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13b,13c-Di-2-pyridyl-5,7,12,13b,13c,14-hexahydro-6*H*,13*H*-5a,6a,12a,13a-tetraazabenz[5,6]azuleno[2,1,8-*ija*]benz[*f*]azulene-6,13-dione methanol hemisolvate

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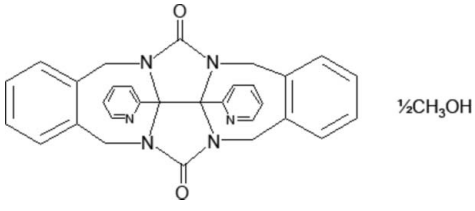
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.074; wR factor = 0.208; data-to-parameter ratio = 12.2.

The title compound, $C_{30}H_{24}N_6O_2\cdot0.5CH_3OH$, a glycoluril derivative with two pyridine substituents on the convex face of the glycoluril system, is an important intermediate for the synthesis of more complex glycoluril derivatives. The compound crystallizes with two independent molecules in the asymmetric unit, one of which exhibits disorder of one benzene ring over two orientations with refined site occupancy factors 0.65 (4):0.35 (4). The crystal structure contains several short C—H···O contacts, and the methanol molecule forms an O—H···O hydrogen bond to one of the glycoluril molecules.

Related literature

For synthetic details, see: Wang *et al.* (2006). For general related literature, see: Adrian & Wilcox (1989); Behrend *et al.* (1905); Freeman *et al.* (1981); Rowan *et al.* (1999); Rebek (2005); Sanderson *et al.* (1989); Wu *et al.* (2002).



Experimental

Crystal data

$C_{30}H_{24}N_6O_2\cdot0.5CH_4O$
M_r = 516.57
Monoclinic, $P2_1/c$
a = 15.0063 (10) Å
b = 20.5919 (13) Å
c = 18.0864 (12) Å
β = 113.405 (1)°
V = 5129.0 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 292 (2) K
0.30 × 0.30 × 0.20 mm

Data collection

Bruker SMART 4K CCD diffractometer
Absorption correction: none
68334 measured reflections
8988 independent reflections
7325 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.208
S = 1.08
8988 reflections
734 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.68 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
O5—H5···O2	0.82	2.02	2.840 (8)	178
C2—H2A···O5^{i}	0.93	2.35	3.279 (9)	174
C22—H22A···O2^{ii}	0.93	2.57	3.462 (4)	160
C43—H43A···O3^{iii}	0.93	2.60	3.249 (5)	128
C46—H46A···O1	0.93	2.59	3.340 (4)	138
C51—H51A···O2^{iv}	0.93	2.52	3.419 (4)	164

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2258).

References

Adrian, J. C. Jr & Wilcox, C. S. (1989). *J. Am. Chem. Soc.* **111**, 8055–8057.
Behrend, R., Meyer, E. & Rusche, F. (1905). *Liebigs Ann. Chem.* **339**, 1–37.
Bruker (2001). *SMART* (Version 5.628) and *SAINT* (Version 6.45). Bruker AXS Inc., Madison, Wisconsin, USA.
Freeman, W. A., Mock, W. L. & Shih, N. Y. (1981). *J. Am. Chem. Soc.* **103**, 7367–7368.
Rebek, J. Jr (2005). *Angew. Chem. Int. Ed.* **44**, 2068–2078.
Rowan, A. E., Elemans, J. A. A. W. & Nolte, R. J. M. (1999). *Acc. Chem. Res.* **32**, 995–1006.
Sanderson, P. E. J., Kilburn, J. D. & Still, W. C. (1989). *J. Am. Chem. Soc.* **111**, 8314–8315.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Wang, Z. G., Zhou, B. H., Chen, Y. F., Yin, G. D., Li, Y. T., Wu, A. X. & Isaacs, L. (2006). *J. Org. Chem.* **71**, 4502–4508.
Wu, A. X., Fettinger, J. C. & Isaacs, L. (2002). *Tetrahedron*, **58**, 9769–9777.

supporting information

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13b,13c-Di-2-pyridyl-5,7,12,13b,13c,14-hexahydro-6*H*,13*H*-5a,6a,12a,13a-tetraazabenz[5,6]azuleno[2,1,8-*ija*]benz[*f*]azulene-6,13-dione methanol hemisolvate

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S1. Comment

In 1905, Behrend synthesized an insoluble polymeric material now known as Behrend's polymer using an acid-induced condensation reaction of glycoluril and formaldehyde (Behrend *et al.*, 1905). The molecular structure was characterized and named as cucurbituril by Mock and co-workers in 1981 (Freeman *et al.*, 1981). Glycoluril and its derivatives have been widely used as building blocks for supramolecular chemistry, for example in the formation of molecular clefts, armatures, bowls, tweezers and clips (Adrian & Wilcox, 1989; Freeman *et al.*, 1981; Rebek, 2005; Sanderson *et al.*, 1989; Rowan *et al.*, 1999; Wu *et al.*, 2002). As a part of our ongoing investigation into glycoluril derivatives, we report the crystal structure of the title compound.

The clip-shaped molecular structure is shown in Fig. 1. It has six fused rings and two pyridine rings. The crystal structure contains several short C—H···O contacts, and the methanol molecule forms an O—H···O hydrogen bond to one of the glycoluril molecules.

S2. Experimental

The compound was synthesized according to a literature procedure (Wang *et al.*, 2006) in 31% isolated yield. Crystals suitable for X-ray analysis were obtained by slow evaporation of a chloroform and methanol solution (20:1 *v*/*v*) at 293 K.

S3. Refinement

One phenyl ring of one molecule is disordered. It was modelled in two orientations, constrained to be regular hexagons, with refined site occupancy factors 0.35 (4):0.65 (4). H atoms bound to C were positioned geometrically and constrained to ride on their parent atoms, with d(C—H) = 0.93–0.97 Å and with U_{iso}(H) = 1.2 or 1.5U_{eq}(C). The H atom of the methanol molecule was placed along the O5···O2 vector with d(O5—H5) = 0.82 Å and refined as riding on O5 with U_{iso}(H) = 1.5U_{eq}(O).

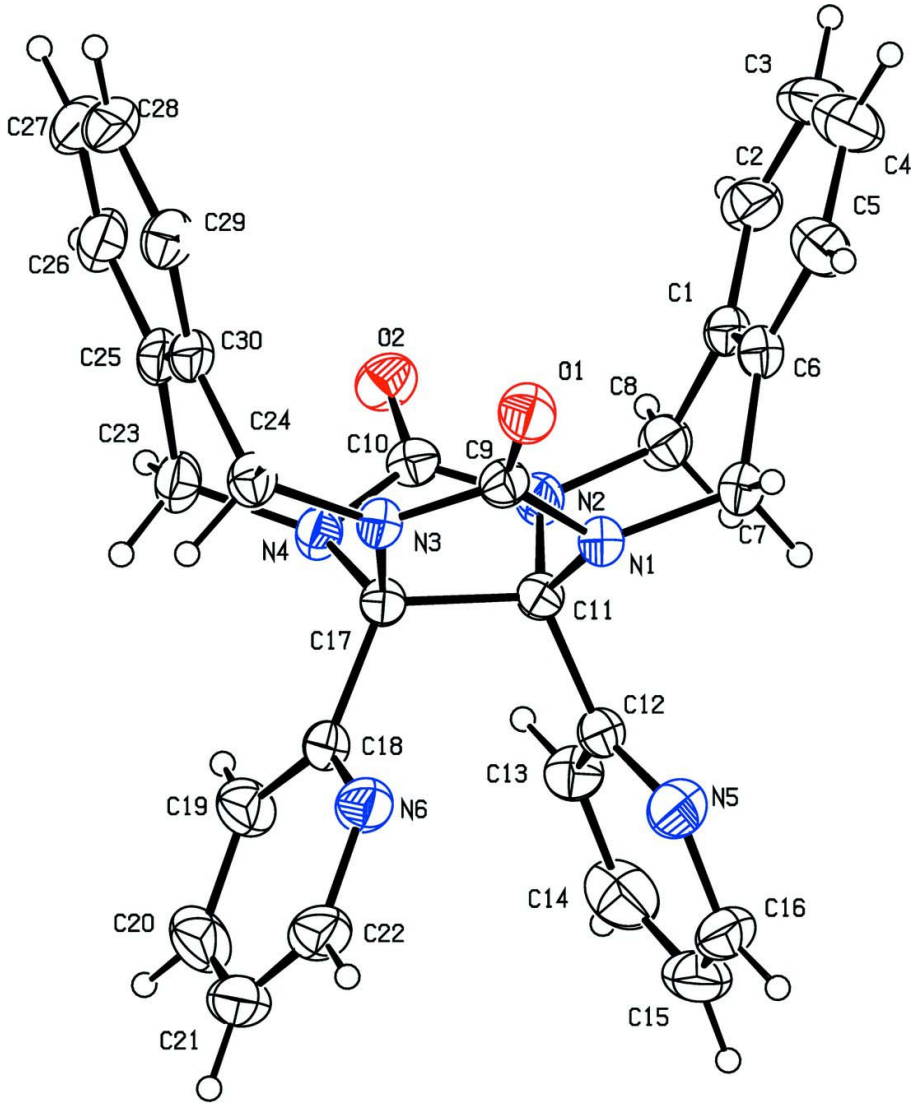

Figure 1

The structure of the non-disordered molecule in the asymmetric unit, with displacement ellipsoids drawn at the 50% proability level for non-H atoms.

13*b*,13*c*-Di-2-pyridyl-5,7,12,13*b*,13*c*,14-hexahydro-6*H*,13*H*- 5a,6a,12a,13a-tetraazabenz[5,6]azuleno[2,1,8-*ija*]benz[*f*]azulene- 6,13-dione methanol hemisolvate

Crystal data

$C_{30}H_{24}N_6O_2 \cdot 0.5CH_4O$
$M_r = 516.57$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 15.0063\ (10)$ Å
$b = 20.5919\ (13)$ Å
$c = 18.0864\ (12)$ Å
$\beta = 113.405\ (1)°$
$V = 5129.0\ (6)$ Å^3
$Z = 8$

$F(000) = 2168$
$D_x = 1.338$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9377 reflections
$\theta = 2.3–24.5°$
$\mu = 0.09$ mm^{-1}
$T = 292$ K
Block, colourless
0.30 × 0.30 × 0.20 mm

Data collection

Bruker SMART 4K CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
68334 measured reflections
8988 independent reflections
7325 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.5°$
$h = -17 \rightarrow 17$
$k = -24 \rightarrow 24$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.074$
$wR(F^2) = 0.208$
$S = 1.08$
8988 reflections
734 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
$w = 1/[\sigma^2(F_o^2) + (0.0999P)^2 + 4.5477P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.68$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ 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}	Occ. (<1)
O1	0.35883 (16)	0.31312 (11)	0.35693 (14)	0.0567 (6)	
O2	0.07810 (17)	0.35765 (11)	0.48148 (13)	0.0545 (6)	
O3	0.39873 (17)	0.60364 (10)	−0.02765 (13)	0.0543 (6)	
O4	0.19640 (17)	0.56407 (12)	0.16797 (17)	0.0685 (7)	
N1	0.20302 (17)	0.35391 (11)	0.29519 (14)	0.0407 (5)	
N2	0.09710 (19)	0.37701 (11)	0.36331 (14)	0.0445 (6)	
N3	0.22538 (17)	0.25952 (11)	0.35859 (14)	0.0392 (5)	
N4	0.09295 (18)	0.27518 (11)	0.40090 (14)	0.0425 (6)	
N5	0.0373 (2)	0.35266 (15)	0.14809 (18)	0.0683 (8)	
N6	0.0988 (2)	0.20264 (13)	0.21698 (16)	0.0559 (7)	
N7	0.41144 (16)	0.52050 (10)	0.06155 (13)	0.0367 (5)	
N8	0.30693 (16)	0.50807 (11)	0.13356 (14)	0.0386 (5)	
N9	0.42379 (18)	0.62206 (11)	0.10440 (14)	0.0428 (6)	
N10	0.35400 (18)	0.60006 (12)	0.20074 (15)	0.0469 (6)	
N11	0.45044 (18)	0.43855 (11)	0.24979 (14)	0.0436 (6)	
N12	0.6039 (2)	0.60347 (15)	0.22005 (18)	0.0622 (8)	
C1	0.1993 (2)	0.47681 (13)	0.39507 (18)	0.0459 (7)	

C2	0.2288 (3)	0.51885 (16)	0.4606 (2)	0.0591 (9)
H2A	0.1878	0.5273	0.4868	0.071*
C3	0.3195 (3)	0.54821 (18)	0.4871 (2)	0.0719 (11)
H3A	0.3386	0.5767	0.5305	0.086*
C4	0.3810 (3)	0.53545 (19)	0.4495 (3)	0.0751 (11)
H4A	0.4414	0.5555	0.4668	0.090*
C5	0.3524 (3)	0.49261 (17)	0.3858 (2)	0.0619 (9)
H5A	0.3947	0.4833	0.3611	0.074*
C6	0.2621 (2)	0.46312 (14)	0.35764 (18)	0.0467 (7)
C7	0.2320 (2)	0.41998 (14)	0.28440 (18)	0.0456 (7)
H7A	0.1781	0.4405	0.2411	0.055*
H7B	0.2856	0.4172	0.2674	0.055*
C8	0.0980 (2)	0.44777 (14)	0.36560 (19)	0.0490 (7)
H8A	0.0670	0.4622	0.4006	0.059*
H8B	0.0601	0.4641	0.3119	0.059*
C9	0.2722 (2)	0.30936 (14)	0.34003 (16)	0.0402 (6)
C10	0.0892 (2)	0.33885 (14)	0.42135 (17)	0.0416 (6)
C11	0.1102 (2)	0.34124 (13)	0.29936 (16)	0.0388 (6)
C12	0.0234 (2)	0.35063 (13)	0.21877 (18)	0.0439 (7)
C13	−0.0634 (2)	0.35483 (15)	0.2217 (2)	0.0498 (7)
H13A	−0.0701	0.3538	0.2706	0.060*
C14	−0.1411 (3)	0.3606 (2)	0.1518 (3)	0.0805 (12)
H14A	−0.2022	0.3632	0.1533	0.097*
C15	−0.1345 (3)	0.36297 (19)	0.0794 (3)	0.0781 (13)
H15A	−0.1900	0.3670	0.0321	0.094*
C16	−0.0452 (3)	0.35929 (18)	0.0773 (2)	0.0694 (11)
H16A	−0.0392	0.3612	0.0281	0.083*
C17	0.1225 (2)	0.26936 (13)	0.33361 (16)	0.0382 (6)
C18	0.0639 (2)	0.21802 (13)	0.27268 (17)	0.0431 (7)
C19	−0.0185 (2)	0.19128 (17)	0.2750 (2)	0.0594 (9)
H19A	−0.0409	0.2033	0.3141	0.071*
C20	−0.0675 (3)	0.1451 (2)	0.2162 (3)	0.0824 (13)
H20A	−0.1234	0.1253	0.2156	0.099*
C21	−0.0318 (3)	0.12943 (19)	0.1588 (3)	0.0781 (13)
H21A	−0.0631	0.0987	0.1193	0.094*
C22	0.0484 (3)	0.15912 (17)	0.1608 (2)	0.0681 (10)
H22A	0.0705	0.1490	0.1209	0.082*
C23	0.1178 (2)	0.22462 (15)	0.46324 (19)	0.0509 (8)
H23A	0.1013	0.1827	0.4368	0.061*
H23B	0.0777	0.2308	0.4935	0.061*
C24	0.2763 (2)	0.20382 (14)	0.40696 (18)	0.0458 (7)
H24A	0.3369	0.1975	0.4006	0.055*
H24B	0.2370	0.1652	0.3870	0.055*
C25	0.2223 (2)	0.22286 (14)	0.52156 (18)	0.0484 (7)
C26	0.2459 (3)	0.22988 (16)	0.6038 (2)	0.0607 (9)
H26A	0.1968	0.2373	0.6219	0.073*
C27	0.3410 (3)	0.22604 (18)	0.6590 (2)	0.0715 (11)
H27A	0.3555	0.2304	0.7137	0.086*

C28	0.4138 (3)	0.21574 (18)	0.6329 (2)	0.0690 (11)
H28A	0.4779	0.2133	0.6700	0.083*
C29	0.3925 (3)	0.20886 (16)	0.5515 (2)	0.0583 (9)
H29A	0.4426	0.2022	0.5344	0.070*
C30	0.2977 (2)	0.21183 (13)	0.49543 (18)	0.0457 (7)
C31	0.2024 (2)	0.46144 (13)	0.00128 (17)	0.0417 (7)
C32	0.1017 (2)	0.46010 (15)	−0.0412 (2)	0.0533 (8)
H32A	0.0616	0.4563	−0.0133	0.064*
C33	0.0610 (3)	0.46441 (16)	−0.1245 (2)	0.0631 (10)
H33A	−0.0060	0.4626	−0.1522	0.076*
C34	0.1189 (3)	0.47125 (17)	−0.1659 (2)	0.0621 (9)
H34A	0.0914	0.4740	−0.2218	0.075*
C35	0.2192 (3)	0.47408 (14)	−0.12473 (18)	0.0519 (8)
H35A	0.2582	0.4795	−0.1534	0.062*
C36	0.2618 (2)	0.46896 (13)	−0.04157 (17)	0.0419 (7)
C37	0.3715 (2)	0.46951 (13)	0.00118 (17)	0.0406 (6)
H37A	0.3923	0.4278	0.0272	0.049*
H37B	0.3989	0.4742	−0.0389	0.049*
C38	0.2450 (2)	0.45412 (14)	0.09123 (17)	0.0436 (7)
H38A	0.1925	0.4500	0.1094	0.052*
H38B	0.2826	0.4143	0.1053	0.052*
C39	0.4092 (2)	0.58470 (13)	0.03871 (17)	0.0387 (6)
C40	0.2763 (2)	0.55784 (14)	0.16627 (18)	0.0446 (7)
C41	0.40525 (19)	0.51493 (13)	0.13935 (16)	0.0356 (6)
C42	0.47331 (19)	0.46232 (12)	0.19148 (16)	0.0352 (6)
C43	0.5554 (2)	0.44407 (15)	0.18015 (18)	0.0480 (7)
H43A	0.5680	0.4607	0.1375	0.058*
C44	0.6183 (2)	0.40048 (17)	0.2335 (2)	0.0569 (8)
H44A	0.6747	0.3877	0.2278	0.068*
C45	0.5969 (2)	0.37631 (15)	0.29470 (19)	0.0534 (8)
H45A	0.6386	0.3473	0.3318	0.064*
C46	0.5128 (2)	0.39569 (15)	0.30031 (18)	0.0506 (8)
H46A	0.4978	0.3783	0.3414	0.061*
C47	0.43119 (19)	0.58581 (12)	0.17507 (16)	0.0368 (6)
C48	0.5309 (2)	0.59353 (13)	0.24317 (18)	0.0432 (7)
C49	0.5433 (3)	0.58788 (17)	0.3217 (2)	0.0641 (10)
H49A	0.4907	0.5801	0.3354	0.077*
C50	0.6374 (4)	0.5941 (2)	0.3814 (2)	0.0857 (14)
H50A	0.6485	0.5909	0.4357	0.103*
C51	0.7112 (3)	0.6047 (2)	0.3587 (3)	0.0900 (15)
H51A	0.7742	0.6093	0.3972	0.108*
C52	0.6927 (3)	0.6086 (2)	0.2786 (3)	0.0837 (13)
H52A	0.7448	0.6153	0.2640	0.100*
C53	0.3372 (3)	0.66679 (18)	0.2196 (3)	0.0716 (11)
H53A	0.3009	0.6635	0.2535	0.086*
H53B	0.4005	0.6839	0.2536	0.086*
C54	0.4285 (3)	0.69218 (14)	0.1041 (2)	0.0551 (8)
H54A	0.4363	0.7071	0.0561	0.066*

H54B	0.4836	0.7073	0.1509	0.066*	
C55	0.2959 (10)	0.7108 (7)	0.1688 (15)	0.044 (4)	0.35 (4)
C56	0.2138 (12)	0.7415 (6)	0.170 (2)	0.080 (6)	0.35 (4)
H56A	0.1945	0.7341	0.2117	0.096*	0.35 (4)
C57	0.1606 (8)	0.7833 (6)	0.107 (3)	0.093 (8)	0.35 (4)
H57A	0.1056	0.8038	0.1078	0.111*	0.35 (4)
C58	0.1894 (11)	0.7943 (7)	0.0442 (19)	0.081 (9)	0.35 (4)
H58A	0.1538	0.8223	0.0025	0.098*	0.35 (4)
C59	0.2715 (16)	0.7636 (8)	0.0434 (12)	0.063 (6)	0.35 (4)
H59A	0.2908	0.7710	0.0012	0.075*	0.35 (4)
C60	0.3248 (12)	0.7219 (9)	0.1057 (11)	0.049 (5)	0.35 (4)
C55′	0.2892 (6)	0.7118 (4)	0.1367 (10)	0.055 (3)	0.65 (4)
C56′	0.2005 (6)	0.7420 (4)	0.1204 (15)	0.087 (5)	0.65 (4)
H56B	0.1668	0.7339	0.1528	0.104*	0.65 (4)
C57′	0.1622 (5)	0.7843 (4)	0.0555 (15)	0.102 (7)	0.65 (4)
H57B	0.1029	0.8045	0.0445	0.122*	0.65 (4)
C58′	0.2126 (11)	0.7965 (4)	0.0070 (11)	0.104 (5)	0.65 (4)
H58B	0.1870	0.8248	−0.0365	0.125*	0.65 (4)
C59′	0.3013 (11)	0.7663 (5)	0.0233 (8)	0.084 (3)	0.65 (4)
H59B	0.3350	0.7745	−0.0092	0.100*	0.65 (4)
C60′	0.3396 (6)	0.7240 (5)	0.0882 (7)	0.052 (2)	0.65 (4)
O5	−0.0697 (6)	0.4504 (5)	0.4616 (6)	0.258 (4)	
H5	−0.0262	0.4241	0.4682	0.387*	
C61	−0.1370 (6)	0.4555 (6)	0.3734 (6)	0.212 (5)	
H61A	−0.2031	0.4517	0.3679	0.318*	
H61B	−0.1227	0.4213	0.3438	0.318*	
H61C	−0.1279	0.4968	0.3527	0.318*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0433 (13)	0.0615 (14)	0.0635 (14)	−0.0031 (10)	0.0194 (11)	0.0080 (11)
O2	0.0630 (14)	0.0619 (14)	0.0436 (12)	0.0062 (11)	0.0264 (11)	−0.0034 (10)
O3	0.0763 (15)	0.0461 (12)	0.0429 (12)	0.0023 (10)	0.0260 (11)	0.0108 (10)
O4	0.0515 (14)	0.0611 (15)	0.105 (2)	−0.0080 (11)	0.0435 (14)	−0.0164 (14)
N1	0.0454 (13)	0.0360 (12)	0.0385 (13)	−0.0021 (10)	0.0144 (11)	0.0030 (10)
N2	0.0605 (16)	0.0339 (12)	0.0416 (13)	0.0004 (11)	0.0230 (12)	−0.0023 (10)
N3	0.0423 (13)	0.0357 (12)	0.0378 (12)	0.0014 (10)	0.0140 (10)	0.0041 (10)
N4	0.0514 (14)	0.0398 (13)	0.0399 (13)	0.0016 (11)	0.0218 (11)	0.0047 (10)
N5	0.087 (2)	0.0580 (18)	0.0514 (17)	0.0009 (15)	0.0179 (16)	0.0024 (14)
N6	0.0739 (19)	0.0483 (15)	0.0435 (15)	0.0020 (13)	0.0210 (14)	−0.0044 (12)
N7	0.0405 (12)	0.0330 (11)	0.0322 (12)	0.0002 (9)	0.0097 (10)	0.0026 (9)
N8	0.0357 (12)	0.0348 (12)	0.0433 (13)	−0.0040 (9)	0.0135 (10)	−0.0010 (10)
N9	0.0537 (14)	0.0300 (12)	0.0423 (13)	−0.0022 (10)	0.0164 (11)	0.0037 (10)
N10	0.0496 (14)	0.0411 (13)	0.0553 (15)	−0.0076 (11)	0.0264 (12)	−0.0116 (11)
N11	0.0489 (14)	0.0401 (13)	0.0392 (13)	−0.0025 (11)	0.0146 (11)	0.0071 (11)
N12	0.0423 (15)	0.0643 (18)	0.0726 (19)	−0.0092 (13)	0.0149 (14)	−0.0130 (15)
C1	0.0575 (18)	0.0294 (14)	0.0404 (16)	0.0046 (12)	0.0085 (14)	0.0043 (12)

C2	0.073 (2)	0.0465 (18)	0.0454 (18)	0.0114 (16)	0.0101 (16)	−0.0056 (14)
C3	0.075 (3)	0.057 (2)	0.058 (2)	−0.0013 (19)	−0.001 (2)	−0.0200 (17)
C4	0.065 (2)	0.063 (2)	0.083 (3)	−0.0108 (19)	0.013 (2)	−0.018 (2)
C5	0.059 (2)	0.0515 (19)	0.072 (2)	−0.0100 (16)	0.0225 (18)	−0.0065 (17)
C6	0.0570 (18)	0.0340 (15)	0.0443 (16)	−0.0008 (13)	0.0152 (14)	0.0050 (12)
C7	0.0538 (17)	0.0419 (15)	0.0413 (16)	−0.0046 (13)	0.0191 (14)	0.0066 (13)
C8	0.0604 (19)	0.0369 (15)	0.0481 (17)	0.0089 (13)	0.0196 (15)	−0.0010 (13)
C9	0.0455 (17)	0.0406 (15)	0.0349 (15)	−0.0006 (12)	0.0163 (13)	−0.0001 (12)
C10	0.0410 (15)	0.0448 (16)	0.0371 (15)	0.0021 (12)	0.0135 (12)	−0.0022 (13)
C11	0.0452 (15)	0.0348 (14)	0.0353 (14)	−0.0004 (12)	0.0147 (12)	−0.0011 (11)
C12	0.0532 (18)	0.0300 (14)	0.0423 (16)	0.0023 (12)	0.0125 (14)	0.0001 (12)
C13	0.0404 (17)	0.0543 (18)	0.0531 (18)	0.0045 (13)	0.0171 (15)	−0.0012 (14)
C14	0.050 (2)	0.066 (2)	0.111 (4)	0.0054 (18)	0.018 (2)	−0.006 (2)
C15	0.069 (3)	0.055 (2)	0.071 (3)	0.0021 (18)	−0.013 (2)	−0.0084 (19)
C16	0.097 (3)	0.057 (2)	0.0376 (18)	0.001 (2)	0.0095 (19)	−0.0014 (15)
C17	0.0431 (15)	0.0342 (14)	0.0366 (14)	−0.0007 (11)	0.0153 (12)	0.0014 (11)
C18	0.0454 (16)	0.0349 (14)	0.0417 (16)	0.0003 (12)	0.0095 (13)	0.0045 (12)
C19	0.0473 (18)	0.059 (2)	0.068 (2)	−0.0096 (15)	0.0185 (16)	0.0004 (17)
C20	0.052 (2)	0.065 (2)	0.104 (3)	−0.0199 (18)	0.004 (2)	0.013 (2)
C21	0.088 (3)	0.049 (2)	0.061 (2)	−0.003 (2)	−0.009 (2)	−0.0051 (18)
C22	0.097 (3)	0.0494 (19)	0.0464 (19)	0.006 (2)	0.0160 (19)	−0.0099 (16)
C23	0.065 (2)	0.0446 (17)	0.0488 (18)	0.0008 (14)	0.0285 (16)	0.0108 (14)
C24	0.0520 (17)	0.0369 (15)	0.0465 (17)	0.0068 (13)	0.0175 (14)	0.0041 (13)
C25	0.068 (2)	0.0330 (14)	0.0429 (17)	0.0021 (14)	0.0203 (15)	0.0084 (12)
C26	0.094 (3)	0.0449 (18)	0.0457 (19)	0.0077 (17)	0.0307 (19)	0.0088 (14)
C27	0.109 (3)	0.053 (2)	0.0367 (18)	0.009 (2)	0.013 (2)	0.0074 (15)
C28	0.079 (3)	0.054 (2)	0.049 (2)	0.0058 (18)	−0.0007 (19)	0.0093 (16)
C29	0.060 (2)	0.0464 (18)	0.057 (2)	0.0100 (15)	0.0111 (17)	0.0105 (15)
C30	0.0580 (19)	0.0316 (14)	0.0410 (16)	0.0053 (13)	0.0128 (14)	0.0059 (12)
C31	0.0425 (15)	0.0289 (13)	0.0448 (16)	−0.0042 (11)	0.0081 (13)	−0.0024 (12)
C32	0.0455 (17)	0.0415 (16)	0.063 (2)	−0.0067 (13)	0.0107 (15)	−0.0044 (14)
C33	0.0502 (19)	0.0500 (19)	0.062 (2)	0.0000 (15)	−0.0062 (17)	−0.0052 (16)
C34	0.066 (2)	0.056 (2)	0.0421 (18)	0.0001 (17)	−0.0025 (17)	−0.0024 (15)
C35	0.065 (2)	0.0390 (16)	0.0425 (17)	−0.0024 (14)	0.0113 (15)	−0.0031 (13)
C36	0.0479 (16)	0.0279 (13)	0.0406 (16)	−0.0001 (11)	0.0078 (13)	−0.0012 (11)
C37	0.0467 (16)	0.0342 (14)	0.0390 (15)	0.0028 (12)	0.0148 (13)	0.0000 (11)
C38	0.0432 (16)	0.0380 (15)	0.0468 (16)	−0.0082 (12)	0.0151 (13)	0.0014 (12)
C39	0.0383 (15)	0.0376 (14)	0.0392 (16)	0.0033 (11)	0.0142 (12)	0.0079 (12)
C40	0.0399 (16)	0.0428 (16)	0.0517 (17)	−0.0038 (12)	0.0187 (14)	−0.0024 (13)
C41	0.0358 (14)	0.0333 (13)	0.0345 (14)	−0.0035 (11)	0.0105 (11)	0.0018 (11)
C42	0.0379 (14)	0.0303 (13)	0.0330 (14)	−0.0017 (11)	0.0094 (11)	0.0006 (11)
C43	0.0475 (17)	0.0503 (17)	0.0453 (17)	0.0058 (14)	0.0176 (14)	0.0098 (14)
C44	0.0470 (18)	0.060 (2)	0.056 (2)	0.0154 (15)	0.0132 (15)	0.0068 (16)
C45	0.0540 (19)	0.0443 (17)	0.0456 (18)	0.0077 (14)	0.0026 (15)	0.0093 (14)
C46	0.061 (2)	0.0446 (17)	0.0381 (16)	−0.0054 (14)	0.0115 (14)	0.0094 (13)
C47	0.0370 (14)	0.0319 (13)	0.0378 (14)	−0.0006 (11)	0.0109 (12)	0.0016 (11)
C48	0.0457 (16)	0.0310 (14)	0.0438 (16)	−0.0021 (12)	0.0080 (13)	0.0001 (12)
C49	0.076 (2)	0.056 (2)	0.0451 (19)	−0.0144 (17)	0.0075 (17)	0.0062 (15)

C50	0.101 (4)	0.063 (2)	0.053 (2)	−0.009 (2)	−0.012 (2)	0.0104 (18)
C51	0.064 (3)	0.064 (3)	0.093 (4)	−0.005 (2)	−0.020 (3)	−0.010 (2)
C52	0.041 (2)	0.085 (3)	0.104 (4)	−0.0090 (19)	0.007 (2)	−0.024 (3)
C53	0.067 (2)	0.053 (2)	0.112 (3)	−0.0120 (18)	0.053 (2)	−0.030 (2)
C54	0.073 (2)	0.0297 (15)	0.0549 (19)	−0.0029 (15)	0.0171 (17)	0.0048 (13)
C55	0.037 (6)	0.033 (5)	0.048 (10)	−0.002 (4)	0.002 (6)	−0.020 (6)
C56	0.055 (7)	0.049 (7)	0.122 (17)	0.000 (5)	0.022 (10)	−0.022 (9)
C57	0.050 (7)	0.059 (8)	0.15 (2)	0.022 (6)	0.024 (10)	0.017 (11)
C58	0.051 (8)	0.044 (8)	0.104 (19)	0.012 (6)	−0.017 (10)	0.014 (9)
C59	0.062 (10)	0.029 (6)	0.057 (9)	0.008 (6)	−0.019 (6)	0.005 (5)
C60	0.052 (9)	0.031 (7)	0.051 (9)	0.015 (7)	0.007 (7)	−0.006 (6)
C55′	0.049 (4)	0.040 (4)	0.059 (8)	0.003 (3)	0.003 (4)	−0.021 (4)
C56′	0.048 (4)	0.056 (4)	0.142 (13)	0.003 (3)	0.023 (5)	−0.028 (6)
C57′	0.054 (5)	0.060 (6)	0.153 (17)	0.011 (4)	0.002 (7)	−0.004 (6)
C58′	0.087 (7)	0.054 (4)	0.118 (9)	0.003 (4)	−0.017 (7)	0.013 (5)
C59′	0.093 (7)	0.043 (4)	0.080 (6)	−0.007 (5)	−0.002 (5)	0.002 (4)
C60′	0.058 (4)	0.029 (3)	0.052 (5)	−0.007 (3)	0.003 (3)	−0.007 (3)
O5	0.199 (7)	0.350 (11)	0.249 (9)	0.050 (7)	0.115 (7)	−0.061 (8)
C61	0.123 (6)	0.364 (16)	0.150 (7)	0.087 (8)	0.056 (6)	0.070 (9)

Geometric parameters (Å, º)

O1—C9	1.213 (3)	C25—C30	1.408 (4)
O2—C10	1.227 (3)	C26—C27	1.381 (6)
O3—C39	1.211 (3)	C26—H26A	0.930
O4—C40	1.219 (4)	C27—C28	1.368 (6)
N1—C9	1.382 (4)	C27—H27A	0.930
N1—C11	1.449 (4)	C28—C29	1.383 (5)
N1—C7	1.465 (4)	C28—H28A	0.930
N2—C10	1.353 (4)	C29—C30	1.382 (5)
N2—C11	1.449 (4)	C29—H29A	0.930
N2—C8	1.458 (4)	C31—C32	1.397 (4)
N3—C9	1.360 (4)	C31—C36	1.402 (4)
N3—C17	1.440 (4)	C31—C38	1.501 (4)
N3—C24	1.461 (4)	C32—C33	1.384 (5)
N4—C10	1.369 (4)	C32—H32A	0.930
N4—C17	1.456 (4)	C33—C34	1.361 (6)
N4—C23	1.470 (4)	C33—H33A	0.930
N5—C12	1.375 (4)	C34—C35	1.390 (5)
N5—C16	1.390 (5)	C34—H34A	0.930
N6—C22	1.340 (4)	C35—C36	1.385 (4)
N6—C18	1.345 (4)	C35—H35A	0.930
N7—C39	1.381 (3)	C36—C37	1.515 (4)
N7—C41	1.451 (3)	C37—H37A	0.970
N7—C37	1.461 (3)	C37—H37B	0.970
N8—C40	1.352 (4)	C38—H38A	0.970
N8—C41	1.444 (3)	C38—H38B	0.970
N8—C38	1.456 (3)	C41—C42	1.530 (4)

N9—C39	1.358 (4)	C41—C47	1.581 (4)
N9—C54	1.446 (4)	C42—C43	1.378 (4)
N9—C47	1.446 (3)	C43—C44	1.380 (4)
N10—C40	1.388 (4)	C43—H43A	0.930
N10—C47	1.439 (4)	C44—C45	1.363 (5)
N10—C53	1.462 (4)	C44—H44A	0.930
N11—C42	1.326 (3)	C45—C46	1.365 (5)
N11—C46	1.346 (4)	C45—H45A	0.930
N12—C48	1.335 (4)	C46—H46A	0.930
N12—C52	1.337 (5)	C47—C48	1.522 (4)
C1—C6	1.390 (5)	C48—C49	1.362 (5)
C1—C2	1.391 (4)	C49—C50	1.402 (6)
C1—C8	1.519 (5)	C49—H49A	0.930
C2—C3	1.389 (5)	C50—C51	1.343 (7)
C2—H2A	0.9300	C50—H50A	0.930
C3—C4	1.372 (6)	C51—C52	1.365 (7)
C3—H3A	0.930	C51—H51A	0.930
C4—C5	1.377 (5)	C52—H52A	0.930
C4—H4A	0.930	C53—C55	1.264 (19)
C5—C6	1.384 (5)	C53—C55′	1.664 (15)
C5—H5A	0.930	C53—H53A	0.970
C6—C7	1.507 (4)	C53—H53B	0.970
C7—H7A	0.970	C54—C60′	1.409 (10)
C7—H7B	0.970	C54—C60	1.683 (17)
C8—H8A	0.970	C54—H54A	0.970
C8—H8B	0.970	C54—H54B	0.970
C11—C12	1.533 (4)	C55—C56	1.390
C11—C17	1.587 (4)	C55—C60	1.390
C12—C13	1.327 (4)	C56—C57	1.390
C13—C14	1.341 (5)	C56—H56A	0.930
C13—H13A	0.930	C57—C58	1.390
C14—C15	1.353 (7)	C57—H57A	0.930
C14—H14A	0.930	C58—C59	1.390
C15—C16	1.358 (6)	C58—H58A	0.930
C15—H15A	0.930	C59—C60	1.390
C16—H16A	0.930	C59—H59A	0.930
C17—C18	1.528 (4)	C55′—C56′	1.390
C18—C19	1.369 (4)	C55′—C60′	1.390
C19—C20	1.398 (6)	C56′—C57′	1.390
C19—H19A	0.930	C56′—H56B	0.930
C20—C21	1.382 (7)	C57′—C58′	1.390
C20—H20A	0.930	C57′—H57B	0.930
C21—C22	1.338 (6)	C58′—C59′	1.390
C21—H21A	0.930	C58′—H58B	0.930
C22—H22A	0.930	C59′—C60′	1.390
C23—C25	1.503 (5)	C59′—H59B	0.930
C23—H23A	0.970	O5—C61	1.515 (10)
C23—H23B	0.970	O5—H5	0.820

C24—C30	1.511 (4)	C61—H61A	0.960
C24—H24A	0.970	C61—H61B	0.960
C24—H24B	0.970	C61—H61C	0.960
C25—C26	1.393 (4)		

C9—N1—C11	111.4 (2)	C36—C31—C38	121.3 (3)
C9—N1—C7	120.1 (2)	C33—C32—C31	120.7 (3)
C11—N1—C7	121.1 (2)	C33—C32—H32A	119.7
C10—N2—C11	113.9 (2)	C31—C32—H32A	119.7
C10—N2—C8	124.1 (2)	C34—C33—C32	120.2 (3)
C11—N2—C8	121.9 (2)	C34—C33—H33A	119.9
C9—N3—C17	114.3 (2)	C32—C33—H33A	119.9
C9—N3—C24	122.8 (2)	C33—C34—C35	120.1 (3)
C17—N3—C24	122.6 (2)	C33—C34—H34A	120.0
C10—N4—C17	111.3 (2)	C35—C34—H34A	120.0
C10—N4—C23	119.7 (2)	C36—C35—C34	120.9 (3)
C17—N4—C23	121.0 (2)	C36—C35—H35A	119.6
C12—N5—C16	116.9 (3)	C34—C35—H35A	119.6
C22—N6—C18	117.0 (3)	C35—C36—C31	119.2 (3)
C39—N7—C41	111.2 (2)	C35—C36—C37	119.5 (3)
C39—N7—C37	120.7 (2)	C31—C36—C37	121.2 (2)
C41—N7—C37	120.2 (2)	N7—C37—C36	115.3 (2)
C40—N8—C41	114.1 (2)	N7—C37—H37A	108.5
C40—N8—C38	123.5 (2)	C36—C37—H37A	108.5
C41—N8—C38	122.2 (2)	N7—C37—H37B	108.5
C39—N9—C54	123.6 (2)	C36—C37—H37B	108.5
C39—N9—C47	114.2 (2)	H37A—C37—H37B	107.5
C54—N9—C47	122.2 (2)	N8—C38—C31	113.7 (2)
C40—N10—C47	111.6 (2)	N8—C38—H38A	108.8
C40—N10—C53	120.1 (3)	C31—C38—H38A	108.8
C47—N10—C53	120.2 (2)	N8—C38—H38B	108.8
C42—N11—C46	116.9 (3)	C31—C38—H38B	108.8
C48—N12—C52	116.6 (4)	H38A—C38—H38B	107.7
C6—C1—C2	119.6 (3)	O3—C39—N9	126.6 (3)
C6—C1—C8	121.8 (3)	O3—C39—N7	125.6 (3)
C2—C1—C8	118.7 (3)	N9—C39—N7	107.7 (2)
C3—C2—C1	120.1 (4)	O4—C40—N8	126.8 (3)
C3—C2—H2A	119.9	O4—C40—N10	125.6 (3)
C1—C2—H2A	119.9	N8—C40—N10	107.5 (2)
C4—C3—C2	120.4 (3)	N8—C41—N7	113.2 (2)
C4—C3—H3A	119.8	N8—C41—C42	112.8 (2)
C2—C3—H3A	119.8	N7—C41—C42	111.7 (2)
C3—C4—C5	119.3 (4)	N8—C41—C47	101.5 (2)
C3—C4—H4A	120.3	N7—C41—C47	102.9 (2)
C5—C4—H4A	120.3	C42—C41—C47	114.0 (2)
C4—C5—C6	121.6 (4)	N11—C42—C43	123.1 (3)
C4—C5—H5A	119.2	N11—C42—C41	115.5 (2)
C6—C5—H5A	119.2	C43—C42—C41	121.2 (2)

C5—C6—C1	119.1 (3)	C42—C43—C44	118.5 (3)
C5—C6—C7	119.3 (3)	C42—C43—H43A	120.8
C1—C6—C7	121.5 (3)	C44—C43—H43A	120.8
N1—C7—C6	115.6 (2)	C45—C44—C43	119.3 (3)
N1—C7—H7A	108.4	C45—C44—H44A	120.4
C6—C7—H7A	108.4	C43—C44—H44A	120.4
N1—C7—H7B	108.4	C44—C45—C46	118.5 (3)
C6—C7—H7B	108.4	C44—C45—H45A	120.8
H7A—C7—H7B	107.5	C46—C45—H45A	120.8
N2—C8—C1	113.6 (2)	N11—C46—C45	123.7 (3)
N2—C8—H8A	108.8	N11—C46—H46A	118.1
C1—C8—H8A	108.8	C45—C46—H46A	118.1
N2—C8—H8B	108.8	N10—C47—N9	113.0 (2)
C1—C8—H8B	108.8	N10—C47—C48	112.1 (2)
H8A—C8—H8B	107.7	N9—C47—C48	111.5 (2)
O1—C9—N3	126.4 (3)	N10—C47—C41	102.8 (2)
O1—C9—N1	125.9 (3)	N9—C47—C41	101.3 (2)
N3—C9—N1	107.7 (2)	C48—C47—C41	115.5 (2)
O2—C10—N2	126.1 (3)	N12—C48—C49	123.4 (3)
O2—C10—N4	125.2 (3)	N12—C48—C47	115.3 (3)
N2—C10—N4	108.7 (2)	C49—C48—C47	121.2 (3)
N1—C11—N2	112.8 (2)	C48—C49—C50	118.3 (4)
N1—C11—C12	113.5 (2)	C48—C49—H49A	120.8
N2—C11—C12	111.2 (2)	C50—C49—H49A	120.8
N1—C11—C17	102.9 (2)	C51—C50—C49	118.7 (4)
N2—C11—C17	101.2 (2)	C51—C50—H50A	120.7
C12—C11—C17	114.3 (2)	C49—C50—H50A	120.7
C13—C12—N5	123.2 (3)	C50—C51—C52	119.3 (4)
C13—C12—C11	116.5 (3)	C50—C51—H51A	120.4
N5—C12—C11	120.2 (3)	C52—C51—H51A	120.4
C12—C13—C14	117.9 (4)	N12—C52—C51	123.7 (4)
C12—C13—H13A	121.0	N12—C52—H52A	118.1
C14—C13—H13A	121.0	C51—C52—H52A	118.1
C13—C14—C15	123.0 (4)	C55—C53—N10	125.7 (10)
C13—C14—H14A	118.5	N10—C53—C55′	111.5 (5)
C15—C14—H14A	118.5	C55—C53—H53A	105.9
C14—C15—C16	118.5 (4)	N10—C53—H53A	105.9
C14—C15—H15A	120.7	C55′—C53—H53A	117.2
C16—C15—H15A	120.7	C55—C53—H53B	105.9
C15—C16—N5	120.4 (4)	N10—C53—H53B	105.9
C15—C16—H16A	119.8	C55′—C53—H53B	109.4
N5—C16—H16A	119.8	H53A—C53—H53B	106.2
N3—C17—N4	113.2 (2)	C60′—C54—N9	114.9 (5)
N3—C17—C18	111.5 (2)	N9—C54—C60	108.4 (8)
N4—C17—C18	112.4 (2)	C60′—C54—H54A	96.4
N3—C17—C11	101.4 (2)	N9—C54—H54A	110.0
N4—C17—C11	102.9 (2)	C60—C54—H54A	110.0
C18—C17—C11	114.8 (2)	C60′—C54—H54B	116.0

N6—C18—C19	123.6 (3)	N9—C54—H54B	110.0
N6—C18—C17	114.8 (3)	C60—C54—H54B	110.0
C19—C18—C17	121.6 (3)	H54A—C54—H54B	108.4
C18—C19—C20	117.2 (4)	C53—C55—C56	119.9 (10)
C18—C19—H19A	121.4	C53—C55—C60	119.5 (10)
C20—C19—H19A	121.4	C56—C55—C60	120.0
C21—C20—C19	119.2 (4)	C57—C56—C55	120.0
C21—C20—H20A	120.4	C57—C56—H56A	120.0
C19—C20—H20A	120.4	C55—C56—H56A	120.0
C22—C21—C20	119.0 (4)	C56—C57—C58	120.0
C22—C21—H21A	120.5	C56—C57—H57A	120.0
C20—C21—H21A	120.5	C58—C57—H57A	120.0
C21—C22—N6	123.9 (4)	C59—C58—C57	120.0
C21—C22—H22A	118.1	C59—C58—H58A	120.0
N6—C22—H22A	118.1	C57—C58—H58A	120.0
N4—C23—C25	115.6 (2)	C60—C59—C58	120.0
N4—C23—H23A	108.4	C60—C59—H59A	120.0
C25—C23—H23A	108.4	C58—C59—H59A	120.0
N4—C23—H23B	108.4	C59—C60—C55	120.0
C25—C23—H23B	108.4	C59—C60—C54	116.6 (8)
H23A—C23—H23B	107.5	C55—C60—C54	123.1 (7)
N3—C24—C30	112.9 (2)	C56′—C55′—C60′	120.0
N3—C24—H24A	109.0	C56′—C55′—C53	118.4 (5)
C30—C24—H24A	109.0	C60′—C55′—C53	121.3 (5)
N3—C24—H24B	109.0	C57′—C56′—C55′	120.0
C30—C24—H24B	109.0	C57′—C56′—H56B	120.0
H24A—C24—H24B	107.8	C55′—C56′—H56B	120.0
C26—C25—C30	118.5 (3)	C56′—C57′—C58′	120.0
C26—C25—C23	120.0 (3)	C56′—C57′—H57B	120.0
C30—C25—C23	121.5 (3)	C58′—C57′—H57B	120.0
C27—C26—C25	121.2 (4)	C59′—C58′—C57′	120.0
C27—C26—H26A	119.4	C59′—C58′—H58B	120.0
C25—C26—H26A	119.4	C57′—C58′—H58B	120.0
C28—C27—C26	119.8 (3)	C58′—C59′—C60′	120.0
C28—C27—H27A	120.1	C58′—C59′—H59B	120.0
C26—C27—H27A	120.1	C60′—C59′—H59B	120.0
C27—C28—C29	120.3 (4)	C59′—C60′—C55′	120.0
C27—C28—H28A	119.8	C59′—C60′—C54	120.3 (6)
C29—C28—H28A	119.8	C55′—C60′—C54	119.7 (6)
C30—C29—C28	120.7 (4)	C61—O5—H5	111.1
C30—C29—H29A	119.6	O5—C61—H61A	109.5
C28—C29—H29A	119.6	O5—C61—H61B	109.5
C29—C30—C25	119.4 (3)	H61A—C61—H61B	109.5
C29—C30—C24	119.8 (3)	O5—C61—H61C	109.5
C25—C30—C24	120.8 (3)	H61A—C61—H61C	109.5
C32—C31—C36	118.9 (3)	H61B—C61—H61C	109.5
C32—C31—C38	119.8 (3)		

C6—C1—C2—C3	1.7 (5)	C31—C36—C37—N7	−59.5 (3)
C8—C1—C2—C3	−177.5 (3)	C40—N8—C38—C31	97.9 (3)
C1—C2—C3—C4	−0.7 (5)	C41—N8—C38—C31	−77.9 (3)
C2—C3—C4—C5	−0.8 (6)	C32—C31—C38—N8	−122.8 (3)
C3—C4—C5—C6	1.4 (6)	C36—C31—C38—N8	57.9 (4)
C4—C5—C6—C1	−0.5 (5)	C54—N9—C39—O3	0.4 (5)
C4—C5—C6—C7	176.1 (3)	C47—N9—C39—O3	178.4 (3)
C2—C1—C6—C5	−1.0 (4)	C54—N9—C39—N7	178.5 (3)
C8—C1—C6—C5	178.1 (3)	C47—N9—C39—N7	−3.5 (3)
C2—C1—C6—C7	−177.5 (3)	C41—N7—C39—O3	−168.4 (3)
C8—C1—C6—C7	1.6 (4)	C37—N7—C39—O3	−19.4 (4)
C9—N1—C7—C6	−72.6 (3)	C41—N7—C39—N9	13.5 (3)
C11—N1—C7—C6	74.7 (3)	C37—N7—C39—N9	162.5 (2)
C5—C6—C7—N1	124.6 (3)	C41—N8—C40—O4	178.8 (3)
C1—C6—C7—N1	−59.0 (4)	C38—N8—C40—O4	2.7 (5)
C10—N2—C8—C1	98.7 (3)	C41—N8—C40—N10	−3.5 (3)
C11—N2—C8—C1	−77.8 (3)	C38—N8—C40—N10	−179.5 (2)
C6—C1—C8—N2	56.8 (4)	C47—N10—C40—O4	−168.9 (3)
C2—C1—C8—N2	−124.1 (3)	C53—N10—C40—O4	−20.3 (5)
C17—N3—C9—O1	177.6 (3)	C47—N10—C40—N8	13.3 (3)
C24—N3—C9—O1	3.6 (4)	C53—N10—C40—N8	161.9 (3)
C17—N3—C9—N1	−5.0 (3)	C40—N8—C41—N7	−115.9 (3)
C24—N3—C9—N1	−179.0 (2)	C38—N8—C41—N7	60.2 (3)
C11—N1—C9—O1	−169.0 (3)	C40—N8—C41—C42	116.0 (3)
C7—N1—C9—O1	−18.8 (4)	C38—N8—C41—C42	−67.9 (3)
C11—N1—C9—N3	13.6 (3)	C40—N8—C41—C47	−6.4 (3)
C7—N1—C9—N3	163.8 (2)	C38—N8—C41—C47	169.7 (2)
C11—N2—C10—O2	179.8 (3)	C39—N7—C41—N8	91.7 (3)
C8—N2—C10—O2	3.1 (5)	C37—N7—C41—N8	−57.5 (3)
C11—N2—C10—N4	−2.0 (3)	C39—N7—C41—C42	−139.6 (2)
C8—N2—C10—N4	−178.7 (3)	C37—N7—C41—C42	71.2 (3)
C17—N4—C10—O2	−170.7 (3)	C39—N7—C41—C47	−16.9 (3)
C23—N4—C10—O2	−21.6 (4)	C37—N7—C41—C47	−166.1 (2)
C17—N4—C10—N2	11.0 (3)	C46—N11—C42—C43	1.4 (4)
C23—N4—C10—N2	160.1 (3)	C46—N11—C42—C41	−175.3 (2)
C9—N1—C11—N2	92.4 (3)	N8—C41—C42—N11	−29.2 (3)
C7—N1—C11—N2	−57.4 (3)	N7—C41—C42—N11	−158.1 (2)
C9—N1—C11—C12	−139.9 (2)	C47—C41—C42—N11	85.8 (3)
C7—N1—C11—C12	70.3 (3)	N8—C41—C42—C43	154.1 (3)
C9—N1—C11—C17	−15.8 (3)	N7—C41—C42—C43	25.2 (3)
C7—N1—C11—C17	−165.6 (2)	C47—C41—C42—C43	−90.9 (3)
C10—N2—C11—N1	−116.0 (3)	N11—C42—C43—C44	−2.1 (5)
C8—N2—C11—N1	60.8 (3)	C41—C42—C43—C44	174.4 (3)
C10—N2—C11—C12	115.1 (3)	C42—C43—C44—C45	0.9 (5)
C8—N2—C11—C12	−68.1 (3)	C43—C44—C45—C46	0.8 (5)
C10—N2—C11—C17	−6.7 (3)	C42—N11—C46—C45	0.4 (4)
C8—N2—C11—C17	170.0 (3)	C44—C45—C46—N11	−1.5 (5)
C16—N5—C12—C13	−0.2 (5)	C40—N10—C47—N9	91.8 (3)

C16—N5—C12—C11	178.1 (3)	C53—N10—C47—N9	−56.8 (4)
N1—C11—C12—C13	−164.8 (3)	C40—N10—C47—C48	−141.1 (2)
N2—C11—C12—C13	−36.2 (3)	C53—N10—C47—C48	70.2 (4)
C17—C11—C12—C13	77.6 (3)	C40—N10—C47—C41	−16.5 (3)
N1—C11—C12—N5	16.9 (4)	C53—N10—C47—C41	−165.1 (3)
N2—C11—C12—N5	145.4 (3)	C39—N9—C47—N10	−115.9 (3)
C17—C11—C12—N5	−100.7 (3)	C54—N9—C47—N10	62.2 (3)
N5—C12—C13—C14	0.7 (5)	C39—N9—C47—C48	116.7 (3)
C11—C12—C13—C14	−177.6 (3)	C54—N9—C47—C48	−65.2 (3)
C12—C13—C14—C15	−0.6 (6)	C39—N9—C47—C41	−6.6 (3)
C13—C14—C15—C16	−0.1 (6)	C54—N9—C47—C41	171.4 (3)
C14—C15—C16—N5	0.6 (6)	N8—C41—C47—N10	13.1 (2)
C12—N5—C16—C15	−0.5 (5)	N7—C41—C47—N10	130.5 (2)
C9—N3—C17—N4	−114.1 (3)	C42—C41—C47—N10	−108.4 (2)
C24—N3—C17—N4	60.0 (3)	N8—C41—C47—N9	−103.9 (2)
C9—N3—C17—C18	118.1 (3)	N7—C41—C47—N9	13.5 (2)
C24—N3—C17—C18	−67.9 (3)	C42—C41—C47—N9	134.6 (2)
C9—N3—C17—C11	−4.6 (3)	N8—C41—C47—C48	135.5 (2)
C24—N3—C17—C11	169.5 (2)	N7—C41—C47—C48	−107.2 (3)
C10—N4—C17—N3	94.1 (3)	C42—C41—C47—C48	13.9 (3)
C23—N4—C17—N3	−54.6 (3)	C52—N12—C48—C49	−0.8 (5)
C10—N4—C17—C18	−138.5 (2)	C52—N12—C48—C47	−178.2 (3)
C23—N4—C17—C18	72.8 (3)	N10—C47—C48—N12	−157.8 (2)
C10—N4—C17—C11	−14.5 (3)	N9—C47—C48—N12	−29.9 (3)
C23—N4—C17—C11	−163.2 (2)	C41—C47—C48—N12	85.0 (3)
N1—C11—C17—N3	11.7 (2)	N10—C47—C48—C49	24.7 (4)
N2—C11—C17—N3	−105.1 (2)	N9—C47—C48—C49	152.6 (3)
C12—C11—C17—N3	135.2 (2)	C41—C47—C48—C49	−92.5 (3)
N1—C11—C17—N4	129.0 (2)	N12—C48—C49—C50	1.3 (5)
N2—C11—C17—N4	12.1 (3)	C47—C48—C49—C50	178.6 (3)
C12—C11—C17—N4	−107.5 (3)	C48—C49—C50—C51	−0.6 (6)
N1—C11—C17—C18	−108.7 (3)	C49—C50—C51—C52	−0.5 (6)
N2—C11—C17—C18	134.5 (2)	C48—N12—C52—C51	−0.4 (6)
C12—C11—C17—C18	14.9 (3)	C50—C51—C52—N12	1.0 (7)
C22—N6—C18—C19	0.7 (5)	C40—N10—C53—C55	−68.9 (10)
C22—N6—C18—C17	−178.2 (3)	C47—N10—C53—C55	77.0 (10)
N3—C17—C18—N6	−40.7 (3)	C40—N10—C53—C55′	−73.7 (5)
N4—C17—C18—N6	−169.0 (2)	C47—N10—C53—C55′	72.2 (5)
C11—C17—C18—N6	73.9 (3)	C39—N9—C54—C60′	93.1 (7)
N3—C17—C18—C19	140.3 (3)	C47—N9—C54—C60′	−84.8 (7)
N4—C17—C18—C19	12.0 (4)	C39—N9—C54—C60	105.9 (7)
C11—C17—C18—C19	−105.1 (3)	C47—N9—C54—C60	−71.9 (7)
N6—C18—C19—C20	0.6 (5)	N10—C53—C55—C56	122.1 (8)
C17—C18—C19—C20	179.4 (3)	C55′—C53—C55—C56	140 (4)
C18—C19—C20—C21	−0.7 (5)	N10—C53—C55—C60	−49.0 (14)
C19—C20—C21—C22	−0.4 (6)	C55′—C53—C55—C60	−31 (4)
C20—C21—C22—N6	1.8 (6)	C53—C55—C56—C57	−171.1 (15)
C18—N6—C22—C21	−1.9 (5)	C60—C55—C56—C57	0.0

C10—N4—C23—C25	−72.3 (4)	C55—C56—C57—C58	0.0
C17—N4—C23—C25	73.8 (4)	C56—C57—C58—C59	0.0
C9—N3—C24—C30	94.1 (3)	C57—C58—C59—C60	0.0
C17—N3—C24—C30	−79.5 (3)	C58—C59—C60—C55	0.0
N4—C23—C25—C26	121.6 (3)	C58—C59—C60—C54	−174.7 (14)
N4—C23—C25—C30	−60.8 (4)	C53—C55—C60—C59	171.1 (15)
C30—C25—C26—C27	−0.1 (5)	C56—C55—C60—C59	0.0
C23—C25—C26—C27	177.6 (3)	C53—C55—C60—C54	−14.6 (18)
C25—C26—C27—C28	0.6 (5)	C56—C55—C60—C54	174.3 (15)
C26—C27—C28—C29	−0.3 (5)	C60′—C54—C60—C59	−2 (4)
C27—C28—C29—C30	−0.4 (5)	N9—C54—C60—C59	−123.3 (8)
C28—C29—C30—C25	0.9 (5)	C60′—C54—C60—C55	−177 (5)
C28—C29—C30—C24	−179.7 (3)	N9—C54—C60—C55	62.2 (11)
C26—C25—C30—C29	−0.6 (4)	C55—C53—C55′—C56′	−42 (4)
C23—C25—C30—C29	−178.2 (3)	N10—C53—C55′—C56′	122.1 (5)
C26—C25—C30—C24	180.0 (3)	C55—C53—C55′—C60′	132 (4)
C23—C25—C30—C24	2.3 (4)	N10—C53—C55′—C60′	−63.4 (6)
N3—C24—C30—C29	−121.8 (3)	C60′—C55′—C56′—C57′	0.0
N3—C24—C30—C25	57.6 (4)	C53—C55′—C56′—C57′	174.6 (7)
C36—C31—C32—C33	1.7 (4)	C55′—C56′—C57′—C58′	0.0
C38—C31—C32—C33	−177.7 (3)	C56′—C57′—C58′—C59′	0.0
C31—C32—C33—C34	−1.2 (5)	C57′—C58′—C59′—C60′	0.0
C32—C33—C34—C35	−0.1 (5)	C58′—C59′—C60′—C55′	0.0
C33—C34—C35—C36	1.1 (5)	C58′—C59′—C60′—C54	178.6 (8)
C34—C35—C36—C31	−0.6 (4)	C56′—C55′—C60′—C59′	0.0
C34—C35—C36—C37	177.6 (3)	C53—C55′—C60′—C59′	−174.4 (7)
C32—C31—C36—C35	−0.7 (4)	C56′—C55′—C60′—C54	−178.6 (8)
C38—C31—C36—C35	178.6 (3)	C53—C55′—C60′—C54	6.9 (8)
C32—C31—C36—C37	−178.9 (3)	N9—C54—C60′—C59′	−121.8 (5)
C38—C31—C36—C37	0.4 (4)	C60—C54—C60′—C59′	174 (5)
C39—N7—C37—C36	−70.8 (3)	N9—C54—C60′—C55′	56.8 (7)
C41—N7—C37—C36	75.5 (3)	C60—C54—C60′—C55′	−7 (4)
C35—C36—C37—N7	122.4 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O2	0.82	2.02	2.840 (8)	178
C2—H2*A*···O5^{i}	0.93	2.35	3.279 (9)	174
C22—H22*A*···O2^{ii}	0.93	2.57	3.462 (4)	160
C43—H43*A*···O3^{iii}	0.93	2.60	3.249 (5)	128
C46—H46*A*···O1	0.93	2.59	3.340 (4)	138
C51—H51*A*···O2^{iv}	0.93	2.52	3.419 (4)	164

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