IUCrData

ISSN 2414-3146

N,*N*′-Bis(pyrazin-2-ylmethyl)pyrazine-2,3-dicarboxamide

Zhaodong Wang,* Yanshuang Yang and Huanzhu Lin

Chongqing Key Laboratory of Environmental Materials & Remediation Technologies, Chongqing University of Arts and Sciences, Yongchuan, Chongqing 402160, People's Republic of China. *Correspondence e-mail: ouwzdong@qq.com

Received 5 October 2016
Accepted 8 October 2016

Edited by J. Simpson, University of Otago, New Zealand

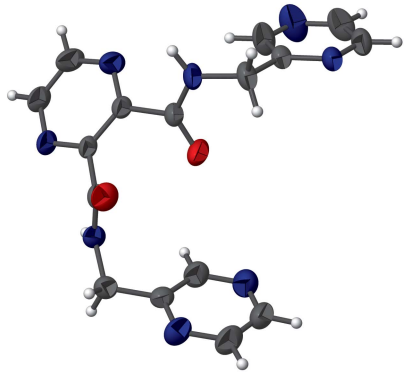
Keywords: crystal structure; pyrazine-based ligand; amide ligand; hydrogen bonds.

CCDC reference: 1504885

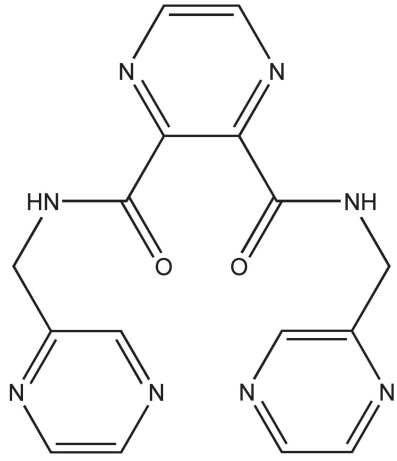
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{16}H_{14}N_8O_2$, was prepared by a condensation reaction between the dimethyl ester of pyrazine-2,3-dicarboxylic acid and an excess of 2-(aminomethyl)pyrazine. The molecule is approximately V-shaped with the central pyrazine ring joined through amide linkages to two pyrazin-2-ylmethyl substituents. In the crystal, molecules are linked by N—H···O, N—H···N and C—H···O hydrogen bonds, forming a three-dimensional framework.

3D view



Chemical scheme



Structure description

Pyrazine-based amide ligands have played a very important role in coordination chemistry (Cati *et al.*, 2004; Cati & Stoeckli-Evans, 2004*a*,*b*; Klingele *et al.*, 2007). The crystal structure of a symmetrical diamide ligand (Cati & Stoeckli-Evans, 2004*c*) and its bi- and tetranuclear copper(II) complexes have also been reported (Cati & Stoeckli-Evans, 2004*d*; Hausmann *et al.*, 2003; Hausmann & Brooker 2004). In order to expand the research scope of this pyrazine-2,3-bisamide ligand system, we have prepared the title compound to investigate its potential use as a ligand in transition metal chemistry.

There are three pyrazine rings in the title compound (Fig. 1). The outer pair are linked to the central pyrazine ring by two CH_2–NH–C(O)–amide units, forming a V-shaped structure. The two outer pyrazine rings are inclined to the central ring by 48.98 (8) and 47.87 (8)°. In the crystal, N—H···O, N—H···N and C—H···O hydrogen bonds generate a three-dimensional framework (Table 1 Fig. 2). The coordination modes of the ligand system could be more flexible than those of previously reported pyrazine-based ligands due to the N atoms of the outer pyrazine rings. These could be used in a bridging sense with the potential to construct metal-organic frameworks.

OPEN ACCESS

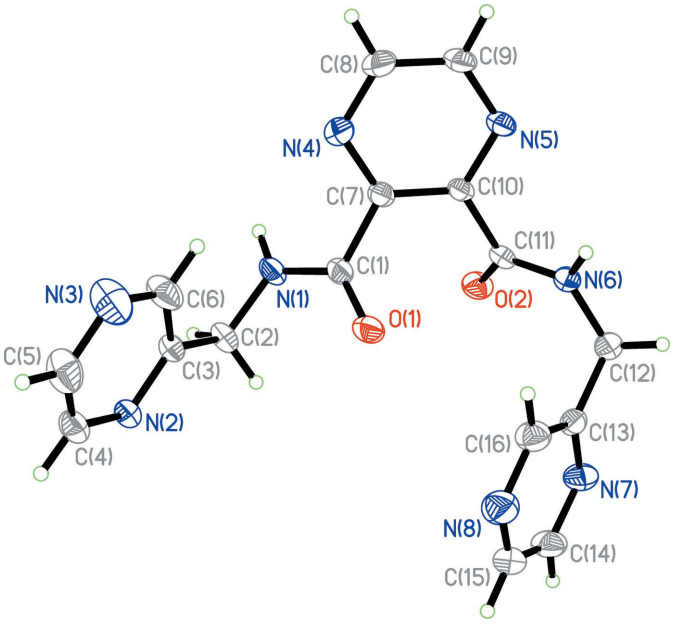


Figure 1
The stucture of the title compound, showing the atom numbering with ellipsoids drawn at the 30% probability level

Synthesis and crystallization

All reagents and solvents for the synthesis were purchased from commercial sources and used as received without further purification. The title compound was prepared by the condensation reaction of the dimethyl ester pyrazine-2,3-dicarboxylate (3.92g, 20mmol) with 2-aminomethylpyrazine (2.18g, 20mmol) under reflux in 15mL of methanol for 12h. Solvent was removed by rotary evaporation to give a light-yellow solid. Recrystallization from CH_3OH solution gave colorless crystals suitable for X-ray analysis.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

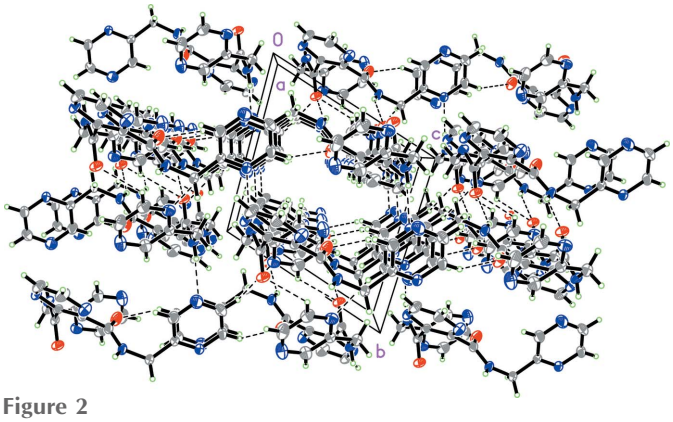


Figure 2
Crystal packing viewed along the *a* axis direction with hydrogen bonds shown as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.86	2.26	2.9940 (18)	144
N6—H6···N2^{ii}	0.86	2.06	2.914 (2)	174
C4—H4···O1^{iii}	0.93	2.52	3.369 (2)	152
C5—H5···N7^{iv}	0.93	2.46	3.359 (3)	162
C14—H14···O2^{v}	0.93	2.56	3.236 (2)	130

Symmetry codes: (i) $-x+1, -y+2, -z+2$; (ii) $x+1, y, z$; (iii) $-x, -y+1, -z+1$; (iv) $x-1, y-1, z$; (v) $-x+1, -y+2, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{14}N_8O_2$
M_r	350.35
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, b, c (Å)	9.1166 (18), 10.022 (2), 10.781 (2)
α, β, γ (°)	116.951 (2), 97.130 (2), 102.195 (2)
V (Å^3)	830.5 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.22 × 0.20 × 0.20
Data collection	
Diffractometer	Bruker SMART APEXII CCD area-detector
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3527, 3527, 2595
R_{int}	0.016
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.653
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.041, 0.132, 1.05
No. of reflections	3527
No. of parameters	235
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.15, −0.17

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

Acknowledgements

The authors would like to thank the Talent Introduction Project of Chongqing University of Arts and Sciences (No. R2012CH12) and the Science and Technology Research Project of Chongqing Municipal Education Committee (No. KJ131215) for financial support.

References

Bruker (2013). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cati, D. S., Ribas, J., Ribas-Ariño, J. & Stoeckli-Evans, H. (2004). *Inorg. Chem.* **43**, 1021–1030.
Cati, D. S. & Stoeckli-Evans, H. (2004*a*). *Acta Cryst.* E**60**, m174–m176.
Cati, D. S. & Stoeckli-Evans, H. (2004*b*). *Acta Cryst.* E**60**, m177–m179.
Cati, D. S. & Stoeckli-Evans, H. (2004*c*). *Acta Cryst.* E**60**, o210–o212.
Cati, D. S. & Stoeckli-Evans, H. (2004*d*). *Acta Cryst.* E**60**, m180–m182.

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Hausmann, J. & Brooker, S. (2004). *Chem. Commun.* pp. 1531–1531.
Hausmann, J., Jameson, G. B. & Brooker, S. (2003). *Chem. Commun.* pp. 2992–2993.
Klingele (née Hausmann), J., Boas, J. F., Pilbrow, J. R., Moubaraki, B., Murray, K. S., Berry, K. J., Hunter, K. A., Jameson, G. B., Boyd, P. D. W. & Brooker, S. (2007). *Dalton Trans.* pp. 633–645.
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 (2016). **1**, x161588 [https://doi.org/10.1107/S2414314616015881]

N,*N*′-Bis(pyrazin-2-ylmethyl)pyrazine-2,3-dicarboxamide

Zhaodong Wang, Yanshuang Yang and Huanzhu Lin

N,*N*′-Bis(pyrazin-2-ylmethyl)pyrazine-2,3-dicarboxamide

Crystal data

$C_{16}H_{14}N_8O_2$
$M_r = 350.35$
Triclinic, $P\overline{1}$
$a = 9.1166$ (18) Å
$b = 10.022$ (2) Å
$c = 10.781$ (2) Å
$\alpha = 116.951$ (2)°
$\beta = 97.130$ (2)°
$\gamma = 102.195$ (2)°
$V = 830.5$ (3) Å^3
$Z = 2$
$F(000) = 364$
$D_x = 1.401$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2164 reflections
$\theta = 2.3$–$26.7°$
$\mu = 0.10$ mm^{-1}
$T = 296$ K
Block, colourless
0.22 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine focus sealed tube
Graphite monochromator
φ and ω scans
3527 measured reflections
3527 independent reflections
2595 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 27.7°$, $\theta_{min} = 2.2°$
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 11$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.132$
$S = 1.05$
3527 reflections
235 parameters
0 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0759P)^2 + 0.0242P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.44574 (13)	0.72355 (16)	0.68709 (12)	0.0573 (4)
O2	0.72074 (13)	1.03109 (13)	0.82972 (12)	0.0489 (3)
N1	0.35307 (13)	0.81613 (15)	0.88449 (13)	0.0423 (3)
H1	0.3740	0.8582	0.9766	0.051*
N2	−0.04468 (13)	0.59908 (14)	0.66043 (13)	0.0385 (3)
N3	0.0331 (2)	0.3695 (2)	0.7075 (2)	0.0799 (6)
N4	0.59054 (16)	0.72344 (19)	0.99377 (15)	0.0525 (4)
N5	0.88003 (14)	0.81968 (16)	0.94792 (13)	0.0432 (3)
N6	0.84472 (14)	0.86414 (16)	0.69834 (13)	0.0398 (3)
H6	0.8840	0.7905	0.6898	0.048*
N7	0.68896 (18)	0.96134 (17)	0.43602 (15)	0.0522 (4)
N8	0.48052 (19)	0.6643 (2)	0.32238 (17)	0.0695 (5)
C1	0.46175 (16)	0.77287 (18)	0.81589 (15)	0.0379 (4)
C2	0.20077 (16)	0.79296 (18)	0.80565 (17)	0.0424 (4)
H2A	0.2119	0.8171	0.7291	0.051*
H2B	0.1541	0.8659	0.8699	0.051*
C3	0.09378 (16)	0.62856 (17)	0.74138 (15)	0.0360 (3)
C4	−0.14353 (18)	0.45555 (19)	0.60466 (18)	0.0490 (4)
H4	−0.2414	0.4312	0.5482	0.059*
C5	−0.1042 (2)	0.3430 (2)	0.6286 (2)	0.0665 (6)
H5	−0.1766	0.2443	0.5879	0.080*
C6	0.1310 (2)	0.5138 (2)	0.7640 (2)	0.0610 (5)
H6A	0.2285	0.5379	0.8211	0.073*
C7	0.60761 (16)	0.78146 (17)	0.90534 (15)	0.0363 (3)
C8	0.7197 (2)	0.7138 (2)	1.0583 (2)	0.0603 (5)
H8	0.7136	0.6766	1.1230	0.072*
C9	0.8614 (2)	0.7568 (2)	1.03266 (18)	0.0517 (4)
H9	0.9465	0.7416	1.0757	0.062*
C10	0.75270 (16)	0.83414 (17)	0.88594 (14)	0.0334 (3)
C11	0.77139 (16)	0.91900 (18)	0.80107 (15)	0.0358 (3)
C12	0.86012 (18)	0.9254 (2)	0.60010 (17)	0.0454 (4)
H12A	0.9436	0.8978	0.5560	0.054*
H12B	0.8884	1.0391	0.6544	0.054*
C13	0.71396 (17)	0.86430 (18)	0.48330 (15)	0.0397 (4)
C14	0.5594 (2)	0.9103 (2)	0.33497 (19)	0.0607 (5)
H14	0.5376	0.9763	0.3013	0.073*
C15	0.4576 (2)	0.7646 (3)	0.2793 (2)	0.0645 (6)
H15	0.3686	0.7342	0.2084	0.077*
C16	0.6093 (2)	0.7170 (2)	0.42593 (19)	0.0561 (5)
H16	0.6294	0.6515	0.4609	0.067*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0428 (7)	0.0868 (9)	0.0372 (6)	0.0231 (6)	0.0054 (5)	0.0262 (6)

O2	0.0534 (7)	0.0522 (7)	0.0530 (7)	0.0304 (6)	0.0143 (5)	0.0292 (6)
N1	0.0275 (6)	0.0489 (8)	0.0373 (7)	0.0097 (6)	0.0023 (5)	0.0130 (6)
N2	0.0311 (6)	0.0396 (7)	0.0400 (7)	0.0083 (6)	0.0054 (5)	0.0178 (6)
N3	0.0610 (11)	0.0556 (10)	0.1300 (16)	0.0145 (9)	0.0086 (11)	0.0567 (11)
N4	0.0498 (8)	0.0739 (10)	0.0550 (9)	0.0270 (7)	0.0203 (7)	0.0436 (8)
N5	0.0369 (7)	0.0564 (8)	0.0423 (7)	0.0211 (6)	0.0057 (6)	0.0273 (7)
N6	0.0383 (7)	0.0510 (8)	0.0449 (7)	0.0226 (6)	0.0131 (6)	0.0311 (6)
N7	0.0629 (9)	0.0526 (9)	0.0490 (8)	0.0239 (7)	0.0090 (7)	0.0301 (7)
N8	0.0603 (10)	0.0770 (12)	0.0580 (10)	−0.0004 (9)	0.0033 (8)	0.0346 (9)
C1	0.0301 (8)	0.0415 (8)	0.0363 (8)	0.0077 (6)	0.0034 (6)	0.0170 (7)
C2	0.0281 (8)	0.0388 (8)	0.0516 (9)	0.0095 (6)	0.0037 (6)	0.0171 (7)
C3	0.0297 (7)	0.0378 (8)	0.0404 (8)	0.0123 (6)	0.0079 (6)	0.0186 (7)
C4	0.0351 (9)	0.0474 (10)	0.0492 (9)	0.0037 (7)	0.0023 (7)	0.0173 (8)
C5	0.0553 (12)	0.0404 (10)	0.0930 (15)	0.0037 (9)	0.0122 (11)	0.0304 (10)
C6	0.0423 (10)	0.0547 (11)	0.0892 (14)	0.0126 (8)	0.0000 (9)	0.0432 (11)
C7	0.0344 (8)	0.0415 (8)	0.0331 (7)	0.0147 (6)	0.0068 (6)	0.0176 (6)
C8	0.0671 (12)	0.0868 (14)	0.0607 (11)	0.0385 (11)	0.0240 (9)	0.0551 (11)
C9	0.0493 (10)	0.0729 (12)	0.0515 (10)	0.0335 (9)	0.0106 (8)	0.0400 (9)
C10	0.0306 (7)	0.0388 (8)	0.0303 (7)	0.0151 (6)	0.0042 (5)	0.0156 (6)
C11	0.0275 (7)	0.0416 (8)	0.0381 (8)	0.0122 (6)	0.0018 (6)	0.0203 (7)
C12	0.0424 (9)	0.0545 (10)	0.0485 (9)	0.0144 (8)	0.0123 (7)	0.0329 (8)
C13	0.0430 (9)	0.0454 (9)	0.0382 (8)	0.0173 (7)	0.0146 (7)	0.0241 (7)
C14	0.0647 (12)	0.0776 (14)	0.0540 (11)	0.0323 (11)	0.0099 (9)	0.0407 (10)
C15	0.0495 (11)	0.0973 (17)	0.0501 (11)	0.0189 (11)	0.0059 (8)	0.0422 (11)
C16	0.0582 (11)	0.0573 (11)	0.0551 (10)	0.0100 (9)	0.0067 (9)	0.0348 (9)

Geometric parameters (Å, º)

O1—C1	1.2210 (17)	C2—C3	1.505 (2)
O2—C11	1.2291 (18)	C2—H2A	0.9700
N1—C1	1.3376 (19)	C2—H2B	0.9700
N1—C2	1.4479 (18)	C3—C6	1.377 (2)
N1—H1	0.8599	C4—C5	1.372 (3)
N2—C4	1.3328 (19)	C4—H4	0.9300
N2—C3	1.3333 (18)	C5—H5	0.9300
N3—C5	1.323 (3)	C6—H6A	0.9300
N3—C6	1.335 (2)	C7—C10	1.395 (2)
N4—C7	1.3312 (19)	C8—C9	1.375 (3)
N4—C8	1.336 (2)	C8—H8	0.9300
N5—C9	1.3327 (19)	C9—H9	0.9300
N5—C10	1.3347 (18)	C10—C11	1.507 (2)
N6—C11	1.3278 (19)	C12—C13	1.509 (2)
N6—C12	1.4523 (18)	C12—H12A	0.9700
N6—H6	0.8601	C12—H12B	0.9700
N7—C14	1.328 (2)	C13—C16	1.379 (2)
N7—C13	1.3311 (19)	C14—C15	1.359 (3)
N8—C15	1.326 (3)	C14—H14	0.9300
N8—C16	1.335 (2)	C15—H15	0.9300

C1—C7	1.5046 (19)	C16—H16	0.9300
C1—N1—C2	120.84 (13)	N4—C7—C10	121.72 (14)
C1—N1—H1	119.6	N4—C7—C1	116.92 (13)
C2—N1—H1	119.6	C10—C7—C1	121.09 (13)
C4—N2—C3	116.98 (14)	N4—C8—C9	122.50 (15)
C5—N3—C6	115.74 (17)	N4—C8—H8	118.8
C7—N4—C8	115.88 (14)	C9—C8—H8	118.7
C9—N5—C10	116.27 (13)	N5—C9—C8	121.79 (15)
C11—N6—C12	121.60 (13)	N5—C9—H9	119.1
C11—N6—H6	119.2	C8—C9—H9	119.1
C12—N6—H6	119.2	N5—C10—C7	121.65 (13)
C14—N7—C13	116.96 (15)	N5—C10—C11	117.86 (12)
C15—N8—C16	115.45 (17)	C7—C10—C11	120.38 (12)
O1—C1—N1	123.67 (14)	O2—C11—N6	124.70 (14)
O1—C1—C7	119.96 (14)	O2—C11—C10	119.93 (13)
N1—C1—C7	116.32 (13)	N6—C11—C10	115.38 (13)
N1—C2—C3	113.38 (13)	N6—C12—C13	113.24 (12)
N1—C2—H2A	108.9	N6—C12—H12A	108.9
C3—C2—H2A	108.9	C13—C12—H12A	108.9
N1—C2—H2B	108.9	N6—C12—H12B	108.9
C3—C2—H2B	108.9	C13—C12—H12B	108.9
H2A—C2—H2B	107.7	H12A—C12—H12B	107.7
N2—C3—C6	120.59 (14)	N7—C13—C16	120.23 (15)
N2—C3—C2	115.50 (13)	N7—C13—C12	116.23 (14)
C6—C3—C2	123.90 (14)	C16—C13—C12	123.54 (15)
N2—C4—C5	121.50 (16)	N7—C14—C15	122.10 (17)
N2—C4—H4	119.3	N7—C14—H14	119.0
C5—C4—H4	119.3	C15—C14—H14	119.0
N3—C5—C4	122.44 (17)	N8—C15—C14	122.35 (17)
N3—C5—H5	118.8	N8—C15—H15	118.8
C4—C5—H5	118.8	C14—C15—H15	118.8
N3—C6—C3	122.74 (17)	N8—C16—C13	122.89 (17)
N3—C6—H6A	118.6	N8—C16—H16	118.6
C3—C6—H6A	118.6	C13—C16—H16	118.6
C2—N1—C1—O1	5.8 (2)	C9—N5—C10—C11	174.14 (14)
C2—N1—C1—C7	−171.79 (12)	N4—C7—C10—N5	4.4 (2)
C1—N1—C2—C3	81.57 (18)	C1—C7—C10—N5	−169.43 (14)
C4—N2—C3—C6	0.5 (2)	N4—C7—C10—C11	−171.71 (14)
C4—N2—C3—C2	−178.48 (14)	C1—C7—C10—C11	14.5 (2)
N1—C2—C3—N2	−176.08 (12)	C12—N6—C11—O2	−5.5 (2)
N1—C2—C3—C6	5.0 (2)	C12—N6—C11—C10	174.84 (12)
C3—N2—C4—C5	−0.5 (2)	N5—C10—C11—O2	−125.27 (15)
C6—N3—C5—C4	0.7 (3)	C7—C10—C11—O2	50.96 (19)
N2—C4—C5—N3	−0.1 (3)	N5—C10—C11—N6	54.42 (18)
C5—N3—C6—C3	−0.6 (3)	C7—C10—C11—N6	−129.35 (15)
N2—C3—C6—N3	0.1 (3)	C11—N6—C12—C13	−77.41 (19)

C2—C3—C6—N3	178.94 (18)	C14—N7—C13—C16	1.2 (2)
C8—N4—C7—C10	−2.3 (2)	C14—N7—C13—C12	−178.84 (14)
C8—N4—C7—C1	171.75 (16)	N6—C12—C13—N7	148.37 (14)
O1—C1—C7—N4	−132.13 (17)	N6—C12—C13—C16	−31.7 (2)
N1—C1—C7—N4	45.6 (2)	C13—N7—C14—C15	−1.4 (3)
O1—C1—C7—C10	42.0 (2)	C16—N8—C15—C14	1.0 (3)
N1—C1—C7—C10	−140.35 (15)	N7—C14—C15—N8	0.3 (3)
C7—N4—C8—C9	−1.7 (3)	C15—N8—C16—C13	−1.2 (3)
C10—N5—C9—C8	−2.0 (3)	N7—C13—C16—N8	0.1 (3)
N4—C8—C9—N5	4.0 (3)	C12—C13—C16—N8	−179.88 (16)
C9—N5—C10—C7	−2.0 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.86	2.26	2.9940 (18)	144
N6—H6···N2^{ii}	0.86	2.06	2.914 (2)	174
C4—H4···O1^{iii}	0.93	2.52	3.369 (2)	152
C5—H5···N7^{iv}	0.93	2.46	3.359 (3)	162
C14—H14···O2^{v}	0.93	2.56	3.236 (2)	130

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