Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-2,4-Dihydroxybenzaldehyde 4-ethylthiosemicarbazone–4,4′-bipyridine–water (4/7/2)

Seik Weng Ng

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

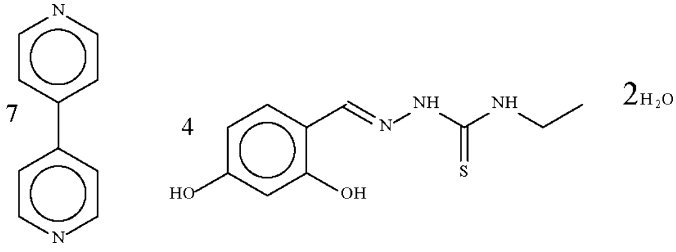
Received 22 July 2009; accepted 3 August 2009

Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.061; wR factor = 0.179; data-to-parameter ratio = 14.6.

The asymmetric unit of the title compound, $7C_{10}H_8N_2\cdot 4C_{10}H_{13}N_3O_2S\cdot 2H_2O$, contains two independent 2,4-hydroxybenzaldehyde 4-ethylthiosemicarbazone molecules, three and a half 4,4′-bipyridine molecules and one water molecule. Two of the 4,4′-bipyridine molecules lie on general positions and the other three on centers of inversion. The two 4,4′-bipyridine molecules on general positions and one of the three on special positions are disordered over two positions each with an occupancy of 0.50. The –NH—C(=S)—NH—NC fragment is close to planar in the two 2,4-hydroxybenzaldehyde 4-ethylthiosemicarbazone molecules (r.m.s. deviations = 0.04 and 0.05 Å). In the crystal, the Schiff base, *N*-heterocycle and water molecules engage in O—H···O, O—H···N and N—H···O hydrogen-bonding interactions, generating a layer structure.

Related literature

4,4′-Bipyridine forms a number of clathrates with diphenols; for the quinol clathrate, see: Oswald *et al.* (2005) and for the 2,2′-biphenol clathrate, see: Lavender *et al.* (1999). For the crystal structure of 2,4-dihydroxybenzaldehyde 4-ethylthiosemicarbazone, see: Tan *et al.* (2008).



Experimental

Crystal data

$7C_{10}H_8N_2\cdot 4C_{10}H_{13}N_3O_2S\cdot 2H_2O$
M_r = 2086.50
Triclinic, $P\overline{1}$
a = 11.7683 (3) Å
b = 15.2531 (4) Å
c = 16.0612 (4) Å
α = 74.639 (2)°
β = 86.561 (2)°
γ = 70.252 (1)°
V = 2615.2 (1) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 153 K
0.20 × 0.20 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.968, T_{max} = 0.992
18419 measured reflections
10042 independent reflections
6419 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.179
S = 1.02
10042 reflections
686 parameters
170 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1o···N8^{i}	0.84 (4)	1.92 (1)	2.755 (5)	171 (5)
O1—H1o···N8′^{i}	0.84 (4)	1.97 (1)	2.798 (5)	171 (5)
O2—H2o···N7	0.84 (4)	1.93 (1)	2.768 (5)	172 (4)
O2—H2o···N7′	0.84 (4)	1.93 (1)	2.766 (5)	178 (4)
O3—H3o···N10^{ii}	0.84 (4)	1.91 (1)	2.751 (5)	178 (5)
O3—H3o···N10′^{ii}	0.84 (4)	1.90 (1)	2.736 (5)	172 (5)
O4—H4o···N11	0.84 (4)	1.90 (1)	2.735 (4)	174 (4)
O1*w*—H1*w*1···O4	0.84 (3)	2.04 (2)	2.843 (4)	160 (5)
O1*w*—H1*w*2···N9	0.84 (3)	1.94 (1)	2.782 (5)	175 (7)
O1*w*—H1*w*2···N9′	0.84 (3)	1.91 (3)	2.722 (5)	160 (7)
N2—H2n···O1*w*	0.88 (3)	1.88 (1)	2.757 (4)	175 (4)

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

The author thanks the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2864).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Lavender, E. S., Ferguson, G. & Glidewell, C. (1999). *Acta Cryst.* C**55**, 430–432.
Oswald, I. D. H., Motherwell, W. D. S. & Parsons, S. (2005). *Acta Cryst.* B**61**, 46–57.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tan, K. W., Ng, C. H., Maah, M. J. & Ng, S. W. (2008). *Acta Cryst.* E**64**, o2123.
Westrip, S. P. (2009). *publCIF*. In preparation.

supporting information

Acta Cryst. (2009). E**65**, o2129 [doi:10.1107/S1600536809030852]

(*E*)-2,4-Dihydroxybenzaldehyde 4-ethylthiosemicarbazone–4,4′-bipyridine–water (4/7/2)

Seik Weng Ng

S1. Experimental

2,4-Dihydroxybenzaldehyde ethylthiosemicarbazone (0.48 g, 2 mmol) and 4,4′-bipyridine (0.15 g, 1 mmol) were dissolved in ethanol (20 ml) and the solution was set aside for a week. The solid material that separated from the solution consisted of small, transparent crystals embedded in some yellow, opaque clumps.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H = 0.95–0.98 Å) and were included in the refinement in the riding-model approximation, with U(H) set to 1.2 to 1.5U_{eq}(C). The amino and water H-atoms were located in a difference Fourier map, and were refined with distance restraints (O—H = 0.84 (1) and N—H = 0.88 (1) Å); their U_{iso} parameters were freely refined.

The two 4,4′-bipyridine molecules on general positions and one of the three on special positions are disordered over two positions. A consideration of the short C···C contacts necessitated a 1:1 type of disorder. For these, the pyridyl rings were refined as rigid hexagons of 1.39 Å sides as attempts to restrain C—C and C—N distances to appropriate values require an excessive number of restraints. The U^{ij} parameters of the primed atoms were set to those of the unprimed ones. Additionally, the anisotropic displacement parameters of the carbon atoms were tightly restrained so that they were almost nearly isotropic. The C21/C21′ atoms have somewhat elongated thermal ellipsoids, which is probably a consequence of the disorder. More than two orientation of the disordered molecules are possible but splitting the molecules further did not yield more meaningful results.

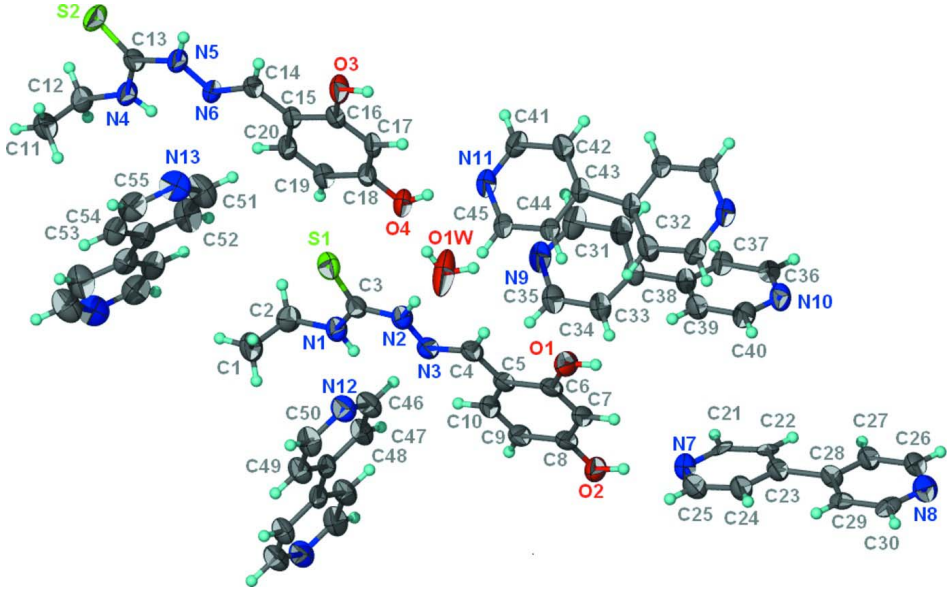


Figure 1

Displacement ellipsoid plot (Barbour, 2001) of $7C_{10}H_8N_2.4C_{10}H_{13}N_3O_2S.2H_2O$ at the 70% probability level; H atoms are drawn as spheres of arbitrary radius. Unlabeled atoms in N11-, N12- and N13-pyridine rings are related to other labeled atoms by the symmetry operation (2 - *x*, 1 - *y*, 1 - *z*), (-*x* - 1, 2 - *y*, 1 - *z*) and (-*x*, 2 - *y*, 2 - *z*), respectively. Only one component of the disordered molecules are shown.

(*E*)-2,4-Dihydroxybenzaldehyde 4-ethylthiosemicarbazone– 4,4′-bipyridine–water (4/7/2)

Crystal data

$7(C_{10}H_8N_2)\cdot4(C_{10}H_{13}N_3O_2S)\cdot2(H_2O)$
$M_r = 2086.50$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 11.7683\ (3)$ Å
$b = 15.2531\ (4)$ Å
$c = 16.0612\ (4)$ Å
$\alpha = 74.639\ (2)°$
$\beta = 86.561\ (2)°$
$\gamma = 70.252\ (1)°$
$V = 2615.2\ (1)$ Å^3
$Z = 1$
$F(000) = 1098$
$D_x = 1.325$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3358 reflections
$\theta = 2.6–26.0°$
$\mu = 0.16$ mm^{-1}
$T = 153$ K
Plate, yellow
0.20 × 0.20 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.968$, $T_{max} = 0.992$
18419 measured reflections
10042 independent reflections
6419 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 26.0°$, $\theta_{min} = 1.3°$
$h = -14\rightarrow15$
$k = -19\rightarrow19$
$l = -20\rightarrow20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.179$
$S = 1.02$
10042 reflections
686 parameters
170 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0783P)^2 + 2.0276P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.66$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	0.25549 (8)	0.84394 (7)	0.73613 (5)	0.0403 (2)	
S2	0.65813 (8)	0.93784 (8)	1.26910 (7)	0.0511 (3)	
O1	0.3505 (2)	0.66350 (18)	0.35305 (17)	0.0458 (6)	
H1O	0.388 (4)	0.645 (3)	0.311 (2)	0.081 (15)*	
O2	−0.0039 (2)	0.74630 (19)	0.17564 (17)	0.0470 (6)	
H2O	0.045 (3)	0.721 (3)	0.141 (2)	0.058 (12)*	
O3	0.9220 (2)	0.67293 (17)	0.94434 (15)	0.0396 (6)	
H3O	0.966 (4)	0.644 (3)	0.910 (3)	0.100 (18)*	
O4	0.6292 (2)	0.70169 (17)	0.73457 (14)	0.0386 (5)	
H4O	0.693 (2)	0.679 (3)	0.710 (2)	0.066 (13)*	
O1w	0.4655 (3)	0.7058 (3)	0.6093 (2)	0.0781 (10)	
H1w1	0.500 (4)	0.716 (3)	0.648 (2)	0.078 (16)*	
H1w2	0.509 (5)	0.696 (5)	0.567 (3)	0.15 (3)*	
N1	0.0466 (2)	0.8843 (2)	0.65301 (18)	0.0353 (6)	
H1N	0.008 (3)	0.877 (3)	0.6112 (16)	0.049 (11)*	
N2	0.2186 (2)	0.7963 (2)	0.59750 (18)	0.0352 (6)	
H2N	0.2980 (10)	0.771 (2)	0.602 (2)	0.049 (11)*	
N3	0.1528 (2)	0.79739 (19)	0.52915 (17)	0.0349 (6)	
N4	0.4992 (2)	0.91841 (19)	1.17051 (18)	0.0343 (6)	
H4N	0.484 (3)	0.904 (3)	1.1236 (14)	0.049 (11)*	
N5	0.6981 (2)	0.8472 (2)	1.14649 (18)	0.0381 (7)	
H5N	0.7738 (13)	0.839 (2)	1.158 (2)	0.042 (10)*	
N6	0.6625 (2)	0.82602 (19)	1.07669 (16)	0.0328 (6)	
C1	−0.0538 (3)	0.8798 (3)	0.7937 (2)	0.0504 (10)	
H1A	−0.0972	0.9212	0.8308	0.076*	
H1B	0.0196	0.8315	0.8244	0.076*	
H1C	−0.1057	0.8472	0.7791	0.076*	
C2	−0.0201 (3)	0.9405 (2)	0.7120 (2)	0.0417 (8)	
H2A	−0.0947	0.9895	0.6818	0.050*	
H2B	0.0297	0.9751	0.7275	0.050*	
C3	0.1660 (3)	0.8421 (2)	0.6589 (2)	0.0313 (7)	
C4	0.2164 (3)	0.7593 (2)	0.4716 (2)	0.0343 (7)	
H4	0.3018	0.7335	0.4791	0.041*	

C5	0.1601 (3)	0.7548 (2)	0.3954 (2)	0.0335 (7)	
C6	0.2295 (3)	0.7075 (2)	0.3359 (2)	0.0362 (8)	
C7	0.1760 (3)	0.7045 (2)	0.2625 (2)	0.0372 (8)	
H7	0.2243	0.6724	0.2228	0.045*	
C8	0.0531 (3)	0.7478 (2)	0.2469 (2)	0.0369 (8)	
C9	−0.0184 (3)	0.7960 (2)	0.3044 (2)	0.0382 (8)	
H9	−0.1030	0.8264	0.2938	0.046*	
C10	0.0360 (3)	0.7987 (2)	0.3766 (2)	0.0371 (8)	
H10	−0.0128	0.8318	0.4155	0.045*	
C11	0.3731 (3)	0.9035 (3)	1.2993 (2)	0.0487 (9)	
H11A	0.3046	0.9408	1.3274	0.073*	
H11B	0.4448	0.8770	1.3379	0.073*	
H11C	0.3542	0.8507	1.2862	0.073*	
C12	0.3977 (3)	0.9690 (2)	1.2159 (2)	0.0384 (8)	
H12A	0.3245	0.9965	1.1775	0.046*	
H12B	0.4147	1.0232	1.2294	0.046*	
C13	0.6134 (3)	0.9001 (2)	1.1925 (2)	0.0345 (7)	
C14	0.7464 (3)	0.7858 (2)	1.0308 (2)	0.0318 (7)	
H14	0.8286	0.7707	1.0467	0.038*	
C15	0.7159 (3)	0.7632 (2)	0.95436 (19)	0.0287 (7)	
C16	0.8071 (3)	0.7075 (2)	0.91068 (19)	0.0291 (7)	
C17	0.7786 (3)	0.6880 (2)	0.83663 (18)	0.0302 (7)	
H17	0.8408	0.6517	0.8065	0.036*	
C18	0.6599 (3)	0.7210 (2)	0.80616 (18)	0.0304 (7)	
C19	0.5682 (3)	0.7757 (2)	0.8492 (2)	0.0338 (7)	
H19	0.4867	0.7987	0.8284	0.041*	
C20	0.5975 (3)	0.7958 (2)	0.9220 (2)	0.0322 (7)	
H20	0.5349	0.8331	0.9512	0.039*	
N7	0.1690 (5)	0.6506 (5)	0.0762 (5)	0.0436 (9)	0.50
C21	0.1857 (6)	0.7027 (3)	−0.0060 (5)	0.0282 (14)	0.50
H21	0.1437	0.7702	−0.0244	0.034*	0.50
C22	0.2638 (7)	0.6562 (5)	−0.0613 (4)	0.0268 (14)	0.50
H22	0.2752	0.6919	−0.1175	0.032*	0.50
C23	0.3253 (5)	0.5576 (5)	−0.0344 (3)	0.0342 (9)	0.50
C24	0.3086 (4)	0.5054 (4)	0.0478 (3)	0.0374 (15)	0.50
H24	0.3506	0.4380	0.0662	0.045*	0.50
C25	0.2304 (5)	0.5519 (4)	0.1031 (3)	0.0419 (15)	0.50
H25	0.2190	0.5163	0.1592	0.050*	0.50
N8	0.5283 (5)	0.4168 (4)	−0.2247 (4)	0.0456 (8)	0.50
C26	0.4775 (4)	0.5168 (4)	−0.2494 (3)	0.0412 (15)	0.50
H26	0.4897	0.5517	−0.3057	0.049*	0.50
C27	0.4088 (4)	0.5656 (3)	−0.1919 (3)	0.0361 (13)	0.50
H27	0.3741	0.6340	−0.2088	0.043*	0.50
C28	0.3910 (5)	0.5145 (5)	−0.1095 (3)	0.0316 (9)	0.50
C29	0.4417 (6)	0.4146 (5)	−0.0848 (4)	0.0283 (14)	0.50
H29	0.4295	0.3796	−0.0285	0.034*	0.50
C30	0.5104 (6)	0.3657 (3)	−0.1423 (5)	0.0322 (15)	0.50
H30	0.5451	0.2974	−0.1254	0.039*	0.50

N7′	0.1582 (5)	0.6579 (5)	0.0664 (5)	0.0436 (9)	0.50
C21′	0.2138 (7)	0.7087 (4)	0.0021 (6)	0.0282 (14)	0.50
H21′	0.1989	0.7753	−0.0039	0.034*	0.50
C22′	0.2911 (6)	0.6620 (5)	−0.0534 (4)	0.0268 (14)	0.50
H22′	0.3291	0.6968	−0.0974	0.032*	0.50
C23′	0.3129 (5)	0.5645 (5)	−0.0447 (4)	0.0342 (9)	0.50
C24′	0.2574 (5)	0.5137 (4)	0.0196 (4)	0.0374 (15)	0.50
H24′	0.2723	0.4471	0.0256	0.045*	0.50
C25′	0.1800 (4)	0.5604 (4)	0.0751 (3)	0.0419 (15)	0.50
H25′	0.1420	0.5257	0.1191	0.050*	0.50
N8′	0.5377 (5)	0.4119 (4)	−0.2166 (5)	0.0456 (8)	0.50
C26′	0.5311 (4)	0.5056 (4)	−0.2200 (3)	0.0412 (15)	0.50
H26′	0.5719	0.5382	−0.2634	0.049*	0.50
C27′	0.4648 (4)	0.5514 (3)	−0.1598 (3)	0.0361 (13)	0.50
H27′	0.4603	0.6154	−0.1621	0.043*	0.50
C28′	0.4051 (5)	0.5037 (5)	−0.0962 (3)	0.0316 (9)	0.50
C29′	0.4117 (6)	0.4101 (5)	−0.0928 (4)	0.0283 (14)	0.50
H29′	0.3709	0.3775	−0.0493	0.034*	0.50
C30′	0.4780 (6)	0.3642 (3)	−0.1530 (5)	0.0322 (15)	0.50
H30′	0.4825	0.3002	−0.1507	0.039*	0.50
N9	0.6183 (5)	0.6645 (4)	0.4755 (3)	0.0440 (18)	0.50
C31	0.6864 (5)	0.7070 (3)	0.4150 (4)	0.0492 (17)	0.50
H31	0.6903	0.7682	0.4152	0.059*	0.50
C32	0.7489 (4)	0.6601 (4)	0.3542 (3)	0.0398 (14)	0.50
H32	0.7954	0.6892	0.3129	0.048*	0.50
C33	0.7432 (5)	0.5706 (4)	0.3540 (4)	0.0336 (12)	0.50
C34	0.6751 (6)	0.5281 (3)	0.4145 (5)	0.0395 (14)	0.50
H34	0.6712	0.4669	0.4143	0.047*	0.50
C35	0.6127 (5)	0.5750 (4)	0.4752 (4)	0.0405 (16)	0.50
H35	0.5661	0.5459	0.5166	0.049*	0.50
N10	0.9335 (5)	0.4261 (4)	0.1649 (3)	0.0391 (8)	0.50
C36	0.8856 (5)	0.5262 (4)	0.1435 (3)	0.0283 (14)	0.50
H36	0.8950	0.5626	0.0871	0.034*	0.50
C37	0.8241 (6)	0.5732 (3)	0.2045 (4)	0.0303 (14)	0.50
H37	0.7914	0.6416	0.1898	0.036*	0.50
C38	0.8105 (5)	0.5199 (3)	0.2869 (4)	0.0314 (10)	0.50
C39	0.8583 (4)	0.4198 (3)	0.3084 (2)	0.0367 (12)	0.50
H39	0.8490	0.3834	0.3648	0.044*	0.50
C40	0.9199 (4)	0.3729 (3)	0.2474 (3)	0.0375 (12)	0.50
H40	0.9526	0.3044	0.2621	0.045*	0.50
N9′	0.6010 (5)	0.6323 (4)	0.4845 (3)	0.0440 (18)	0.50
C31′	0.6291 (5)	0.6924 (3)	0.4109 (4)	0.0492 (17)	0.50
H31′	0.6067	0.7596	0.4057	0.059*	0.50
C32′	0.6898 (5)	0.6541 (4)	0.3448 (3)	0.0398 (14)	0.50
H32′	0.7090	0.6952	0.2945	0.048*	0.50
C33′	0.7225 (6)	0.5558 (4)	0.3525 (4)	0.0336 (12)	0.50
C34′	0.6944 (6)	0.4957 (3)	0.4261 (5)	0.0395 (14)	0.50
H34′	0.7167	0.4285	0.4313	0.047*	0.50

C35′	0.6337 (6)	0.5340 (3)	0.4922 (4)	0.0405 (16)	0.50
H35′	0.6145	0.4929	0.5425	0.049*	0.50
N10′	0.9211 (5)	0.4325 (5)	0.1540 (3)	0.0391 (8)	0.50
C36′	0.9193 (5)	0.5226 (4)	0.1589 (3)	0.0283 (14)	0.50
H36′	0.9624	0.5566	0.1187	0.034*	0.50
C37′	0.8547 (6)	0.5630 (3)	0.2227 (4)	0.0303 (14)	0.50
H37′	0.8535	0.6246	0.2260	0.036*	0.50
C38′	0.7917 (5)	0.5134 (4)	0.2815 (4)	0.0314 (10)	0.50
C39′	0.7934 (4)	0.4233 (3)	0.2767 (3)	0.0367 (12)	0.50
H39′	0.7504	0.3894	0.3169	0.044*	0.50
C40′	0.8581 (4)	0.3829 (3)	0.2129 (3)	0.0375 (12)	0.50
H40′	0.8593	0.3213	0.2096	0.045*	0.50
N11	0.8292 (4)	0.6185 (4)	0.6505 (3)	0.0392 (10)	0.50
C41	0.9111 (4)	0.6576 (3)	0.6025 (3)	0.0398 (12)	0.50
H41	0.9174	0.7158	0.6092	0.048*	0.50
C42	0.9836 (4)	0.6115 (3)	0.5446 (3)	0.0398 (12)	0.50
H42	1.0395	0.6382	0.5118	0.048*	0.50
C43	0.9743 (5)	0.5263 (3)	0.5348 (3)	0.0299 (11)	0.50
C44	0.8924 (5)	0.4872 (3)	0.5828 (4)	0.0270 (14)	0.50
H44	0.8861	0.4290	0.5761	0.032*	0.50
C45	0.8199 (5)	0.5334 (4)	0.6407 (3)	0.0289 (14)	0.50
H45	0.7640	0.5067	0.6735	0.035*	0.50
N11′	0.8058 (5)	0.6149 (4)	0.6454 (3)	0.0392 (10)	0.50
C41′	0.8322 (4)	0.6690 (3)	0.5672 (3)	0.0398 (12)	0.50
H41′	0.8013	0.7375	0.5535	0.048*	0.50
C42′	0.9038 (4)	0.6229 (3)	0.5089 (2)	0.0398 (12)	0.50
H42′	0.9218	0.6599	0.4555	0.048*	0.50
C43′	0.9490 (5)	0.5228 (3)	0.5289 (3)	0.0299 (11)	0.50
C44′	0.9227 (5)	0.4687 (3)	0.6071 (4)	0.0270 (14)	0.50
H44′	0.9536	0.4002	0.6207	0.032*	0.50
C45′	0.8511 (5)	0.5147 (4)	0.6653 (3)	0.0289 (14)	0.50
H45′	0.8330	0.4777	0.7188	0.035*	0.50
N12	−0.1892 (2)	0.9088 (2)	0.56896 (19)	0.0413 (7)	
C46	−0.2279 (3)	0.9691 (2)	0.4914 (2)	0.0421 (8)	
H46	−0.1695	0.9873	0.4538	0.050*	
C47	−0.3472 (3)	1.0070 (2)	0.4621 (2)	0.0392 (8)	
H47	−0.3688	1.0504	0.4064	0.047*	
C48	−0.4356 (3)	0.9812 (2)	0.51455 (19)	0.0306 (7)	
C49	−0.3957 (3)	0.9185 (2)	0.5956 (2)	0.0389 (8)	
H49	−0.4518	0.8986	0.6344	0.047*	
C50	−0.2754 (3)	0.8854 (2)	0.6193 (2)	0.0424 (8)	
H50	−0.2515	0.8431	0.6752	0.051*	
N13	0.3191 (3)	0.8864 (2)	1.0389 (2)	0.0523 (8)	
C51	0.2705 (4)	0.9350 (3)	0.9600 (3)	0.0574 (11)	
H51	0.3237	0.9394	0.9135	0.069*	
C52	0.1478 (3)	0.9795 (3)	0.9415 (2)	0.0518 (10)	
H52	0.1192	1.0120	0.8837	0.062*	
C53	0.0666 (3)	0.9762 (2)	1.0080 (2)	0.0371 (8)	

C54	0.1158 (3)	0.9245 (2)	1.0897 (2)	0.0417 (8)
H54	0.0647	0.9185	1.1374	0.050*
C55	0.2396 (3)	0.8816 (3)	1.1015 (2)	0.0479 (9)
H55	0.2704	0.8462	1.1583	0.057*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0363 (5)	0.0501 (5)	0.0309 (4)	−0.0126 (4)	0.0032 (4)	−0.0077 (4)
S2	0.0350 (5)	0.0713 (7)	0.0654 (6)	−0.0167 (5)	0.0108 (4)	−0.0514 (6)
O1	0.0297 (13)	0.0553 (15)	0.0630 (17)	−0.0139 (11)	0.0150 (12)	−0.0363 (14)
O2	0.0386 (14)	0.0649 (17)	0.0536 (16)	−0.0230 (13)	0.0166 (12)	−0.0379 (14)
O3	0.0308 (13)	0.0503 (14)	0.0357 (13)	−0.0050 (11)	0.0080 (10)	−0.0203 (11)
O4	0.0421 (14)	0.0454 (14)	0.0293 (12)	−0.0104 (11)	0.0074 (11)	−0.0181 (11)
O1w	0.0499 (17)	0.119 (3)	0.0486 (17)	0.0148 (16)	−0.0045 (14)	−0.0489 (18)
N1	0.0298 (15)	0.0382 (15)	0.0381 (16)	−0.0083 (12)	0.0068 (12)	−0.0155 (13)
N2	0.0273 (15)	0.0398 (16)	0.0415 (16)	−0.0101 (13)	0.0082 (13)	−0.0184 (13)
N3	0.0344 (15)	0.0342 (15)	0.0413 (16)	−0.0155 (12)	0.0108 (13)	−0.0152 (12)
N4	0.0313 (15)	0.0362 (15)	0.0380 (16)	−0.0069 (12)	0.0050 (12)	−0.0202 (13)
N5	0.0276 (15)	0.0537 (18)	0.0433 (16)	−0.0146 (14)	0.0090 (13)	−0.0302 (14)
N6	0.0349 (15)	0.0376 (15)	0.0318 (14)	−0.0140 (12)	0.0082 (12)	−0.0180 (12)
C1	0.049 (2)	0.050 (2)	0.056 (2)	−0.0161 (18)	0.0219 (19)	−0.0252 (19)
C2	0.0370 (19)	0.0362 (18)	0.053 (2)	−0.0072 (15)	0.0079 (16)	−0.0210 (16)
C3	0.0333 (17)	0.0275 (16)	0.0330 (17)	−0.0116 (13)	0.0083 (14)	−0.0077 (13)
C4	0.0281 (16)	0.0324 (17)	0.0469 (19)	−0.0127 (14)	0.0117 (15)	−0.0165 (15)
C5	0.0312 (17)	0.0316 (16)	0.0450 (19)	−0.0164 (14)	0.0145 (15)	−0.0169 (15)
C6	0.0297 (17)	0.0357 (18)	0.054 (2)	−0.0185 (14)	0.0170 (15)	−0.0222 (16)
C7	0.0340 (18)	0.0379 (18)	0.052 (2)	−0.0184 (15)	0.0189 (16)	−0.0266 (16)
C8	0.0322 (18)	0.0412 (19)	0.049 (2)	−0.0210 (15)	0.0143 (15)	−0.0221 (16)
C9	0.0296 (17)	0.0421 (19)	0.050 (2)	−0.0160 (15)	0.0116 (15)	−0.0213 (16)
C10	0.0342 (18)	0.0355 (18)	0.049 (2)	−0.0144 (15)	0.0177 (15)	−0.0229 (16)
C11	0.044 (2)	0.054 (2)	0.054 (2)	−0.0196 (18)	0.0175 (18)	−0.0236 (19)
C12	0.0279 (17)	0.0398 (19)	0.047 (2)	−0.0040 (14)	0.0074 (15)	−0.0218 (16)
C13	0.0311 (18)	0.0344 (17)	0.0412 (18)	−0.0124 (14)	0.0120 (14)	−0.0157 (15)
C14	0.0295 (16)	0.0340 (17)	0.0360 (17)	−0.0129 (14)	0.0096 (14)	−0.0146 (14)
C15	0.0322 (17)	0.0278 (15)	0.0276 (16)	−0.0114 (13)	0.0086 (13)	−0.0096 (13)
C16	0.0280 (16)	0.0273 (15)	0.0298 (16)	−0.0087 (13)	0.0087 (13)	−0.0063 (13)
C17	0.0347 (17)	0.0282 (16)	0.0251 (16)	−0.0073 (13)	0.0108 (13)	−0.0089 (13)
C18	0.0398 (18)	0.0288 (16)	0.0223 (15)	−0.0128 (14)	0.0074 (13)	−0.0059 (13)
C19	0.0321 (17)	0.0368 (18)	0.0304 (17)	−0.0095 (14)	0.0037 (14)	−0.0084 (14)
C20	0.0332 (17)	0.0287 (16)	0.0320 (17)	−0.0060 (13)	0.0102 (14)	−0.0114 (13)
N7	0.0465 (19)	0.051 (2)	0.040 (2)	−0.0195 (16)	0.0131 (16)	−0.0216 (17)
C21	0.029 (3)	0.0387 (19)	0.031 (2)	−0.0195 (18)	−0.007 (2)	−0.0192 (16)
C22	0.029 (3)	0.0328 (18)	0.0305 (19)	−0.0228 (19)	0.000 (2)	−0.0115 (16)
C23	0.0334 (19)	0.0347 (18)	0.037 (2)	−0.0138 (15)	0.0092 (16)	−0.0115 (16)
C24	0.038 (3)	0.036 (2)	0.038 (3)	−0.012 (2)	0.009 (2)	−0.010 (2)
C25	0.039 (3)	0.049 (2)	0.041 (3)	−0.020 (3)	0.008 (2)	−0.011 (2)
N8	0.0305 (17)	0.0496 (19)	0.059 (2)	−0.0095 (14)	0.0123 (15)	−0.0260 (17)

C26	0.035 (3)	0.050 (2)	0.046 (3)	−0.019 (3)	0.014 (2)	−0.020 (2)
C27	0.036 (3)	0.035 (2)	0.039 (3)	−0.013 (2)	0.009 (2)	−0.011 (2)
C28	0.0282 (19)	0.031 (2)	0.034 (2)	−0.0096 (16)	0.0049 (16)	−0.0064 (17)
C29	0.022 (3)	0.0336 (18)	0.0334 (19)	−0.0144 (19)	−0.004 (2)	−0.0079 (16)
C30	0.021 (4)	0.0358 (18)	0.043 (3)	−0.0053 (18)	−0.012 (2)	−0.0167 (17)
N7′	0.0465 (19)	0.051 (2)	0.040 (2)	−0.0195 (16)	0.0131 (16)	−0.0216 (17)
C21′	0.029 (3)	0.0387 (19)	0.031 (2)	−0.0195 (18)	−0.007 (2)	−0.0192 (16)
C22′	0.029 (3)	0.0328 (18)	0.0305 (19)	−0.0228 (19)	0.000 (2)	−0.0115 (16)
C23′	0.0334 (19)	0.0347 (18)	0.037 (2)	−0.0138 (15)	0.0092 (16)	−0.0115 (16)
C24′	0.038 (3)	0.036 (2)	0.038 (3)	−0.012 (2)	0.009 (2)	−0.010 (2)
C25′	0.039 (3)	0.049 (2)	0.041 (3)	−0.020 (3)	0.008 (2)	−0.011 (2)
N8′	0.0305 (17)	0.0496 (19)	0.059 (2)	−0.0095 (14)	0.0123 (15)	−0.0260 (17)
C26′	0.035 (3)	0.050 (2)	0.046 (3)	−0.019 (3)	0.014 (2)	−0.020 (2)
C27′	0.036 (3)	0.035 (2)	0.039 (3)	−0.013 (2)	0.009 (2)	−0.011 (2)
C28′	0.0282 (19)	0.031 (2)	0.034 (2)	−0.0096 (16)	0.0049 (16)	−0.0064 (17)
C29′	0.022 (3)	0.0336 (18)	0.0334 (19)	−0.0144 (19)	−0.004 (2)	−0.0079 (16)
C30′	0.021 (4)	0.0358 (18)	0.043 (3)	−0.0053 (18)	−0.012 (2)	−0.0167 (17)
N9	0.040 (3)	0.050 (5)	0.037 (2)	−0.005 (3)	0.0048 (17)	−0.017 (3)
C31	0.053 (4)	0.046 (3)	0.040 (2)	−0.002 (3)	0.000 (3)	−0.017 (2)
C32	0.044 (4)	0.036 (2)	0.034 (2)	−0.006 (2)	0.005 (3)	−0.0093 (18)
C33	0.031 (2)	0.034 (2)	0.0305 (16)	−0.0044 (16)	0.0036 (16)	−0.0078 (15)
C34	0.038 (3)	0.041 (3)	0.036 (2)	−0.008 (3)	0.009 (2)	−0.012 (3)
C35	0.035 (3)	0.044 (4)	0.037 (3)	−0.006 (3)	0.005 (2)	−0.012 (3)
N10	0.0273 (17)	0.0492 (19)	0.0391 (18)	−0.0054 (14)	0.0060 (14)	−0.0194 (16)
C36	0.014 (3)	0.043 (2)	0.026 (2)	−0.006 (2)	−0.008 (2)	−0.0083 (18)
C37	0.025 (3)	0.0354 (19)	0.027 (3)	−0.0045 (19)	−0.005 (2)	−0.0084 (18)
C38	0.028 (2)	0.0308 (18)	0.0309 (18)	−0.0036 (16)	0.0038 (16)	−0.0097 (15)
C39	0.038 (3)	0.037 (2)	0.035 (2)	−0.012 (2)	0.0079 (19)	−0.0095 (18)
C40	0.037 (3)	0.036 (2)	0.038 (3)	−0.007 (2)	0.002 (2)	−0.015 (2)
N9′	0.040 (3)	0.050 (5)	0.037 (2)	−0.005 (3)	0.0048 (17)	−0.017 (3)
C31′	0.053 (4)	0.046 (3)	0.040 (2)	−0.002 (3)	0.000 (3)	−0.017 (2)
C32′	0.044 (4)	0.036 (2)	0.034 (2)	−0.006 (2)	0.005 (3)	−0.0093 (18)
C33′	0.031 (2)	0.034 (2)	0.0305 (16)	−0.0044 (16)	0.0036 (16)	−0.0078 (15)
C34′	0.038 (3)	0.041 (3)	0.036 (2)	−0.008 (3)	0.009 (2)	−0.012 (3)
C35′	0.035 (3)	0.044 (4)	0.037 (3)	−0.006 (3)	0.005 (2)	−0.012 (3)
N10′	0.0273 (17)	0.0492 (19)	0.0391 (18)	−0.0054 (14)	0.0060 (14)	−0.0194 (16)
C36′	0.014 (3)	0.043 (2)	0.026 (2)	−0.006 (2)	−0.008 (2)	−0.0083 (18)
C37′	0.025 (3)	0.0354 (19)	0.027 (3)	−0.0045 (19)	−0.005 (2)	−0.0084 (18)
C38′	0.028 (2)	0.0308 (18)	0.0309 (18)	−0.0036 (16)	0.0038 (16)	−0.0097 (15)
C39′	0.038 (3)	0.037 (2)	0.035 (2)	−0.012 (2)	0.0079 (19)	−0.0095 (18)
C40′	0.037 (3)	0.036 (2)	0.038 (3)	−0.007 (2)	0.002 (2)	−0.015 (2)
N11	0.039 (2)	0.0432 (18)	0.0345 (16)	−0.0080 (16)	0.0083 (16)	−0.0186 (14)
C41	0.048 (3)	0.038 (2)	0.037 (3)	−0.012 (2)	0.004 (2)	−0.018 (2)
C42	0.042 (3)	0.035 (2)	0.040 (3)	−0.011 (2)	0.015 (2)	−0.011 (2)
C43	0.034 (3)	0.0284 (17)	0.0249 (17)	−0.0060 (17)	0.0060 (18)	−0.0089 (14)
C44	0.026 (3)	0.031 (2)	0.022 (3)	−0.007 (2)	−0.002 (2)	−0.007 (2)
C45	0.024 (3)	0.040 (3)	0.019 (3)	−0.006 (2)	−0.005 (2)	−0.007 (2)
N11′	0.039 (2)	0.0432 (18)	0.0345 (16)	−0.0080 (16)	0.0083 (16)	−0.0186 (14)

C41′	0.048 (3)	0.038 (2)	0.037 (3)	−0.012 (2)	0.004 (2)	−0.018 (2)
C42′	0.042 (3)	0.035 (2)	0.040 (3)	−0.011 (2)	0.015 (2)	−0.011 (2)
C43′	0.034 (3)	0.0284 (17)	0.0249 (17)	−0.0060 (17)	0.0060 (18)	−0.0089 (14)
C44′	0.026 (3)	0.031 (2)	0.022 (3)	−0.007 (2)	−0.002 (2)	−0.007 (2)
C45′	0.024 (3)	0.040 (3)	0.019 (3)	−0.006 (2)	−0.005 (2)	−0.007 (2)
N12	0.0373 (16)	0.0340 (15)	0.0480 (17)	−0.0078 (13)	0.0078 (13)	−0.0100 (14)
C46	0.040 (2)	0.0369 (19)	0.046 (2)	−0.0140 (16)	0.0115 (16)	−0.0058 (16)
C47	0.0399 (19)	0.0386 (19)	0.0350 (18)	−0.0133 (15)	0.0096 (15)	−0.0046 (15)
C48	0.0381 (17)	0.0237 (15)	0.0291 (16)	−0.0082 (13)	0.0102 (14)	−0.0101 (13)
C49	0.040 (2)	0.0306 (17)	0.0397 (19)	−0.0101 (15)	0.0097 (15)	−0.0031 (15)
C50	0.045 (2)	0.0338 (18)	0.0377 (19)	−0.0054 (16)	0.0063 (16)	−0.0023 (15)
N13	0.0445 (18)	0.0436 (18)	0.058 (2)	−0.0058 (15)	−0.0058 (16)	−0.0050 (16)
C51	0.047 (2)	0.049 (2)	0.059 (3)	−0.0063 (19)	0.009 (2)	−0.002 (2)
C52	0.048 (2)	0.048 (2)	0.042 (2)	−0.0030 (18)	−0.0010 (17)	−0.0004 (17)
C53	0.0438 (19)	0.0246 (16)	0.0378 (18)	−0.0048 (14)	−0.0017 (15)	−0.0076 (14)
C54	0.045 (2)	0.0373 (19)	0.0386 (19)	−0.0060 (16)	0.0005 (16)	−0.0135 (16)
C55	0.051 (2)	0.043 (2)	0.042 (2)	−0.0086 (18)	−0.0096 (18)	−0.0064 (17)

Geometric parameters (Å, º)

S1—C3	1.686 (3)	C26′—C27′	1.39
S2—C13	1.667 (3)	C26′—H26′	0.95
O1—C6	1.363 (4)	C27′—C28′	1.39
O1—H1O	0.84 (4)	C27′—H27′	0.95
O2—C8	1.370 (4)	C28′—C29′	1.39
O2—H2O	0.84 (4)	C29′—C30′	1.39
O3—C16	1.361 (4)	C29′—H29′	0.95
O3—H3O	0.84 (4)	C30′—H30′	0.95
O4—C18	1.357 (4)	N9—C31	1.39
O4—H4O	0.84 (4)	N9—C35	1.39
O1w—H1w1	0.84 (3)	C31—C32	1.39
O1w—H1w2	0.84 (3)	C31—H31	0.95
N1—C3	1.331 (4)	C32—C33	1.39
N1—C2	1.461 (4)	C32—H32	0.95
N1—H1N	0.88 (3)	C33—C34	1.39
N2—C3	1.357 (4)	C33—C38	1.520 (5)
N2—N3	1.374 (4)	C34—C35	1.39
N2—H2N	0.88 (3)	C34—H34	0.95
N3—C4	1.288 (4)	C35—H35	0.95
N4—C13	1.327 (4)	N10—C36	1.39
N4—C12	1.458 (4)	N10—C40	1.39
N4—H4N	0.88 (3)	C36—C37	1.39
N5—C13	1.369 (4)	C36—H36	0.95
N5—N6	1.370 (4)	C37—C38	1.39
N5—H5N	0.88 (3)	C37—H37	0.95
N6—C14	1.280 (4)	C38—C39	1.39
C1—C2	1.510 (5)	C39—C40	1.39
C1—H1A	0.98	C39—H39	0.95

C1—H1B	0.98	C40—H40	0.95
C1—H1C	0.98	N9′—C31′	1.39
C2—H2A	0.99	N9′—C35′	1.39
C2—H2B	0.99	C31′—C32′	1.39
C4—C5	1.454 (5)	C31′—H31′	0.95
C4—H4	0.95	C32′—C33′	1.39
C5—C10	1.398 (5)	C32′—H32′	0.95
C5—C6	1.405 (4)	C33′—C34′	1.39
C6—C7	1.388 (5)	C33′—C38′	1.517 (5)
C7—C8	1.379 (4)	C34′—C35′	1.39
C7—H7	0.95	C34′—H34′	0.95
C8—C9	1.396 (4)	C35′—H35′	0.95
C9—C10	1.375 (5)	N10′—C36′	1.39
C9—H9	0.95	N10′—C40′	1.39
C10—H10	0.95	C36′—C37′	1.39
C11—C12	1.521 (5)	C36′—H36′	0.95
C11—H11A	0.98	C37′—C38′	1.39
C11—H11B	0.98	C37′—H37′	0.95
C11—H11C	0.98	C38′—C39′	1.39
C12—H12A	0.99	C39′—C40′	1.39
C12—H12B	0.99	C39′—H39′	0.95
C14—C15	1.455 (4)	C40′—H40′	0.95
C14—H14	0.95	N11—C41	1.39
C15—C20	1.393 (4)	N11—C45	1.39
C15—C16	1.405 (4)	C41—C42	1.39
C16—C17	1.385 (4)	C41—H41	0.95
C17—C18	1.385 (4)	C42—C43	1.39
C17—H17	0.95	C42—H42	0.95
C18—C19	1.395 (4)	C43—C44	1.39
C19—C20	1.376 (4)	C43—C43^{i}	1.525 (8)
C19—H19	0.95	C44—C45	1.39
C20—H20	0.95	C44—H44	0.95
N7—C21	1.39	C45—H45	0.95
N7—C25	1.39	N11′—C41′	1.39
C21—C22	1.39	N11′—C45′	1.39
C21—H21	0.95	C41′—C42′	1.39
C22—C23	1.39	C41′—H41′	0.95
C22—H22	0.95	C42′—C43′	1.39
C23—C24	1.39	C42′—H42′	0.95
C23—C28	1.565 (6)	C43′—C44′	1.39
C24—C25	1.39	C43′—C43′^{i}	1.558 (8)
C24—H24	0.95	C44′—C45′	1.39
C25—H25	0.95	C44′—H44′	0.95
N8—C26	1.39	C45′—H45′	0.95
N8—C30	1.39	N12—C46	1.336 (4)
C26—C27	1.39	N12—C50	1.343 (4)
C26—H26	0.95	C46—C47	1.384 (5)
C27—C28	1.39	C46—H46	0.95

C27—H27	0.95	C47—C48	1.394 (4)
C28—C29	1.39	C47—H47	0.95
C29—C30	1.39	C48—C49	1.392 (4)
C29—H29	0.95	C48—C48^{ii}	1.484 (6)
C30—H30	0.95	C49—C50	1.373 (5)
N7′—C21′	1.39	C49—H49	0.95
N7′—C25′	1.39	C50—H50	0.95
C21′—C22′	1.39	N13—C51	1.335 (5)
C21′—H21′	0.95	N13—C55	1.337 (5)
C22′—C23′	1.39	C51—C52	1.384 (5)
C22′—H22′	0.95	C51—H51	0.95
C23′—C24′	1.39	C52—C53	1.392 (5)
C23′—C28′	1.530 (6)	C52—H52	0.95
C24′—C25′	1.39	C53—C54	1.383 (5)
C24′—H24′	0.95	C53—C53^{iii}	1.494 (7)
C25′—H25′	0.95	C54—C55	1.381 (5)
N8′—C26′	1.39	C54—H54	0.95
N8′—C30′	1.39	C55—H55	0.95

C6—O1—H1O	113 (3)	N8′—C26′—H26′	120.0
C8—O2—H2O	113 (3)	C26′—C27′—C28′	120.0
C16—O3—H3O	106 (4)	C26′—C27′—H27′	120.0
C18—O4—H4O	108 (3)	C28′—C27′—H27′	120.0
H1w1—O1w—H1w2	114 (5)	C29′—C28′—C27′	120.0
C3—N1—C2	122.8 (3)	C29′—C28′—C23′	121.6 (5)
C3—N1—H1N	117 (2)	C27′—C28′—C23′	117.1 (5)
C2—N1—H1N	120 (2)	C28′—C29′—C30′	120.0
C3—N2—N3	121.8 (3)	C28′—C29′—H29′	120.0
C3—N2—H2N	115 (2)	C30′—C29′—H29′	120.0
N3—N2—H2N	123 (2)	C29′—C30′—N8′	120.0
C4—N3—N2	114.8 (3)	C29′—C30′—H30′	120.0
C13—N4—C12	122.8 (3)	N8′—C30′—H30′	120.0
C13—N4—H4N	119 (2)	C31—N9—C35	120.0
C12—N4—H4N	118 (2)	C32—C31—N9	120.0
C13—N5—N6	119.8 (3)	C32—C31—H31	120.0
C13—N5—H5N	116 (2)	N9—C31—H31	120.0
N6—N5—H5N	123 (2)	C31—C32—C33	120.0
C14—N6—N5	116.7 (3)	C31—C32—H32	120.0
C2—C1—H1A	109.5	C33—C32—H32	120.0
C2—C1—H1B	109.5	C32—C33—C34	120.0
H1A—C1—H1B	109.5	C32—C33—C38	119.9 (4)
C2—C1—H1C	109.5	C34—C33—C38	120.1 (4)
H1A—C1—H1C	109.5	C33—C34—C35	120.0
H1B—C1—H1C	109.5	C33—C34—H34	120.0
N1—C2—C1	113.3 (3)	C35—C34—H34	120.0
N1—C2—H2A	108.9	C34—C35—N9	120.0
C1—C2—H2A	108.9	C34—C35—H35	120.0
N1—C2—H2B	108.9	N9—C35—H35	120.0

C1—C2—H2B	108.9	C36—N10—C40	120.0
H2A—C2—H2B	107.7	N10—C36—C37	120.0
N1—C3—N2	117.4 (3)	N10—C36—H36	120.0
N1—C3—S1	124.2 (2)	C37—C36—H36	120.0
N2—C3—S1	118.4 (2)	C36—C37—C38	120.0
N3—C4—C5	121.4 (3)	C36—C37—H37	120.0
N3—C4—H4	119.3	C38—C37—H37	120.0
C5—C4—H4	119.3	C39—C38—C37	120.0
C10—C5—C6	116.9 (3)	C39—C38—C33	119.7 (4)
C10—C5—C4	122.2 (3)	C37—C38—C33	120.3 (4)
C6—C5—C4	120.9 (3)	C38—C39—C40	120.0
O1—C6—C7	121.0 (3)	C38—C39—H39	120.0
O1—C6—C5	118.1 (3)	C40—C39—H39	120.0
C7—C6—C5	120.9 (3)	C39—C40—N10	120.0
C8—C7—C6	120.3 (3)	C39—C40—H40	120.0
C8—C7—H7	119.9	N10—C40—H40	120.0
C6—C7—H7	119.9	C31′—N9′—C35′	120.0
O2—C8—C7	122.4 (3)	C32′—C31′—N9′	120.0
O2—C8—C9	117.3 (3)	C32′—C31′—H31′	120.0
C7—C8—C9	120.3 (3)	N9′—C31′—H31′	120.0
C10—C9—C8	118.7 (3)	C31′—C32′—C33′	120.0
C10—C9—H9	120.6	C31′—C32′—H32′	120.0
C8—C9—H9	120.6	C33′—C32′—H32′	120.0
C9—C10—C5	122.9 (3)	C32′—C33′—C34′	120.0
C9—C10—H10	118.6	C32′—C33′—C38′	120.1 (4)
C5—C10—H10	118.6	C34′—C33′—C38′	119.9 (4)
C12—C11—H11A	109.5	C35′—C34′—C33′	120.0
C12—C11—H11B	109.5	C35′—C34′—H34′	120.0
H11A—C11—H11B	109.5	C33′—C34′—H34′	120.0
C12—C11—H11C	109.5	C34′—C35′—N9′	120.0
H11A—C11—H11C	109.5	C34′—C35′—H35′	120.0
H11B—C11—H11C	109.5	N9′—C35′—H35′	120.0
N4—C12—C11	112.5 (3)	C36′—N10′—C40′	120.0
N4—C12—H12A	109.1	N10′—C36′—C37′	120.0
C11—C12—H12A	109.1	N10′—C36′—H36′	120.0
N4—C12—H12B	109.1	C37′—C36′—H36′	120.0
C11—C12—H12B	109.1	C38′—C37′—C36′	120.0
H12A—C12—H12B	107.8	C38′—C37′—H37′	120.0
N4—C13—N5	115.5 (3)	C36′—C37′—H37′	120.0
N4—C13—S2	124.9 (2)	C37′—C38′—C39′	120.0
N5—C13—S2	119.5 (2)	C37′—C38′—C33′	120.6 (4)
N6—C14—C15	120.0 (3)	C39′—C38′—C33′	119.4 (4)
N6—C14—H14	120.0	C40′—C39′—C38′	120.0
C15—C14—H14	120.0	C40′—C39′—H39′	120.0
C20—C15—C16	118.2 (3)	C38′—C39′—H39′	120.0
C20—C15—C14	121.8 (3)	C39′—C40′—N10′	120.0
C16—C15—C14	120.1 (3)	C39′—C40′—H40′	120.0
O3—C16—C17	122.0 (3)	N10′—C40′—H40′	120.0

O3—C16—C15	117.9 (3)	C41—N11—C45	120.0
C17—C16—C15	120.1 (3)	N11—C41—C42	120.0
C16—C17—C18	120.5 (3)	N11—C41—H41	120.0
C16—C17—H17	119.8	C42—C41—H41	120.0
C18—C17—H17	119.8	C41—C42—C43	120.0
O4—C18—C17	121.7 (3)	C41—C42—H42	120.0
O4—C18—C19	118.1 (3)	C43—C42—H42	120.0
C17—C18—C19	120.2 (3)	C44—C43—C42	120.0
C20—C19—C18	119.0 (3)	C44—C43—C43^{i}	109.9 (7)
C20—C19—H19	120.5	C42—C43—C43^{i}	129.1 (7)
C18—C19—H19	120.5	C45—C44—C43	120.0
C19—C20—C15	122.1 (3)	C45—C44—H44	120.0
C19—C20—H20	118.9	C43—C44—H44	120.0
C15—C20—H20	118.9	C44—C45—N11	120.0
C21—N7—C25	120.0	C44—C45—H45	120.0
N7—C21—C22	120.0	N11—C45—H45	120.0
N7—C21—H21	120.0	C41′—N11′—C45′	120.0
C22—C21—H21	120.0	C42′—C41′—N11′	120.0
C21—C22—C23	120.0	C42′—C41′—H41′	120.0
C21—C22—H22	120.0	N11′—C41′—H41′	120.0
C23—C22—H22	120.0	C41′—C42′—C43′	120.0
C24—C23—C22	120.0	C41′—C42′—H42′	120.0
C24—C23—C28	126.3 (5)	C43′—C42′—H42′	120.0
C22—C23—C28	112.9 (5)	C42′—C43′—C44′	120.0
C25—C24—C23	120.0	C42′—C43′—C43′^{i}	116.6 (6)
C25—C24—H24	120.0	C44′—C43′—C43′^{i}	122.0 (6)
C23—C24—H24	120.0	C43′—C44′—C45′	120.0
C24—C25—N7	120.0	C43′—C44′—H44′	120.0
C24—C25—H25	120.0	C45′—C44′—H44′	120.0
N7—C25—H25	120.0	C44′—C45′—N11′	120.0
C26—N8—C30	120.0	C44′—C45′—H45′	120.0
C27—C26—N8	120.0	N11′—C45′—H45′	120.0
C27—C26—H26	120.0	C46—N12—C50	115.3 (3)
N8—C26—H26	120.0	N12—C46—C47	124.4 (3)
C26—C27—C28	120.0	N12—C46—H46	117.8
C26—C27—H27	120.0	C47—C46—H46	117.8
C28—C27—H27	120.0	C46—C47—C48	119.7 (3)
C29—C28—C27	120.0	C46—C47—H47	120.1
C29—C28—C23	112.9 (5)	C48—C47—H47	120.1
C27—C28—C23	126.9 (5)	C49—C48—C47	116.0 (3)
C28—C29—C30	120.0	C49—C48—C48^{ii}	122.3 (3)
C28—C29—H29	120.0	C47—C48—C48^{ii}	121.7 (3)
C30—C29—H29	120.0	C50—C49—C48	120.0 (3)
C29—C30—N8	120.0	C50—C49—H49	120.0
C29—C30—H30	120.0	C48—C49—H49	120.0
N8—C30—H30	120.0	N12—C50—C49	124.5 (3)
C21′—N7′—C25′	120.0	N12—C50—H50	117.7
C22′—C21′—N7′	120.0	C49—C50—H50	117.7

C22′—C21′—H21′	120.0	C51—N13—C55	115.0 (3)
N7′—C21′—H21′	120.0	N13—C51—C52	124.5 (4)
C21′—C22′—C23′	120.0	N13—C51—H51	117.7
C21′—C22′—H22′	120.0	C52—C51—H51	117.7
C23′—C22′—H22′	120.0	C51—C52—C53	119.7 (3)
C24′—C23′—C22′	120.0	C51—C52—H52	120.2
C24′—C23′—C28′	115.3 (5)	C53—C52—H52	120.2
C22′—C23′—C28′	124.5 (5)	C54—C53—C52	116.3 (3)
C23′—C24′—C25′	120.0	C54—C53—C53^{iii}	121.7 (4)
C23′—C24′—H24′	120.0	C52—C53—C53^{iii}	122.0 (4)
C25′—C24′—H24′	120.0	C55—C54—C53	119.7 (3)
C24′—C25′—N7′	120.0	C55—C54—H54	120.2
C24′—C25′—H25′	120.0	C53—C54—H54	120.2
N7′—C25′—H25′	120.0	N13—C55—C54	124.8 (3)
C26′—N8′—C30′	120.0	N13—C55—H55	117.6
C27′—C26′—N8′	120.0	C54—C55—H55	117.6
C27′—C26′—H26′	120.0		

C3—N2—N3—C4	173.4 (3)	C24′—C23′—C28′—C27′	−178.5 (3)
C13—N5—N6—C14	172.5 (3)	C22′—C23′—C28′—C27′	−4.4 (6)
C3—N1—C2—C1	−86.2 (4)	C27′—C28′—C29′—C30′	0.0
C2—N1—C3—N2	−176.4 (3)	C23′—C28′—C29′—C30′	166.7 (6)
C2—N1—C3—S1	2.2 (4)	C28′—C29′—C30′—N8′	0.0
N3—N2—C3—N1	3.3 (4)	C26′—N8′—C30′—C29′	0.0
N3—N2—C3—S1	−175.4 (2)	C35—N9—C31—C32	0.0
N2—N3—C4—C5	180.0 (3)	N9—C31—C32—C33	0.0
N3—C4—C5—C10	5.7 (5)	C31—C32—C33—C34	0.0
N3—C4—C5—C6	−175.8 (3)	C31—C32—C33—C38	179.4 (6)
C10—C5—C6—O1	−179.5 (3)	C32—C33—C34—C35	0.0
C4—C5—C6—O1	1.9 (4)	C38—C33—C34—C35	−179.4 (6)
C10—C5—C6—C7	−0.4 (5)	C33—C34—C35—N9	0.0
C4—C5—C6—C7	−179.0 (3)	C31—N9—C35—C34	0.0
O1—C6—C7—C8	178.8 (3)	C40—N10—C36—C37	0.0
C5—C6—C7—C8	−0.2 (5)	N10—C36—C37—C38	0.0
C6—C7—C8—O2	−179.6 (3)	C36—C37—C38—C39	0.0
C6—C7—C8—C9	0.7 (5)	C36—C37—C38—C33	−179.9 (6)
O2—C8—C9—C10	179.9 (3)	C32—C33—C38—C39	146.9 (4)
C7—C8—C9—C10	−0.4 (5)	C34—C33—C38—C39	−33.7 (6)
C8—C9—C10—C5	−0.3 (5)	C32—C33—C38—C37	−33.2 (6)
C6—C5—C10—C9	0.7 (5)	C34—C33—C38—C37	146.2 (3)
C4—C5—C10—C9	179.3 (3)	C37—C38—C39—C40	0.0
C13—N4—C12—C11	−80.2 (4)	C33—C38—C39—C40	179.9 (6)
C12—N4—C13—N5	176.5 (3)	C38—C39—C40—N10	0.0
C12—N4—C13—S2	−4.5 (5)	C36—N10—C40—C39	0.0
N6—N5—C13—N4	7.0 (4)	C35′—N9′—C31′—C32′	0.0
N6—N5—C13—S2	−172.1 (2)	N9′—C31′—C32′—C33′	0.0
N5—N6—C14—C15	−178.4 (3)	C31′—C32′—C33′—C34′	0.0
N6—C14—C15—C20	9.0 (5)	C31′—C32′—C33′—C38′	−178.4 (6)

N6—C14—C15—C16	−171.3 (3)	C32′—C33′—C34′—C35′	0.0
C20—C15—C16—O3	−177.7 (3)	C38′—C33′—C34′—C35′	178.4 (6)
C14—C15—C16—O3	2.7 (4)	C33′—C34′—C35′—N9′	0.0
C20—C15—C16—C17	1.3 (4)	C31′—N9′—C35′—C34′	0.0
C14—C15—C16—C17	−178.4 (3)	C40′—N10′—C36′—C37′	0.0
O3—C16—C17—C18	177.3 (3)	N10′—C36′—C37′—C38′	0.0
C15—C16—C17—C18	−1.6 (4)	C36′—C37′—C38′—C39′	0.0
C16—C17—C18—O4	−179.0 (3)	C36′—C37′—C38′—C33′	178.9 (6)
C16—C17—C18—C19	1.0 (4)	C32′—C33′—C38′—C37′	19.7 (7)
O4—C18—C19—C20	179.8 (3)	C34′—C33′—C38′—C37′	−158.7 (3)
C17—C18—C19—C20	−0.2 (4)	C32′—C33′—C38′—C39′	−161.4 (4)
C18—C19—C20—C15	−0.1 (5)	C34′—C33′—C38′—C39′	20.3 (6)
C16—C15—C20—C19	−0.4 (4)	C37′—C38′—C39′—C40′	0.0
C14—C15—C20—C19	179.2 (3)	C33′—C38′—C39′—C40′	−178.9 (6)
C25—N7—C21—C22	0.0	C38′—C39′—C40′—N10′	0.0
N7—C21—C22—C23	0.0	C36′—N10′—C40′—C39′	0.0
C21—C22—C23—C24	0.0	C45—N11—C41—C42	0.0
C21—C22—C23—C28	170.0 (5)	N11—C41—C42—C43	0.0
C22—C23—C24—C25	0.0	C41—C42—C43—C44	0.0
C28—C23—C24—C25	−168.5 (6)	C41—C42—C43—C43^{i}	167.5 (10)
C23—C24—C25—N7	0.0	C42—C43—C44—C45	0.0
C21—N7—C25—C24	0.0	C43^{i}—C43—C44—C45	−169.7 (8)
C30—N8—C26—C27	0.0	C43—C44—C45—N11	0.0
N8—C26—C27—C28	0.0	C41—N11—C45—C44	0.0
C26—C27—C28—C29	0.0	C45′—N11′—C41′—C42′	0.0
C26—C27—C28—C23	174.9 (6)	N11′—C41′—C42′—C43′	0.0
C24—C23—C28—C29	−4.5 (6)	C41′—C42′—C43′—C44′	0.0
C22—C23—C28—C29	−173.7 (3)	C41′—C42′—C43′—C43′^{i}	−166.4 (9)
C24—C23—C28—C27	−179.7 (3)	C42′—C43′—C44′—C45′	0.0
C22—C23—C28—C27	11.1 (6)	C43′^{i}—C43′—C44′—C45′	165.6 (10)
C27—C28—C29—C30	0.0	C43′—C44′—C45′—N11′	0.0
C23—C28—C29—C30	−175.6 (5)	C41′—N11′—C45′—C44′	0.0
C28—C29—C30—N8	0.0	C50—N12—C46—C47	0.1 (5)
C26—N8—C30—C29	0.0	N12—C46—C47—C48	−0.9 (5)
C25′—N7′—C21′—C22′	0.0	C46—C47—C48—C49	1.0 (5)
N7′—C21′—C22′—C23′	0.0	C46—C47—C48—C48^{ii}	−179.2 (4)
C21′—C22′—C23′—C24′	0.0	C47—C48—C49—C50	−0.4 (5)
C21′—C22′—C23′—C28′	−173.9 (6)	C48^{ii}—C48—C49—C50	179.8 (4)
C22′—C23′—C24′—C25′	0.0	C46—N12—C50—C49	0.5 (5)
C28′—C23′—C24′—C25′	174.5 (5)	C48—C49—C50—N12	−0.4 (5)
C23′—C24′—C25′—N7′	0.0	C55—N13—C51—C52	0.8 (6)
C21′—N7′—C25′—C24′	0.0	N13—C51—C52—C53	1.1 (7)
C30′—N8′—C26′—C27′	0.0	C51—C52—C53—C54	−2.2 (5)
N8′—C26′—C27′—C28′	0.0	C51—C52—C53—C53^{iii}	179.3 (4)
C26′—C27′—C28′—C29′	0.0	C52—C53—C54—C55	1.4 (5)
C26′—C27′—C28′—C23′	−167.3 (5)	C53^{iii}—C53—C54—C55	179.9 (4)

C24′—C23′—C28′—C29′	14.4 (6)	C51—N13—C55—C54	−1.7 (6)
C22′—C23′—C28′—C29′	−171.4 (4)	C53—C54—C55—N13	0.5 (6)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1o···N8^{iv}	0.84 (4)	1.92 (1)	2.755 (5)	171 (5)
O1—H1o···N8′^{iv}	0.84 (4)	1.97 (1)	2.798 (5)	171 (5)
O2—H2o···N7	0.84 (4)	1.93 (1)	2.768 (5)	172 (4)
O2—H2o···N7′	0.84 (4)	1.93 (1)	2.766 (5)	178 (4)
O3—H3o···N10^{i}	0.84 (4)	1.91 (1)	2.751 (5)	178 (5)
O3—H3o···N10′^{i}	0.84 (4)	1.90 (1)	2.736 (5)	172 (5)
O4—H4o···N11	0.84 (4)	1.90 (1)	2.735 (4)	174 (4)
O1*w*—H1*w*1···O4	0.84 (3)	2.04 (2)	2.843 (4)	160 (5)
O1*w*—H1*w*2···N9	0.84 (3)	1.94 (1)	2.782 (5)	175 (7)
O1*w*—H1*w*2···N9′	0.84 (3)	1.91 (3)	2.722 (5)	160 (7)
N1—H1n···N12	0.88 (3)	2.31 (3)	3.024 (4)	138 (3)
N2—H2n···O1*w*	0.88 (3)	1.88 (1)	2.757 (4)	175 (4)
N4—H4n···N13	0.88 (3)	2.56 (2)	3.315 (4)	144 (3)
N5—H5n···O2^{v}	0.88 (1)	2.51 (2)	3.325 (4)	155 (3)

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