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2-[(2-{Bis[2-(2-hydroxy-5-nitrobenzylideneamino)ethyl]amino}ethyl)iminomethyl]-4-nitrophenol acetonitrile monosolvate

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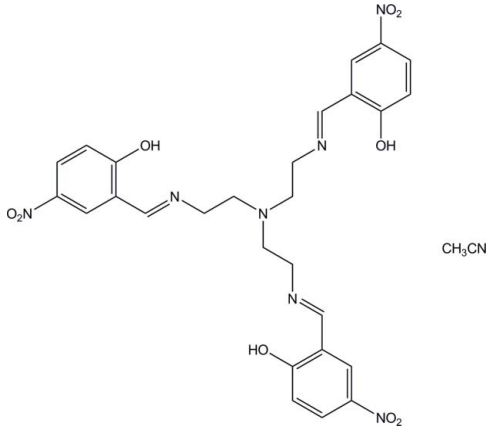
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.005 Å; R factor = 0.062; wR factor = 0.175; data-to-parameter ratio = 13.6.

In the title compound, $C_{27}H_{27}N_7O_9\cdot CH_3CN$, the three nitro groups of the polydentate tripodal Schiff base are located approximately parallel to their respective carrier benzene rings, making dihedral angles of 3.9 (4), 5.0 (4) and 6.3 (4)°. Intramolecular O—H···N hydrogen bonds between the hydroxy O atoms and the imine N atoms, with O···N distances in the range 2.607 (3)–2.665 (3) Å, form nearly planar six-membered rings. In the crystal, weak intermolecular C—H···O and C—H···N hydrogen bonds occur and several intra- and intermolecular π–π interactions are present between adjacent benzene rings, with a shortest centroid–centroid distance of 3.507 (2) Å.

Related literature

For the crystal structure of tris{2-[(5-bromosalicylidene)amino]ethyl}amine, see: Kanesato *et al.* (2001).



Experimental

Crystal data

$C_{27}H_{27}N_7O_9\cdot C_2H_3N$
M_r = 634.61
Triclinic, $P\overline{1}$
a = 10.6097 (9) Å
b = 11.8168 (9) Å
c = 12.8003 (10) Å
α = 79.054 (2)°
β = 68.293 (2)°
γ = 88.527 (2)°
V = 1462.1 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 200 K
0.32 × 0.13 × 0.11 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.846, T_{max} = 0.988
9227 measured reflections
5688 independent reflections
3102 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.175
S = 1.03
5688 reflections
419 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*O*···N2	0.84	1.87	2.627 (3)	149
O4—H4*O*···N4	0.84	1.92	2.665 (3)	147
O7—H7*O*···N6	0.84	1.85	2.607 (3)	149
C1—H1*A*···N8^{i}	0.99	2.56	3.369 (5)	139
C1—H1*B*···O4^{ii}	0.99	2.41	3.290 (4)	148
C2—H2*B*···O2^{iii}	0.99	2.44	3.300 (4)	146
C3—H3···O7	0.95	2.53	3.297 (4)	138
C6—H6···N8^{iv}	0.95	2.49	3.360 (5)	153
C9—H9···O7	0.95	2.55	3.328 (4)	139
C11—H11*A*···O5^{v}	0.99	2.40	3.331 (4)	157
C12—H12···O5^{v}	0.95	2.54	3.339 (4)	142
C16—H16···O6^{vi}	0.95	2.51	3.330 (4)	145
C25—H25···O9^{vii}	0.95	2.48	3.359 (4)	153

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; 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 *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2631).

References

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Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

Acta Cryst. (2010). E**66**, o3222–o3223 [https://doi.org/10.1107/S1600536810047185]

2-[(2-{Bis[2-(2-hydroxy-5-nitrobenzylideneamino)ethyl]amino}ethyl)iminomethyl]-4-nitrophenol acetonitrile monosolvate

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S1. Comment

The title compound, $C_{27}H_{27}N_7O_9.CH_3CN$, consists of a polydentate tripodal Schiff base and an acetonitrile solvent molecule (Fig. 1). The Schiff base can act as a tribasic hexa- or heptadentate ligand, that is, the N_3O_3 or N_4O_3 donor atoms can coordinate to a metal ion or metal ions. In the crystal structure, the Schiff base reveals an approximate threefold axis, when viewed down the apical amine N atom (N1) through the plane formed by the atoms C1, C10 and C19, and three nitro groups are located approximately parallel to their respective carrier benzene rings. The N—C bond lengths and the C—N—C bond angles indicate that the apical N1 atom is sp^3-hybridized [d(N1—C) = 1.470 (4)–1.480 (4) Å; <C—N1—C = 109.5 (2)–111.7 (2)°] and the other imine N atoms (N2, N4, N6) are sp^2-hybridized [d(N═C) = 1.291 (4)–1.307 (4) Å and d(N—C) = 1.460 (4)–1.469 (4) Å; <C—N—C = 122.7 (3)–123.9 (3)°]. The compound displays strong intramolecular O—H···N hydrogen bonds between the hydroxy O atoms and the imine N atoms with d(O···N) = 2.607 (3)–2.665 (3) Å thus forming a nearly planar six-membered ring (Fig. 2, Table 1). There are also weak intermolecular C—H···O and C—H···N hydrogen bonds with d(C···O) = 3.290 (4)–3.359 (4) Å and d(C···N) = 3.360 (5)–3.369 (5) Å. Moreover, several intra- and intermolecular π–π interactions between the adjacent benzene rings are present, with a shortest ring centroid-centroid distance of 3.507 (2) Å, and the dihedral angle between the ring planes is 5.1 (2)°.

S2. Experimental

Tris(2-aminoethyl)amine (0.7305 g, 4.995 mmol) and 5-nitrosalicylaldehyde (2.5077 g, 15.005 mmol) in EtOH (30 ml) were stirred for 3 h at room temperature. The precipitate was then separated by filtration, washed with ether, and dried at 50 °C, to give a yellow powder (2.9135 g). Crystals suitable for X-ray analysis were obtained by slow evaporation from a CH_3CN solution.

S3. Refinement

H atoms were positioned geometrically and allowed to ride on their respective parent atoms [C—H = 0.95 Å (CH), 0.99 Å (CH_2) or 0.98 Å (CH_3) and O—H = 0.84 Å, and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C, O)].

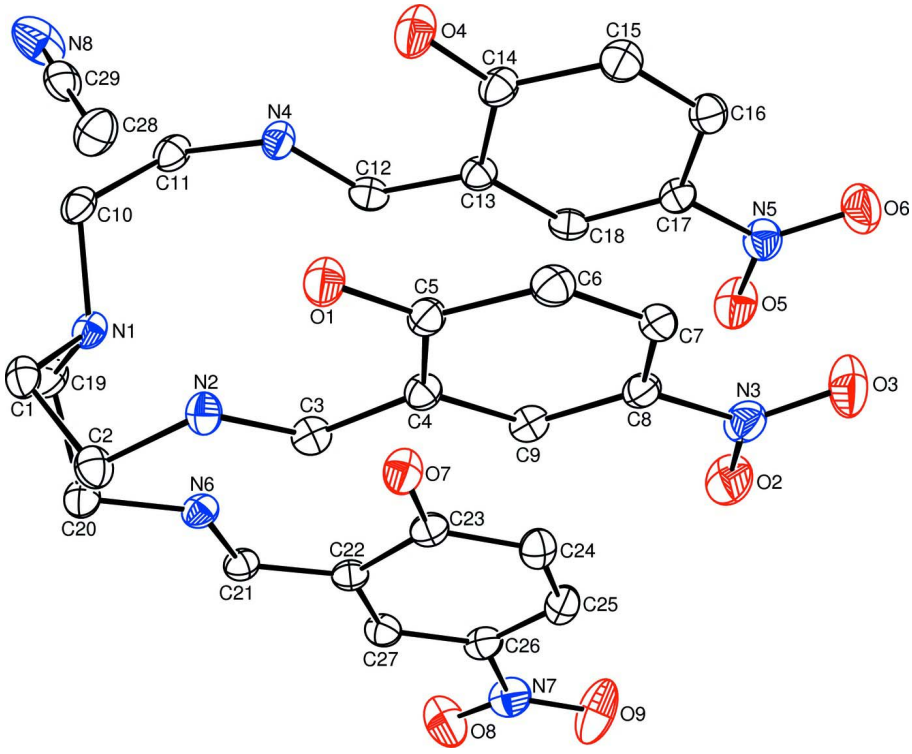

Figure 1

The structure of the title compound, with displacement ellipsoids drawn at the 40% probability level. H atoms are omitted for clarity.

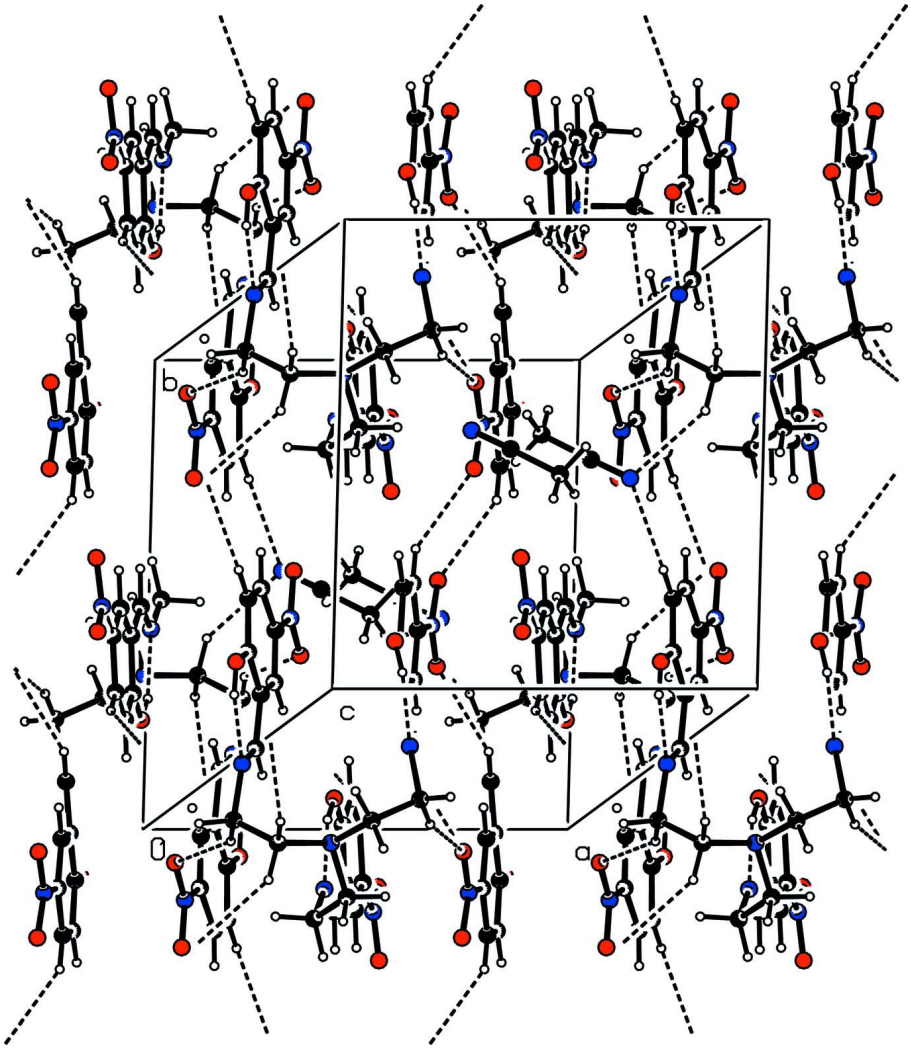

Figure 2

View of the unit-cell contents of the title compound. Hydrogen-bond interactions are drawn with dashed lines.

2-[(2-{Bis[2-(2-hydroxy-5- nitrobenzylideneamino)ethyl]amino}ethyl)iminomethyl]-4-nitrophenol acetonitrile monosolvate

Crystal data

$C_{27}H_{27}N_7O_9 \cdot C_2H_3N$
$M_r = 634.61$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 10.6097\ (9)$ Å
$b = 11.8168\ (9)$ Å
$c = 12.8003\ (10)$ Å
$\alpha = 79.054\ (2)°$
$\beta = 68.293\ (2)°$
$\gamma = 88.527\ (2)°$
$V = 1462.1\ (2)$ Å^3
$Z = 2$
$F(000) = 664$
$D_x = 1.441$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2046 reflections
$\theta = 2.2–25.2°$
$\mu = 0.11$ mm^{-1}
$T = 200$ K
Block, yellow
0.32 × 0.13 × 0.11 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.846, T_{max} = 0.988
9227 measured reflections
5688 independent reflections
3102 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 26.0°, θ_{min} = 2.1°
h = −13→13
k = −14→13
l = −15→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.175
S = 1.03
5688 reflections
419 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0681P)^2 + 0.0868P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness 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 threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	1.1867 (3)	−0.08637 (18)	0.09431 (18)	0.0448 (6)
H1O	1.1937	−0.0154	0.0928	0.067*
O2	0.8123 (3)	−0.2527 (2)	0.6061 (2)	0.0621 (8)
O3	0.8574 (3)	−0.4164 (2)	0.5510 (2)	0.0703 (8)
O4	0.8089 (3)	−0.13330 (19)	0.10667 (19)	0.0595 (8)
H4O	0.7975	−0.0622	0.0925	0.089*
O5	0.4797 (3)	−0.2368 (2)	0.6310 (2)	0.0608 (8)
O6	0.4974 (3)	−0.4093 (2)	0.5958 (2)	0.0658 (8)
O7	0.7816 (3)	0.09156 (19)	0.4737 (2)	0.0502 (7)
H7O	0.8186	0.1513	0.4252	0.075*
O8	0.4783 (3)	0.3083 (3)	0.9032 (2)	0.0830 (10)
O9	0.4522 (3)	0.1281 (3)	0.9805 (2)	0.0913 (11)
N1	0.9339 (3)	0.2871 (2)	0.1376 (2)	0.0324 (6)
N2	1.1352 (3)	0.1102 (2)	0.1657 (2)	0.0352 (6)
N3	0.8708 (3)	−0.3096 (3)	0.5314 (3)	0.0458 (7)
N4	0.7479 (3)	0.0770 (2)	0.1510 (2)	0.0354 (7)

N5	0.5246 (3)	−0.3041 (3)	0.5642 (3)	0.0455 (7)
N6	0.8372 (3)	0.3109 (2)	0.3871 (2)	0.0361 (7)
N7	0.4978 (3)	0.2066 (3)	0.8974 (3)	0.0566 (9)
C1	1.0834 (3)	0.3037 (3)	0.0811 (3)	0.0381 (8)
H1A	1.1088	0.3867	0.0680	0.046*
H1B	1.1120	0.2813	0.0052	0.046*
C2	1.1598 (3)	0.2351 (2)	0.1490 (3)	0.0381 (8)
H2A	1.2583	0.2544	0.1084	0.046*
H2B	1.1319	0.2575	0.2248	0.046*
C3	1.0620 (3)	0.0478 (3)	0.2639 (3)	0.0336 (8)
H3	1.0176	0.0862	0.3256	0.040*
C4	1.0438 (3)	−0.0738 (3)	0.2849 (3)	0.0308 (7)
C5	1.1155 (3)	−0.1371 (3)	0.1946 (3)	0.0324 (8)
C6	1.1040 (3)	−0.2603 (3)	0.2275 (3)	0.0389 (8)
H6	1.1518	−0.3051	0.1721	0.047*
C7	1.0271 (3)	−0.3153 (3)	0.3355 (3)	0.0377 (8)
H7	1.0223	−0.3971	0.3547	0.045*
C8	0.9544 (3)	−0.2504 (3)	0.4190 (3)	0.0343 (8)
C9	0.9646 (3)	−0.1321 (3)	0.3945 (3)	0.0323 (7)
H9	0.9175	−0.0898	0.4524	0.039*
C10	0.8749 (3)	0.2637 (3)	0.0560 (3)	0.0366 (8)
H10A	0.9375	0.2169	0.0044	0.044*
H10B	0.8665	0.3378	0.0083	0.044*
C11	0.7373 (3)	0.2011 (3)	0.1132 (3)	0.0381 (8)
H11A	0.6827	0.2347	0.1803	0.046*
H11B	0.6896	0.2122	0.0589	0.046*
C12	0.7036 (3)	0.0251 (3)	0.2570 (3)	0.0345 (8)
H12	0.6720	0.0712	0.3140	0.041*
C13	0.6991 (3)	−0.0966 (3)	0.2943 (3)	0.0317 (7)
C14	0.7506 (4)	−0.1716 (3)	0.2118 (3)	0.0388 (8)
C15	0.7267 (4)	−0.2933 (3)	0.2577 (3)	0.0448 (9)
H15	0.7619	−0.3456	0.2071	0.054*
C16	0.6556 (3)	−0.3364 (3)	0.3706 (3)	0.0401 (8)
H16	0.6389	−0.4173	0.3977	0.048*
C17	0.6071 (3)	−0.2591 (3)	0.4471 (3)	0.0329 (8)
C18	0.6295 (3)	−0.1425 (3)	0.4103 (3)	0.0329 (8)
H18	0.5977	−0.0925	0.4637	0.039*
C19	0.8723 (4)	0.3882 (3)	0.1849 (3)	0.0386 (8)
H19A	0.7726	0.3806	0.2069	0.046*
H19B	0.9062	0.4583	0.1245	0.046*
C20	0.9027 (4)	0.4034 (3)	0.2889 (3)	0.0394 (8)
H20A	1.0021	0.4043	0.2694	0.047*
H20B	0.8703	0.4785	0.3093	0.047*
C21	0.7671 (3)	0.3281 (3)	0.4904 (3)	0.0359 (8)
H21	0.7578	0.4052	0.5025	0.043*
C22	0.7048 (3)	0.2381 (3)	0.5843 (3)	0.0344 (8)
C23	0.7173 (3)	0.1188 (3)	0.5718 (3)	0.0378 (8)
C24	0.6567 (3)	0.0327 (3)	0.6723 (3)	0.0436 (9)

H24	0.6650	−0.0463	0.6666	0.052*
C25	0.5872 (3)	0.0604 (3)	0.7763 (3)	0.0446 (9)
H25	0.5484	0.0012	0.8421	0.054*
C26	0.5731 (3)	0.1773 (3)	0.7858 (3)	0.0419 (9)
C27	0.6304 (3)	0.2637 (3)	0.6931 (3)	0.0400 (8)
H27	0.6201	0.3419	0.7018	0.048*
N8	0.2620 (4)	0.5168 (3)	0.1139 (4)	0.0889 (14)
C28	0.4516 (4)	0.4093 (4)	0.1626 (4)	0.0712 (13)
H28A	0.4123	0.3395	0.2205	0.107*
H28B	0.4975	0.4587	0.1924	0.107*
H28C	0.5173	0.3877	0.0931	0.107*
C29	0.3459 (4)	0.4707 (3)	0.1358 (3)	0.0549 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0633 (17)	0.0335 (13)	0.0328 (14)	0.0035 (13)	−0.0115 (12)	−0.0087 (11)
O2	0.072 (2)	0.0692 (18)	0.0320 (15)	−0.0026 (15)	−0.0059 (13)	−0.0066 (14)
O3	0.084 (2)	0.0460 (17)	0.0602 (19)	−0.0093 (15)	−0.0123 (16)	0.0112 (15)
O4	0.094 (2)	0.0395 (14)	0.0328 (15)	0.0145 (15)	−0.0087 (14)	−0.0099 (12)
O5	0.0694 (19)	0.0594 (17)	0.0357 (15)	0.0137 (14)	0.0020 (13)	−0.0127 (14)
O6	0.084 (2)	0.0493 (17)	0.0494 (17)	−0.0176 (15)	−0.0115 (15)	0.0005 (14)
O7	0.0633 (18)	0.0406 (14)	0.0415 (15)	0.0102 (13)	−0.0127 (13)	−0.0110 (12)
O8	0.103 (3)	0.076 (2)	0.0479 (18)	−0.0262 (19)	0.0065 (16)	−0.0275 (17)
O9	0.087 (2)	0.101 (2)	0.0378 (17)	0.029 (2)	0.0163 (16)	0.0149 (17)
N1	0.0443 (17)	0.0281 (14)	0.0238 (14)	0.0091 (12)	−0.0097 (13)	−0.0097 (12)
N2	0.0330 (16)	0.0322 (15)	0.0364 (16)	0.0019 (13)	−0.0102 (13)	−0.0029 (13)
N3	0.0440 (19)	0.054 (2)	0.0355 (18)	−0.0034 (16)	−0.0133 (15)	−0.0012 (16)
N4	0.0418 (17)	0.0320 (15)	0.0285 (15)	0.0032 (13)	−0.0096 (13)	−0.0040 (13)
N5	0.0449 (19)	0.0459 (19)	0.0407 (18)	0.0015 (16)	−0.0109 (15)	−0.0072 (16)
N6	0.0416 (17)	0.0404 (16)	0.0278 (15)	0.0046 (13)	−0.0122 (13)	−0.0117 (13)
N7	0.041 (2)	0.080 (3)	0.039 (2)	−0.0021 (19)	−0.0043 (16)	−0.010 (2)
C1	0.046 (2)	0.0312 (18)	0.0274 (18)	−0.0015 (16)	−0.0009 (16)	−0.0082 (15)
C2	0.042 (2)	0.0310 (18)	0.039 (2)	0.0011 (16)	−0.0113 (17)	−0.0085 (16)
C3	0.0307 (19)	0.0388 (19)	0.0314 (18)	0.0094 (15)	−0.0090 (15)	−0.0130 (16)
C4	0.0327 (19)	0.0322 (18)	0.0291 (18)	0.0045 (15)	−0.0124 (15)	−0.0086 (15)
C5	0.0358 (19)	0.0339 (18)	0.0292 (19)	0.0031 (15)	−0.0150 (16)	−0.0044 (16)
C6	0.045 (2)	0.0383 (19)	0.038 (2)	0.0089 (17)	−0.0175 (17)	−0.0149 (17)
C7	0.050 (2)	0.0295 (17)	0.042 (2)	0.0021 (16)	−0.0262 (19)	−0.0080 (16)
C8	0.0319 (19)	0.042 (2)	0.0293 (18)	−0.0027 (16)	−0.0135 (15)	−0.0030 (16)
C9	0.0291 (18)	0.0388 (19)	0.0294 (18)	0.0076 (15)	−0.0106 (15)	−0.0087 (15)
C10	0.050 (2)	0.0351 (18)	0.0230 (17)	0.0054 (16)	−0.0114 (16)	−0.0071 (15)
C11	0.048 (2)	0.0372 (19)	0.0299 (18)	0.0109 (17)	−0.0148 (17)	−0.0092 (16)
C12	0.0306 (19)	0.041 (2)	0.036 (2)	0.0067 (15)	−0.0126 (16)	−0.0181 (17)
C13	0.0313 (19)	0.0330 (18)	0.0311 (18)	0.0050 (15)	−0.0101 (15)	−0.0106 (15)
C14	0.045 (2)	0.040 (2)	0.032 (2)	0.0098 (17)	−0.0143 (17)	−0.0088 (17)
C15	0.059 (2)	0.038 (2)	0.038 (2)	0.0122 (18)	−0.0147 (19)	−0.0171 (17)
C16	0.045 (2)	0.0348 (19)	0.043 (2)	0.0058 (16)	−0.0190 (18)	−0.0093 (17)

C17	0.0287 (19)	0.0395 (19)	0.0290 (18)	0.0020 (15)	−0.0091 (15)	−0.0068 (16)
C18	0.0311 (19)	0.0385 (19)	0.0328 (19)	0.0054 (15)	−0.0123 (15)	−0.0152 (16)
C19	0.057 (2)	0.0314 (18)	0.0251 (18)	0.0093 (16)	−0.0133 (16)	−0.0057 (15)
C20	0.050 (2)	0.0349 (19)	0.0307 (19)	0.0044 (17)	−0.0093 (16)	−0.0114 (16)
C21	0.040 (2)	0.0373 (19)	0.034 (2)	0.0062 (16)	−0.0170 (17)	−0.0110 (16)
C22	0.0310 (19)	0.041 (2)	0.036 (2)	0.0057 (15)	−0.0157 (16)	−0.0123 (17)
C23	0.0314 (19)	0.046 (2)	0.039 (2)	0.0077 (16)	−0.0170 (17)	−0.0101 (18)
C24	0.037 (2)	0.042 (2)	0.050 (2)	0.0075 (17)	−0.0165 (18)	−0.0051 (19)
C25	0.030 (2)	0.053 (2)	0.044 (2)	0.0003 (17)	−0.0125 (17)	0.0064 (19)
C26	0.032 (2)	0.061 (2)	0.032 (2)	0.0016 (18)	−0.0102 (16)	−0.0096 (18)
C27	0.037 (2)	0.047 (2)	0.037 (2)	−0.0007 (17)	−0.0115 (17)	−0.0134 (18)
N8	0.091 (3)	0.051 (2)	0.151 (4)	0.020 (2)	−0.069 (3)	−0.033 (2)
C28	0.069 (3)	0.075 (3)	0.066 (3)	0.011 (3)	−0.029 (2)	0.000 (2)
C29	0.063 (3)	0.039 (2)	0.063 (3)	0.003 (2)	−0.022 (2)	−0.014 (2)

Geometric parameters (Å, º)

O1—C5	1.262 (4)	C9—H9	0.9500
O1—H1O	0.8400	C10—C11	1.507 (4)
O2—N3	1.239 (3)	C10—H10A	0.9900
O3—N3	1.240 (4)	C10—H10B	0.9900
O4—C14	1.252 (4)	C11—H11A	0.9900
O4—H4O	0.8400	C11—H11B	0.9900
O5—N5	1.236 (3)	C12—C13	1.422 (4)
O6—N5	1.238 (3)	C12—H12	0.9500
O7—C23	1.286 (4)	C13—C18	1.396 (4)
O7—H7O	0.8400	C13—C14	1.450 (4)
O8—N7	1.227 (4)	C14—C15	1.436 (4)
O9—N7	1.222 (4)	C15—C16	1.361 (4)
N1—C10	1.470 (4)	C15—H15	0.9500
N1—C19	1.471 (4)	C16—C17	1.414 (4)
N1—C1	1.480 (4)	C16—H16	0.9500
N2—C3	1.297 (4)	C17—C18	1.366 (4)
N2—C2	1.466 (4)	C18—H18	0.9500
N3—C8	1.432 (4)	C19—C20	1.523 (4)
N4—C12	1.291 (4)	C19—H19A	0.9900
N4—C11	1.469 (4)	C19—H19B	0.9900
N5—C17	1.431 (4)	C20—H20A	0.9900
N6—C21	1.307 (4)	C20—H20B	0.9900
N6—C20	1.460 (4)	C21—C22	1.406 (4)
N7—C26	1.458 (4)	C21—H21	0.9500
C1—C2	1.514 (4)	C22—C27	1.410 (4)
C1—H1A	0.9900	C22—C23	1.446 (4)
C1—H1B	0.9900	C23—C24	1.424 (5)
C2—H2A	0.9900	C24—C25	1.359 (5)
C2—H2B	0.9900	C24—H24	0.9500
C3—C4	1.416 (4)	C25—C26	1.410 (5)
C3—H3	0.9500	C25—H25	0.9500

C4—C9	1.393 (4)	C26—C27	1.364 (4)
C4—C5	1.457 (4)	C27—H27	0.9500
C5—C6	1.432 (4)	N8—C29	1.126 (5)
C6—C7	1.360 (4)	C28—C29	1.432 (5)
C6—H6	0.9500	C28—H28A	0.9800
C7—C8	1.413 (4)	C28—H28B	0.9800
C7—H7	0.9500	C28—H28C	0.9800
C8—C9	1.372 (4)		

C5—O1—H1O	109.5	H11A—C11—H11B	107.9
C14—O4—H4O	109.5	N4—C12—C13	124.4 (3)
C23—O7—H7O	109.5	N4—C12—H12	117.8
C10—N1—C19	109.5 (2)	C13—C12—H12	117.8
C10—N1—C1	110.2 (2)	C18—C13—C12	118.3 (3)
C19—N1—C1	111.7 (2)	C18—C13—C14	120.7 (3)
C3—N2—C2	122.7 (3)	C12—C13—C14	120.6 (3)
O2—N3—O3	121.7 (3)	O4—C14—C15	121.7 (3)
O2—N3—C8	119.1 (3)	O4—C14—C13	122.4 (3)
O3—N3—C8	119.2 (3)	C15—C14—C13	115.9 (3)
C12—N4—C11	123.9 (3)	C16—C15—C14	122.6 (3)
O5—N5—O6	121.5 (3)	C16—C15—H15	118.7
O5—N5—C17	119.3 (3)	C14—C15—H15	118.7
O6—N5—C17	119.2 (3)	C15—C16—C17	119.0 (3)
C21—N6—C20	123.8 (3)	C15—C16—H16	120.5
O9—N7—O8	122.7 (3)	C17—C16—H16	120.5
O9—N7—C26	118.5 (4)	C18—C17—C16	121.7 (3)
O8—N7—C26	118.8 (3)	C18—C17—N5	119.3 (3)
N1—C1—C2	113.8 (3)	C16—C17—N5	118.9 (3)
N1—C1—H1A	108.8	C17—C18—C13	120.0 (3)
C2—C1—H1A	108.8	C17—C18—H18	120.0
N1—C1—H1B	108.8	C13—C18—H18	120.0
C2—C1—H1B	108.8	N1—C19—C20	113.4 (3)
H1A—C1—H1B	107.7	N1—C19—H19A	108.9
N2—C2—C1	112.5 (3)	C20—C19—H19A	108.9
N2—C2—H2A	109.1	N1—C19—H19B	108.9
C1—C2—H2A	109.1	C20—C19—H19B	108.9
N2—C2—H2B	109.1	H19A—C19—H19B	107.7
C1—C2—H2B	109.1	N6—C20—C19	111.5 (3)
H2A—C2—H2B	107.8	N6—C20—H20A	109.3
N2—C3—C4	124.2 (3)	C19—C20—H20A	109.3
N2—C3—H3	117.9	N6—C20—H20B	109.3
C4—C3—H3	117.9	C19—C20—H20B	109.3
C9—C4—C3	119.2 (3)	H20A—C20—H20B	108.0
C9—C4—C5	120.8 (3)	N6—C21—C22	123.3 (3)
C3—C4—C5	119.9 (3)	N6—C21—H21	118.4
O1—C5—C6	122.0 (3)	C22—C21—H21	118.4
O1—C5—C4	122.0 (3)	C21—C22—C27	119.9 (3)
C6—C5—C4	116.0 (3)	C21—C22—C23	121.0 (3)

C7—C6—C5	122.2 (3)	C27—C22—C23	119.1 (3)
C7—C6—H6	118.9	O7—C23—C24	121.3 (3)
C5—C6—H6	118.9	O7—C23—C22	121.2 (3)
C6—C7—C8	119.9 (3)	C24—C23—C22	117.5 (3)
C6—C7—H7	120.0	C25—C24—C23	121.9 (3)
C8—C7—H7	120.0	C25—C24—H24	119.1
C9—C8—C7	121.0 (3)	C23—C24—H24	119.1
C9—C8—N3	119.8 (3)	C24—C25—C26	119.6 (3)
C7—C8—N3	119.3 (3)	C24—C25—H25	120.2
C8—C9—C4	120.1 (3)	C26—C25—H25	120.2
C8—C9—H9	119.9	C27—C26—C25	121.3 (3)
C4—C9—H9	119.9	C27—C26—N7	119.3 (3)
N1—C10—C11	113.3 (3)	C25—C26—N7	119.5 (3)
N1—C10—H10A	108.9	C26—C27—C22	120.6 (3)
C11—C10—H10A	108.9	C26—C27—H27	119.7
N1—C10—H10B	108.9	C22—C27—H27	119.7
C11—C10—H10B	108.9	C29—C28—H28A	109.5
H10A—C10—H10B	107.7	C29—C28—H28B	109.5
N4—C11—C10	111.9 (3)	H28A—C28—H28B	109.5
N4—C11—H11A	109.2	C29—C28—H28C	109.5
C10—C11—H11A	109.2	H28A—C28—H28C	109.5
N4—C11—H11B	109.2	H28B—C28—H28C	109.5
C10—C11—H11B	109.2	N8—C29—C28	178.4 (4)

C10—N1—C1—C2	−131.8 (3)	C13—C14—C15—C16	2.5 (5)
C19—N1—C1—C2	106.1 (3)	C14—C15—C16—C17	−2.2 (5)
C3—N2—C2—C1	−107.8 (3)	C15—C16—C17—C18	0.1 (5)
N1—C1—C2—N2	62.4 (3)	C15—C16—C17—N5	175.8 (3)
C2—N2—C3—C4	−175.4 (3)	O5—N5—C17—C18	−2.8 (4)
N2—C3—C4—C9	178.9 (3)	O6—N5—C17—C18	175.5 (3)
N2—C3—C4—C5	3.2 (5)	O5—N5—C17—C16	−178.6 (3)
C9—C4—C5—O1	179.2 (3)	O6—N5—C17—C16	−0.3 (4)
C3—C4—C5—O1	−5.1 (5)	C16—C17—C18—C13	1.6 (5)
C9—C4—C5—C6	−2.9 (4)	N5—C17—C18—C13	−174.1 (3)
C3—C4—C5—C6	172.7 (3)	C12—C13—C18—C17	171.2 (3)
O1—C5—C6—C7	−179.9 (3)	C14—C13—C18—C17	−1.2 (4)
C4—C5—C6—C7	2.3 (4)	C10—N1—C19—C20	167.1 (3)
C5—C6—C7—C8	0.5 (5)	C1—N1—C19—C20	−70.5 (3)
C6—C7—C8—C9	−2.8 (5)	C21—N6—C20—C19	−132.3 (3)
C6—C7—C8—N3	178.3 (3)	N1—C19—C20—N6	−67.3 (4)
O2—N3—C8—C9	−2.3 (4)	C20—N6—C21—C22	−179.8 (3)
O3—N3—C8—C9	176.7 (3)	N6—C21—C22—C27	−179.8 (3)
O2—N3—C8—C7	176.6 (3)	N6—C21—C22—C23	1.2 (5)
O3—N3—C8—C7	−4.5 (4)	C21—C22—C23—O7	−2.5 (5)
C7—C8—C9—C4	2.2 (4)	C27—C22—C23—O7	178.4 (3)
N3—C8—C9—C4	−179.0 (3)	C21—C22—C23—C24	177.0 (3)
C3—C4—C9—C8	−174.9 (3)	C27—C22—C23—C24	−2.0 (4)
C5—C4—C9—C8	0.8 (4)	O7—C23—C24—C25	−179.3 (3)

C19—N1—C10—C11	−79.5 (3)	C22—C23—C24—C25	1.2 (5)
C1—N1—C10—C11	157.2 (2)	C23—C24—C25—C26	0.5 (5)
C12—N4—C11—C10	114.6 (3)	C24—C25—C26—C27	−1.5 (5)
N1—C10—C11—N4	−78.8 (3)	C24—C25—C26—N7	179.6 (3)
C11—N4—C12—C13	172.5 (3)	O9—N7—C26—C27	−176.8 (3)
N4—C12—C13—C18	−170.1 (3)	O8—N7—C26—C27	6.0 (5)
N4—C12—C13—C14	2.4 (5)	O9—N7—C26—C25	2.1 (5)
C18—C13—C14—O4	177.3 (3)	O8—N7—C26—C25	−175.1 (3)
C12—C13—C14—O4	5.0 (5)	C25—C26—C27—C22	0.6 (5)
C18—C13—C14—C15	−0.8 (4)	N7—C26—C27—C22	179.5 (3)
C12—C13—C14—C15	−173.0 (3)	C21—C22—C27—C26	−177.9 (3)
O4—C14—C15—C16	−175.5 (3)	C23—C22—C27—C26	1.2 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*···N2	0.84	1.87	2.627 (3)	149
O4—H4*O*···N4	0.84	1.92	2.665 (3)	147
O7—H7*O*···N6	0.84	1.85	2.607 (3)	149
C1—H1*A*···N8^{i}	0.99	2.56	3.369 (5)	139
C1—H1*B*···O4^{ii}	0.99	2.41	3.290 (4)	148
C2—H2*B*···O2^{iii}	0.99	2.44	3.300 (4)	146
C3—H3···O7	0.95	2.53	3.297 (4)	138
C6—H6···N8^{iv}	0.95	2.49	3.360 (5)	153
C9—H9···O7	0.95	2.55	3.328 (4)	139
C11—H11*A*···O5^{v}	0.99	2.40	3.331 (4)	157
C12—H12···O5^{v}	0.95	2.54	3.339 (4)	142
C16—H16···O6^{vi}	0.95	2.51	3.330 (4)	145
C25—H25···O9^{vii}	0.95	2.48	3.359 (4)	153

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