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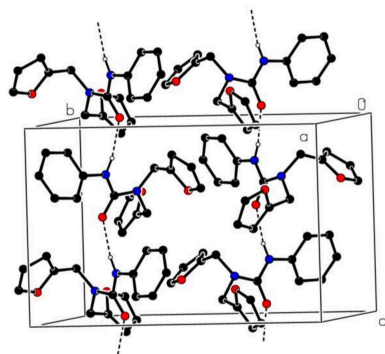
Synthesis and structure of 1,1-bis(furan-2-ylmethyl)-3-phenylurea

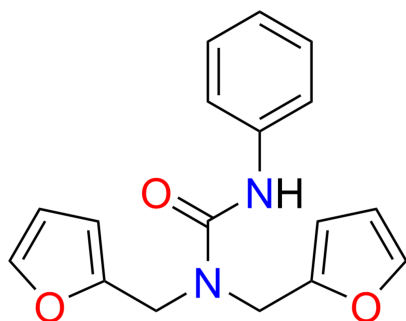
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The asymmetric unit of the title compound, C₁₇H₁₆N₂O₃, contains two molecules, *A* and *B*, with one furan ring in each molecule showing ‘flip’ rotational disorder. In the crystal, N—H⋯O hydrogen bonds link the molecules into separate infinite ⋯*A*⋯*A*⋯*A*⋯ and ⋯*B*⋯*B*⋯*B*⋯ chains propagating along the *c*-axis direction. Weak C—H⋯π interactions may help to consolidate the packing. Hirshfeld surface analysis revealed that the most important contributions to the packing are from H⋯H, H⋯C/C⋯H and H⋯O/O⋯H interactions with very similar surface percentages for both molecules.

1. Chemical context

Difurfurylamine, C₁₀H₁₁NO₂ (DiFAM), is a common precursor chemical with potential applications in pharmaceuticals (Zhuang *et al.*, 2022). Particular interest is invested into the use of DiFAM in the synthesis of dithiocarbamates: such compounds exhibit antitumor activity, are used as catalysts and biocides (Tamilvanan *et al.*, 2017), and thallium(III) *N,N*-difurfuryldithiocarbamate has an oxidative property towards organic molecules, is used within anti-alcohol medicine and possesses significant fungicidal activity (Gomathi *et al.*, 2023). Ureas themselves have shown a wide variety of applications and their antimicrobial (Resende *et al.*, 2023) and antibacterial (Yang *et al.*, 2025) activities have led to applications such as soporifics and local and general anaesthetics or herbicides (fenuron, isoproturon, sulfentrazone) and pesticides (diuron, monolinuron, chlorotoluron). The combination of the urea skeleton with a pharmaceutically promising difurfurylamine structure may lead to a new library of biologically active compounds. Interest in difurfurylamine chemistry was sparked in us in 2019, when difurfurylethers, alkenes and amides showed good selectivity in reactions with electron-deficient dimethyl acetylenedicarboxylate, producing kinetically and thermodynamically controlled diepoxynaphthalenes (Borisova *et al.*, 2018). We decided to introduce different substituents to the nitrogen atom of difurfurylamine, in this case transforming it into the title compound, C₁₇H₁₆N₂O₃ (**3**) and we now describe its structure.





2. Structural commentary

The asymmetric unit of **(3)** contains two molecules, *A* and *B* (Fig. 1). In each molecule, one furan ring is well ordered, while the other [the furan *B* ring (O3/C8–C11) in *A* and the *D* ring (O5/C20–C23) in *B*] is disordered over two sets of sites related by a 'flip' (~180°) rotation about the C–C bond connecting it to the adjacent methylene group. In molecule *A*, the ordered furan, *A* (O2/C3–C6) and phenyl, *C* (C12–C17) rings are oriented at a dihedral angle of *A/C* = 87.60 (19)°. In molecule *B*, the furan, *E* (O6/C25–C28) and phenyl, *F* (C29–C34) rings are oriented at a dihedral angle of *E/F* = 74.12 (17)°. There are no significant differences between the bond distances in *A* and *B* but the C7–N1–C1 [121.20 (19)°], C1–N1–C2 [115.52 (19)°], O1–C1–N2 [123.1 (2)°] and C1–N1–C12 [125.8 (2)°] bond angles in *A* are slightly different from the corresponding C18–N3–C24 [123.74 (19)°], C19–N3–C18 [118.2 (2)°], O4–C18–N4 [122.2 (2)°] and C18–N4–C29 [124.1 (2)°] bond angles in *B*.

3. Supramolecular features

In the crystal, N–H...O hydrogen bonds (Table 1) link the molecules into separate infinite ...*A*...*A*...*A*... and

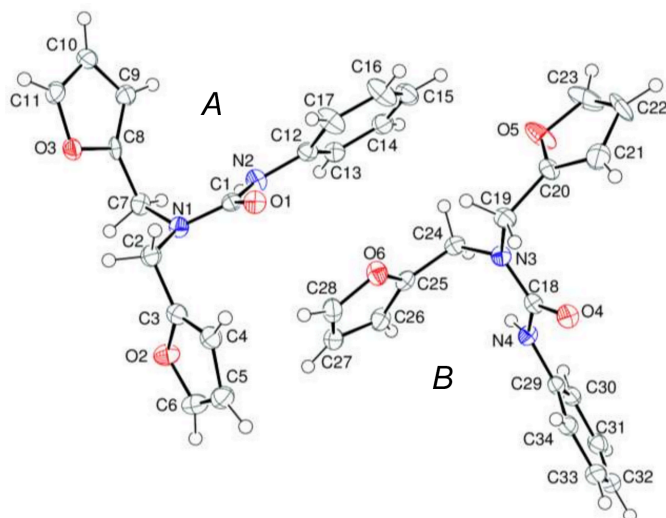


Figure 1

The asymmetric unit of **(3)** with 50% probability ellipsoids. Only the major disorder components of the disordered furan rings are shown.

Table 1

Hydrogen-bond geometry (Å, °).

*Cg*1, *Cg*5 and *Cg*6 are the centroids of the O2/C3–C6, C12–C17 and C29–C34 rings, respectively.

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
N2–H2N...O1 ⁱ	0.90	2.03	2.917 (2)	168
N4–H4N...O4 ⁱⁱ	0.90	2.07	2.955 (2)	167
C6–H6... <i>Cg</i> 6 ⁱⁱⁱ	0.95	2.62	3.482	150
C24–H24B... <i>Cg</i> 5 ^{iv}	0.99	2.90	3.789	149
C28–H28... <i>Cg</i> 1 ^{iv}	0.95	2.82	3.658	148

Symmetry codes: (i) $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (ii) $x, -y + \frac{3}{2}, z - \frac{1}{2}$; (iii) $-x, y + \frac{1}{2}, -z + \frac{1}{2}$; (iv) $x, y - 1, z$.

...*B*...*B*...*B*... *C*(4) chains propagating along the *c*-axis direction (Fig. 2). Adjacent molecules are generated by *c*-glide symmetry in each case. Weak C–H... π interactions may help to consolidate the packing.

4. Hirshfeld surface analysis

For visualizing the intermolecular interactions in the crystal of **(3)**, Hirshfeld surface (HS) analyses were carried out using *Crystal Explorer 17.5* (Spackman *et al.*, 2021). Only the major disorder components of furan rings were taken into account for the analysis. In the HSs plotted over d_{norm} (Fig. 3*a,b*), the red spots indicate their roles as the respective donors and/or acceptors in hydrogen bonding, as discussed above. The overall two-dimensional fingerprint plots are shown in Fig. 4*a* and 5*a* and those delineated into the different contact types are illustrated in Fig. 4(*b*)–(*j*) and Fig. 5(*b*)–(*j*) for molecules *A* and *B*, respectively. Their contributions to the HSs are presented in Table 2. Comparison of their percentages shows that there are no significant differences, implying that their environments in the crystal are very similar.

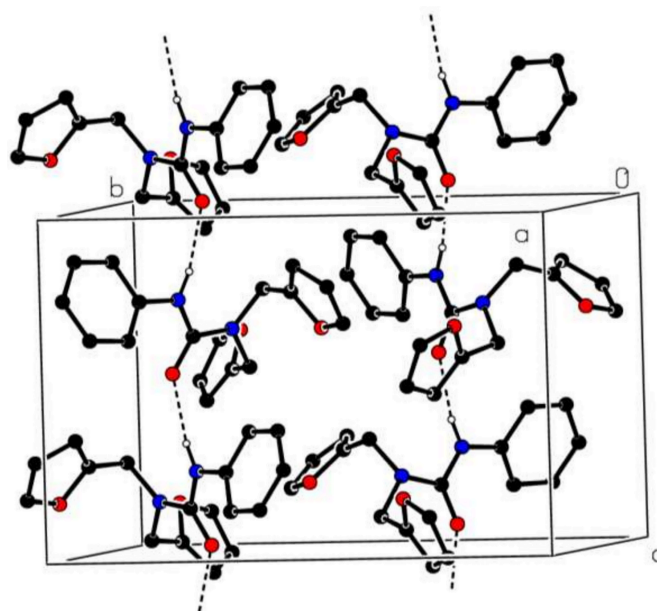


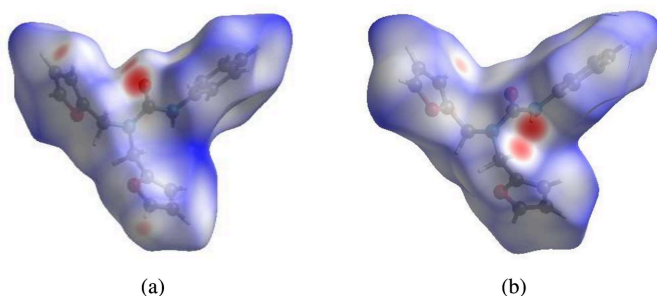
Figure 2

A partial packing diagram of **(3)** with N–H...O hydrogen bonds shown as dashed lines. Non-bonding H atoms have been omitted for clarity.

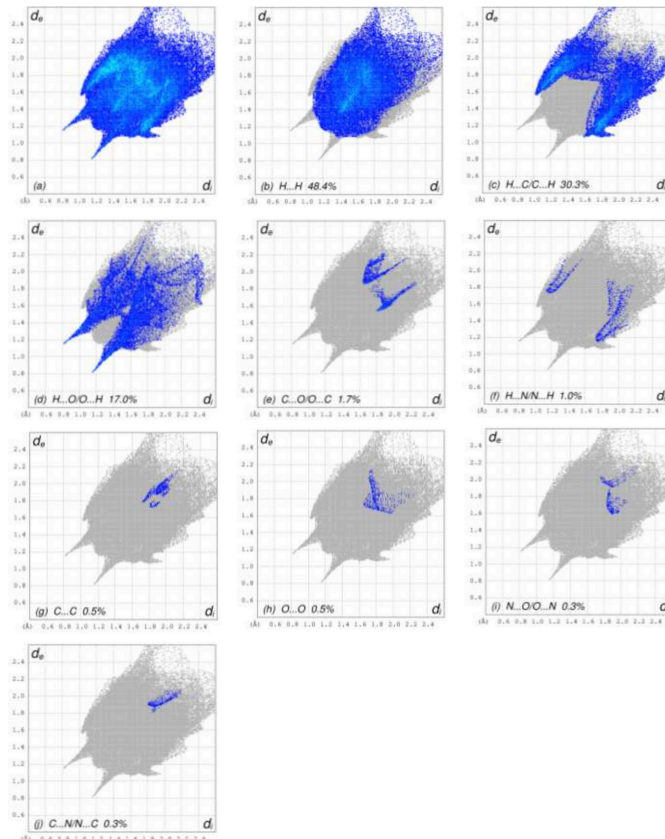
Table 2

Comparison of the Hirshfeld fingerprint percentages for molecules *A* and *B*.

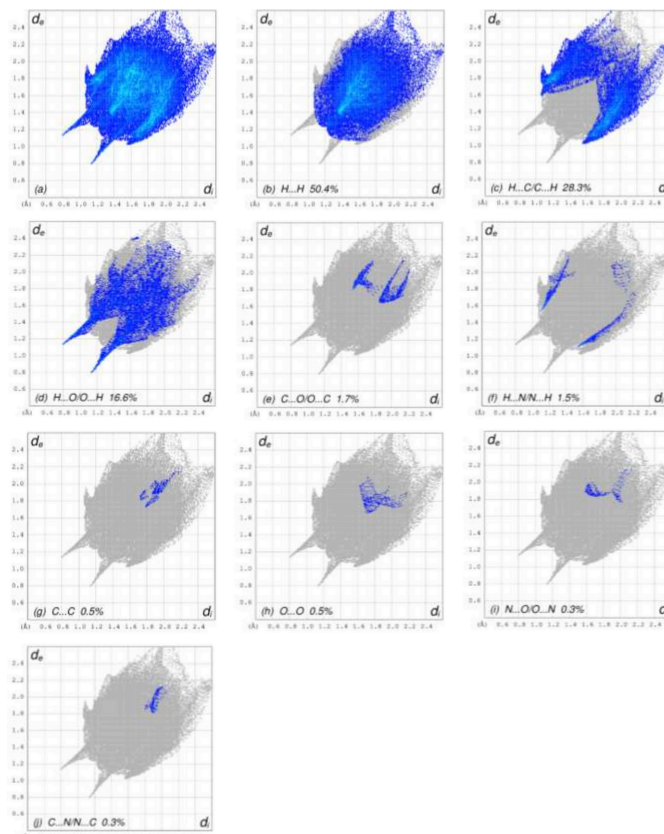
Contact	<i>A</i>	<i>B</i>
H...H	48.4	50.4
H...C/C...H	30.3	28.3
H...O/O...H	17.0	16.6
C...O/O...C	1.7	1.7
H...N/N...H	1.0	1.5
C...C	0.5	0.5
O...O	0.5	0.5
O...N/N...O	0.3	0.3
C...N/N...C	0.3	0.3

**Figure 3**

Views of the three-dimensional Hirshfeld surfaces for molecules (*a*) *A* and (*b*) *B* plotted over d_{norm} in the ranges from -0.49 to 1.58 a.u. and -0.53 to 1.56 a.u., respectively.

**Figure 4**

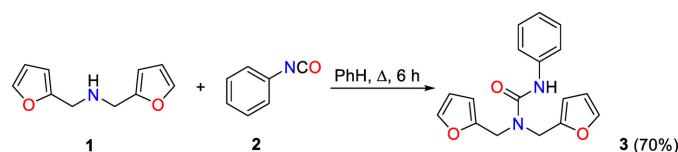
The full two-dimensional fingerprint plots for molecule *A*, showing all interactions, and delineated into different contact types.

**Figure 5**

The full two-dimensional fingerprint plots for molecule *B*, showing all interactions, and delineated into different contact types.

5. Synthesis and crystallization

Bis(furan-2-yl)amine (**1**) (100 mg, 0.56 mmol) was dissolved in benzene (5 ml) at room temperature (Fig. 6). Isocyanatobenzene (**2**) (74 mg, 0.62 mmol) was added to the solution and the reaction was refluxed for 6 h (progress monitored by TLC). The resulting mixture was cooled, and the formation of a solid was observed. The crystals were filtered off, washed with diethyl ether (3×5 ml), dried under vacuum, and then air. The target compound (**3**) did not require further purification; yield 70%, 117.1 mg, colourless crystals, m.p. 381–383 K. Single crystals of (**3**) were grown from a solvent mixture of ethanol and *N,N*-dimethylformamide. IR (KBr), ν (cm^{-1}): 1725, ^1H NMR (700 MHz, DMSO- d_6 , 273 K) (J Hz): δ 8.57 (s, 1H), 7.60 (dd, $J = 0.9, 1.9$ Hz, 2H), 7.47 (d, $J = 7.6$ Hz, 2H), 7.24 (t, $J = 7.6$ Hz, 2H), 6.96 (t, $J = 7.6$ Hz, 1H), 6.40 (dd, $J = 1.9, 3.10$ Hz, 2H), 6.31 (d, $J = 3.10$ Hz, 2H), 4.54 (s, 4H) ppm. ^{13}C [^1H] NMR (150.9 MHz, DMSO- d_6 , 273 K): δ 155.17,

**Figure 6**

Reaction scheme for obtaining (**3**).

151.67 (2C), 143.12 (2C), 140.66, 128.79 (2C), 122.53, 120.35 (2C), 110.95 (2C), 108.55 (2C), 42.72 (2C) ppm. MS (ESI) m/z : 296 $[M + H]^+$.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The hydrogen atom positions were calculated geometrically at distances of N—H = 0.90 and C—H = 0.95–0.99 Å and refined using a riding model while applying the constraint $U_{iso}(H) = 1.2 \times U_{eq}(C, N)$. One furan ring in each molecule was found to be disordered over two sets of sites and they were refined with site occupancy ratios of 0.6:0.4 in each case.

Acknowledgements

The authors' contributions are as follows. Conceptualization, TH and ANB; synthesis, DFM and DMS; X-ray analysis, VNK and TH; Hirshfeld surface analysis, TH; founding, KIH; writing (review and editing of the manuscript) TH, KIH and IHM, supervision, TH and ANB.

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Table 3

Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{16}N_2O_3$
M_r	296.32
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	20.1286 (2), 14.76143 (13), 9.97556 (8)
β (°)	92.8316 (8)
V (Å ³)	2960.39 (5)
Z	8
Radiation type	Cu $K\alpha$
μ (mm ^{−1})	0.76
Crystal size (mm)	0.39 × 0.15 × 0.09
Data collection	
Diffraction	Micro-focus sealed X-ray tube
Absorption correction	Gaussian (<i>CrysAlis PRO</i> ; Rigaku OD, 2024)
T_{min}, T_{max}	0.708, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	34327, 6425, 5840
R_{int}	0.039
$(\sin \theta/\lambda)_{max}$ (Å ^{−1})	0.639
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.068, 0.181, 1.09
No. of reflections	6425
No. of parameters	439
No. of restraints	20
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å ^{−3})	0.59, −0.50

Computer programs: *CrysAlis PRO* (Rigaku OD, 2024), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b) and *SHELXTL* (Sheldrick, 2008).

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Acta Cryst. (2026). E82 [https://doi.org/10.1107/S2056989026010029]

Synthesis and structure of 1,1-bis(furan-2-ylmethyl)-3-phenylurea

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Computing details

1,1-Bis(furan-2-ylmethyl)-3-phenylurea

Crystal data

$C_{17}H_{16}N_2O_3$

$M_r = 296.32$

Monoclinic, $P2_1/c$

$a = 20.1286$ (2) Å

$b = 14.76143$ (13) Å

$c = 9.97556$ (8) Å

$\beta = 92.8316$ (8)°

$V = 2960.39$ (5) Å³

$Z = 8$

$F(000) = 1248$

$D_x = 1.330$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 21717 reflections

$\theta = 2.3$ – 79.8°

$\mu = 0.76$ mm⁻¹

$T = 100$ K

Plate, colourless

$0.39 \times 0.15 \times 0.09$ mm

Data collection

Micro-focus sealed X-ray tube
diffractometer

Detector resolution: φ and ω pixels mm⁻¹

Rigaku XtaLAB Synergy-S, HyPix-6000HE
area-detector scans

Absorption correction: gaussian
(CrysAlisPro; Rigaku OD, 2024)

$T_{\min} = 0.708$, $T_{\max} = 1.000$

34327 measured reflections

6425 independent reflections

5840 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 79.9^\circ$, $\theta_{\min} = 2.2^\circ$

$h = -25 \rightarrow 25$

$k = -18 \rightarrow 18$

$l = -11 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.068$

$wR(F^2) = 0.181$

$S = 1.09$

6425 reflections

439 parameters

20 restraints

Primary atom site location: difference Fourier
map

Secondary atom site location: difference Fourier
map

Hydrogen site location: mixed

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.053P)^2 + 5.931P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.59$ e Å⁻³

$\Delta\rho_{\min} = -0.50$ e Å⁻³

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.73597 (8)	0.26382 (12)	0.41633 (16)	0.0238 (4)	
O2	0.93019 (9)	0.19490 (13)	0.33124 (17)	0.0292 (4)	
O3	0.72614 (15)	−0.0280 (2)	0.2890 (4)	0.0258 (7)	0.6
O3'	0.6514 (4)	0.0572 (6)	0.1365 (9)	0.0369 (11)	0.4
N1	0.78252 (10)	0.16531 (14)	0.2722 (2)	0.0228 (4)	
N2	0.70445 (10)	0.27093 (14)	0.19393 (19)	0.0235 (4)	
H2N	0.718467	0.254403	0.113215	0.028*	
C1	0.73968 (11)	0.23500 (15)	0.3017 (2)	0.0199 (4)	
C2	0.82410 (12)	0.13022 (17)	0.3869 (2)	0.0251 (5)	
H2A	0.840890	0.069373	0.364152	0.030*	
H2B	0.796338	0.123743	0.465540	0.030*	
C3	0.88149 (12)	0.19036 (16)	0.4231 (2)	0.0230 (5)	
C4	0.89752 (12)	0.24433 (17)	0.5291 (2)	0.0256 (5)	
H4	0.872377	0.253027	0.606158	0.031*	
C5	0.95961 (13)	0.28578 (18)	0.5027 (3)	0.0291 (5)	
H5	0.983903	0.327312	0.558896	0.035*	
C6	0.97693 (13)	0.25468 (19)	0.3831 (3)	0.0308 (6)	
H6	1.016140	0.271663	0.340117	0.037*	
C7	0.76556 (12)	0.09991 (16)	0.1638 (2)	0.0243 (5)	
H7A	0.805435	0.063271	0.146441	0.029*	
H7B	0.752968	0.133674	0.080602	0.029*	
C8	0.7098 (3)	0.0376 (6)	0.1960 (9)	0.0177 (9)	0.6
C9	0.6447 (3)	0.0330 (4)	0.1597 (7)	0.0289 (13)	0.6
H9A	0.620994	0.071540	0.097844	0.035*	0.6
C10	0.61794 (19)	−0.0410 (3)	0.2322 (5)	0.0270 (10)	0.6
H10A	0.573193	−0.061697	0.227695	0.032*	0.6
C11	0.66879 (19)	−0.0753 (3)	0.3076 (4)	0.0283 (9)	0.6
H11A	0.665511	−0.125728	0.366157	0.034*	0.6
C8'	0.7112 (6)	0.0382 (13)	0.202 (2)	0.0369 (11)	0.4
C9'	0.7046 (4)	−0.0346 (8)	0.2804 (12)	0.0369 (11)	0.4
H9B	0.738754	−0.062110	0.335402	0.044*	0.4
C10'	0.6368 (4)	−0.0636 (6)	0.2663 (10)	0.0369 (11)	0.4
H10B	0.617100	−0.113450	0.309578	0.044*	0.4
C11'	0.6071 (4)	−0.0056 (5)	0.1794 (8)	0.0369 (11)	0.4
H11B	0.561389	−0.007764	0.151164	0.044*	0.4
C12	0.65565 (12)	0.33951 (16)	0.1991 (2)	0.0228 (5)	
C13	0.64293 (12)	0.39180 (17)	0.0844 (2)	0.0257 (5)	
H13	0.667850	0.381825	0.007354	0.031*	
C14	0.59412 (13)	0.45840 (18)	0.0818 (3)	0.0293 (5)	

H14	0.585419	0.492990	0.002518	0.035*	
C15	0.55810 (13)	0.4746 (2)	0.1940 (3)	0.0338 (6)	
H15	0.525088	0.520663	0.192665	0.041*	
C16	0.57075 (15)	0.4232 (2)	0.3076 (3)	0.0422 (7)	
H16	0.546302	0.434362	0.384976	0.051*	
C17	0.61871 (14)	0.3551 (2)	0.3112 (3)	0.0367 (6)	
H17	0.626191	0.319475	0.389759	0.044*	
O4	0.77404 (9)	0.78394 (12)	0.46392 (16)	0.0252 (4)	
O5	0.5736 (2)	0.6855 (4)	0.3492 (5)	0.0407 (13)	0.6
O5'	0.5886 (6)	0.7549 (10)	0.5176 (14)	0.085 (3)	0.4
O6	0.77833 (9)	0.48633 (12)	0.33707 (18)	0.0283 (4)	
N3	0.72207 (10)	0.67406 (14)	0.3382 (2)	0.0224 (4)	
N4	0.80378 (10)	0.76368 (13)	0.24824 (19)	0.0216 (4)	
H4N	0.790312	0.742669	0.166683	0.026*	
C18	0.76737 (11)	0.74267 (16)	0.3562 (2)	0.0202 (4)	
C19	0.68232 (12)	0.64849 (17)	0.4523 (3)	0.0260 (5)	
H18A	0.709067	0.660842	0.536268	0.031*	
H19B	0.674325	0.582352	0.448025	0.031*	
C20	0.6152 (7)	0.6961 (14)	0.4609 (11)	0.0221 (15)	0.6
C21	0.5916 (3)	0.7523 (6)	0.5522 (7)	0.0411 (18)	0.6
H21A	0.613037	0.771586	0.634353	0.049*	0.6
C22	0.5269 (3)	0.7769 (4)	0.4983 (7)	0.048 (2)	0.6
H22A	0.495344	0.814112	0.539855	0.058*	0.6
C23	0.5196 (3)	0.7374 (5)	0.3783 (7)	0.055 (2)	0.6
H23A	0.481364	0.744451	0.319557	0.066*	0.6
C20'	0.6202 (11)	0.693 (2)	0.441 (2)	0.0221 (15)	0.4
C21'	0.5714 (8)	0.6598 (13)	0.359 (2)	0.085 (3)	0.4
H21B	0.577458	0.611488	0.297609	0.102*	0.4
C22'	0.5093 (8)	0.7047 (10)	0.374 (2)	0.085 (3)	0.4
H22B	0.467395	0.692505	0.329415	0.102*	0.4
C23'	0.5250 (7)	0.7681 (13)	0.466 (2)	0.085 (3)	0.4
H23B	0.496192	0.815340	0.491410	0.102*	0.4
C24	0.72054 (12)	0.61143 (16)	0.2234 (2)	0.0235 (5)	
H24A	0.722451	0.646887	0.139457	0.028*	
H24B	0.677921	0.577832	0.220027	0.028*	
C25	0.77646 (12)	0.54547 (15)	0.2306 (2)	0.0222 (5)	
C26	0.82809 (13)	0.53027 (17)	0.1531 (3)	0.0287 (5)	
H26	0.838036	0.561857	0.073551	0.034*	
C27	0.86519 (13)	0.45689 (18)	0.2143 (3)	0.0300 (5)	
H27	0.904671	0.430420	0.183513	0.036*	
C28	0.83324 (13)	0.43317 (18)	0.3230 (3)	0.0294 (5)	
H28	0.846796	0.385737	0.382614	0.035*	
C29	0.85387 (11)	0.83123 (16)	0.2496 (2)	0.0214 (5)	
C30	0.86190 (12)	0.88096 (17)	0.1319 (3)	0.0253 (5)	
H30	0.833586	0.869805	0.054597	0.030*	
C31	0.91132 (13)	0.94669 (17)	0.1280 (3)	0.0297 (5)	
H31	0.916819	0.979950	0.047775	0.036*	
C32	0.95276 (13)	0.96400 (18)	0.2409 (3)	0.0320 (6)	

H32	0.985742	1.009955	0.238964	0.038*
C33	0.94535 (13)	0.91352 (19)	0.3560 (3)	0.0312 (6)
H33	0.973921	0.924638	0.432916	0.037*
C34	0.89680 (12)	0.84679 (17)	0.3611 (3)	0.0260 (5)
H34	0.892900	0.811896	0.440347	0.031*

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0288 (9)	0.0276 (9)	0.0151 (7)	0.0001 (7)	0.0024 (6)	0.0014 (6)
O2	0.0275 (9)	0.0370 (10)	0.0234 (8)	−0.0056 (8)	0.0036 (7)	0.0004 (7)
O3	0.0258 (17)	0.0215 (16)	0.0304 (16)	0.0044 (14)	0.0038 (16)	0.0046 (13)
O3'	0.032 (2)	0.042 (3)	0.038 (2)	−0.009 (2)	0.0042 (17)	−0.0037 (19)
N1	0.0245 (10)	0.0221 (10)	0.0218 (10)	0.0007 (8)	−0.0001 (8)	0.0000 (8)
N2	0.0293 (10)	0.0281 (10)	0.0133 (8)	0.0035 (8)	0.0018 (7)	−0.0012 (7)
C1	0.0206 (10)	0.0212 (11)	0.0179 (10)	−0.0029 (8)	0.0015 (8)	0.0009 (8)
C2	0.0255 (12)	0.0230 (11)	0.0264 (12)	0.0005 (9)	−0.0017 (9)	0.0052 (9)
C3	0.0230 (11)	0.0249 (11)	0.0210 (11)	0.0022 (9)	0.0014 (9)	0.0069 (9)
C4	0.0266 (12)	0.0259 (12)	0.0242 (11)	0.0019 (9)	−0.0003 (9)	0.0026 (9)
C5	0.0292 (12)	0.0277 (12)	0.0298 (12)	−0.0029 (10)	−0.0051 (10)	0.0038 (10)
C6	0.0254 (12)	0.0360 (14)	0.0307 (13)	−0.0075 (10)	−0.0004 (10)	0.0049 (11)
C7	0.0293 (12)	0.0236 (11)	0.0203 (11)	0.0005 (9)	0.0045 (9)	−0.0015 (9)
C8	0.029 (2)	0.0126 (18)	0.012 (2)	0.0080 (16)	0.0002 (16)	−0.0028 (15)
C9	0.025 (3)	0.023 (3)	0.037 (3)	−0.001 (2)	−0.006 (2)	−0.004 (2)
C10	0.022 (2)	0.026 (3)	0.033 (3)	0.0009 (19)	0.004 (2)	−0.005 (2)
C11	0.030 (2)	0.021 (2)	0.035 (2)	−0.0007 (18)	0.012 (2)	0.0022 (17)
C8'	0.032 (2)	0.042 (3)	0.038 (2)	−0.009 (2)	0.0042 (17)	−0.0037 (19)
C9'	0.032 (2)	0.042 (3)	0.038 (2)	−0.009 (2)	0.0042 (17)	−0.0037 (19)
C10'	0.032 (2)	0.042 (3)	0.038 (2)	−0.009 (2)	0.0042 (17)	−0.0037 (19)
C11'	0.032 (2)	0.042 (3)	0.038 (2)	−0.009 (2)	0.0042 (17)	−0.0037 (19)
C12	0.0212 (11)	0.0260 (11)	0.0210 (11)	−0.0011 (9)	−0.0016 (8)	−0.0026 (9)
C13	0.0256 (12)	0.0278 (12)	0.0239 (11)	−0.0008 (9)	0.0016 (9)	0.0013 (9)
C14	0.0264 (12)	0.0280 (12)	0.0330 (13)	−0.0015 (10)	−0.0028 (10)	0.0052 (10)
C15	0.0243 (12)	0.0366 (14)	0.0401 (15)	0.0064 (11)	−0.0027 (11)	−0.0027 (12)
C16	0.0338 (14)	0.064 (2)	0.0292 (14)	0.0192 (14)	0.0037 (11)	−0.0027 (13)
C17	0.0317 (14)	0.0534 (18)	0.0251 (13)	0.0133 (13)	0.0033 (10)	0.0053 (12)
O4	0.0304 (9)	0.0293 (9)	0.0161 (7)	0.0015 (7)	0.0027 (6)	−0.0009 (6)
O5	0.0256 (17)	0.074 (4)	0.0217 (16)	0.023 (2)	−0.0078 (13)	−0.003 (2)
O5'	0.053 (4)	0.076 (5)	0.123 (7)	0.017 (3)	−0.020 (4)	−0.046 (5)
O6	0.0329 (9)	0.0257 (9)	0.0268 (9)	0.0035 (7)	0.0059 (7)	0.0060 (7)
N3	0.0246 (10)	0.0230 (10)	0.0198 (9)	0.0002 (8)	0.0045 (7)	0.0016 (7)
N4	0.0258 (10)	0.0229 (10)	0.0161 (9)	−0.0023 (8)	0.0019 (7)	−0.0018 (7)
C18	0.0221 (11)	0.0223 (11)	0.0162 (10)	0.0042 (8)	0.0012 (8)	0.0028 (8)
C19	0.0238 (11)	0.0273 (12)	0.0271 (12)	0.0016 (9)	0.0051 (9)	0.0069 (10)
C20	0.024 (2)	0.0258 (17)	0.017 (4)	0.0033 (15)	0.011 (3)	0.003 (3)
C21	0.045 (4)	0.032 (3)	0.049 (4)	−0.009 (3)	0.030 (3)	−0.005 (3)
C22	0.038 (3)	0.030 (3)	0.081 (5)	0.023 (2)	0.038 (3)	0.025 (3)
C23	0.020 (3)	0.079 (6)	0.065 (4)	0.021 (3)	−0.010 (3)	0.017 (5)

C20'	0.024 (2)	0.0258 (17)	0.017 (4)	0.0033 (15)	0.011 (3)	0.003 (3)
C21'	0.053 (4)	0.076 (5)	0.123 (7)	0.017 (3)	−0.020 (4)	−0.046 (5)
C22'	0.053 (4)	0.076 (5)	0.123 (7)	0.017 (3)	−0.020 (4)	−0.046 (5)
C23'	0.053 (4)	0.076 (5)	0.123 (7)	0.017 (3)	−0.020 (4)	−0.046 (5)
C24	0.0237 (11)	0.0239 (11)	0.0227 (11)	−0.0027 (9)	−0.0006 (9)	0.0010 (9)
C25	0.0267 (11)	0.0191 (11)	0.0205 (11)	−0.0047 (9)	0.0001 (9)	0.0015 (8)
C26	0.0348 (13)	0.0250 (12)	0.0269 (12)	−0.0032 (10)	0.0083 (10)	−0.0023 (10)
C27	0.0286 (12)	0.0256 (12)	0.0358 (14)	0.0001 (10)	0.0034 (10)	−0.0074 (10)
C28	0.0340 (13)	0.0234 (12)	0.0304 (13)	0.0044 (10)	−0.0019 (10)	−0.0013 (10)
C29	0.0204 (10)	0.0203 (11)	0.0237 (11)	0.0025 (8)	0.0038 (8)	−0.0017 (9)
C30	0.0223 (11)	0.0242 (11)	0.0295 (12)	0.0025 (9)	0.0022 (9)	0.0029 (9)
C31	0.0263 (12)	0.0244 (12)	0.0390 (14)	0.0024 (10)	0.0071 (10)	0.0060 (10)
C32	0.0234 (12)	0.0237 (12)	0.0492 (16)	−0.0013 (10)	0.0036 (11)	−0.0022 (11)
C33	0.0244 (12)	0.0319 (13)	0.0371 (14)	−0.0006 (10)	0.0000 (10)	−0.0096 (11)
C34	0.0234 (11)	0.0285 (12)	0.0262 (12)	0.0032 (9)	0.0013 (9)	−0.0033 (10)

Geometric parameters (Å, °)

O1—C1	1.226 (3)	O4—C18	1.236 (3)
O2—C6	1.372 (3)	O5—C20	1.369 (3)
O2—C3	1.376 (3)	O5—C23	1.373 (3)
O3—C11	1.370 (3)	O5'—C20'	1.368 (3)
O3—C8	1.371 (3)	O5'—C23'	1.372 (3)
O3'—C11'	1.369 (3)	O6—C28	1.368 (3)
O3'—C8'	1.371 (3)	O6—C25	1.374 (3)
N1—C1	1.383 (3)	N3—C18	1.369 (3)
N1—C7	1.477 (3)	N3—C24	1.471 (3)
N1—C2	1.478 (3)	N3—C19	1.472 (3)
N2—C1	1.366 (3)	N4—C18	1.368 (3)
N2—C12	1.413 (3)	N4—C29	1.418 (3)
N2—H2N	0.8999	N4—H4N	0.8999
C2—C3	1.487 (3)	C19—C20'	1.414 (14)
C2—H2A	0.9900	C19—C20	1.530 (9)
C2—H2B	0.9900	C19—H18A	0.9900
C3—C4	1.350 (4)	C19—H19B	0.9900
C4—C5	1.428 (4)	C20—C21	1.336 (3)
C4—H4	0.9500	C21—C22	1.432 (3)
C5—C6	1.341 (4)	C21—H21A	0.9500
C5—H5	0.9500	C22—C23	1.333 (3)
C6—H6	0.9500	C22—H22A	0.9500
C7—C8'	1.487 (12)	C23—H23A	0.9500
C7—C8	1.499 (7)	C20'—C21'	1.340 (3)
C7—H7A	0.9900	C21'—C22'	1.429 (3)
C7—H7B	0.9900	C21'—H21B	0.9500
C8—C9	1.343 (3)	C22'—C23'	1.338 (3)
C9—C10	1.430 (3)	C22'—H22B	0.9500
C9—H9A	0.9500	C23'—H23B	0.9500
C10—C11	1.339 (3)	C24—C25	1.487 (3)

C10—H10A	0.9500	C24—H24A	0.9900
C11—H11A	0.9500	C24—H24B	0.9900
C8'—C9'	1.341 (3)	C25—C26	1.345 (3)
C9'—C10'	1.429 (3)	C26—C27	1.435 (4)
C9'—H9B	0.9500	C26—H26	0.9500
C10'—C11'	1.339 (3)	C27—C28	1.335 (4)
C10'—H10B	0.9500	C27—H27	0.9500
C11'—H11B	0.9500	C28—H28	0.9500
C12—C17	1.392 (4)	C29—C34	1.394 (3)
C12—C13	1.393 (3)	C29—C30	1.401 (3)
C13—C14	1.389 (4)	C30—C31	1.391 (4)
C13—H13	0.9500	C30—H30	0.9500
C14—C15	1.384 (4)	C31—C32	1.392 (4)
C14—H14	0.9500	C31—H31	0.9500
C15—C16	1.377 (4)	C32—C33	1.383 (4)
C15—H15	0.9500	C32—H32	0.9500
C16—C17	1.393 (4)	C33—C34	1.390 (4)
C16—H16	0.9500	C33—H33	0.9500
C17—H17	0.9500	C34—H34	0.9500
C6—O2—C3	106.2 (2)	C20—O5—C23	102.9 (6)
C11—O3—C8	106.0 (4)	C20'—O5'—C23'	109.5 (14)
C11'—O3'—C8'	106.4 (8)	C28—O6—C25	106.06 (19)
C1—N1—C7	121.20 (19)	C18—N3—C24	123.74 (19)
C1—N1—C2	115.52 (19)	C18—N3—C19	118.2 (2)
C7—N1—C2	116.07 (19)	C24—N3—C19	116.70 (19)
C1—N2—C12	125.8 (2)	C18—N4—C29	124.1 (2)
C1—N2—H2N	115.2	C18—N4—H4N	119.0
C12—N2—H2N	118.4	C29—N4—H4N	115.7
O1—C1—N2	123.1 (2)	O4—C18—N4	122.2 (2)
O1—C1—N1	121.6 (2)	O4—C18—N3	121.4 (2)
N2—C1—N1	115.2 (2)	N4—C18—N3	116.3 (2)
N1—C2—C3	112.47 (19)	C20'—C19—N3	109.2 (14)
N1—C2—H2A	109.1	N3—C19—C20	116.2 (8)
C3—C2—H2A	109.1	N3—C19—H18A	108.2
N1—C2—H2B	109.1	C20—C19—H18A	108.2
C3—C2—H2B	109.1	N3—C19—H19B	108.2
H2A—C2—H2B	107.8	C20—C19—H19B	108.2
C4—C3—O2	110.0 (2)	H18A—C19—H19B	107.4
C4—C3—C2	134.0 (2)	C21—C20—O5	113.8 (7)
O2—C3—C2	116.0 (2)	C21—C20—C19	131.9 (6)
C3—C4—C5	106.6 (2)	O5—C20—C19	114.1 (5)
C3—C4—H4	126.7	C20—C21—C22	104.4 (6)
C5—C4—H4	126.7	C20—C21—H21A	127.8
C6—C5—C4	106.6 (2)	C22—C21—H21A	127.8
C6—C5—H5	126.7	C23—C22—C21	106.5 (5)
C4—C5—H5	126.7	C23—C22—H22A	126.7
C5—C6—O2	110.6 (2)	C21—C22—H22A	126.7

C5—C6—H6	124.7	C22—C23—O5	112.4 (5)
O2—C6—H6	124.7	C22—C23—H23A	123.8
N1—C7—C8'	111.0 (11)	O5—C23—H23A	123.8
N1—C7—C8	113.1 (5)	C21'—C20'—O5'	103.9 (16)
N1—C7—H7A	109.0	C21'—C20'—C19	119.8 (14)
C8—C7—H7A	109.0	O5'—C20'—C19	134.3 (11)
N1—C7—H7B	109.0	C20'—C21'—C22'	112.6 (16)
C8—C7—H7B	109.0	C20'—C21'—H21B	123.7
H7A—C7—H7B	107.8	C22'—C21'—H21B	123.7
C9—C8—O3	110.2 (5)	C23'—C22'—C21'	102.6 (15)
C9—C8—C7	134.8 (4)	C23'—C22'—H22B	128.7
O3—C8—C7	114.9 (4)	C21'—C22'—H22B	128.7
C8—C9—C10	106.8 (5)	C22'—C23'—O5'	110.1 (15)
C8—C9—H9A	126.6	C22'—C23'—H23B	124.9
C10—C9—H9A	126.6	O5'—C23'—H23B	124.9
C11—C10—C9	106.1 (4)	N3—C24—C25	112.77 (19)
C11—C10—H10A	127.0	N3—C24—H24A	109.0
C9—C10—H10A	127.0	C25—C24—H24A	109.0
C10—C11—O3	110.9 (3)	N3—C24—H24B	109.0
C10—C11—H11A	124.5	C25—C24—H24B	109.0
O3—C11—H11A	124.5	H24A—C24—H24B	107.8
C9'—C8'—O3'	109.3 (10)	C26—C25—O6	110.3 (2)
C9'—C8'—C7	137.5 (8)	C26—C25—C24	133.3 (2)
O3'—C8'—C7	113.1 (7)	O6—C25—C24	116.4 (2)
C8'—C9'—C10'	107.8 (9)	C25—C26—C27	106.4 (2)
C8'—C9'—H9B	126.1	C25—C26—H26	126.8
C10'—C9'—H9B	126.1	C27—C26—H26	126.8
C11'—C10'—C9'	105.4 (8)	C28—C27—C26	106.4 (2)
C11'—C10'—H10B	127.3	C28—C27—H27	126.8
C9'—C10'—H10B	127.3	C26—C27—H27	126.8
C10'—C11'—O3'	111.1 (8)	C27—C28—O6	110.9 (2)
C10'—C11'—H11B	124.5	C27—C28—H28	124.5
O3'—C11'—H11B	124.5	O6—C28—H28	124.5
C17—C12—C13	118.9 (2)	C34—C29—C30	119.3 (2)
C17—C12—N2	123.2 (2)	C34—C29—N4	122.4 (2)
C13—C12—N2	117.8 (2)	C30—C29—N4	118.2 (2)
C14—C13—C12	120.6 (2)	C31—C30—C29	120.1 (2)
C14—C13—H13	119.7	C31—C30—H30	120.0
C12—C13—H13	119.7	C29—C30—H30	120.0
C15—C14—C13	120.3 (2)	C30—C31—C32	120.4 (2)
C15—C14—H14	119.8	C30—C31—H31	119.8
C13—C14—H14	119.8	C32—C31—H31	119.8
C16—C15—C14	119.1 (3)	C33—C32—C31	119.2 (2)
C16—C15—H15	120.4	C33—C32—H32	120.4
C14—C15—H15	120.4	C31—C32—H32	120.4
C15—C16—C17	121.2 (3)	C32—C33—C34	121.1 (3)
C15—C16—H16	119.4	C32—C33—H33	119.4
C17—C16—H16	119.4	C34—C33—H33	119.4

C12—C17—C16	119.7 (3)	C33—C34—C29	119.8 (2)
C12—C17—H17	120.1	C33—C34—H34	120.1
C16—C17—H17	120.1	C29—C34—H34	120.1
C12—N2—C1—O1	−5.6 (4)	C29—N4—C18—O4	2.5 (4)
C12—N2—C1—N1	177.0 (2)	C29—N4—C18—N3	−178.5 (2)
C7—N1—C1—O1	147.7 (2)	C24—N3—C18—O4	−167.8 (2)
C2—N1—C1—O1	−1.4 (3)	C19—N3—C18—O4	−1.7 (3)
C7—N1—C1—N2	−34.8 (3)	C24—N3—C18—N4	13.2 (3)
C2—N1—C1—N2	176.1 (2)	C19—N3—C18—N4	179.3 (2)
C1—N1—C2—C3	−77.9 (3)	C18—N3—C19—C20'	96.2 (10)
C7—N1—C2—C3	131.4 (2)	C24—N3—C19—C20'	−96.6 (10)
C6—O2—C3—C4	−0.7 (3)	C18—N3—C19—C20	92.1 (7)
C6—O2—C3—C2	178.9 (2)	C24—N3—C19—C20	−100.7 (7)
N1—C2—C3—C4	111.0 (3)	C23—O5—C20—C21	−2.2 (19)
N1—C2—C3—O2	−68.4 (3)	C23—O5—C20—C19	−176.8 (13)
O2—C3—C4—C5	0.3 (3)	N3—C19—C20—C21	−116 (2)
C2—C3—C4—C5	−179.1 (3)	N3—C19—C20—O5	57.0 (18)
C3—C4—C5—C6	0.2 (3)	O5—C20—C21—C22	3.4 (19)
C4—C5—C6—O2	−0.7 (3)	C19—C20—C21—C22	176.8 (19)
C3—O2—C6—C5	0.9 (3)	C20—C21—C22—C23	−3.2 (13)
C1—N1—C7—C8'	−71.0 (6)	C21—C22—C23—O5	2.1 (8)
C2—N1—C7—C8'	77.9 (5)	C20—O5—C23—C22	−0.1 (12)
C1—N1—C7—C8	−69.6 (3)	C23'—O5'—C20'—C21'	−10 (3)
C2—N1—C7—C8	79.4 (3)	C23'—O5'—C20'—C19	−173 (3)
C11—O3—C8—C9	1.2 (10)	N3—C19—C20'—C21'	81 (3)
C11—O3—C8—C7	178.1 (7)	N3—C19—C20'—O5'	−118 (4)
N1—C7—C8—C9	102.4 (12)	O5'—C20'—C21'—C22'	5 (3)
N1—C7—C8—O3	−73.4 (9)	C19—C20'—C21'—C22'	171 (2)
O3—C8—C9—C10	−1.0 (11)	C20'—C21'—C22'—C23'	2 (3)
C7—C8—C9—C10	−177.0 (10)	C21'—C22'—C23'—O5'	−8 (3)
C8—C9—C10—C11	0.4 (8)	C20'—O5'—C23'—C22'	12 (3)
C9—C10—C11—O3	0.3 (6)	C18—N3—C24—C25	72.2 (3)
C8—O3—C11—C10	−1.0 (7)	C19—N3—C24—C25	−94.2 (2)
C11'—O3'—C8'—C9'	1 (2)	C28—O6—C25—C26	−0.1 (3)
C11'—O3'—C8'—C7	178.7 (14)	C28—O6—C25—C24	−179.9 (2)
N1—C7—C8'—C9'	−77 (3)	N3—C24—C25—C26	−116.6 (3)
N1—C7—C8'—O3'	106.3 (17)	N3—C24—C25—O6	63.1 (3)
O3'—C8'—C9'—C10'	−1 (2)	O6—C25—C26—C27	−0.1 (3)
C7—C8'—C9'—C10'	−178 (3)	C24—C25—C26—C27	179.6 (2)
C8'—C9'—C10'—C11'	0.1 (18)	C25—C26—C27—C28	0.3 (3)
C9'—C10'—C11'—O3'	0.6 (12)	C26—C27—C28—O6	−0.4 (3)
C8'—O3'—C11'—C10'	−1.1 (16)	C25—O6—C28—C27	0.3 (3)
C1—N2—C12—C17	−25.9 (4)	C18—N4—C29—C34	37.3 (3)
C1—N2—C12—C13	156.1 (2)	C18—N4—C29—C30	−145.6 (2)
C17—C12—C13—C14	−0.1 (4)	C34—C29—C30—C31	−1.5 (4)
N2—C12—C13—C14	178.0 (2)	N4—C29—C30—C31	−178.7 (2)
C12—C13—C14—C15	1.0 (4)	C29—C30—C31—C32	−0.5 (4)

C13—C14—C15—C16	−0.8 (4)	C30—C31—C32—C33	1.6 (4)
C14—C15—C16—C17	−0.3 (5)	C31—C32—C33—C34	−0.8 (4)
C13—C12—C17—C16	−1.0 (4)	C32—C33—C34—C29	−1.2 (4)
N2—C12—C17—C16	−179.0 (3)	C30—C29—C34—C33	2.4 (4)
C15—C16—C17—C12	1.2 (5)	N4—C29—C34—C33	179.4 (2)

Hydrogen-bond geometry ($\text{\AA}$, $^\circ$)

*Cg*1, *Cg*5 and *Cg*6 are the centroids of the O2/C3—C6, C12—C17 and C29—C34 rings, respectively.

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N2—H2N $\cdots$ O1 ⁱ	0.90	2.03	2.917 (2)	168
N4—H4N $\cdots$ O4 ⁱⁱ	0.90	2.07	2.955 (2)	167
C6—H6 $\cdots$ Cg6 ⁱⁱⁱ	0.95	2.62	3.482	150
C24—H24B $\cdots$ Cg5 ^{iv}	0.99	2.90	3.789	149
C28—H28 $\cdots$ Cg1 ^{iv}	0.95	2.82	3.658	148

Symmetry codes: (i) $x, -y+1/2, z-1/2$; (ii) $x, -y+3/2, z-1/2$; (iii) $-x, y+1/2, -z+1/2$; (iv) $x, y-1, z$.