (2)	0.42530 (15)	0.76680 (16)	0.0329 (4)
H1	0.068 (3)	0.482 (2)	0.7613 (18)	0.029 (7)*
N2	0.1427 (3)	0.34020 (17)	0.59789 (16)	0.0402 (5)
H2	0.123 (3)	0.286 (2)	0.6050 (18)	0.029 (7)*
N3	0.4495 (2)	0.33712 (17)	0.6762 (2)	0.0458 (6)
H3	0.456 (3)	0.279 (2)	0.687 (2)	0.042 (8)*
N4	0.3719 (3)	0.42382 (16)	0.84436 (18)	0.0449 (6)
H4	0.381 (3)	0.480 (2)	0.837 (2)	0.048 (9)*
C1	−0.0451 (3)	0.3835 (2)	0.6988 (3)	0.0572 (9)
H1A	−0.0622	0.3153	0.7154	0.069*
H1B	−0.1342	0.4197	0.7033	0.069*
C2	0.0026 (3)	0.3900 (2)	0.5994 (3)	0.0578 (9)
H2A	0.0117	0.4584	0.5806	0.069*
H2B	−0.0672	0.3586	0.5538	0.069*
C3	0.2129 (5)	0.3580 (2)	0.5075 (2)	0.0687 (11)
H3A	0.2255	0.4283	0.4990	0.082*
H3B	0.1516	0.3339	0.4527	0.082*
C4	0.3572 (5)	0.3081 (3)	0.5081 (3)	0.0863 (15)
H4A	0.3460	0.2400	0.5274	0.104*
H4B	0.3886	0.3083	0.4422	0.104*
C5	0.4711 (5)	0.3539 (3)	0.5739 (3)	0.0800 (14)
H5A	0.5640	0.3274	0.5592	0.096*
H5B	0.4727	0.4242	0.5617	0.096*
C6	0.5594 (3)	0.3852 (2)	0.7434 (4)	0.0789 (14)
H6A	0.6518	0.3529	0.7393	0.095*
H6B	0.5699	0.4537	0.7254	0.095*
C7	0.5134 (4)	0.3782 (2)	0.8436 (3)	0.0702 (12)
H7A	0.5085	0.3098	0.8634	0.084*
H7B	0.5815	0.4121	0.8885	0.084*
C8	0.3033 (4)	0.4087 (2)	0.9361 (2)	0.0646 (10)
H8A	0.3638	0.4370	0.9892	0.078*
H8B	0.2955	0.3386	0.9481	0.078*
C9	0.1575 (5)	0.4538 (2)	0.9350 (2)	0.0707 (11)
H9A	0.1256	0.4521	1.0008	0.085*
H9B	0.1648	0.5224	0.9163	0.085*
C10	0.0464 (4)	0.4057 (2)	0.8686 (2)	0.0572 (9)
H10A	0.0492	0.3352	0.8797	0.069*
H10B	−0.0481	0.4292	0.8834	0.069*

O1	0.5464 (3)	0.36267 (15)	0.13842 (18)	0.0472 (5)
H1O	0.478 (5)	0.365 (3)	0.144 (3)	0.061 (15)*
H2O	0.587 (5)	0.356 (3)	0.189 (3)	0.077 (14)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.02275 (9)	0.02002 (9)	0.02342 (9)	−0.00007 (6)	0.00437 (6)	0.00080 (6)
I1	0.04594 (11)	0.03372 (10)	0.04233 (11)	−0.00799 (7)	−0.00412 (7)	0.00678 (7)
I2	0.04463 (11)	0.03331 (10)	0.04076 (11)	0.00051 (6)	0.00623 (7)	0.00246 (6)
N1	0.0341 (11)	0.0208 (10)	0.0455 (12)	0.0008 (8)	0.0143 (9)	−0.0006 (8)
N2	0.0591 (15)	0.0290 (11)	0.0312 (11)	0.0031 (10)	−0.0070 (10)	−0.0049 (9)
N3	0.0349 (12)	0.0260 (11)	0.0794 (18)	0.0015 (9)	0.0249 (11)	0.0021 (11)
N4	0.0533 (14)	0.0213 (10)	0.0564 (15)	0.0045 (9)	−0.0226 (11)	−0.0058 (10)
C1	0.0233 (13)	0.0366 (14)	0.112 (3)	−0.0024 (10)	0.0032 (15)	−0.0162 (16)
C2	0.0521 (18)	0.0468 (16)	0.069 (2)	0.0100 (13)	−0.0336 (16)	−0.0153 (15)
C3	0.133 (3)	0.0510 (17)	0.0224 (13)	0.0114 (19)	0.0033 (17)	−0.0005 (12)
C4	0.159 (4)	0.059 (2)	0.048 (2)	0.025 (3)	0.065 (3)	0.0053 (17)
C5	0.087 (3)	0.058 (2)	0.103 (3)	0.0159 (19)	0.074 (3)	0.016 (2)
C6	0.0208 (13)	0.0350 (15)	0.181 (5)	−0.0028 (11)	0.007 (2)	−0.017 (2)
C7	0.0479 (18)	0.0402 (17)	0.116 (3)	0.0065 (13)	−0.041 (2)	−0.0198 (18)
C8	0.123 (3)	0.0407 (15)	0.0269 (14)	0.0178 (18)	−0.0203 (17)	−0.0052 (12)
C9	0.138 (3)	0.0493 (18)	0.0264 (14)	0.036 (2)	0.0204 (18)	0.0019 (13)
C10	0.077 (2)	0.0404 (15)	0.0597 (19)	0.0200 (15)	0.0493 (17)	0.0140 (14)
O1	0.0459 (14)	0.0437 (12)	0.0510 (14)	−0.0005 (10)	−0.0045 (11)	0.0059 (9)

Geometric parameters (Å, º)

Pd1—N1	2.0307 (19)	C3—H3A	0.9800
Pd1—N3	2.034 (2)	C3—H3B	0.9800
Pd1—N2	2.038 (2)	C4—C5	1.498 (6)
Pd1—N4	2.044 (2)	C4—H4A	0.9800
N1—C10	1.466 (3)	C4—H4B	0.9800
N1—C1	1.487 (4)	C5—H5A	0.9800
N1—H1	0.78 (3)	C5—H5B	0.9800
N2—C3	1.477 (4)	C6—C7	1.487 (6)
N2—C2	1.485 (4)	C6—H6A	0.9800
N2—H2	0.78 (3)	C6—H6B	0.9800
N3—C5	1.467 (5)	C7—H7A	0.9800
N3—C6	1.497 (5)	C7—H7B	0.9800
N3—H3	0.82 (3)	C8—C9	1.503 (5)
N4—C7	1.471 (4)	C8—H8A	0.9800
N4—C8	1.480 (4)	C8—H8B	0.9800
N4—H4	0.78 (3)	C9—C10	1.499 (5)
C1—C2	1.482 (5)	C9—H9A	0.9800
C1—H1A	0.9800	C9—H9B	0.9800
C1—H1B	0.9800	C10—H10A	0.9800
C2—H2A	0.9800	C10—H10B	0.9800

C2—H2B	0.9800	O1—H1O	0.65 (4)
C3—C4	1.518 (6)	O1—H2O	0.79 (4)
N1—Pd1—N3	179.11 (9)	C4—C3—H3B	109.3
N1—Pd1—N2	85.15 (9)	H3A—C3—H3B	107.9
N3—Pd1—N2	95.45 (10)	C5—C4—C3	114.5 (3)
N1—Pd1—N4	94.14 (10)	C5—C4—H4A	108.6
N3—Pd1—N4	85.24 (11)	C3—C4—H4A	108.6
N2—Pd1—N4	178.61 (9)	C5—C4—H4B	108.6
C10—N1—C1	113.6 (3)	C3—C4—H4B	108.6
C10—N1—Pd1	115.25 (18)	H4A—C4—H4B	107.6
C1—N1—Pd1	106.87 (16)	N3—C5—C4	112.6 (3)
C10—N1—H1	106.2 (18)	N3—C5—H5A	109.1
C1—N1—H1	109.3 (19)	C4—C5—H5A	109.1
Pd1—N1—H1	105.2 (19)	N3—C5—H5B	109.1
C3—N2—C2	112.6 (3)	C4—C5—H5B	109.1
C3—N2—Pd1	115.2 (2)	H5A—C5—H5B	107.8
C2—N2—Pd1	106.56 (17)	C7—C6—N3	108.8 (3)
C3—N2—H2	113.1 (19)	C7—C6—H6A	109.9
C2—N2—H2	103 (2)	N3—C6—H6A	109.9
Pd1—N2—H2	105.7 (19)	C7—C6—H6B	109.9
C5—N3—C6	113.5 (3)	N3—C6—H6B	109.9
C5—N3—Pd1	115.8 (2)	H6A—C6—H6B	108.3
C6—N3—Pd1	105.6 (2)	N4—C7—C6	107.5 (3)
C5—N3—H3	109 (2)	N4—C7—H7A	110.2
C6—N3—H3	106 (2)	C6—C7—H7A	110.2
Pd1—N3—H3	106 (2)	N4—C7—H7B	110.2
C7—N4—C8	113.2 (3)	C6—C7—H7B	110.2
C7—N4—Pd1	107.0 (2)	H7A—C7—H7B	108.5
C8—N4—Pd1	116.4 (2)	N4—C8—C9	112.6 (2)
C7—N4—H4	108 (2)	N4—C8—H8A	109.1
C8—N4—H4	108 (2)	C9—C8—H8A	109.1
Pd1—N4—H4	104 (2)	N4—C8—H8B	109.1
C2—C1—N1	108.6 (2)	C9—C8—H8B	109.1
C2—C1—H1A	110.0	H8A—C8—H8B	107.8
N1—C1—H1A	110.0	C10—C9—C8	114.9 (2)
C2—C1—H1B	110.0	C10—C9—H9A	108.5
N1—C1—H1B	110.0	C8—C9—H9A	108.5
H1A—C1—H1B	108.4	C10—C9—H9B	108.5
C1—C2—N2	108.1 (2)	C8—C9—H9B	108.5
C1—C2—H2A	110.1	H9A—C9—H9B	107.5
N2—C2—H2A	110.1	N1—C10—C9	112.1 (3)
C1—C2—H2B	110.1	N1—C10—H10A	109.2
N2—C2—H2B	110.1	C9—C10—H10A	109.2
H2A—C2—H2B	108.4	N1—C10—H10B	109.2
N2—C3—C4	111.8 (3)	C9—C10—H10B	109.2
N2—C3—H3A	109.3	H10A—C10—H10B	107.9
C4—C3—H3A	109.3	H1O—O1—H2O	109 (5)

N2—C3—H3B	109.3		
C10—N1—C1—C2	−168.5 (2)	C5—N3—C6—C7	170.2 (3)
Pd1—N1—C1—C2	−40.3 (2)	Pd1—N3—C6—C7	42.3 (3)
N1—C1—C2—N2	55.7 (3)	C8—N4—C7—C6	171.1 (2)
C3—N2—C2—C1	−169.2 (3)	Pd1—N4—C7—C6	41.7 (3)
Pd1—N2—C2—C1	−42.0 (3)	N3—C6—C7—N4	−57.1 (3)
C2—N2—C3—C4	178.9 (3)	C7—N4—C8—C9	−178.7 (3)
Pd1—N2—C3—C4	56.5 (3)	Pd1—N4—C8—C9	−54.2 (3)
N2—C3—C4—C5	−72.1 (4)	N4—C8—C9—C10	68.8 (4)
C6—N3—C5—C4	−178.0 (3)	C1—N1—C10—C9	−176.7 (2)
Pd1—N3—C5—C4	−55.7 (3)	Pd1—N1—C10—C9	59.6 (3)
C3—C4—C5—N3	71.6 (4)	C8—C9—C10—N1	−72.0 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H2*O*···I1	0.79 (4)	2.63 (5)	3.405 (3)	169 (4)
O1—H1*O*···I2	0.65 (4)	2.91 (4)	3.549 (3)	168 (5)
N2—H2···O1^{i}	0.78 (3)	2.22 (3)	2.993 (3)	173 (3)
N3—H3···I2^{ii}	0.82 (3)	3.02 (3)	3.656 (2)	137 (2)
N4—H4···O1^{iii}	0.78 (3)	2.29 (3)	3.035 (3)	161 (3)

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