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Crystal structure and Hirshfeld surface analysis of 2,6-bis[1-(prop-2-yn-1-yl)-1*H*-benzo[*d*]imidazol-2-yl]pyridine 0.144-hydrate

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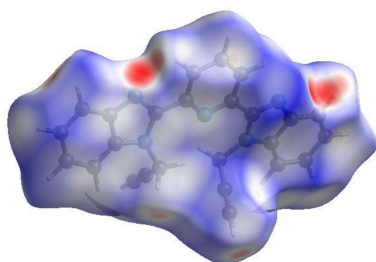
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The title compound, C₂₅H₁₇N₅·0.144H₂O, contains two substituted benzimidazole ring systems bridged over a pyridine ring and a disordered non-coordinating water molecule. In the crystal, O—H...N and C—H...O hydrogen-bonding interactions link the molecules into infinite chains parallel to [101]. Furthermore, π – π stacking interactions between the imidazole rings and between the pyridine and imidazole rings of adjacent molecules with inter-centroid distances of 3.6371 (4), 3.9872 (5), 3.4916 (4) and 3.6648 (4) Å are present, but C—H... π (ring) interactions are not observed. Hirshfeld surface analysis of the crystal structure indicates that the most important contributions for the crystal packing are from H...H (39.3%), H...C/C...H (35.9%) and H...N/N...H (9.1%).

1. Chemical context

Heterocyclic compounds occupy a central position in medicinal chemistry as they are key elements in the search for the and development of new bioactive molecules for the pharmaceutical industry (Vitaku *et al.*, 2014). Among them, nitrogen-containing heterocycles exhibit a wide range of biological activities due to their structural similarities with numerous natural and synthetic molecules already known for their pharmacological properties (Tahlan *et al.*, 2019). In this context, benzimidazole is a noteworthy representative and an important pharmacophore and scaffold in medicinal chemistry (Al-Ghulikah *et al.*, 2023). This core structure is frequently employed as a basis for designing therapeutic molecules of pharmaceutical and biological interests. Benzimidazole derivatives have demonstrated a broad spectrum of biological activities, including antihistaminic, antiulcer, antibacterial, antiparasitic, anticancer, antiviral, anti-inflammatory, anti-oxidant and antidiabetic properties (Saber *et al.*, 2021; Leonard *et al.*, 2006; Reddy *et al.*, 2005).

Building on our previous work on benzimidazole-based systems (Missiouei *et al.*, 2022; Moussaif *et al.*, 2025), we now report the synthesis, structure and Hirshfeld surface of the title compound, C₂₅H₁₇N₅·0.144H₂O. The molecular and crystal structure of this compound was established unambiguously by single-crystal X-ray diffraction. To gain deeper insight into its supramolecular features, a Hirshfeld surface analysis was undertaken, which enabled the identification and quanti-



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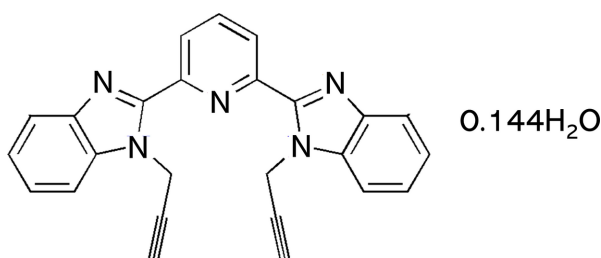
Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O1A-H1A\cdots N3^{iv}$	0.87	2.14	2.923 (16)	149
$O1A-H1B\cdots N5$	0.87	2.37	3.178 (17)	154
$O1B-H1C\cdots N3^{iv}$	0.87	2.21	2.96 (3)	144
$O1B-H1D\cdots N5$	0.87	2.24	3.07 (4)	161
$C9-H9\cdots O1A^i$	0.95	2.16	2.891 (15)	133
$C9-H9\cdots O1B^i$	0.95	2.15	2.93 (3)	139
$C19-H19\cdots O1A$	0.95	2.36	3.086 (18)	133

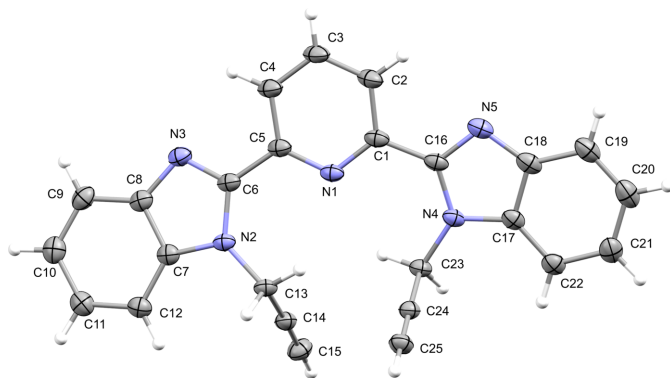
Symmetry codes: (i) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$; (iv) $x + \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$.

fication of the key intermolecular interactions governing the organization of the crystal structure.

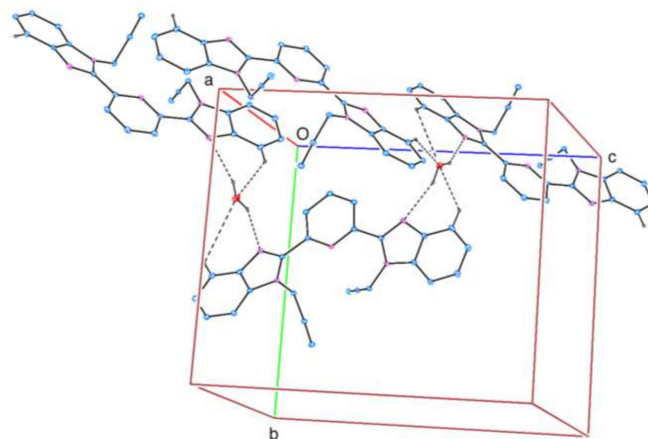


2. Structural commentary

The title compound contains two benzimidazole entities bridged over a pyridine ring, two propyl moieties and a disordered non-coordinating water molecule (Fig. 1). The dihedral angles between the imidazole rings (B , $N2/N3/C6-C8$; D , $N4/N5/C16-C18$) and the benzene rings (C , $C7-C12$; E , $C17-C22$) of the heterocyclic moieties, are $B/C = 1.93 (4)^\circ$ and $D/E = 0.97 (5)^\circ$. Thus, the two benzimidazole rings are almost planar. The central pyridine ring (A , $N1/C1-C5$) is oriented at dihedral angles of $11.77 (4)$ and $6.64 (3)^\circ$, respectively, to the mean plane of the benzimidazole rings B and D . The dihedral angle between the two benzimidazole ring systems BD and DE is $18.26 (3)^\circ$. There are no unusual bond lengths or interbond angles in the molecule.

**Figure 1**

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level. The disordered water molecule is not shown for clarity.

**Figure 2**

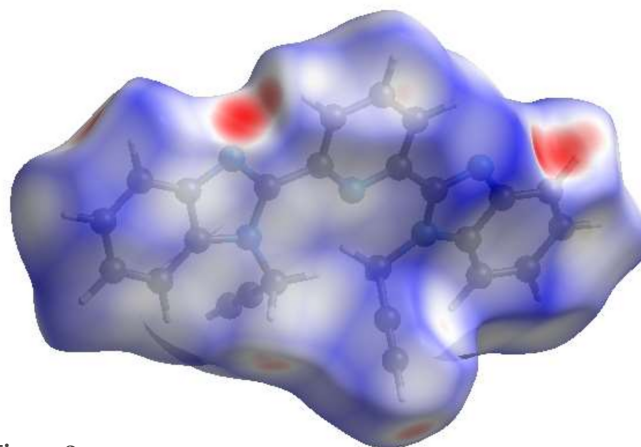
A partial packing diagram of the title compound with intermolecular $O-H\cdots N$ and $C-H\cdots O$ hydrogen bonds shown as dashed lines. Hydrogen atoms not involved in these interactions have been omitted.

3. Supramolecular features

In the crystal, $O-H\cdots N$ and $C-H\cdots O$ hydrogen-bonding interactions (Table 1) between the non-coordinating water molecule and the benzimidazole rings link molecules into infinite chains extending parallel to $[101]$ (Fig. 2). Furthermore, $\pi-\pi$ stacking interactions between the B rings [centroid-to-centroid distance = $3.6371 (4) \text{ \AA}$, $\alpha = 0.04 (3)^\circ$, slippage = $1.150 \text{ \AA}$], B and D rings [centroid-to-centroid distance = $3.9872 (5) \text{ \AA}$, $\alpha = 1.34 (5)^\circ$, slippage = $1.957 \text{ \AA}$], D rings [centroid-to-centroid distance = $3.4916 (4) \text{ \AA}$, $\alpha = 0.00 (5)^\circ$, slippage = $0.947 \text{ \AA}$] and A and E rings [centroid-to-centroid distance = $3.6648 (4) \text{ \AA}$, $\alpha = 6.81 (5)^\circ$, slippage = $1.461 \text{ \AA}$] of adjacent molecules may help to consolidate the packing. $C-H\cdots\pi(\text{ring})$ interactions are not observed.

4. Hirshfeld surface analysis

In order to visualize and quantify the intermolecular interactions in the crystal, a Hirshfeld surface (HS) analysis was

**Figure 3**

View of the three-dimensional Hirshfeld surface plotted over d_{norm} .

Table 2

Selected interatomic distances (Å).

H9...O1A ⁱ	2.16	C12...C14	3.376 (2)
H4...O1A ⁱⁱ	2.62	C16...C17 ^{vi}	3.3783 (19)
O1A...H19	2.36	C1...H23B	2.89
O1B...H1D	0.87	H1A...C4 ^{iv}	2.85
H9...O1B ⁱ	2.15	C5...H13B	2.87
H13A...O1B ⁱⁱⁱ	2.37	C13...H23B	2.86
O1B...H1C	0.87	C14...H23B	2.78
N1...N4	2.9842 (16)	C18...H1B	2.56
N1...C13	3.0587 (18)	C18...H1D	2.60
N1...N2	3.0092 (16)	C19...H1B	2.38
N1...C23	3.012 (2)	C19...H1D	2.64
N1...H23B	2.34	C22...H23A	2.87
N1...H13B	2.36	C23...H13B	2.85
H1A...N3 ^{iv}	2.14	C24...H13B	2.74
N3...H25 ^v	2.46	H1A...H19	2.37
H1C...N3 ^{iv}	2.21	H4...H1A ⁱⁱ	1.96
N3...H4	2.44	H1B...H19	1.83
N5...H2	2.45	H4...H1C ⁱⁱ	2.37
N5...H1B	2.37	H1C...H1D	1.38
N5...H1D	2.24	H1D...H19	2.21
C2...C22 ^{vi}	3.381 (2)	H13B...H23B	2.19
C5...C21 ^{vi}	3.392 (2)	H19...H1A	2.37
C5...C20 ^{vi}	3.386 (2)		

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

carried out using *CrystalExplorer* (Spackman *et al.*, 2021) following the protocol of Tan *et al.* (2019) after non-consideration of the partially occupied water molecule. Fig. 3 shows the contact distances where the bright-red spots correspond to the respective donors and/or acceptors noted above; numerical values of contact distances are collated in Table 2.

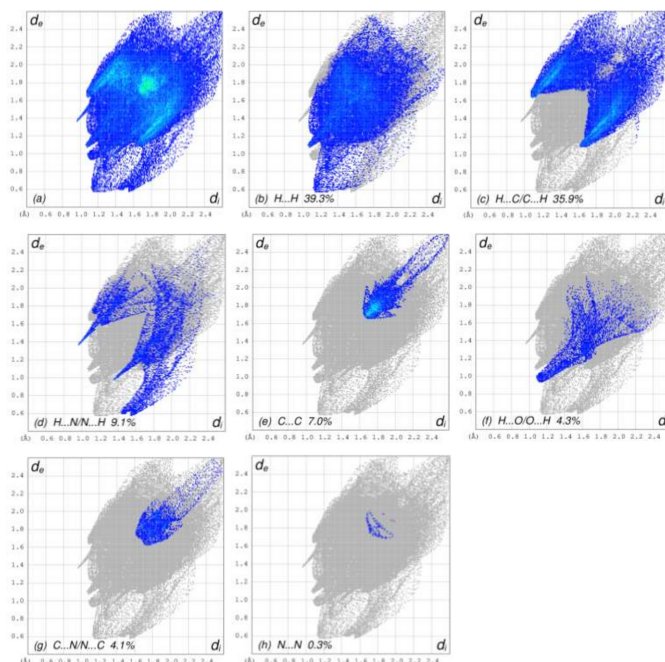


Figure 4

Two-dimensional fingerprint plots, showing (a) all interactions, and delineated into (b) H...H, (c) H...C/C...H, (d) H...N/N...H, (e) C...C, (f) H...O/O...H, (g) C...N/N...C and (h) N...N interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface contacts.

According to the two-dimensional fingerprint plots (McKinnon *et al.*, 2007), the H...H, H...C/C...H and H...N/N...H contacts make the most significant contributions to the HS, at 39.3%, 35.9%, 14.5% and 9.1%, respectively (Table 2, Fig. 4).

5. Database survey

A search of the Cambridge Structural Database (CSD, July 2025 update; Groom *et al.*, 2016) revealed several entries closely related to the title compound, a derivative of 2-(6-(1H-benzo[d]imidazol-2-yl)pyridin-2-yl)-1H-benzo[d]imidazole. The most relevant analogs are illustrated in Fig. 5 and include compounds **I** (CSD refcode DIXNUU; Liu *et al.*, 2007), **II** (MOTGEI; Chen *et al.*, 2009), **III** (WAKJID01; Gong *et al.*, 2012), **IV** (VAPTEN; Gong *et al.*, 2012), **V** (VAPTEN; Gong *et al.*, 2012), and **VI** (VAPVEP; Gong *et al.*, 2012). A detailed comparative analysis of these structures and the title compound highlights both common structural characteristics and distinctive features.

Core molecular geometry. All compounds display a benzimidazole-pyridine-benzimidazole framework that remains essentially planar. Compounds **I–III**, exhibit classical N–H...N or N–H...O hydrogen bonds between the constituents, which leads to the formation of supramolecular chains or layers. In the title compound, however, the hydrogen-bonding network is more compact and directional, leading to a more compact packing arrangement than in the other analogues.

Influence of metal coordination. Compounds **IV–VI** feature coordination to metals, which significantly alters both struc-

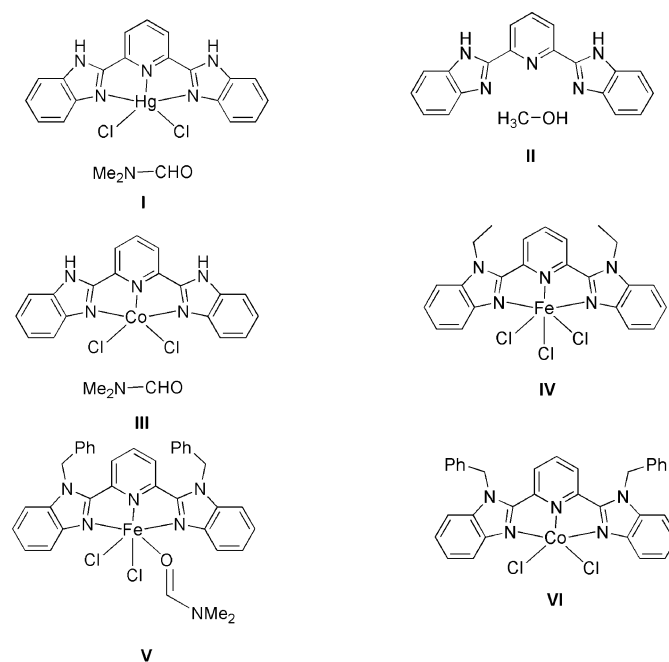


Figure 5

Structures closely related to the title compound according to a CSD search.

tural details within the organic ligands and in the supramolecular packing modes.

Crystal packing and π - π stacking. In all of the above related structures, π - π stacking appears to play a significant role in consolidating the crystal structure. Notably, the title compound exhibits slightly shorter centroid-to-centroid distances compared to its metal-coordinating analogues, indicating stronger intermolecular π - π interactions. This comparative structural analysis demonstrates that the title compound, although it shares a common molecular framework with its analogues, has a unique supramolecular organization that is governed by the absence of metal coordination and the predominance of hydrogen-bonding and π - π stacking interactions. These observations provide insight into the impact of structural modifications on molecular packing and overall crystal architectures.

6. Synthesis and crystallization

The synthesis of the title compound is shown schematically in Fig. 6. In a 100 ml round-bottom flask, 2,6-bis(1*H*-benzo[d]imidazol-2-yl)pyridine (**1**) (0.30 g, 0.96 mmol) was combined with potassium carbonate (K_2CO_3) (0.34 g, 2.49 mmol) in 10 ml of dimethylformamide (DMF). The mixture was stirred at room temperature for 15 min to ensure homogenization. Subsequently, propargyl bromide (**2**) (0.20 ml, 2.30 mmol) was added dropwise under continuous stirring. The progress of the reaction was monitored by thin-layer chromatography (TLC), and stirring was continued for 8 h at room temperature. After completion, the solvent was removed under reduced pressure using a rotary evaporator. The resulting crude residue was extracted with ethyl acetate and water. The organic layer was collected, dried over anhydrous sodium sulfate, and filtered. Purification by recrystallization from ethanol solution afforded the title compound (**3**) in 75% yield (Fig. 6). 1H NMR (500 MHz, $DMSO-d_6$) δ (ppm): 9.30 (*d*; $J = 7.9$ Hz; 2H; CH_{pyr}); 9.11 (*dd*; $J = 8.2, 7.6$ Hz; 1H; CH_{pyr}); 8.69–8.20 (*m*; 8H; CH_{Ar}); 6.68 (*d*; 4H; $J = 2.5$ Hz; $-NCH_2$); 4.23 (*t*; 2H; $-CCH$).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. After refinement of the organic molecule ($R1/wR2 = 0.0528/0.1440$), relatively high residual electron density was located between molecules, pointing to an underoccupied oxygen atom of a water molecule. Additional positional disorder using SIMU restraints was introduced, and the occupancies independently refined. The ‘add-

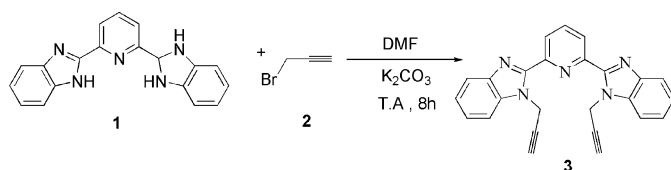


Figure 6
Reaction scheme for obtaining the title compound.

Table 3

Experimental details.

Crystal data	
Chemical formula	$C_{25}H_{17}N_5 \cdot 0.144H_2O$
M_r	390.04
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	160
a, b, c (Å)	9.6858 (2), 13.7005 (2), 15.0908 (3)
β (°)	102.943 (2)
V (Å ³)	1951.68 (6)
Z	4
Radiation type	Cu $K\alpha$
μ (mm ⁻¹)	0.65
Crystal size (mm)	0.22 $\times$ 0.15 $\times$ 0.07
Data collection	
Diffractometer	XtaLAB Synergy, Dualflex, HyPix
Absorption correction	Analytical [CrysAlis PRO (Rigaku OD, 2024) using a multifaceted crystal model (Clark & Reid, 1995)]
T_{min}, T_{max}	0.900, 0.965
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	21779, 4122, 3852
R_{int}	0.022
$(\sin \theta/\lambda)_{max}$ (Å ⁻¹)	0.633
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.043, 0.102, 1.07
No. of reflections	4122
No. of parameters	298
No. of restraints	6
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å ⁻³)	0.26, -0.17

Computer programs: CrysAlis PRO (Rigaku OD, 2024), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b), OLEX2 (Dolomanov *et al.*, 2009) and publCIF (Westrip, 2010).

H' tool in OLEX2 (Dolomanov *et al.*, 2009) was used for the H atoms of the water molecules, with a fixed O—H distance of 0.87 Å and $U_{iso}(H) = 1.5U_{eq}(O)$. Other hydrogen atom positions were calculated geometrically at CH = 0.95 Å and CH₂ = 0.99 Å and refined using a riding model with $U_{iso}(H) = 1.2U_{eq}(C)$.

Funding information

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supporting information

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Mohamed Ait Idar, Yousri El Barkaoui, Tuncer Hökelek, Olivier Blacque, Hassan Cherkaoui and Ahmed Moussaif

Computing details

2,6-Bis[1-(prop-2-yn-1-yl)-1H-benzo[d]imidazol-2-yl]pyridine 0.144-hydrate

Crystal data

$C_{25}H_{17}N_5 \cdot 0.144H_2O$

$M_r = 390.04$

Monoclinic, $P2_1/n$

$a = 9.6858$ (2) Å

$b = 13.7005$ (2) Å

$c = 15.0908$ (3) Å

$\beta = 102.943$ (2)°

$V = 1951.68$ (6) Å³

$Z = 4$

$F(000) = 814$

$D_x = 1.327$ Mg m⁻³

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

Cell parameters from 13938 reflections

$\theta = 3.0\text{--}78.9^\circ$

$\mu = 0.65$ mm⁻¹

$T = 160$ K

Plate, colourless

$0.22 \times 0.15 \times 0.07$ mm

Data collection

XtaLAB Synergy, Dualflex, HyPix
diffractometer

Radiation source: micro-focus sealed X-ray
tube, PhotonJet (Cu) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm⁻¹

ω scans

Absorption correction: analytical

[CrysAlisPro (Rigaku OD, 2024) using a
multifaceted crystal model (Clark & Reid,
1995)]

$T_{\min} = 0.900$, $T_{\max} = 0.965$

21779 measured reflections

4122 independent reflections

3852 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.022$

$\theta_{\max} = 77.4^\circ$, $\theta_{\min} = 4.4^\circ$

$h = -12 \rightarrow 12$

$k = -16 \rightarrow 17$

$l = -19 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.102$

$S = 1.07$

4122 reflections

298 parameters

6 restraints

Primary atom site location: dual

Hydrogen site location: mixed

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 0.7695P]$

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

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

$\Delta\rho_{\max} = 0.26$ e Å⁻³

$\Delta\rho_{\min} = -0.16$ e Å⁻³

Extinction correction: SHELXL-2019/2
 (Sheldrick, 2015b),
 $F_c^* = k F_c [1 + 0.001 x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.00117 (14)

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)
O1A	0.5569 (16)	0.8667 (13)	0.4309 (17)	0.053 (5)	0.094 (6)
H1A	0.631531	0.888804	0.414429	0.079*	0.094 (6)
H1B	0.580600	0.808201	0.451140	0.079*	0.094 (6)
O1B	0.586 (3)	0.877 (3)	0.479 (3)	0.055 (6)	0.050 (5)
H1C	0.649154	0.890204	0.448456	0.083*	0.050 (5)
H1D	0.586936	0.813222	0.483298	0.083*	0.050 (5)
N1	0.46815 (11)	0.54778 (8)	0.72961 (8)	0.0360 (3)	
N2	0.47168 (11)	0.42143 (8)	0.89321 (8)	0.0353 (2)	
N3	0.26310 (12)	0.49018 (9)	0.89744 (8)	0.0422 (3)	
N4	0.64889 (11)	0.52420 (8)	0.59477 (8)	0.0356 (2)	
N5	0.55002 (13)	0.66248 (9)	0.52978 (8)	0.0437 (3)	
C1	0.46243 (14)	0.61188 (10)	0.66146 (10)	0.0386 (3)	
C2	0.36641 (17)	0.68994 (11)	0.64552 (11)	0.0476 (4)	
H2	0.367614	0.735284	0.598073	0.057*	
C3	0.27084 (18)	0.69972 (12)	0.69953 (11)	0.0518 (4)	
H3	0.204134	0.751643	0.689551	0.062*	
C4	0.27245 (17)	0.63337 (11)	0.76859 (10)	0.0477 (4)	
H4	0.206054	0.638148	0.806157	0.057*	
C5	0.37380 (14)	0.55902 (10)	0.78210 (9)	0.0382 (3)	
C6	0.37072 (14)	0.49008 (10)	0.85648 (9)	0.0372 (3)	
C7	0.42394 (14)	0.37547 (10)	0.96244 (9)	0.0372 (3)	
C8	0.29418 (15)	0.41936 (10)	0.96437 (10)	0.0404 (3)	
C9	0.21911 (17)	0.39074 (12)	1.02873 (11)	0.0485 (4)	
H9	0.130887	0.419885	1.030806	0.058*	
C10	0.27767 (18)	0.31844 (12)	1.08943 (11)	0.0509 (4)	
H10	0.229306	0.298193	1.134536	0.061*	
C11	0.40679 (17)	0.27434 (11)	1.08590 (11)	0.0480 (4)	
H11	0.443327	0.224444	1.128418	0.058*	
C12	0.48269 (15)	0.30134 (11)	1.02228 (10)	0.0427 (3)	
H12	0.569882	0.271054	1.019571	0.051*	
C13	0.60936 (13)	0.39938 (10)	0.87291 (10)	0.0368 (3)	
H13A	0.683742	0.402283	0.929774	0.044*	
H13B	0.631548	0.449353	0.830796	0.044*	
C14	0.61060 (14)	0.30259 (10)	0.83176 (10)	0.0391 (3)	
C15	0.61316 (17)	0.22284 (12)	0.80235 (12)	0.0507 (4)	

H15	0.615212	0.158724	0.778700	0.061*
C16	0.55498 (14)	0.59963 (10)	0.59699 (9)	0.0378 (3)
C17	0.70860 (14)	0.54168 (10)	0.52136 (9)	0.0387 (3)
C18	0.64509 (15)	0.62793 (11)	0.48159 (10)	0.0429 (3)
C19	0.68005 (17)	0.66414 (12)	0.40296 (11)	0.0511 (4)
H19	0.636823	0.721620	0.374186	0.061*
C20	0.77899 (17)	0.61388 (13)	0.36863 (11)	0.0547 (4)
H20	0.804802	0.637632	0.315445	0.066*
C21	0.84308 (16)	0.52855 (13)	0.40971 (11)	0.0503 (4)
H21	0.911830	0.496311	0.384139	0.060*
C22	0.80837 (15)	0.49030 (12)	0.48667 (10)	0.0445 (3)
H22	0.850488	0.431997	0.514347	0.053*
C23	0.68304 (13)	0.43763 (9)	0.65209 (9)	0.0353 (3)
H23A	0.688719	0.380165	0.613293	0.042*
H23B	0.606340	0.425733	0.684480	0.042*
C24	0.81836 (14)	0.44900 (10)	0.71894 (9)	0.0377 (3)
C25	0.92809 (15)	0.45846 (12)	0.77189 (11)	0.0470 (4)
H25	1.016051	0.466048	0.814336	0.056*

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

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.027 (7)	0.053 (7)	0.080 (13)	0.000 (5)	0.016 (8)	0.010 (9)
O1B	0.032 (10)	0.053 (10)	0.081 (15)	0.001 (8)	0.014 (11)	0.002 (13)
N1	0.0326 (5)	0.0309 (5)	0.0393 (6)	0.0027 (4)	−0.0030 (4)	−0.0031 (5)
N2	0.0305 (5)	0.0328 (5)	0.0391 (6)	0.0029 (4)	0.0006 (4)	−0.0035 (5)
N3	0.0374 (6)	0.0421 (6)	0.0450 (6)	0.0055 (5)	0.0046 (5)	−0.0088 (5)
N4	0.0299 (5)	0.0328 (5)	0.0404 (6)	−0.0006 (4)	0.0000 (4)	0.0046 (5)
N5	0.0413 (6)	0.0350 (6)	0.0482 (7)	−0.0020 (5)	−0.0036 (5)	0.0063 (5)
C1	0.0368 (7)	0.0310 (6)	0.0416 (7)	0.0028 (5)	−0.0047 (5)	−0.0025 (5)
C2	0.0534 (9)	0.0372 (7)	0.0456 (8)	0.0131 (6)	−0.0030 (7)	0.0011 (6)
C3	0.0571 (9)	0.0442 (8)	0.0480 (8)	0.0242 (7)	−0.0012 (7)	−0.0042 (7)
C4	0.0480 (8)	0.0453 (8)	0.0452 (8)	0.0172 (7)	0.0005 (6)	−0.0066 (6)
C5	0.0357 (7)	0.0348 (7)	0.0395 (7)	0.0062 (5)	−0.0014 (5)	−0.0075 (5)
C6	0.0331 (6)	0.0345 (6)	0.0395 (7)	0.0050 (5)	−0.0014 (5)	−0.0091 (5)
C7	0.0360 (7)	0.0343 (7)	0.0382 (7)	−0.0034 (5)	0.0015 (5)	−0.0075 (5)
C8	0.0385 (7)	0.0374 (7)	0.0423 (7)	−0.0009 (6)	0.0032 (6)	−0.0108 (6)
C9	0.0456 (8)	0.0477 (8)	0.0539 (9)	−0.0032 (6)	0.0145 (7)	−0.0141 (7)
C10	0.0580 (9)	0.0463 (8)	0.0501 (9)	−0.0118 (7)	0.0159 (7)	−0.0094 (7)
C11	0.0546 (9)	0.0417 (8)	0.0453 (8)	−0.0078 (7)	0.0058 (7)	−0.0020 (6)
C12	0.0410 (7)	0.0382 (7)	0.0449 (8)	−0.0012 (6)	0.0014 (6)	−0.0020 (6)
C13	0.0286 (6)	0.0339 (6)	0.0443 (7)	0.0026 (5)	0.0010 (5)	0.0000 (6)
C14	0.0332 (6)	0.0384 (7)	0.0448 (7)	0.0050 (5)	0.0069 (6)	0.0031 (6)
C15	0.0524 (9)	0.0385 (8)	0.0637 (10)	0.0051 (7)	0.0185 (8)	−0.0041 (7)
C16	0.0332 (6)	0.0307 (6)	0.0430 (7)	−0.0011 (5)	−0.0051 (5)	0.0017 (5)
C17	0.0305 (6)	0.0407 (7)	0.0399 (7)	−0.0081 (5)	−0.0024 (5)	0.0038 (6)
C18	0.0372 (7)	0.0387 (7)	0.0455 (8)	−0.0104 (6)	−0.0060 (6)	0.0067 (6)
C19	0.0462 (8)	0.0495 (9)	0.0505 (9)	−0.0148 (7)	−0.0041 (7)	0.0135 (7)

C20	0.0472 (9)	0.0660 (10)	0.0455 (8)	−0.0238 (8)	−0.0008 (7)	0.0105 (8)
C21	0.0363 (7)	0.0656 (10)	0.0463 (8)	−0.0139 (7)	0.0036 (6)	0.0009 (7)
C22	0.0335 (7)	0.0503 (8)	0.0459 (8)	−0.0062 (6)	0.0009 (6)	0.0041 (7)
C23	0.0325 (6)	0.0296 (6)	0.0410 (7)	0.0018 (5)	0.0022 (5)	0.0040 (5)
C24	0.0340 (7)	0.0351 (7)	0.0430 (7)	0.0042 (5)	0.0064 (6)	0.0048 (6)
C25	0.0358 (7)	0.0521 (9)	0.0488 (8)	0.0023 (6)	0.0006 (6)	0.0066 (7)

Geometric parameters (Å, °)

O1A—H1A	0.8700	C9—H9	0.9500
O1A—H1B	0.8697	C9—C10	1.382 (2)
O1B—H1C	0.8704	C10—H10	0.9500
O1B—H1D	0.8701	C10—C11	1.401 (2)
N1—C1	1.3441 (18)	C11—H11	0.9500
N1—C5	1.3456 (18)	C11—C12	1.384 (2)
N2—C6	1.3804 (16)	C12—H12	0.9500
N2—C7	1.3850 (18)	C13—H13A	0.9900
N2—C13	1.4650 (17)	C13—H13B	0.9900
N3—C6	1.3257 (18)	C13—C14	1.4654 (19)
N3—C8	1.3841 (19)	C14—C15	1.182 (2)
N4—C16	1.3821 (17)	C15—H15	0.9500
N4—C17	1.3805 (18)	C17—C18	1.403 (2)
N4—C23	1.4616 (16)	C17—C22	1.389 (2)
N5—C16	1.3231 (18)	C18—C19	1.396 (2)
N5—C18	1.379 (2)	C19—H19	0.9500
C1—C2	1.4022 (19)	C19—C20	1.372 (3)
C1—C16	1.473 (2)	C20—H20	0.9500
C2—H2	0.9500	C20—C21	1.401 (2)
C2—C3	1.370 (2)	C21—H21	0.9500
C3—H3	0.9500	C21—C22	1.383 (2)
C3—C4	1.380 (2)	C22—H22	0.9500
C4—H4	0.9500	C23—H23A	0.9900
C4—C5	1.3975 (19)	C23—H23B	0.9900
C5—C6	1.472 (2)	C23—C24	1.4722 (18)
C7—C8	1.3995 (19)	C24—C25	1.185 (2)
C7—C12	1.393 (2)	C25—H25	0.9500
C8—C9	1.394 (2)		
H9···O1A ⁱ	2.16	C12···C14	3.376 (2)
H4···O1A ⁱⁱ	2.62	C16···C17 ^{vi}	3.3783 (19)
O1A···H19	2.36	C1···H23B	2.89
O1B···H1D	0.87	H1A···C4 ^{iv}	2.85
H9···O1B ⁱ	2.15	C5···H13B	2.87
H13A···O1B ⁱⁱⁱ	2.37	C13···H23B	2.86
O1B···H1C	0.87	C14···H23B	2.78
N1···N4	2.9842 (16)	C18···H1B	2.56
N1···C13	3.0587 (18)	C18···H1D	2.60
N1···N2	3.0092 (16)	C19···H1B	2.38

N1...C23	3.012 (2)	C19...H1D	2.64
N1...H23B	2.34	C22...H23A	2.87
N1...H13B	2.36	C23...H13B	2.85
H1A...N3 ^{iv}	2.14	C24...H13B	2.74
N3...H25 ^v	2.46	H1A...H19	2.37
H1C...N3 ^{iv}	2.21	H4...H1A ⁱⁱ	1.96
N3...H4	2.44	H1B...H19	1.83
N5...H2	2.45	H4...H1C ⁱⁱ	2.37
N5...H1B	2.37	H1C...H1D	1.38
N5...H1D	2.24	H1D...H19	2.21
C2...C22 ^{vi}	3.381 (2)	H13B...H23B	2.19
C5...C21 ^{vi}	3.392 (2)	H19...H1A	2.37
C5...C20 ^{vi}	3.386 (2)		
H1A—O1A—H1B	104.5	C12—C11—C10	121.92 (15)
H1C—O1B—H1D	104.5	C12—C11—H11	119.0
C1—N1—C5	117.30 (11)	C7—C12—H12	121.9
C6—N2—C7	106.65 (11)	C11—C12—C7	116.18 (14)
C6—N2—C13	130.75 (12)	C11—C12—H12	121.9
C7—N2—C13	122.50 (11)	N2—C13—H13A	109.3
C6—N3—C8	105.92 (11)	N2—C13—H13B	109.3
C16—N4—C23	130.94 (12)	N2—C13—C14	111.68 (11)
C17—N4—C16	106.62 (11)	H13A—C13—H13B	107.9
C17—N4—C23	122.40 (11)	C14—C13—H13A	109.3
C16—N5—C18	105.49 (12)	C14—C13—H13B	109.3
N1—C1—C2	122.79 (14)	C15—C14—C13	177.08 (16)
N1—C1—C16	120.28 (12)	C14—C15—H15	180.0
C2—C1—C16	116.88 (13)	N4—C16—C1	127.05 (12)
C1—C2—H2	120.6	N5—C16—N4	112.28 (13)
C3—C2—C1	118.89 (15)	N5—C16—C1	120.61 (12)
C3—C2—H2	120.6	N4—C17—C18	105.48 (13)
C2—C3—H3	120.3	N4—C17—C22	131.78 (13)
C2—C3—C4	119.33 (14)	C22—C17—C18	122.73 (14)
C4—C3—H3	120.3	N5—C18—C17	110.13 (13)
C3—C4—H4	120.7	N5—C18—C19	130.10 (14)
C3—C4—C5	118.62 (15)	C19—C18—C17	119.75 (15)
C5—C4—H4	120.7	C18—C19—H19	121.1
N1—C5—C4	123.03 (14)	C20—C19—C18	117.73 (15)
N1—C5—C6	120.51 (11)	C20—C19—H19	121.1
C4—C5—C6	116.44 (13)	C19—C20—H20	119.0
N2—C6—C5	127.57 (12)	C19—C20—C21	121.95 (15)
N3—C6—N2	111.95 (13)	C21—C20—H20	119.0
N3—C6—C5	120.47 (12)	C20—C21—H21	119.3
N2—C7—C8	105.87 (12)	C22—C21—C20	121.39 (16)
N2—C7—C12	131.57 (13)	C22—C21—H21	119.3
C12—C7—C8	122.56 (14)	C17—C22—H22	121.8
N3—C8—C7	109.61 (13)	C21—C22—C17	116.43 (15)
N3—C8—C9	130.06 (14)	C21—C22—H22	121.8

C9—C8—C7	120.32 (14)	N4—C23—H23A	109.3
C8—C9—H9	121.2	N4—C23—H23B	109.3
C10—C9—C8	117.58 (15)	N4—C23—C24	111.62 (11)
C10—C9—H9	121.2	H23A—C23—H23B	108.0
C9—C10—H10	119.3	C24—C23—H23A	109.3
C9—C10—C11	121.43 (15)	C24—C23—H23B	109.3
C11—C10—H10	119.3	C25—C24—C23	179.18 (16)
C10—C11—H11	119.0	C24—C25—H25	180.0
N1—C1—C2—C3	−2.3 (2)	C8—N3—C6—N2	0.42 (15)
N1—C1—C16—N4	3.0 (2)	C8—N3—C6—C5	−178.67 (11)
N1—C1—C16—N5	−179.95 (12)	C8—C7—C12—C11	−1.4 (2)
N1—C5—C6—N2	13.2 (2)	C8—C9—C10—C11	−0.9 (2)
N1—C5—C6—N3	−167.82 (12)	C9—C10—C11—C12	0.6 (2)
N2—C7—C8—N3	0.31 (15)	C10—C11—C12—C7	0.5 (2)
N2—C7—C8—C9	−178.53 (12)	C12—C7—C8—N3	179.96 (12)
N2—C7—C12—C11	178.18 (13)	C12—C7—C8—C9	1.1 (2)
N3—C8—C9—C10	−178.54 (14)	C13—N2—C6—N3	−176.46 (12)
N4—C17—C18—N5	0.41 (15)	C13—N2—C6—C5	2.6 (2)
N4—C17—C18—C19	−178.00 (12)	C13—N2—C7—C8	176.56 (11)
N4—C17—C22—C21	178.90 (13)	C13—N2—C7—C12	−3.1 (2)
N5—C18—C19—C20	−179.42 (14)	C16—N4—C17—C18	−0.70 (14)
C1—N1—C5—C4	−0.05 (19)	C16—N4—C17—C22	−179.75 (14)
C1—N1—C5—C6	178.26 (11)	C16—N4—C23—C24	−102.55 (15)
C1—C2—C3—C4	0.7 (2)	C16—N5—C18—C17	0.06 (15)
C2—C1—C16—N4	−174.45 (13)	C16—N5—C18—C19	178.26 (14)
C2—C1—C16—N5	2.62 (19)	C16—C1—C2—C3	175.04 (14)
C2—C3—C4—C5	1.1 (2)	C17—N4—C16—N5	0.79 (15)
C3—C4—C5—N1	−1.5 (2)	C17—N4—C16—C1	178.07 (12)
C3—C4—C5—C6	−179.86 (13)	C17—N4—C23—C24	80.13 (15)
C4—C5—C6—N2	−168.33 (13)	C17—C18—C19—C20	−1.4 (2)
C4—C5—C6—N3	10.61 (19)	C18—N5—C16—N4	−0.53 (15)
C5—N1—C1—C2	1.97 (19)	C18—N5—C16—C1	−178.00 (11)
C5—N1—C1—C16	−175.30 (11)	C18—C17—C22—C21	0.0 (2)
C6—N2—C7—C8	−0.05 (14)	C18—C19—C20—C21	0.5 (2)
C6—N2—C7—C12	−179.67 (14)	C19—C20—C21—C22	0.6 (2)
C6—N2—C13—C14	−113.23 (15)	C20—C21—C22—C17	−0.9 (2)
C6—N3—C8—C7	−0.45 (15)	C22—C17—C18—N5	179.57 (12)
C6—N3—C8—C9	178.24 (14)	C22—C17—C18—C19	1.2 (2)
C7—N2—C6—N3	−0.23 (15)	C23—N4—C16—N5	−176.85 (12)
C7—N2—C6—C5	178.78 (12)	C23—N4—C16—C1	0.4 (2)
C7—N2—C13—C14	71.06 (15)	C23—N4—C17—C18	177.19 (11)
C7—C8—C9—C10	0.0 (2)	C23—N4—C17—C22	−1.9 (2)

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

Hydrogen-bond geometry (Å, °)

<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>
O1 <i>A</i> —H1 <i>A</i> $\cdots$ N3 ^{iv}	0.87	2.14	2.923 (16)	149
O1 <i>A</i> —H1 <i>B</i> $\cdots$ N5	0.87	2.37	3.178 (17)	154
O1 <i>B</i> —H1 <i>C</i> $\cdots$ N3 ^{iv}	0.87	2.21	2.96 (3)	144
O1 <i>B</i> —H1 <i>D</i> $\cdots$ N5	0.87	2.24	3.07 (4)	161
C9—H9 $\cdots$ O1 <i>A</i> ⁱ	0.95	2.16	2.891 (15)	133
C9—H9 $\cdots$ O1 <i>B</i> ⁱ	0.95	2.15	2.93 (3)	139
C19—H19 $\cdots$ O1 <i>A</i>	0.95	2.36	3.086 (18)	133

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