Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(−)-*N*,*N*′-Bis[(1*S*,2*R*,5*S*)-6,6-dimethylbicyclo[3.1.1]heptan-2-ylmethyl]pyridine-2,6-dicarboxamide monohydrate

Sylvain Bernès,[a]* Francisco Javier Pérez-Flores[b] and René Gutiérrez[b]

[a]DEP Facultad de Ciencias Químicas, UANL, Guerrero y Progreso S/N, Col. Treviño, 64570 Monterrey, NL, Mexico, and [b]Laboratorio de Síntesis de Complejos, Facultad de Ciencias Químicas, Universidad Autónoma de Puebla, A.P. 1067, 72001 Puebla, Pue., Mexico
Correspondence e-mail: sylvain_bernes@Hotmail.com

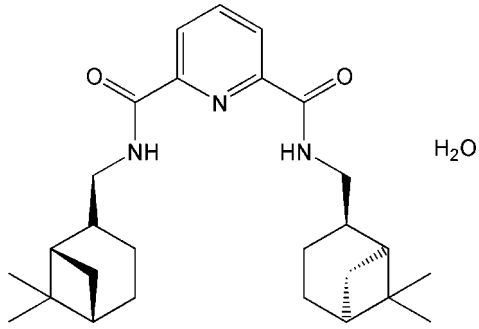
Received 16 April 2008; accepted 8 May 2008

Key indicators: single-crystal X-ray study; T = 298 K, P = 101 kPa; mean σ(C–C) = 0.004 Å; R factor = 0.040; wR factor = 0.107; data-to-parameter ratio = 10.0.

The title compound, $C_{27}H_{39}N_3O_2\cdot H_2O$, is a chiral pyridine-2,6-dicarboxamide derivative including *cis*-myrtanyl groups as amine substituents. The pyridine-2,6-dicarboxamide core approximates C_2 point symmetry and a solvent water molecule lies on the pseudo-twofold axis. The water molecule serves both as acceptor and donor for efficient hydrogen bonds involving N—H and C═O functional groups as donor and acceptor groups, respectively. As a result, each water molecule in the crystal structure is tetrahedrally bonded to three symmetry-related molecules, forming a three-dimensional supramolecular network. Such an arrangement is a common feature found in the majority of X-ray-characterized symmetrically substituted pyridine-2,6-dicarboxamide derivatives.

Related literature

For background to the solvent–free synthesis used for the preparation of the title compound, see: Tanaka & Toda (2000); Vázquez *et al.* (2004); Tovar *et al.* (2007); Pérez-Flores & Gutiérrez (2008). For hydrates of pyridine-2,6-dicarboxamide derivatives, see: Yu *et al.* (1999); Qi *et al.* (2002); Jain *et al.* (2004); Odriozola *et al.* (2004).



Experimental

Crystal data

$C_{27}H_{39}N_3O_2\cdot H_2O$
M_r = 455.63
Monoclinic, $P2_1$
a = 6.8476 (11) Å
b = 12.1101 (14) Å
c = 16.012 (2) Å
β = 91.173 (15)°
V = 1327.5 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 298 (1) K
0.6 × 0.6 × 0.2 mm

Data collection

Bruker P4 diffractometer
Absorption correction: none
6391 measured reflections
3180 independent reflections
2580 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
3 standard reflections
every 97 reflections
intensity decay: 2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.107
S = 1.04
3180 reflections
319 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O3	0.82 (2)	2.16 (2)	2.941 (3)	160 (2)
N3—H3···O3	0.86 (3)	2.19 (3)	3.017 (3)	159 (2)
O3—H31···O2^{i}	0.87 (5)	1.90 (5)	2.756 (3)	167 (4)
O3—H32···O1^{ii}	0.90 (4)	1.86 (5)	2.754 (3)	171 (3)

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

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *POV-RAY* (Cason, 2004); software used to prepare material for publication: *SHELXL97*.

Partial support from VIEP–UAP (14/G/NAT/05) is acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2088).

References

Cason, C. J. (2004). *POV-RAY for Windows*. Persistence of Vision Raytracer Pty Ltd, Victoria, Australia.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Jain, S. L., Bhattacharyya, P., Milton, H. L., Slawin, A. M. Z., Crayston, J. A. & Woollins, J. D. (2004). *J. Chem. Soc. Dalton Trans.*, pp. 862–871.

Odriozola, I., Kyritsakas, N. & Lehn, J.-M. (2004). *Chem. Commun.* pp. 62–63.

Pérez-Flores, F. J. & Gutiérrez, R. (2008). *Synthesis.* Submitted.

Qi, J. Y., Chen, J., Yang, Q. Y., Zhou, Z. Y. & Chan, A. S. C. (2002). *Acta Cryst.* E**58**, o1232–o1233.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Siemens (1996). *XSCANS*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Tanaka, K. & Toda, F. (2000). *Chem. Rev.* **100**, 1025–1074.

Tovar, A., Peña, U., Hernández, G., Portillo, R. & Gutiérrez, R. (2007). *Synthesis*, pp. 22–24.

Vázquez, J., Bernès, S., Reyes, Y., Moya, M., Sharma, P., Alvarez, C. & Gutiérrez, R. (2004). *Synthesis*, pp. 1955–1958.

Yu, Q., Baroni, T. E., Borovik, A. S., Liable-Sands, L., Yap, G. P. A. & Rheingold, A. L. (1999). *Chem. Commun.* pp. 1467–1468.

supporting information

Acta Cryst. (2008). E**64**, o1078–o1079 [doi:10.1107/S1600536808013652]

(−)-*N*,*N*′-Bis[(1*S*,2*R*,5*S*)-6,6-dimethyl-bicyclo[3.1.1]heptan-2-ylmethyl]-pyridine-2,6-dicarboxamide monohydrate

Sylvain Bernès, Francisco Javier Pérez-Flores and René Gutiérrez

S1. Comment

Nowadays, reactions conducted in the absence of solvents under mild reaction conditions are becoming an important method in laboratories worldwide as an environment–friendly technique for the efficient syntheses of organic molecules. The main advantages of solvent-free organic synthesis are shorter reaction times, minimum waste and generally higher yields, operational simplicity as well as reduction of thermal degradative byproducts along with cleaner work–up (Tanaka & Toda, 2000). As part of an ongoing program aiming to develop simpler and eco–friendly methods for organic transformations under solvent–free conditions (Tovar *et al.*, 2007; Vázquez *et al.*, 2004), we engaged the preparation of chiral pincer ligands (Pérez-Flores & Gutiérrez, 2008). The title compound resulted from this research, by introducing chiral *cis*–myrtanyl groups as amine substituents.

The X–ray characterized monohydrate has the expected molecular geometry (Fig. 1). The pyridine–2,6–dicarboxamide core approximates a C_2 point symmetry, with the pseudo 2–fold axis passing through N1 and C24. The guest water molecule O3 is placed on the pseudo 2–fold axis and is involved in two N—H···O hydrogen bonds within the asymmetric unit (Fig. 1 and Table 1, lines 1 and 2). The same water molecule is a donor group for two C═O···H intermolecular hydrogen bonds of relatively strong strength (Table 1, lines 3 and 4). As a consequence, a three–dimensional supramolecular structure is formed in the crystal structure, with water molecules being bonded in a tetrahedral arrangement (Fig. 2) to three symmetry–related molecules. Such a feature seems to be common for symmetrically substituted pyridine–2,6–dicarboxamide derivatives. These compounds are generally crystallized as hydrates, and, at least for X–ray characterized compounds, water molecules form hydrogen bonds similar to those observed in the title molecule (*e.g.* Yu *et al.*, 1999; Qi *et al.*, 2002; Jain *et al.*, 2004; Odriozola *et al.*, 2004).

S2. Experimental

Under solvent–free conditions, (-)-*cis*–myrtanylamine (0.38 g, 2.5 mmol) and 2,6–pyridinedicarbonyl dichloride (0.30 g, 1.5 mmol) were mixed at room temperature, giving a white solid. The crude was recrystallized from EtOH affording the corresponding dicarboxamide (98% yield).

S3. Refinement

C–bonded H atoms were placed in idealized positions and refined with a riding model approximation. Constrained C—H distances: 0.93 (aromatic CH), 0.96 (methyl CH_3), 0.97 (methylene CH_2) or 0.98 Å (methine CH). Isotropic displacement parameters: U_{iso}= 1.5U_{eq}(carrier C atom) for methyl groups and U_{iso} = 1.2U_{eq}(carrier C atom) otherwise. Methyl groups were considered as rigid rotating groups. Other H atoms (amine groups and water molecule) were found in a difference map and refined freely. Measured Friedel pairs (287) were merged.

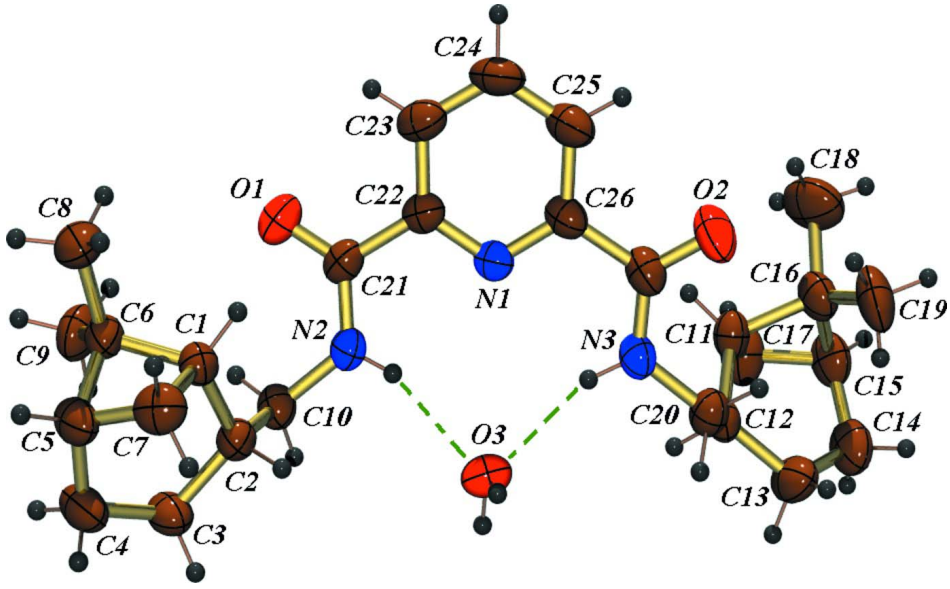


Figure 1

The asymmetric unit for the title compound, with the numbering scheme. Displacement ellipsoids for non–H atoms are drawn at the 40% probability level. H atoms are presented as small spheres of arbitrary radius. Dashed bonds are hydrogen bonds involving N—H groups as donor groups.

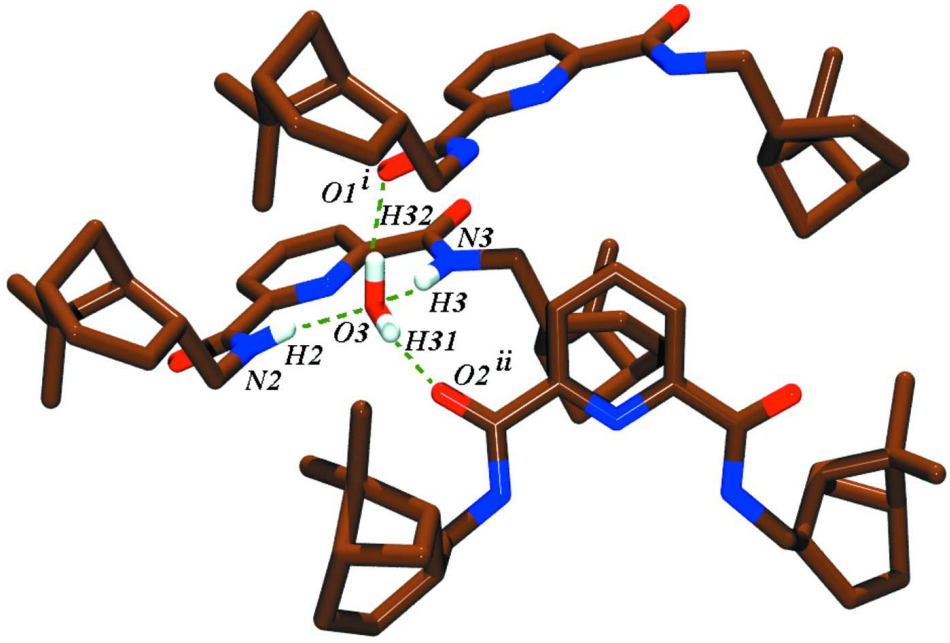


Figure 2

A part of the packing structure for the title compound, showing the four H bonds formed by a water molecule, in a tetrahedral geometry (dashed bonds). Symmetry codes: (*i*) 1+*x*, *y*, *z*; (*ii*) 2-*x*, 1/2+*y*, 2-*z*. H atoms not involved in hydrogen bonds have been omitted for clarity.

(-)-*N*,*N*′-Bis[(1*S*,2*R*,5*S*)-6,6-dimethyl- bicyclo[3.1.1]heptan-2-ylmethyl]pyridine-2,6-dicarboxamide monohydrate

Crystal data

$C_{27}H_{39}N_3O_2{\cdot}H_2O$
$M_r = 455.63$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 6.8476\ (11)$ Å
$b = 12.1101\ (14)$ Å
$c = 16.012\ (2)$ Å
$\beta = 91.173\ (15)°$
$V = 1327.5\ (3)$ Å^3
$Z = 2$
$F(000) = 496$
$D_x = 1.140$ Mg m^{-3}
Melting point = 398–401 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 60 reflections
$\theta = 4.6–12.5°$
$\mu = 0.07$ mm^{-1}
$T = 298$ K
Plate, colourless
0.6 × 0.6 × 0.2 mm

Data collection

Bruker P4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
6391 measured reflections
3180 independent reflections
2580 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.1°$
$h = -8\rightarrow6$
$k = -1\rightarrow15$
$l = -20\rightarrow20$
3 standard reflections every 97 reflections
intensity decay: 2%

Refinement

Refinement on F^2
Least-squares matrix: Full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.107$
$S = 1.04$
3180 reflections
319 parameters
1 restraint
0 constraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 0.0694P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.037 (7)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.6298 (3)	0.24506 (16)	0.95082 (10)	0.0520 (4)
N2	0.5268 (3)	0.37490 (17)	0.81930 (11)	0.0558 (5)
H2	0.636 (4)	0.374 (2)	0.8414 (14)	0.049 (6)*
N3	0.9690 (3)	0.29137 (18)	1.03447 (12)	0.0576 (5)
H3	0.926 (3)	0.330 (2)	0.9926 (17)	0.058 (7)*
O1	0.2258 (2)	0.29762 (18)	0.81492 (11)	0.0731 (5)
O2	0.9360 (4)	0.13912 (17)	1.11410 (13)	0.0893 (6)
C1	0.4749 (3)	0.30035 (19)	0.63787 (13)	0.0529 (5)
H1A	0.4642	0.2424	0.6803	0.063*
C2	0.5749 (3)	0.4058 (2)	0.66831 (13)	0.0556 (5)
H2A	0.7122	0.3869	0.6793	0.067*

C3	0.5733 (5)	0.4954 (2)	0.59935 (16)	0.0724 (7)
H3A	0.7040	0.5257	0.5955	0.087*
H3B	0.4877	0.5549	0.6162	0.087*
C4	0.5059 (6)	0.4548 (3)	0.51078 (18)	0.0866 (9)
H4A	0.4046	0.5037	0.4893	0.104*
H4B	0.6154	0.4589	0.4734	0.104*
C5	0.4292 (4)	0.3386 (2)	0.51132 (15)	0.0737 (7)
H5A	0.3843	0.3108	0.4567	0.088*
C6	0.2858 (3)	0.3192 (2)	0.58333 (14)	0.0598 (5)
C7	0.5754 (4)	0.2641 (2)	0.55738 (16)	0.0737 (7)
H7A	0.7103	0.2878	0.5536	0.088*
H7B	0.5613	0.1862	0.5445	0.088*
C8	0.1734 (5)	0.2103 (3)	0.5733 (2)	0.0834 (8)
H8A	0.0857	0.2152	0.5259	0.125*
H8B	0.1003	0.1964	0.6227	0.125*
H8C	0.2641	0.1510	0.5650	0.125*
C9	0.1402 (4)	0.4092 (3)	0.6038 (2)	0.0831 (8)
H9A	0.0386	0.4109	0.5617	0.125*
H9B	0.2057	0.4793	0.6057	0.125*
H9C	0.0843	0.3943	0.6572	0.125*
C10	0.4962 (4)	0.45157 (19)	0.74996 (14)	0.0597 (5)
H10A	0.5610	0.5209	0.7630	0.072*
H10B	0.3576	0.4663	0.7430	0.072*
C11	0.9333 (3)	0.3930 (2)	1.20404 (14)	0.0620 (6)
H11A	0.8147	0.3610	1.1788	0.074*
C12	1.0879 (3)	0.4242 (2)	1.14197 (14)	0.0604 (6)
H12A	1.0340	0.4855	1.1088	0.072*
C13	1.2723 (5)	0.4692 (3)	1.18754 (18)	0.0875 (9)
H13A	1.3080	0.5385	1.1616	0.105*
H13B	1.3784	0.4177	1.1788	0.105*
C14	1.2550 (5)	0.4890 (3)	1.28285 (19)	0.0920 (10)
H14A	1.3606	0.4507	1.3119	0.110*
H14B	1.2692	0.5673	1.2942	0.110*
C15	1.0623 (5)	0.4496 (3)	1.31641 (16)	0.0800 (8)
H15A	1.0455	0.4611	1.3764	0.096*
C16	1.0091 (4)	0.3322 (3)	1.28420 (15)	0.0737 (7)
C17	0.8967 (5)	0.4939 (3)	1.26053 (18)	0.0884 (9)
H17A	0.9245	0.5645	1.2346	0.106*
H17B	0.7699	0.4942	1.2865	0.106*
C18	0.8353 (7)	0.2817 (5)	1.3307 (2)	0.1257 (16)
H18A	0.8757	0.2633	1.3868	0.189*
H18B	0.7917	0.2162	1.3022	0.189*
H18C	0.7305	0.3343	1.3322	0.189*
C19	1.1675 (6)	0.2462 (3)	1.2809 (2)	0.0988 (11)
H19A	1.2788	0.2762	1.2533	0.148*
H19B	1.1201	0.1829	1.2505	0.148*
H19C	1.2047	0.2245	1.3366	0.148*
C20	1.1400 (3)	0.3334 (2)	1.08030 (14)	0.0637 (6)

H20A	1.2328	0.3622	1.0409	0.076*
H20B	1.2024	0.2731	1.1104	0.076*
C21	0.3935 (3)	0.3035 (2)	0.84428 (13)	0.0548 (5)
C22	0.4577 (3)	0.22560 (19)	0.91276 (13)	0.0540 (5)
C23	0.3379 (4)	0.1377 (2)	0.93392 (16)	0.0700 (7)
H23A	0.2180	0.1270	0.9068	0.084*
C24	0.4017 (5)	0.0662 (3)	0.99674 (19)	0.0831 (8)
H24A	0.3253	0.0063	1.0120	0.100*
C25	0.5781 (5)	0.0851 (2)	1.03575 (16)	0.0756 (7)
H25A	0.6238	0.0381	1.0777	0.091*
C26	0.6875 (4)	0.17521 (19)	1.01182 (13)	0.0580 (5)
C27	0.8771 (4)	0.2008 (2)	1.05701 (14)	0.0625 (6)
O3	0.9282 (3)	0.42517 (19)	0.87612 (13)	0.0712 (5)
H31	0.970 (6)	0.493 (4)	0.871 (2)	0.103 (12)*
H32	1.020 (6)	0.384 (4)	0.851 (2)	0.111 (13)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0609 (10)	0.0502 (9)	0.0449 (8)	−0.0055 (8)	0.0042 (7)	−0.0049 (8)
N2	0.0580 (10)	0.0596 (11)	0.0496 (9)	−0.0052 (9)	−0.0080 (8)	−0.0031 (8)
N3	0.0677 (11)	0.0598 (11)	0.0448 (9)	0.0007 (10)	−0.0059 (8)	−0.0011 (9)
O1	0.0527 (8)	0.0858 (12)	0.0805 (11)	−0.0020 (9)	−0.0054 (8)	−0.0119 (10)
O2	0.1293 (17)	0.0611 (11)	0.0760 (12)	0.0033 (12)	−0.0296 (11)	0.0112 (10)
C1	0.0608 (11)	0.0457 (10)	0.0521 (10)	0.0023 (10)	−0.0015 (9)	−0.0007 (9)
C2	0.0563 (11)	0.0551 (12)	0.0553 (11)	−0.0027 (10)	−0.0024 (9)	−0.0011 (10)
C3	0.0943 (18)	0.0577 (14)	0.0651 (14)	−0.0191 (14)	−0.0012 (13)	0.0020 (12)
C4	0.127 (2)	0.0740 (17)	0.0585 (14)	−0.0293 (18)	−0.0019 (15)	0.0070 (13)
C5	0.1024 (19)	0.0689 (16)	0.0497 (12)	−0.0215 (15)	−0.0041 (12)	−0.0041 (12)
C6	0.0670 (13)	0.0535 (12)	0.0584 (12)	−0.0066 (11)	−0.0102 (9)	0.0019 (10)
C7	0.0846 (16)	0.0659 (15)	0.0711 (15)	−0.0043 (13)	0.0138 (13)	−0.0193 (13)
C8	0.0909 (18)	0.0741 (18)	0.0846 (18)	−0.0257 (16)	−0.0117 (15)	−0.0012 (15)
C9	0.0700 (15)	0.0827 (18)	0.096 (2)	0.0101 (15)	−0.0225 (14)	−0.0004 (16)
C10	0.0726 (13)	0.0508 (12)	0.0553 (11)	−0.0009 (11)	−0.0083 (10)	−0.0044 (10)
C11	0.0634 (13)	0.0698 (15)	0.0523 (11)	0.0122 (11)	−0.0061 (9)	−0.0083 (11)
C12	0.0732 (14)	0.0547 (13)	0.0527 (11)	0.0017 (11)	−0.0121 (10)	0.0028 (10)
C13	0.0916 (19)	0.100 (2)	0.0709 (16)	−0.0322 (18)	−0.0087 (14)	−0.0056 (16)
C14	0.118 (2)	0.088 (2)	0.0689 (16)	−0.0129 (19)	−0.0287 (16)	−0.0106 (15)
C15	0.105 (2)	0.0843 (19)	0.0502 (12)	0.0075 (17)	−0.0120 (12)	−0.0118 (13)
C16	0.0944 (17)	0.0774 (18)	0.0491 (12)	−0.0003 (15)	−0.0076 (11)	0.0035 (12)
C17	0.102 (2)	0.092 (2)	0.0702 (16)	0.0267 (18)	−0.0074 (14)	−0.0248 (16)
C18	0.154 (3)	0.151 (4)	0.0734 (19)	−0.040 (3)	0.026 (2)	0.006 (2)
C19	0.145 (3)	0.0752 (19)	0.0743 (17)	0.023 (2)	−0.0426 (19)	0.0044 (16)
C20	0.0604 (12)	0.0768 (17)	0.0539 (11)	0.0021 (12)	−0.0013 (9)	−0.0068 (12)
C21	0.0500 (11)	0.0603 (12)	0.0542 (11)	−0.0010 (10)	0.0036 (8)	−0.0155 (10)
C22	0.0580 (12)	0.0564 (12)	0.0481 (10)	−0.0066 (10)	0.0098 (9)	−0.0131 (9)
C23	0.0706 (15)	0.0743 (16)	0.0654 (14)	−0.0205 (13)	0.0127 (11)	−0.0153 (13)
C24	0.101 (2)	0.0730 (17)	0.0755 (16)	−0.0347 (16)	0.0194 (15)	0.0035 (14)

C25	0.108 (2)	0.0615 (15)	0.0573 (13)	−0.0187 (15)	0.0054 (13)	0.0036 (12)
C26	0.0786 (14)	0.0501 (11)	0.0456 (10)	−0.0036 (11)	0.0059 (10)	−0.0029 (9)
C27	0.0860 (16)	0.0513 (12)	0.0501 (11)	0.0064 (12)	−0.0031 (11)	−0.0034 (10)
O3	0.0613 (10)	0.0674 (12)	0.0853 (12)	−0.0080 (10)	0.0110 (8)	−0.0047 (10)

Geometric parameters (Å, º)

N1—C22	1.337 (3)	C11—C17	1.544 (4)
N1—C26	1.345 (3)	C11—C16	1.559 (3)
N2—C21	1.325 (3)	C11—H11A	0.9800
N2—C10	1.459 (3)	C12—C20	1.525 (4)
N2—H2	0.82 (2)	C12—C13	1.545 (4)
N3—C27	1.318 (3)	C12—H12A	0.9800
N3—C20	1.461 (3)	C13—C14	1.552 (4)
N3—H3	0.86 (3)	C13—H13A	0.9700
O1—C21	1.234 (3)	C13—H13B	0.9700
O2—C27	1.242 (3)	C14—C15	1.512 (5)
C1—C2	1.524 (3)	C14—H14A	0.9700
C1—C7	1.537 (3)	C14—H14B	0.9700
C1—C6	1.564 (3)	C15—C17	1.527 (4)
C1—H1A	0.9800	C15—C16	1.553 (4)
C2—C10	1.528 (3)	C15—H15A	0.9800
C2—C3	1.548 (3)	C16—C19	1.506 (5)
C2—H2A	0.9800	C16—C18	1.543 (5)
C3—C4	1.562 (4)	C17—H17A	0.9700
C3—H3A	0.9700	C17—H17B	0.9700
C3—H3B	0.9700	C18—H18A	0.9600
C4—C5	1.502 (4)	C18—H18B	0.9600
C4—H4A	0.9700	C18—H18C	0.9600
C4—H4B	0.9700	C19—H19A	0.9600
C5—C7	1.526 (4)	C19—H19B	0.9600
C5—C6	1.548 (4)	C19—H19C	0.9600
C5—H5A	0.9800	C20—H20A	0.9700
C6—C9	1.518 (4)	C20—H20B	0.9700
C6—C8	1.534 (4)	C21—C22	1.505 (3)
C7—H7A	0.9700	C22—C23	1.390 (4)
C7—H7B	0.9700	C23—C24	1.391 (4)
C8—H8A	0.9600	C23—H23A	0.9300
C8—H8B	0.9600	C24—C25	1.368 (4)
C8—H8C	0.9600	C24—H24A	0.9300
C9—H9A	0.9600	C25—C26	1.382 (4)
C9—H9B	0.9600	C25—H25A	0.9300
C9—H9C	0.9600	C26—C27	1.506 (3)
C10—H10A	0.9700	O3—H31	0.87 (5)
C10—H10B	0.9700	O3—H32	0.90 (4)
C11—C12	1.514 (4)		
C22—N1—C26	117.60 (19)	C20—C12—C13	111.1 (2)

C21—N2—C10	123.8 (2)	C11—C12—H12A	106.5
C21—N2—H2	119.2 (17)	C20—C12—H12A	106.5
C10—N2—H2	116.9 (17)	C13—C12—H12A	106.5
C27—N3—C20	122.3 (2)	C12—C13—C14	116.2 (3)
C27—N3—H3	120.2 (17)	C12—C13—H13A	108.2
C20—N3—H3	117.4 (17)	C14—C13—H13A	108.2
C2—C1—C7	107.55 (19)	C12—C13—H13B	108.2
C2—C1—C6	114.69 (18)	C14—C13—H13B	108.2
C7—C1—C6	87.32 (18)	H13A—C13—H13B	107.4
C2—C1—H1A	114.7	C15—C14—C13	112.7 (2)
C7—C1—H1A	114.7	C15—C14—H14A	109.1
C6—C1—H1A	114.7	C13—C14—H14A	109.1
C1—C2—C10	114.43 (19)	C15—C14—H14B	109.1
C1—C2—C3	111.28 (17)	C13—C14—H14B	109.1
C10—C2—C3	111.0 (2)	H14A—C14—H14B	107.8
C1—C2—H2A	106.5	C14—C15—C17	108.9 (3)
C10—C2—H2A	106.5	C14—C15—C16	111.8 (3)
C3—C2—H2A	106.5	C17—C15—C16	87.8 (2)
C2—C3—C4	115.1 (2)	C14—C15—H15A	115.1
C2—C3—H3A	108.5	C17—C15—H15A	115.1
C4—C3—H3A	108.5	C16—C15—H15A	115.1
C2—C3—H3B	108.5	C19—C16—C18	107.8 (3)
C4—C3—H3B	108.5	C19—C16—C15	118.8 (3)
H3A—C3—H3B	107.5	C18—C16—C15	112.3 (3)
C5—C4—C3	112.8 (2)	C19—C16—C11	121.7 (2)
C5—C4—H4A	109.0	C18—C16—C11	109.7 (3)
C3—C4—H4A	109.0	C15—C16—C11	85.0 (2)
C5—C4—H4B	109.0	C15—C17—C11	86.4 (2)
C3—C4—H4B	109.0	C15—C17—H17A	114.3
H4A—C4—H4B	107.8	C11—C17—H17A	114.3
C4—C5—C7	109.3 (2)	C15—C17—H17B	114.3
C4—C5—C6	111.9 (2)	C11—C17—H17B	114.2
C7—C5—C6	88.31 (19)	H17A—C17—H17B	111.4
C4—C5—H5A	114.8	C16—C18—H18A	109.5
C7—C5—H5A	114.8	C16—C18—H18B	109.5
C6—C5—H5A	114.8	H18A—C18—H18B	109.5
C9—C6—C8	108.1 (2)	C16—C18—H18C	109.5
C9—C6—C5	118.8 (2)	H18A—C18—H18C	109.5
C8—C6—C5	112.2 (2)	H18B—C18—H18C	109.5
C9—C6—C1	121.6 (2)	C16—C19—H19A	109.5
C8—C6—C1	109.9 (2)	C16—C19—H19B	109.5
C5—C6—C1	84.67 (18)	H19A—C19—H19B	109.5
C5—C7—C1	86.32 (19)	C16—C19—H19C	109.5
C5—C7—H7A	114.3	H19A—C19—H19C	109.5
C1—C7—H7A	114.3	H19B—C19—H19C	109.5
C5—C7—H7B	114.3	N3—C20—C12	112.38 (19)
C1—C7—H7B	114.3	N3—C20—H20A	109.1
H7A—C7—H7B	111.4	C12—C20—H20A	109.1

C6—C8—H8A	109.5	N3—C20—H20B	109.1
C6—C8—H8B	109.5	C12—C20—H20B	109.1
H8A—C8—H8B	109.5	H20A—C20—H20B	107.9
C6—C8—H8C	109.5	O1—C21—N2	124.4 (2)
H8A—C8—H8C	109.5	O1—C21—C22	119.8 (2)
H8B—C8—H8C	109.5	N2—C21—C22	115.86 (18)
C6—C9—H9A	109.5	N1—C22—C23	122.9 (2)
C6—C9—H9B	109.5	N1—C22—C21	117.59 (19)
H9A—C9—H9B	109.5	C23—C22—C21	119.5 (2)
C6—C9—H9C	109.5	C22—C23—C24	118.4 (2)
H9A—C9—H9C	109.5	C22—C23—H23A	120.8
H9B—C9—H9C	109.5	C24—C23—H23A	120.8
N2—C10—C2	111.88 (19)	C25—C24—C23	119.2 (3)
N2—C10—H10A	109.2	C25—C24—H24A	120.4
C2—C10—H10A	109.2	C23—C24—H24A	120.4
N2—C10—H10B	109.2	C24—C25—C26	118.9 (3)
C2—C10—H10B	109.2	C24—C25—H25A	120.5
H10A—C10—H10B	107.9	C26—C25—H25A	120.5
C12—C11—C17	108.1 (2)	N1—C26—C25	123.0 (2)
C12—C11—C16	115.7 (2)	N1—C26—C27	117.2 (2)
C17—C11—C16	86.95 (19)	C25—C26—C27	119.7 (2)
C12—C11—H11A	114.3	O2—C27—N3	123.5 (2)
C17—C11—H11A	114.3	O2—C27—C26	119.6 (2)
C16—C11—H11A	114.3	N3—C27—C26	116.9 (2)
C11—C12—C20	114.9 (2)	H31—O3—H32	103 (4)
C11—C12—C13	110.7 (2)		

C7—C1—C2—C10	−178.2 (2)	C14—C15—C16—C18	168.8 (3)
C6—C1—C2—C10	86.5 (2)	C17—C15—C16—C18	−81.7 (3)
C7—C1—C2—C3	54.9 (3)	C14—C15—C16—C11	−81.9 (2)
C6—C1—C2—C3	−40.3 (3)	C17—C15—C16—C11	27.6 (2)
C1—C2—C3—C4	−10.1 (4)	C12—C11—C16—C19	−39.6 (4)
C10—C2—C3—C4	−138.8 (3)	C17—C11—C16—C19	−148.2 (3)
C2—C3—C4—C5	7.6 (4)	C12—C11—C16—C18	−166.7 (3)
C3—C4—C5—C7	−50.0 (4)	C17—C11—C16—C18	84.7 (3)
C3—C4—C5—C6	46.1 (4)	C12—C11—C16—C15	81.3 (3)
C4—C5—C6—C9	40.2 (3)	C17—C11—C16—C15	−27.3 (2)
C7—C5—C6—C9	150.4 (2)	C14—C15—C17—C11	84.4 (3)
C4—C5—C6—C8	167.5 (3)	C16—C15—C17—C11	−27.8 (2)
C7—C5—C6—C8	−82.3 (2)	C12—C11—C17—C15	−88.3 (2)
C4—C5—C6—C1	−83.1 (2)	C16—C11—C17—C15	27.7 (2)
C7—C5—C6—C1	27.07 (17)	C27—N3—C20—C12	−97.3 (3)
C2—C1—C6—C9	−39.5 (3)	C11—C12—C20—N3	55.0 (3)
C7—C1—C6—C9	−147.6 (3)	C13—C12—C20—N3	−178.4 (2)
C2—C1—C6—C8	−167.1 (2)	C10—N2—C21—O1	−4.0 (3)
C7—C1—C6—C8	84.8 (2)	C10—N2—C21—C22	175.63 (19)
C2—C1—C6—C5	81.2 (2)	C26—N1—C22—C23	0.4 (3)
C7—C1—C6—C5	−26.87 (18)	C26—N1—C22—C21	−179.78 (18)

C4—C5—C7—C1	85.2 (3)	O1—C21—C22—N1	−170.6 (2)
C6—C5—C7—C1	−27.51 (18)	N2—C21—C22—N1	9.8 (3)
C2—C1—C7—C5	−87.9 (2)	O1—C21—C22—C23	9.3 (3)
C6—C1—C7—C5	27.22 (18)	N2—C21—C22—C23	−170.4 (2)
C21—N2—C10—C2	−95.3 (3)	N1—C22—C23—C24	−0.9 (4)
C1—C2—C10—N2	62.7 (2)	C21—C22—C23—C24	179.2 (2)
C3—C2—C10—N2	−170.3 (2)	C22—C23—C24—C25	0.5 (4)
C17—C11—C12—C20	−179.3 (2)	C23—C24—C25—C26	0.3 (4)
C16—C11—C12—C20	85.2 (3)	C22—N1—C26—C25	0.6 (3)
C17—C11—C12—C13	53.8 (3)	C22—N1—C26—C27	−177.24 (19)
C16—C11—C12—C13	−41.6 (3)	C24—C25—C26—N1	−0.9 (4)
C11—C12—C13—C14	−8.2 (4)	C24—C25—C26—C27	176.8 (3)
C20—C12—C13—C14	−137.1 (3)	C20—N3—C27—O2	−6.1 (4)
C12—C13—C14—C15	5.6 (5)	C20—N3—C27—C26	171.8 (2)
C13—C14—C15—C17	−48.7 (4)	N1—C26—C27—O2	179.6 (2)
C13—C14—C15—C16	46.6 (4)	C25—C26—C27—O2	1.8 (3)
C14—C15—C16—C19	41.7 (3)	N1—C26—C27—N3	1.6 (3)
C17—C15—C16—C19	151.2 (3)	C25—C26—C27—N3	−176.2 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O3	0.82 (2)	2.16 (2)	2.941 (3)	160 (2)
N3—H3···O3	0.86 (3)	2.19 (3)	3.017 (3)	159 (2)
O3—H31···O2^{i}	0.87 (5)	1.90 (5)	2.756 (3)	167 (4)
O3—H32···O1^{ii}	0.90 (4)	1.86 (5)	2.754 (3)	171 (3)

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