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Synthesis and crystal structure of 1,3-bis{[*N*,*N*-bis(2-hydroxyethyl)amino]methyl}-5-{[(4,6-dimethylpyridin-2-yl)amino]methyl}-2,4,6-triethylbenzene

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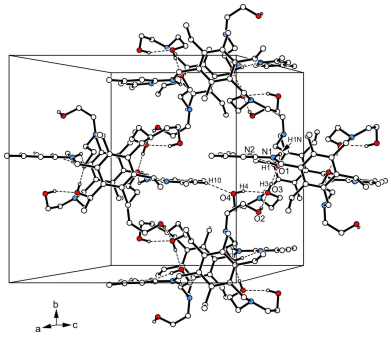
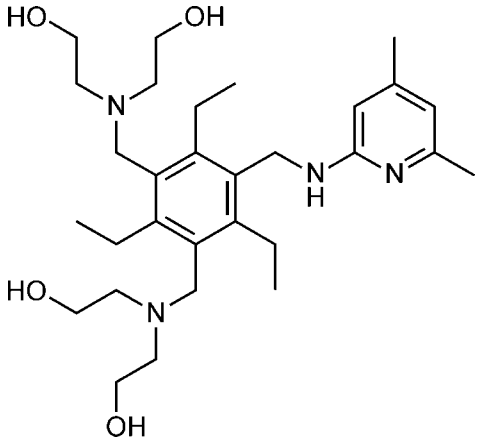
In the crystal structure of the title compound, $C_{30}H_{50}N_4O_4$, the two bis(hydroxyethyl)amino moieties and the 2,4-dimethylpyridinylamino unit of the molecule are located on one side of the central benzene ring, while the ethyl substituents are oriented in the opposite direction. The dihedral angle between the planes of the aromatic rings is 73.6 (1)°. The conformation of the molecule is stabilized by intramolecular O—H···O (1.86–2.12 Å) and C—H···N (2.40, 2.54 Å) hydrogen bonds. Dimers of inversion-related molecules represent the basic supramolecular entities of the crystal structure. They are further connected *via* O—H···O hydrogen bonding into undulating layers extending parallel to the crystallographic *bc* plane. Interlayer interaction is accomplished by weak C—H···π contacts.

1. Chemical context

The 1,3,5-trisubstituted 2,4,6-trialkylbenzene scaffold has shown to be valuable for the construction of various artificial receptors (Hennrich & Anslyn, 2002). In the course of our research work, we have successfully used this molecular scaffold in the design of acyclic and macrocyclic receptors for neutral (Mazik, 2009, 2012; Lippe & Mazik, 2015; Lippe *et al.*, 2015; Amrhein *et al.*, 2016; Koch *et al.*, 2016; Amrhein & Mazik, 2021; Köhler *et al.*, 2020, 2021) and ionic substrates (Geffert *et al.*, 2013; Stapf *et al.*, 2015; Schulze *et al.*, 2018). Our studies on the molecular recognition of carbohydrates have shown that the participation of different types of recognition groups in the complexation of the substrate favourably influences the binding process (Stapf *et al.*, 2020*a*,*b*; Kaiser *et al.*, 2019). Such a combination of two types of recognition units, namely heterocyclic and hydroxy groups, is realised in the triethylbenzene-based title compound **1** (see also Stapf *et al.*, 2020*a*). The design of the receptors consisting of the aforementioned recognition units was inspired by the nature of the protein binding sites involved in the interactions stabilizing the crystalline protein–carbohydrate complexes (Quiocho, 1989). For example, 2-aminopyridine can be considered as a heterocyclic analogue of the asparagine/glutamine primary amide side chain. Furthermore, it should be noted that the formation of intramolecular interactions is also one of the factors influencing the binding properties of a receptor molecule (Rosien *et al.*, 2013). Intramolecular interactions can also be observed in the crystal structure of **1**.





2. Structural commentary

In the title molecule, the structure of which is shown in Fig. 1, the functionalized side arms are arranged on one side of the central benzene ring, while the ethyl substituents are oriented in the opposite direction. One of the bis(hydroxyethyl)amino moieties is disordered over two positions [s.o.f. 0.879 (2)/0.121 (2)]. The interplanar angle between the aromatic rings is 73.6 (1)°. Within the molecule, three hydroxy groups create a continuous pattern of O—H···O hydrogen bonds [d(H···O) 1.86–2.12 Å]. The amino nitrogen atoms N3 and N4 are involved in intramolecular C—H···N hydrogen bonding [d(H···N) 2.40, 2.54 Å]. The crystal structure contains four potentially solvent-accessible voids with a total volume of 110 Å^3 per unit cell (Spek, 2015). The void volume of 27.5 Å^3 and the maximum residual electron density of 0.55 e Å^{-3} indicate that the voids could be partially occupied by water molecules.

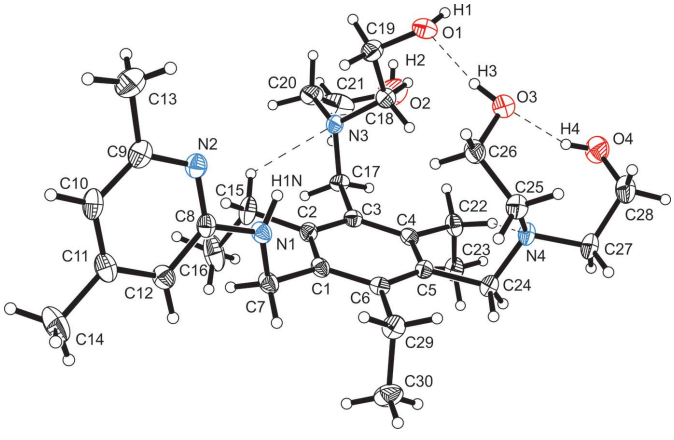


Figure 1
Perspective view of the molecular structure of the title compound including atom numbering. Anisotropic displacement ellipsoids are drawn at a 50% probability level. Dashed lines represent hydrogen-bonding interactions. For the sake of clarity, only the major position of the disordered bis(hydroxyethyl)amino moiety is shown.

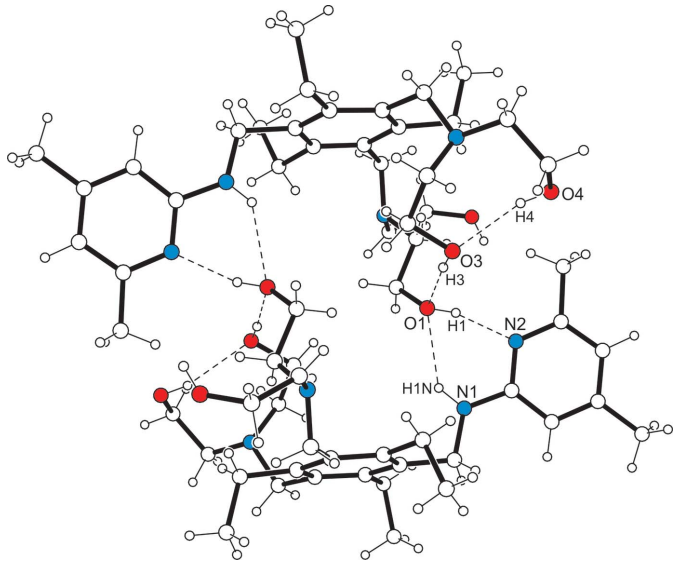


Figure 2
Structure of the molecular dimer including the numbering of atoms involved in hydrogen-bonding interactions. For the sake of clarity, only the major position of the disordered hydroxyethyl moiety is shown. Hydrogen bonds are shown as dashed lines.

3. Supramolecular features

As depicted in Fig. 2 and Fig. 3, the crystal structure is constructed of inversion-symmetric molecular dimers held together by O—H···N and N—H···O hydrogen bonding [d(H···N) 1.89 (2) Å; d(H···O) 2.19 (2) Å; graph set $R_2^2(6)$ (Etter, 1990; Bernstein *et al.*, 1995)]. These dimers are further assembled *via* O—H···O [d(H···O) 1.99 (2) Å] and C—H···O [d(H···O) 2.45 Å] bonds (Desiraju & Steiner, 1999)

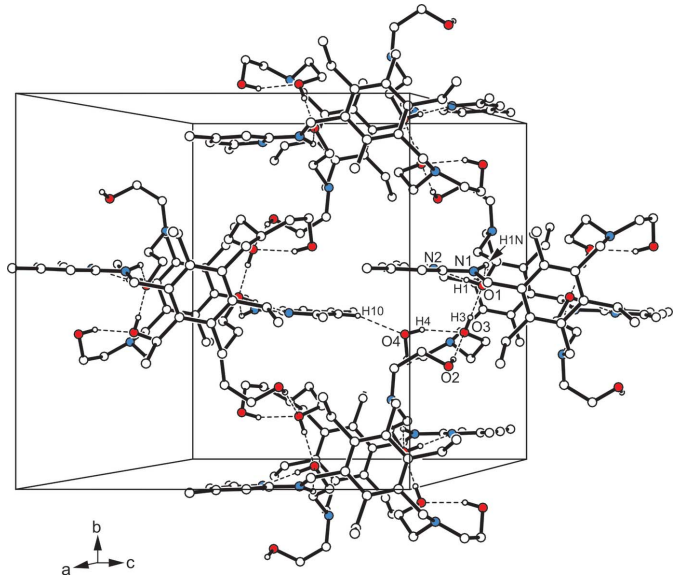


Figure 3
Packing excerpt of the title compound with numbering of coordinating atoms. Oxygen atoms are displayed as red, nitrogen atoms as blue circles. Hydrogen atoms excluded from intermolecular interactions are omitted for clarity. Hydrogen bonds are shown as broken lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2*A*—H2*A*···O4^{i}	0.84	1.99	2.763 (18)	152
O1*A*—H1*A*···N2^{ii}	0.84	1.99	2.832 (12)	178
C18*A*—H18*D*···O2*A*	0.99	2.46	3.08 (2)	120
O2—H2···O3^{i}	0.87 (2)	1.99 (2)	2.828 (2)	162 (3)
O1—H1···N2^{ii}	0.87 (2)	1.89 (2)	2.7449 (19)	167 (3)
N1—H1*N*···O1^{ii}	0.89 (2)	2.19 (2)	3.014 (2)	152.0 (17)
C22—H22*A*···N4	0.99	2.40	3.152 (2)	132
C18—H18*A*···O2	0.99	2.39	3.106 (3)	128
C15—H15*A*···N3	0.99	2.54	3.282 (3)	131
C13—H13*A*···O2*A*^{iii}	0.98	2.33	3.220 (14)	151
C10—H10···O4^{iv}	0.95	2.45	3.365 (2)	161
O4—H4···O3	0.86 (2)	2.12 (2)	2.9200 (18)	155 (3)
O3—H3···O1*A*	0.87 (2)	1.93 (2)	2.727 (13)	152 (3)
O3—H3···O1	0.87 (2)	1.86 (2)	2.7156 (19)	172 (3)

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

into layers extending parallel to the crystallographic *bc* plane (Fig. 4). As the layer surfaces are defined by the ethyl groups of the molecules, interlayer association is restricted to weak C—H···π contacts (Nishio *et al.*, 1995). Information regarding non-covalent bonding present in the crystal is found in Table 1.

4. Database survey

A search in the Cambridge Structural Database (CSD, Version 5.43, update November 2021; Groom *et al.*, 2016) for 2,4,6-triethylbenzene derivatives bearing the (4,6-dimethylpyridin-2-yl)aminomethyl unit gave eight hits. In the crystal structures of the monohydrate and the methanol solvate of {1-[(3,5-bis{[(4,6-dimethylpyridin-2-yl)amino]methyl}-2,4,6-triethylbenzyl)amino]cyclopentyl}methanol (CADTAG, CADTEK; Stapf *et al.*, 2020*a*), the host molecules reveal similar geometries with an alternating arrangement of the substituents above and below the plane of the central benzene ring. The crystals of these solvates are composed of inversion-symmetric dimers of 1:1 host–guest complexes held together by O—H···N and N—H···O hydrogen bonds.

In the case of the ethanol solvate of 1,3,5-tris[(4,6-dimethylpyridin-2-yl)aminomethyl]-2,4,6-triethylbenzene (RAJZAE; Mazik *et al.*, 2004), dimers of host–guest units stabilized by O—H···N$_{pyr}$ and N—H···O bonds represent the basic supramolecular aggregates. The latter compound is also capable of forming crystalline complexes with methyl β-D-glucopyranoside (LAJZOP; Köhler *et al.*, 2020). This crystal structure (acetonitrile tetrasolvate monohydrate) contains two structurally different 2:1 receptor-carbohydrate complexes in which the sugar substrate is located in a cavity formed by the functionalized side arms of a pair of receptor molecules.

In the crystal structure of 1-{[*N*-(1,10-phenanthrolin-2-ylcarbonyl)amino]methyl}-3,5-bis{[(4,6-dimethylpyridin-2-yl)-amino]methyl}-2,4,6-triethylbenzene (ROKJEH; Mazik *et al.*, 2008), three water molecules are accommodated in the binding pocket created by the heterocyclic units (one phenanthrolinyl and two pyridinyl groups) of the host molecule. This host–water aggregate is stabilized by O—H···O, N—H···O and O—H···N hydrogen bonds. In a similar way, two water molecules and one ethanol molecule are accommodated in the binding pocket of 1,3-bis{[*N*-(1,10-phenanthrolin-2-ylcarbonyl)amino]methyl}-5-{[(4,6-dimethylpyridin-2-yl)amino]methyl}-2,4,6-triethylbenzene (TUGVEX; Mazik *et al.*, 2009), containing one pyridinyl and two phenanthrolinyl groups.

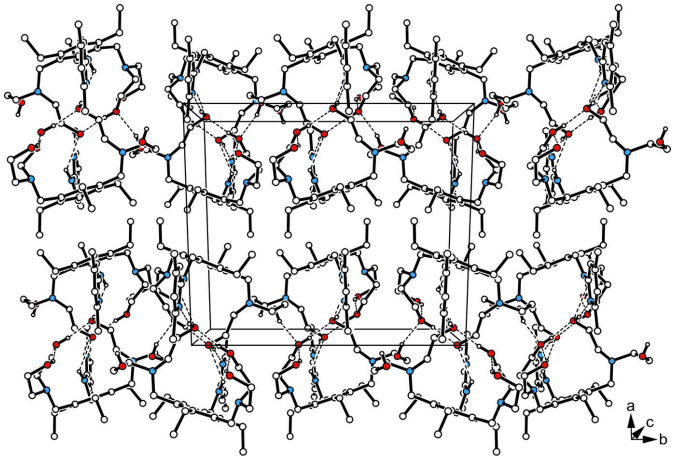


Figure 4
Packing diagram of the title compound viewed down the *c* axis. Oxygen atoms are displayed as red, nitrogen atoms as blue circles. Hydrogen-bonding interactions are shown as dashed lines.

5. Synthesis and crystallization

A mixture of diethanolamine (0.18 mL, 0.20 g, 1.88 mmol), THF (10 mL) and potassium carbonate (86 mg, 0.62 mmol) was stirred at room temperature for 30 minutes. After that, a solution of 1,3-bis(bromomethyl)-5-{[(4,6-dimethylpyridin-2-yl)amino]methyl}-2,4,6-triethylbenzene (150 mg, 0.31 mmol) in 10 mL of THF/CH_3CN (1:1) was added dropwise and the resulting mixture was stirred at room temperature and under light exclusion (the progress of the reaction was monitored by TLC). After filtration, the solvents were removed under reduced pressure and the residual yellow oil was treated with a THF/water mixture. Then the THF was evaporated and the resulting oil was separated from the water. The oil was again dissolved in THF and dried over $MgSO_4$. By addition of *n*-hexane, the product was precipitated as a white solid (58% yield, 95 mg, 0.18 mmol). *Analysis data*: m.p. = 408 K; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.14 (*t*, *J* = 7.5 Hz, 3H, C*H*$_3$), 1.19 (*t*, *J* = 7.5 Hz, 6H, C*H*$_3$), 2.29 (*s*, 3H, C*H*$_3$), 2.35 (*s*, 3H, C*H*$_3$), 2.63 (*t*, *J* = 5.0 Hz, 8H, C*H*$_2$), 2.80 (*q*, *J* = 7.5 Hz, 4H, C*H*$_2$), 3.26 (*q*, *J* = 7.5 Hz, 2H, C*H*$_2$), 3.50 (*t*, *J* = 5.0 Hz, 8H, C*H*$_2$), 3.77 (*s*, 4H, C*H*$_2$), 4.22 (*d*, *J* = 4.0 Hz, 2H, C*H*$_2$), 4.60 (*br*, 1H, N*H*), 6.17 (*s*, 1H, Ar*H*), 6.38 (*s*, 1H, Ar*H*); ^{13}C NMR (126 MHz, $CDCl_3$) δ 15.7, 16.5, 21.2, 21.3, 22.9, 23.8, 40.8, 52.3, 55.2, 59.7, 102.7, 114.1, 131.6, 132.6, 143.5, 145.8, 149.7, 156.2, 158.0; MS (ESI): *m*/*z* calculated for $C_{30}H_{51}N_4O_4$: 531.4 [*M* + H]$^+$, found 531.4; R_f = 0.50 (Al_2O_3, $CHCl_3$/CH_3OH 7:1). Crystals of the title compound suitable for X-ray analysis were obtained as

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{30}H_{50}N_4O_4$
M_r	530.74
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	14.2508 (3), 15.3046 (4), 15.2593 (3)
β (°)	113.3107 (13)
V (Å^3)	3056.43 (12)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.20 × 0.13 × 0.12
Data collection	
Diffractometer	Bruker Kappa APEXII with CCD area detector
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	22458, 6881, 4968
R_{int}	0.031
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.647
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.049, 0.144, 1.03
No. of reflections	6881
No. of parameters	431
No. of restraints	290
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.55, −0.26

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SHELXS97* and *SHELXTL* (Sheldrick, 2008), *SHELXL2018/3* (Sheldrick, 2015) and *ORTEP-3 for Windows* (Farrugia, 2012).

colourless blocks by diffusion of *n*-hexane into a solution of the compound in THF.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Carbon-bound hydrogen atoms and protons of the minor (12%) positions of the disordered OH groups (H1*A*, H2*A*) were positioned geometrically and allowed to ride on their respective parent atoms, with C—H = 0.95 Å (aromatic) and 0.99 Å (methylene) and U_{iso}(H) = 1.2 U_{eq}(C), and O—H = 0.84 Å (OH) and C—H = 0.98 Å (methyl) and U_{iso}(H) = 1.5 U_{eq}(C,O), respectively. The protons of the N—H and O—H (undisordered or the main positions) were located from the residual electron density map and refined with U_{iso}(H) bound to the parent atom (1.2, for NH and 1.5 for OH) with distance restraints for the OH bonds (SADI). The refinement of the disordered $N(CH_2CH_2OH)_2$ group was performed using geometry (SAME) and U_{ij} (SIMU, RIGU) restrains implemented in *SHELXL* (Sheldrick, 2015). The refined proportion of the two positions is 88:12%. The maximum residual peak of 0.55 e Å^{-3} is located inside a 27.5 Å^3 void and can be refined as a partially occupied water molecule (∼6%); however, due to the low occupancy, it was not included in the final refinement.

Funding information

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supporting information

Acta Cryst. (2022). E**78**, 825-828 [https://doi.org/10.1107/S2056989022007411]

Synthesis and crystal structure of 1,3-bis{[*N*,*N*-bis(2-hydroxyethyl)amino]-methyl}-5-{[(4,6-dimethylpyridin-2-yl)amino]methyl}-2,4,6-triethylbenzene

Manuel Stapf, Ute Schmidt, Wilhelm Seichter and Monika Mazik

Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

2-{[(3-{[Bis(2-hydroxyethyl)amino]methyl}-5-{[(4,6-dimethylpyridin-2-yl)amino]methyl}-2,4,6-triethylphenyl)methyl](2-hydroxyethyl)amino}ethan-1-ol

Crystal data

$C_{30}H_{50}N_4O_4$
$M_r = 530.74$
Monoclinic, $P2_1/c$
$a = 14.2508$ (3) Å
$b = 15.3046$ (4) Å
$c = 15.2593$ (3) Å
$\beta = 113.3107$ (13)°
$V = 3056.43$ (12) Å^3
$Z = 4$
$F(000) = 1160$
$D_x = 1.153$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5468 reflections
$\theta = 2.7$–$28.2°$
$\mu = 0.08$ mm^{-1}
$T = 100$ K
Rod, colourless
0.20 × 0.13 × 0.12 mm

Data collection

Bruker Kappa APEXII with CCD area detector diffractometer
Radiation source: fine-focus sealed X-Ray tube
phi and ω scans
22458 measured reflections
6881 independent reflections
4968 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 27.4°$, $\theta_{min} = 3.1°$
$h = -18\rightarrow17$
$k = -19\rightarrow12$
$l = -19\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.144$
$S = 1.03$
6881 reflections
431 parameters
290 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
$w = 1/[\sigma^2(F_o^2) + (0.0773P)^2 + 0.7833P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ 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}	Occ. (<1)
O3	0.06213 (10)	0.37633 (9)	0.20622 (9)	0.0317 (3)	
H3	0.0307 (19)	0.4220 (14)	0.1746 (17)	0.066 (8)*	
O4	0.14319 (11)	0.36916 (9)	0.41431 (9)	0.0344 (3)	
H4	0.133 (2)	0.3843 (18)	0.3569 (14)	0.075 (9)*	
N1	0.27113 (11)	0.44288 (9)	−0.07113 (9)	0.0208 (3)	
H1N	0.2143 (16)	0.4558 (13)	−0.0622 (13)	0.031 (5)*	
N2	0.15327 (11)	0.43433 (8)	−0.22623 (9)	0.0228 (3)	
N4	0.27759 (10)	0.34248 (8)	0.32090 (9)	0.0205 (3)	
C1	0.35480 (12)	0.49260 (10)	0.09155 (10)	0.0187 (3)	
C2	0.31562 (12)	0.57360 (10)	0.10358 (11)	0.0194 (3)	
C3	0.30179 (12)	0.59065 (9)	0.18829 (11)	0.0181 (3)	
C4	0.32351 (11)	0.52609 (10)	0.25834 (10)	0.0173 (3)	
C5	0.35813 (12)	0.44317 (9)	0.24358 (10)	0.0174 (3)	
C6	0.37449 (11)	0.42637 (10)	0.16047 (10)	0.0177 (3)	
C7	0.36773 (12)	0.47485 (11)	−0.00056 (11)	0.0221 (3)	
H7A	0.387939	0.529168	−0.023767	0.027*	
H7B	0.421962	0.430698	0.010295	0.027*	
C8	0.25215 (12)	0.43984 (9)	−0.16689 (11)	0.0193 (3)	
C9	0.12953 (14)	0.42942 (11)	−0.32090 (12)	0.0267 (4)	
C10	0.20380 (14)	0.43045 (11)	−0.35800 (12)	0.0275 (4)	
H10	0.185041	0.427754	−0.425011	0.033*	
C11	0.30622 (14)	0.43547 (11)	−0.29619 (12)	0.0273 (4)	
C12	0.33091 (13)	0.43981 (10)	−0.19916 (11)	0.0233 (3)	
H12	0.400235	0.442732	−0.155317	0.028*	
C13	0.01792 (15)	0.42276 (15)	−0.38339 (14)	0.0410 (5)	
H13A	0.009330	0.414806	−0.449864	0.062*	
H13B	−0.016851	0.476413	−0.377828	0.062*	
H13C	−0.011639	0.372699	−0.363317	0.062*	
C14	0.38925 (17)	0.43524 (14)	−0.33408 (15)	0.0413 (5)	
H14A	0.396002	0.376396	−0.356233	0.062*	
H14B	0.454117	0.452731	−0.283249	0.062*	
H14C	0.371554	0.476451	−0.387372	0.062*	
C15	0.28910 (15)	0.64292 (11)	0.02668 (12)	0.0301 (4)	
H15A	0.232164	0.678992	0.028352	0.036*	
H15B	0.265364	0.614087	−0.036485	0.036*	
C16	0.37980 (19)	0.70256 (13)	0.03842 (16)	0.0468 (6)	
H16A	0.435285	0.667612	0.033810	0.070*	
H16B	0.403792	0.731212	0.100914	0.070*	
H16C	0.358268	0.746977	−0.011954	0.070*	

C17	0.25925 (12)	0.67846 (10)	0.20094 (12)	0.0220 (3)	
H17A	0.275654	0.688014	0.269611	0.026*	0.879 (2)
H17B	0.292937	0.725243	0.179147	0.026*	0.879 (2)
H17C	0.295174	0.699548	0.267252	0.026*	0.121 (2)
H17D	0.268160	0.722174	0.157027	0.026*	0.121 (2)
N3	0.14881 (13)	0.68479 (11)	0.14808 (13)	0.0210 (4)	0.879 (2)
C18	0.09000 (15)	0.61856 (13)	0.17436 (13)	0.0257 (4)	0.879 (2)
H18A	0.067231	0.643267	0.222641	0.031*	0.879 (2)
H18B	0.134482	0.567761	0.203334	0.031*	0.879 (2)
C19	−0.0016 (2)	0.5886 (3)	0.0894 (3)	0.0291 (8)	0.879 (2)
H19A	−0.050599	0.637513	0.064946	0.035*	0.879 (2)
H19B	0.019809	0.569992	0.037941	0.035*	0.879 (2)
O1	−0.04980 (13)	0.51746 (9)	0.11566 (11)	0.0291 (4)	0.879 (2)
H1	−0.0746 (19)	0.5386 (16)	0.1552 (16)	0.044*	0.879 (2)
C20	0.11081 (16)	0.77397 (12)	0.14305 (16)	0.0307 (5)	0.879 (2)
H20A	0.035285	0.772108	0.111954	0.037*	0.879 (2)
H20B	0.135047	0.808013	0.100913	0.037*	0.879 (2)
C21	0.1402 (2)	0.82323 (15)	0.2360 (2)	0.0441 (6)	0.879 (2)
H21A	0.215140	0.831891	0.264051	0.053*	0.879 (2)
H21B	0.107765	0.881661	0.222582	0.053*	0.879 (2)
O2	0.11150 (16)	0.78077 (12)	0.30323 (13)	0.0529 (5)	0.879 (2)
H2	0.0511 (16)	0.801 (2)	0.292 (2)	0.088 (12)*	0.879 (2)
N3A	0.1503 (6)	0.6654 (8)	0.1793 (8)	0.028 (3)	0.121 (2)
C18A	0.0841 (8)	0.6365 (8)	0.0834 (7)	0.022 (2)	0.121 (2)
H18C	0.119291	0.590229	0.062551	0.026*	0.121 (2)
H18D	0.071014	0.686176	0.038571	0.026*	0.121 (2)
C19A	−0.0162 (11)	0.6017 (19)	0.0800 (16)	0.025 (4)	0.121 (2)
H19C	−0.061027	0.650470	0.081917	0.030*	0.121 (2)
H19D	−0.051601	0.568134	0.020504	0.030*	0.121 (2)
O1A	0.0064 (10)	0.5463 (9)	0.1615 (10)	0.051 (3)	0.121 (2)
H1A	−0.040111	0.550860	0.181942	0.076*	0.121 (2)
C20A	0.1121 (12)	0.7331 (9)	0.2246 (10)	0.050 (3)	0.121 (2)
H20C	0.152433	0.732286	0.294424	0.060*	0.121 (2)
H20D	0.039976	0.720561	0.213106	0.060*	0.121 (2)
C21A	0.1192 (18)	0.8226 (10)	0.1859 (13)	0.066 (5)	0.121 (2)
H21C	0.106339	0.867512	0.226497	0.079*	0.121 (2)
H21D	0.189389	0.831435	0.189293	0.079*	0.121 (2)
O2A	0.0491 (14)	0.8342 (12)	0.0907 (11)	0.087 (5)	0.121 (2)
H2A	−0.010553	0.825947	0.087201	0.130*	0.121 (2)
C22	0.31398 (13)	0.54528 (10)	0.35242 (11)	0.0229 (3)	
H22A	0.299154	0.490307	0.378670	0.027*	
H22B	0.256090	0.585763	0.340778	0.027*	
C23	0.41152 (14)	0.58586 (11)	0.42515 (11)	0.0261 (4)	
H23A	0.469352	0.546753	0.435284	0.039*	
H23B	0.404018	0.594612	0.485670	0.039*	
H23C	0.423840	0.642269	0.401207	0.039*	
C24	0.37421 (12)	0.37309 (10)	0.31879 (11)	0.0205 (3)	
H24A	0.410543	0.322946	0.305591	0.025*	

H24B	0.417870	0.396841	0.382251	0.025*
C25	0.21403 (13)	0.29319 (11)	0.23560 (11)	0.0246 (4)
H25A	0.182595	0.243188	0.255098	0.029*
H25B	0.258086	0.269381	0.204862	0.029*
C26	0.13072 (13)	0.34762 (11)	0.16397 (12)	0.0265 (4)
H26A	0.160910	0.398788	0.145041	0.032*
H26B	0.093041	0.312534	0.106152	0.032*
C27	0.29400 (14)	0.29730 (11)	0.41001 (12)	0.0264 (4)
H27A	0.344684	0.330023	0.463986	0.032*
H27B	0.322171	0.238398	0.408799	0.032*
C28	0.19565 (15)	0.28872 (12)	0.42530 (13)	0.0320 (4)
H28A	0.150549	0.245962	0.379068	0.038*
H28B	0.211491	0.265817	0.490291	0.038*
C29	0.41588 (13)	0.33905 (10)	0.14462 (12)	0.0246 (4)
H29A	0.389151	0.326780	0.075312	0.029*
H29B	0.391172	0.292370	0.175083	0.029*
C30	0.53240 (14)	0.33718 (12)	0.18528 (13)	0.0336 (4)
H30A	0.555528	0.279991	0.172831	0.050*
H30B	0.559255	0.347470	0.254260	0.050*
H30C	0.557266	0.382886	0.154862	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0266 (7)	0.0379 (7)	0.0313 (7)	0.0018 (6)	0.0122 (6)	0.0082 (6)
O4	0.0409 (8)	0.0418 (8)	0.0244 (7)	0.0039 (6)	0.0170 (6)	0.0003 (6)
N1	0.0216 (7)	0.0258 (7)	0.0169 (7)	−0.0023 (6)	0.0096 (6)	−0.0033 (5)
N2	0.0267 (8)	0.0207 (7)	0.0218 (7)	0.0009 (5)	0.0103 (6)	0.0008 (5)
N4	0.0258 (7)	0.0192 (6)	0.0153 (6)	−0.0026 (5)	0.0068 (6)	0.0014 (5)
C1	0.0180 (7)	0.0219 (7)	0.0168 (7)	−0.0045 (6)	0.0077 (6)	−0.0025 (6)
C2	0.0188 (8)	0.0188 (7)	0.0194 (8)	−0.0033 (6)	0.0063 (6)	0.0013 (6)
C3	0.0167 (7)	0.0153 (7)	0.0221 (8)	−0.0014 (6)	0.0075 (6)	−0.0011 (6)
C4	0.0169 (7)	0.0172 (7)	0.0188 (7)	−0.0023 (6)	0.0084 (6)	−0.0027 (6)
C5	0.0178 (7)	0.0169 (7)	0.0168 (7)	−0.0005 (6)	0.0062 (6)	0.0000 (6)
C6	0.0167 (7)	0.0182 (7)	0.0183 (7)	−0.0008 (6)	0.0069 (6)	−0.0036 (6)
C7	0.0215 (8)	0.0286 (8)	0.0180 (8)	−0.0041 (7)	0.0096 (7)	−0.0028 (6)
C8	0.0261 (8)	0.0147 (7)	0.0185 (7)	0.0000 (6)	0.0104 (7)	0.0001 (6)
C9	0.0325 (10)	0.0221 (8)	0.0232 (8)	−0.0002 (7)	0.0088 (7)	−0.0002 (6)
C10	0.0395 (10)	0.0268 (9)	0.0174 (8)	−0.0027 (7)	0.0127 (8)	−0.0005 (6)
C11	0.0360 (10)	0.0255 (8)	0.0261 (9)	−0.0049 (7)	0.0184 (8)	−0.0036 (7)
C12	0.0246 (9)	0.0254 (8)	0.0210 (8)	−0.0029 (7)	0.0101 (7)	−0.0038 (6)
C13	0.0350 (11)	0.0513 (12)	0.0294 (10)	0.0004 (9)	0.0047 (9)	0.0011 (9)
C14	0.0483 (13)	0.0514 (12)	0.0367 (11)	−0.0127 (10)	0.0301 (10)	−0.0116 (9)
C15	0.0458 (11)	0.0221 (8)	0.0234 (8)	−0.0009 (8)	0.0145 (8)	0.0052 (7)
C16	0.0773 (17)	0.0288 (10)	0.0509 (13)	−0.0146 (10)	0.0429 (13)	−0.0012 (9)
C17	0.0218 (8)	0.0189 (7)	0.0255 (8)	0.0010 (6)	0.0093 (7)	−0.0014 (6)
N3	0.0214 (9)	0.0169 (8)	0.0247 (9)	0.0047 (6)	0.0092 (7)	0.0066 (7)
C18	0.0249 (10)	0.0268 (10)	0.0260 (10)	0.0008 (8)	0.0109 (8)	0.0009 (8)

C19	0.0286 (13)	0.0305 (17)	0.0260 (13)	0.0004 (12)	0.0085 (10)	0.0001 (10)
O1	0.0237 (8)	0.0311 (8)	0.0342 (8)	0.0008 (6)	0.0130 (7)	−0.0026 (6)
C20	0.0276 (11)	0.0183 (9)	0.0487 (13)	0.0081 (8)	0.0178 (10)	0.0071 (9)
C21	0.0336 (14)	0.0276 (11)	0.0711 (18)	0.0065 (10)	0.0207 (14)	−0.0135 (12)
O2	0.0628 (13)	0.0487 (10)	0.0471 (10)	0.0246 (9)	0.0218 (10)	−0.0080 (8)
N3A	0.025 (5)	0.018 (5)	0.035 (5)	−0.001 (4)	0.006 (4)	−0.002 (4)
C18A	0.013 (4)	0.020 (5)	0.027 (5)	0.003 (4)	0.002 (4)	−0.004 (4)
C19A	0.019 (6)	0.021 (7)	0.033 (7)	−0.005 (5)	0.008 (5)	−0.014 (5)
O1A	0.026 (6)	0.069 (8)	0.053 (7)	0.011 (6)	0.011 (5)	0.021 (6)
C20A	0.046 (6)	0.042 (5)	0.054 (6)	0.008 (5)	0.010 (5)	−0.012 (5)
C21A	0.060 (8)	0.047 (7)	0.077 (8)	0.007 (6)	0.013 (7)	0.004 (6)
O2A	0.077 (10)	0.096 (11)	0.080 (8)	0.027 (8)	0.023 (7)	0.017 (7)
C22	0.0326 (9)	0.0182 (7)	0.0236 (8)	−0.0005 (7)	0.0174 (7)	−0.0018 (6)
C23	0.0374 (10)	0.0228 (8)	0.0195 (8)	0.0007 (7)	0.0126 (7)	−0.0035 (6)
C24	0.0243 (8)	0.0183 (7)	0.0173 (7)	0.0015 (6)	0.0066 (7)	0.0012 (6)
C25	0.0284 (9)	0.0214 (8)	0.0227 (8)	−0.0037 (7)	0.0088 (7)	−0.0026 (6)
C26	0.0259 (9)	0.0329 (9)	0.0197 (8)	−0.0065 (7)	0.0077 (7)	−0.0001 (7)
C27	0.0341 (10)	0.0245 (8)	0.0195 (8)	−0.0002 (7)	0.0094 (7)	0.0060 (6)
C28	0.0398 (11)	0.0338 (10)	0.0249 (9)	−0.0032 (8)	0.0153 (8)	0.0042 (7)
C29	0.0306 (9)	0.0212 (8)	0.0230 (8)	0.0032 (7)	0.0118 (7)	−0.0040 (6)
C30	0.0323 (10)	0.0331 (9)	0.0348 (10)	0.0130 (8)	0.0128 (8)	−0.0021 (8)

Geometric parameters (Å, º)

O3—C26	1.436 (2)	C18—C19	1.502 (4)
O3—H3	0.867 (16)	C18—H18A	0.9900
O4—C28	1.415 (2)	C18—H18B	0.9900
O4—H4	0.863 (16)	C19—O1	1.427 (3)
N1—C8	1.3775 (19)	C19—H19A	0.9900
N1—C7	1.456 (2)	C19—H19B	0.9900
N1—H1N	0.89 (2)	O1—H1	0.873 (16)
N2—C8	1.343 (2)	C20—C21	1.512 (3)
N2—C9	1.349 (2)	C20—H20A	0.9900
N4—C27	1.4595 (19)	C20—H20B	0.9900
N4—C25	1.466 (2)	C21—O2	1.404 (3)
N4—C24	1.467 (2)	C21—H21A	0.9900
C1—C2	1.401 (2)	C21—H21B	0.9900
C1—C6	1.407 (2)	O2—H2	0.865 (17)
C1—C7	1.513 (2)	N3A—C18A	1.461 (9)
C2—C3	1.407 (2)	N3A—C20A	1.466 (9)
C2—C15	1.515 (2)	C18A—C19A	1.507 (10)
C3—C4	1.397 (2)	C18A—H18C	0.9900
C3—C17	1.518 (2)	C18A—H18D	0.9900
C4—C5	1.412 (2)	C19A—O1A	1.433 (10)
C4—C22	1.523 (2)	C19A—H19C	0.9900
C5—C6	1.4010 (19)	C19A—H19D	0.9900
C5—C24	1.521 (2)	O1A—H1A	0.8400
C6—C29	1.518 (2)	C20A—C21A	1.511 (10)

C7—H7A	0.9900	C20A—H20C	0.9900
C7—H7B	0.9900	C20A—H20D	0.9900
C8—C12	1.393 (2)	C21A—O2A	1.411 (10)
C9—C10	1.384 (2)	C21A—H21C	0.9900
C9—C13	1.500 (3)	C21A—H21D	0.9900
C10—C11	1.392 (3)	O2A—H2A	0.8400
C10—H10	0.9500	C22—C23	1.526 (2)
C11—C12	1.381 (2)	C22—H22A	0.9900
C11—C14	1.509 (2)	C22—H22B	0.9900
C12—H12	0.9500	C23—H23A	0.9800
C13—H13A	0.9800	C23—H23B	0.9800
C13—H13B	0.9800	C23—H23C	0.9800
C13—H13C	0.9800	C24—H24A	0.9900
C14—H14A	0.9800	C24—H24B	0.9900
C14—H14B	0.9800	C25—C26	1.507 (2)
C14—H14C	0.9800	C25—H25A	0.9900
C15—C16	1.534 (3)	C25—H25B	0.9900
C15—H15A	0.9900	C26—H26A	0.9900
C15—H15B	0.9900	C26—H26B	0.9900
C16—H16A	0.9800	C27—C28	1.515 (2)
C16—H16B	0.9800	C27—H27A	0.9900
C16—H16C	0.9800	C27—H27B	0.9900
C17—N3	1.460 (2)	C28—H28A	0.9900
C17—N3A	1.468 (8)	C28—H28B	0.9900
C17—H17A	0.9900	C29—C30	1.526 (2)
C17—H17B	0.9900	C29—H29A	0.9900
C17—H17C	0.9900	C29—H29B	0.9900
C17—H17D	0.9900	C30—H30A	0.9800
N3—C20	1.459 (2)	C30—H30B	0.9800
N3—C18	1.469 (2)	C30—H30C	0.9800

C26—O3—H3	106.9 (18)	H19A—C19—H19B	108.2
C28—O4—H4	102.7 (19)	C19—O1—H1	106.4 (17)
C8—N1—C7	121.81 (13)	N3—C20—C21	117.25 (18)
C8—N1—H1N	111.1 (12)	N3—C20—H20A	108.0
C7—N1—H1N	117.4 (12)	C21—C20—H20A	108.0
C8—N2—C9	118.51 (14)	N3—C20—H20B	108.0
C27—N4—C25	113.46 (12)	C21—C20—H20B	108.0
C27—N4—C24	111.47 (13)	H20A—C20—H20B	107.2
C25—N4—C24	113.60 (12)	O2—C21—C20	113.70 (19)
C2—C1—C6	120.77 (13)	O2—C21—H21A	108.8
C2—C1—C7	118.81 (13)	C20—C21—H21A	108.8
C6—C1—C7	120.24 (13)	O2—C21—H21B	108.8
C1—C2—C3	119.50 (13)	C20—C21—H21B	108.8
C1—C2—C15	120.56 (13)	H21A—C21—H21B	107.7
C3—C2—C15	119.94 (14)	C21—O2—H2	104 (2)
C4—C3—C2	120.16 (13)	C18A—N3A—C20A	118.3 (9)
C4—C3—C17	120.50 (13)	C18A—N3A—C17	118.2 (8)

C2—C3—C17	119.29 (13)	C20A—N3A—C17	110.8 (8)
C3—C4—C5	119.95 (13)	N3A—C18A—C19A	111.7 (11)
C3—C4—C22	120.61 (13)	N3A—C18A—H18C	109.3
C5—C4—C22	119.40 (13)	C19A—C18A—H18C	109.3
C6—C5—C4	120.22 (13)	N3A—C18A—H18D	109.3
C6—C5—C24	121.63 (13)	C19A—C18A—H18D	109.3
C4—C5—C24	118.13 (12)	H18C—C18A—H18D	107.9
C5—C6—C1	119.28 (13)	O1A—C19A—C18A	107.1 (10)
C5—C6—C29	121.33 (13)	O1A—C19A—H19C	110.3
C1—C6—C29	119.37 (12)	C18A—C19A—H19C	110.3
N1—C7—C1	108.68 (12)	O1A—C19A—H19D	110.3
N1—C7—H7A	110.0	C18A—C19A—H19D	110.3
C1—C7—H7A	110.0	H19C—C19A—H19D	108.5
N1—C7—H7B	110.0	C19A—O1A—H1A	109.5
C1—C7—H7B	110.0	N3A—C20A—C21A	111.2 (12)
H7A—C7—H7B	108.3	N3A—C20A—H20C	109.4
N2—C8—N1	115.51 (13)	C21A—C20A—H20C	109.4
N2—C8—C12	122.62 (14)	N3A—C20A—H20D	109.4
N1—C8—C12	121.84 (15)	C21A—C20A—H20D	109.4
N2—C9—C10	121.94 (16)	H20C—C20A—H20D	108.0
N2—C9—C13	115.99 (15)	O2A—C21A—C20A	112.8 (12)
C10—C9—C13	122.07 (16)	O2A—C21A—H21C	109.0
C9—C10—C11	119.35 (15)	C20A—C21A—H21C	109.0
C9—C10—H10	120.3	O2A—C21A—H21D	109.0
C11—C10—H10	120.3	C20A—C21A—H21D	109.0
C12—C11—C10	118.89 (15)	H21C—C21A—H21D	107.8
C12—C11—C14	120.34 (17)	C21A—O2A—H2A	109.5
C10—C11—C14	120.76 (15)	C4—C22—C23	111.64 (13)
C11—C12—C8	118.68 (16)	C4—C22—H22A	109.3
C11—C12—H12	120.7	C23—C22—H22A	109.3
C8—C12—H12	120.7	C4—C22—H22B	109.3
C9—C13—H13A	109.5	C23—C22—H22B	109.3
C9—C13—H13B	109.5	H22A—C22—H22B	108.0
H13A—C13—H13B	109.5	C22—C23—H23A	109.5
C9—C13—H13C	109.5	C22—C23—H23B	109.5
H13A—C13—H13C	109.5	H23A—C23—H23B	109.5
H13B—C13—H13C	109.5	C22—C23—H23C	109.5
C11—C14—H14A	109.5	H23A—C23—H23C	109.5
C11—C14—H14B	109.5	H23B—C23—H23C	109.5
H14A—C14—H14B	109.5	N4—C24—C5	112.30 (13)
C11—C14—H14C	109.5	N4—C24—H24A	109.1
H14A—C14—H14C	109.5	C5—C24—H24A	109.1
H14B—C14—H14C	109.5	N4—C24—H24B	109.1
C2—C15—C16	112.70 (16)	C5—C24—H24B	109.1
C2—C15—H15A	109.1	H24A—C24—H24B	107.9
C16—C15—H15A	109.1	N4—C25—C26	113.08 (14)
C2—C15—H15B	109.1	N4—C25—H25A	109.0
C16—C15—H15B	109.1	C26—C25—H25A	109.0

H15A—C15—H15B	107.8	N4—C25—H25B	109.0
C15—C16—H16A	109.5	C26—C25—H25B	109.0
C15—C16—H16B	109.5	H25A—C25—H25B	107.8
H16A—C16—H16B	109.5	O3—C26—C25	108.92 (13)
C15—C16—H16C	109.5	O3—C26—H26A	109.9
H16A—C16—H16C	109.5	C25—C26—H26A	109.9
H16B—C16—H16C	109.5	O3—C26—H26B	109.9
N3—C17—C3	112.70 (14)	C25—C26—H26B	109.9
N3A—C17—C3	106.9 (5)	H26A—C26—H26B	108.3
N3—C17—H17A	109.1	N4—C27—C28	111.58 (14)
C3—C17—H17A	109.1	N4—C27—H27A	109.3
N3—C17—H17B	109.1	C28—C27—H27A	109.3
C3—C17—H17B	109.1	N4—C27—H27B	109.3
H17A—C17—H17B	107.8	C28—C27—H27B	109.3
N3A—C17—H17C	110.3	H27A—C27—H27B	108.0
C3—C17—H17C	110.3	O4—C28—C27	112.57 (14)
N3A—C17—H17D	110.3	O4—C28—H28A	109.1
C3—C17—H17D	110.3	C27—C28—H28A	109.1
H17C—C17—H17D	108.6	O4—C28—H28B	109.1
C20—N3—C17	112.80 (16)	C27—C28—H28B	109.1
C20—N3—C18	114.82 (16)	H28A—C28—H28B	107.8
C17—N3—C18	114.22 (15)	C6—C29—C30	112.38 (14)
N3—C18—C19	111.71 (17)	C6—C29—H29A	109.1
N3—C18—H18A	109.3	C30—C29—H29A	109.1
C19—C18—H18A	109.3	C6—C29—H29B	109.1
N3—C18—H18B	109.3	C30—C29—H29B	109.1
C19—C18—H18B	109.3	H29A—C29—H29B	107.9
H18A—C18—H18B	107.9	C29—C30—H30A	109.5
O1—C19—C18	109.9 (2)	C29—C30—H30B	109.5
O1—C19—H19A	109.7	H30A—C30—H30B	109.5
C18—C19—H19A	109.7	C29—C30—H30C	109.5
O1—C19—H19B	109.7	H30A—C30—H30C	109.5
C18—C19—H19B	109.7	H30B—C30—H30C	109.5

C6—C1—C2—C3	3.9 (2)	N2—C8—C12—C11	1.1 (2)
C7—C1—C2—C3	179.11 (14)	N1—C8—C12—C11	179.13 (15)
C6—C1—C2—C15	−176.63 (15)	C1—C2—C15—C16	−88.89 (19)
C7—C1—C2—C15	−1.5 (2)	C3—C2—C15—C16	90.55 (19)
C1—C2—C3—C4	−2.3 (2)	C4—C3—C17—N3	−99.98 (17)
C15—C2—C3—C4	178.26 (15)	C2—C3—C17—N3	77.67 (18)
C1—C2—C3—C17	−179.95 (14)	C4—C3—C17—N3A	−77.6 (6)
C15—C2—C3—C17	0.6 (2)	C2—C3—C17—N3A	100.1 (6)
C2—C3—C4—C5	−0.8 (2)	C3—C17—N3—C20	−167.61 (15)
C17—C3—C4—C5	176.79 (14)	C3—C17—N3—C18	58.88 (19)
C2—C3—C4—C22	176.88 (14)	C20—N3—C18—C19	83.7 (3)
C17—C3—C4—C22	−5.5 (2)	C17—N3—C18—C19	−143.8 (2)
C3—C4—C5—C6	2.4 (2)	N3—C18—C19—O1	173.3 (2)
C22—C4—C5—C6	−175.35 (14)	C17—N3—C20—C21	−52.4 (2)

C3—C4—C5—C24	−176.09 (14)	C18—N3—C20—C21	80.8 (2)
C22—C4—C5—C24	6.2 (2)	N3—C20—C21—O2	−56.8 (3)
C4—C5—C6—C1	−0.8 (2)	C3—C17—N3A—C18A	−61.6 (11)
C24—C5—C6—C1	177.64 (14)	C3—C17—N3A—C20A	157.2 (8)
C4—C5—C6—C29	177.43 (14)	C20A—N3A—C18A—C19A	−58.0 (19)
C24—C5—C6—C29	−4.1 (2)	C17—N3A—C18A—C19A	163.7 (14)
C2—C1—C6—C5	−2.4 (2)	N3A—C18A—C19A—O1A	−45 (3)
C7—C1—C6—C5	−177.49 (14)	C18A—N3A—C20A—C21A	−77.9 (15)
C2—C1—C6—C29	179.35 (14)	C17—N3A—C20A—C21A	63.2 (15)
C7—C1—C6—C29	4.2 (2)	N3A—C20A—C21A—O2A	70 (2)
C8—N1—C7—C1	165.06 (14)	C3—C4—C22—C23	−85.93 (18)
C2—C1—C7—N1	−85.21 (17)	C5—C4—C22—C23	91.81 (17)
C6—C1—C7—N1	90.00 (17)	C27—N4—C24—C5	−162.08 (12)
C9—N2—C8—N1	−178.75 (14)	C25—N4—C24—C5	68.22 (16)
C9—N2—C8—C12	−0.6 (2)	C6—C5—C24—N4	−108.97 (16)
C7—N1—C8—N2	−161.06 (14)	C4—C5—C24—N4	69.50 (18)
C7—N1—C8—C12	20.8 (2)	C27—N4—C25—C26	132.61 (15)
C8—N2—C9—C10	−0.4 (2)	C24—N4—C25—C26	−98.70 (15)
C8—N2—C9—C13	179.58 (15)	N4—C25—C26—O3	−63.25 (17)
N2—C9—C10—C11	0.9 (2)	C25—N4—C27—C28	−66.62 (18)
C13—C9—C10—C11	−179.07 (17)	C24—N4—C27—C28	163.61 (13)
C9—C10—C11—C12	−0.4 (2)	N4—C27—C28—O4	−50.53 (19)
C9—C10—C11—C14	179.00 (16)	C5—C6—C29—C30	−88.66 (18)
C10—C11—C12—C8	−0.6 (2)	C1—C6—C29—C30	89.57 (18)
C14—C11—C12—C8	−179.97 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2*A*—H2*A*···O4^{i}	0.84	1.99	2.763 (18)	152
O1*A*—H1*A*···N2^{ii}	0.84	1.99	2.832 (12)	178
C18*A*—H18*D*···O2*A*	0.99	2.46	3.08 (2)	120
O2—H2···O3^{i}	0.87 (2)	1.99 (2)	2.828 (2)	162 (3)
O1—H1···N2^{ii}	0.87 (2)	1.89 (2)	2.7449 (19)	167 (3)
N1—H1*N*···O1^{ii}	0.89 (2)	2.19 (2)	3.014 (2)	152.0 (17)
C22—H22*A*···N4	0.99	2.40	3.152 (2)	132
C18—H18*A*···O2	0.99	2.39	3.106 (3)	128
C15—H15*A*···N3	0.99	2.54	3.282 (3)	131
C13—H13*A*···O2*A*^{iii}	0.98	2.33	3.220 (14)	151
C10—H10···O4^{iv}	0.95	2.45	3.365 (2)	161
O4—H4···O3	0.86 (2)	2.12 (2)	2.9200 (18)	155 (3)
O3—H3···O1*A*	0.87 (2)	1.93 (2)	2.727 (13)	152 (3)
O3—H3···O1	0.87 (2)	1.86 (2)	2.7156 (19)	172 (3)

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