



Crystal structure of 1,4-bis[5-(2-methoxyphenyl)-2H-tetrazol-2-yl]butane

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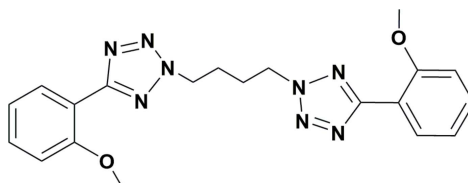
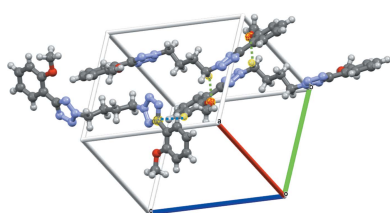
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The title compound, $C_{20}H_{22}N_8O_2$, was synthesized by the coupling reaction of a sodium tetrazolate salt and dibromobutane in a molar ratio of 2:1. The reaction can produce several possible regioisomers and the title compound was separated as the major product. The X-ray crystallographic study confirmed that the title compound crystallizes in the monoclinic $P2_1/c$ space group and possesses a bridging butylene group that connects two identical phenyl tetrazole moieties. The butylene group is attached not to the first but the second nitrogen atoms of both tetrazole rings. The dihedral angles between the phenyl groups and the adjacent tetrazolyl rings are $5.32(6)$ and $15.37(7)^\circ$. In the crystal, the molecules form centrosymmetric dimers through $C-H \cdots O$ hydrogen bonds between a $C-H$ group of the butylene linker and the O atom of a methoxy group.

1. Chemical context

Tetrazole ligands have four nitrogen atoms in their five-membered rings and the lone pairs of these nitrogen atoms are useful for coordination bonds with metal ions (Zhao *et al.*, 2008). Tetrazole has a variety of binding modes with metal ions, which results in the unusual formation of high-dimensional metal–organic frameworks (MOFs) or coordination polymers (Karaghiosoff *et al.*, 2009; Liu *et al.*, 2013). Valuable mono-, bis- and polytetrazole ligands for the formation of MOFs and coordination polymers have been also reported (Boland *et al.*, 2013; Fan *et al.*, 2016; Tăbăcaru *et al.*, 2018; Zhao *et al.*, 2016). As an extension of a project on the study of self-assembly behaviour in solution, we designed a ditetrazolyl chelate ligand possessing a butane bridge. It is worth noting that tetrazole has two different resonance structures in which the hydrogen atoms are located at either the N1 or N2 positions. In many cases, this results in the formation of several products (Lee *et al.*, 2017). It is therefore essential to study the molecular structure of synthesized tetrazole complexes by X-ray crystallography.



The title compound was isolated as an intermediate in the middle of the synthetic route for a chelate ligand. The reaction between the sodium salt of tetrazole and 1,4-dibromobutane gave three isomeric products (Fig. 1). Using column chromatography, the major product was isolated and its molecular structure was determined unambiguously by X-ray crystal-



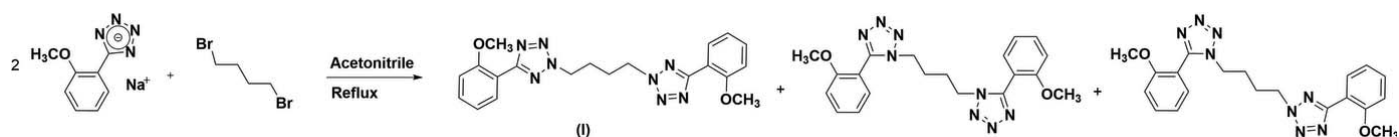


Figure 1
Synthesis of the title compound (I).

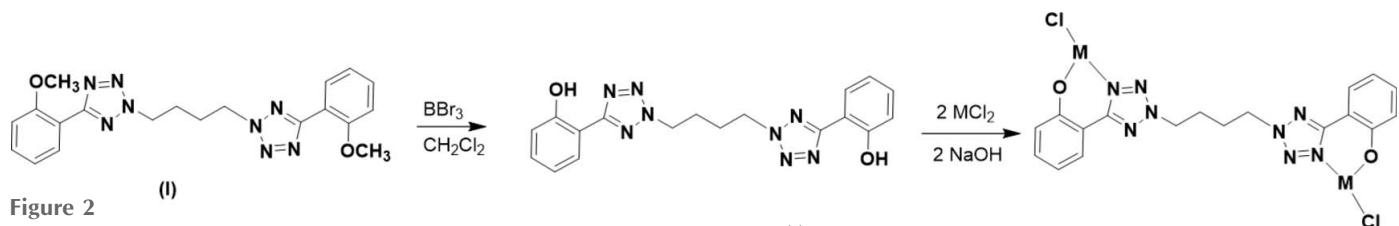


Figure 2
Synthetic route of the desired dinuclear metal complexes from the title compound (I).

lography. This compound is a useful precursor for the synthesis of dinuclear metal complexes with the expectation of synergetic effects of two metal centers (Fig. 2). Herein, we report the synthesis and crystal structure of this compound.

2. Structural commentary

The reaction yielded three isomeric products as described in Section 5, *Synthesis and crystallization*, and the structural analysis confirms the formation of the desired major product. The molecular structure of the title compound is shown in Fig. 3. There are no unusual bond lengths or angles. The title compound possesses two identical phenyl tetrazole fragments, connected by a butyl (C17–C20) bridge. The butyl group is

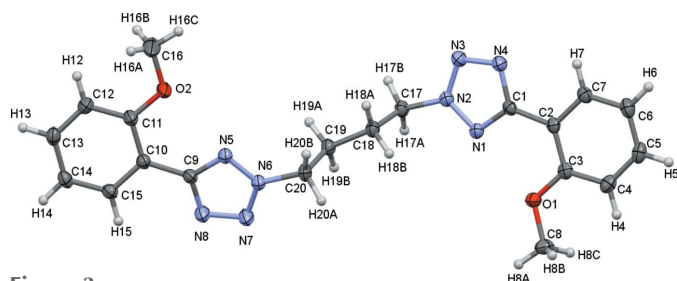


Figure 3
A view of the molecular structure of the title compound, with the atom labelling and 30% probability displacement ellipsoids.

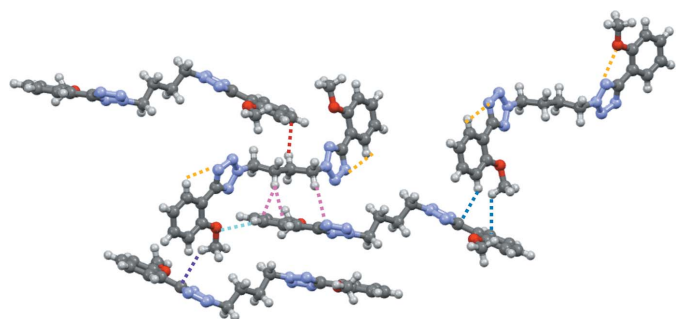


Figure 4
A plot showing the intramolecular C–H...N hydrogen bonding (dashed yellow lines) and short contacts between molecules (dashed pink, sky-blue and blue lines).

attached to the second N atom of both tetrazole rings (N2 and N6, Fig. 3). The dihedral angles between the phenyl group and tetrazolyl ring are somewhat different in the two phenyl-tetrazolyl groups. One phenyltetrazolyl group (N1–N4/C1–C7) is almost planar with an angle of 5.32 (6)° between the mean planes of the rings. However, the other phenyltetrazolyl group (N5–N8/C9–C15) is tilted with a dihedral angle of 15.37 (7)°.

Two intramolecular C–H...N hydrogen bonds (Table 1) occur, which are shown as yellow dashed lines in Fig. 4. These interactions may contribute to the planarity of the phenyl-tetrazolyl units.

3. Supramolecular features

The two phenyltetrazolyl fragments exhibit different intermolecular interactions. The tilted fragment (N5–N8/C9–C15) interacts with the butyl bridge of a glide-related molecule through C19–H19A...C14ⁱⁱ [H...A = 2.812 (2) Å; symmetry code: (ii) $x, -y + \frac{3}{2}, z + \frac{1}{2}$], C19–H19A...C15ⁱⁱ [H...A = 2.895 (2) Å] and C17–H17B...N8ⁱⁱ [H...A = 2.729 (2) Å] contacts (Fig. 4, pink dashed lines). There is an additional weak C14ⁱⁱ–H14ⁱⁱ...O2 interaction [H...A = 2.624 (2) Å] between the same pair of molecules, which is indicated by a sky-blue dashed line in Fig. 4. The bridging butyl group forms a further C18–H18B...C5ⁱⁱⁱ [H...C = 2.738 (2) Å; symmetry code: (iii) $x, -y + \frac{1}{2}, z - \frac{1}{2}$] close contact (Fig. 4, red dashed line) with a molecule generated by an adjacent glide plane. The planar fragments of screw-related molecules form C4–H4...C1^{iv} [H...A = 2.692 (2) Å; symmetry code: (iv) $-x + 2, y - \frac{1}{2}, -z + \frac{1}{2}$] and C8–H8C...C7^{iv} [H...A = 2.828 (2) Å] close contacts, which are indicated by blue dashed lines in the right-

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

D–H...A	D–H	H...A	D...A	D–H...A
C7–H7...N4	0.95	2.48	2.8371 (16)	102
C15–H15...N8	0.95	2.53	2.8586 (17)	101
C17–H17A...O1 ⁱ	0.99	2.58	3.4337 (15)	144

Symmetry code: (i) $-x + 2, -y + 1, -z$.

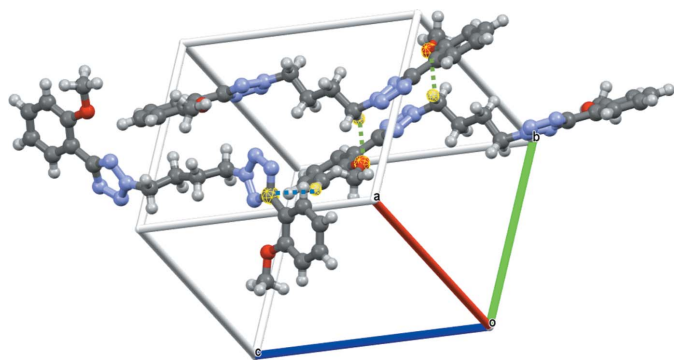


Figure 5
A plot showing the short contacts between molecules (dashed green and blue lines).

hand side of Fig. 4 (for clarity a different reference molecule was used for the illustration of this contact). It is interesting that the C1 atom has another close C—H...C contact from the opposite side of the aromatic plane (Fig. 4, purple dashed lines), C16—H16A...C1^v [H...C = 2.798 (2) Å; symmetry code: (v) $-x + 1, y + \frac{1}{2}, -z - \frac{1}{2}$]. There is one notable close contact, C17—H17A...O1ⁱ that can be considered a weak hydrogen bond, which is indicated by green dashed line in Fig. 5. This contact forms a dimeric rectangle between two molecules. This rectangle extends in the *c*-axis direction by the short interactions described above.

To provide an overall view of the weak interactions between the molecules, a Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) was performed with *CrystalExplorer17* (Turner *et al.*, 2017). The Hirshfeld surface was calculated using a standard (high) surface resolution with the three-

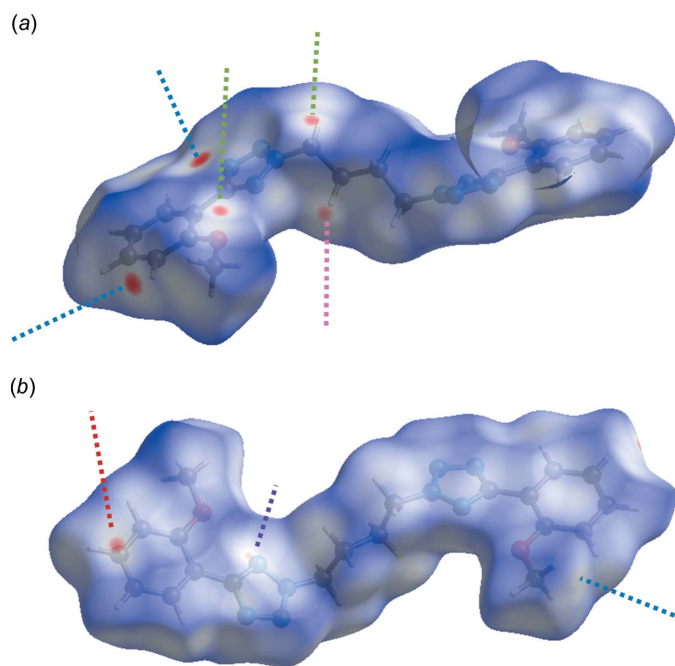


Figure 6
 d_{norm} mapped on the Hirshfeld surface for visualizing the intermolecular interactions. (a) front side, (b) back side.

dimensional (3D) d_{norm} surface plotted over a fixed colour scale of -0.1339 (red) to 1.4773 a.u. (blue). The 3D d_{norm} surface of the title complex is shown in Fig. 6a and 6b. The red spots indicate short contacts, *i.e.*, negative d_{norm} values on the surface, which highlight the most important weak interactions: C17—H17A...O1ⁱ hydrogen bond (green dashed line), C4—H4...C1^{iv} contact (blue in Fig. 6a), C18—H18B...C5ⁱⁱⁱ (pink in Fig. 6a, red in Fig. 6b) and C16—H16A...C1^v (blue in Fig. 6b).

4. Database survey

A search of the Cambridge Structural Database (CSD Version 5.40, November 2018; Groom *et al.*, 2016) for bis(tetrazolyl)alkane fragments provided four hits with a methylene bridge [SAVPAJ, SAVPIR (Freis *et al.*, 2017), OYIWOK02 (Feng, Qiu *et al.*, 2016) and UMOJEN (Feng, Bi *et al.*, 2016)] and two with a propylene bridge (SIBFIV, SIBFUH; Wurzenberger *et al.*, 2018). The butylene-bridged examples include a bistetrazolyl copper complex (SIBGIW; Wurzenberger *et al.*, 2018) and three bis(pyridyltetrazolyl)silver complexes (QOKBAV, QOKBEZ, QOKBID; Wang *et al.*, 2014). All of the above bis(tetrazolyl)alkane structures are metal complexes. It is worth noting that interesting metal-free cyclic bistetrazolyl compounds have been reported (VELPUZ, VELPOT; Voitekhovich *et al.*, 2012) in which the bis(tetrazolyl)butane fragment is part of a ring.

5. Synthesis and crystallization

The synthesis scheme for the title compound is represented in Fig. 1. The sodium salt of 5-(2-methoxyphenyl)-1*H*-tetrazole (495 mg, 2.5 mmol) and dibromobutane (150 μ l, 1.25 mmol) were dissolved in acetonitrile and refluxed for 2 d. The resulting white solid was filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography on silica gel using hexane:acetone (1:1) as eluent. Three isomeric compounds were obtained, as shown in Fig. 1. The major product (I) (yield = 35%) was recrystallized in ethanol by the slow evaporation method and yielded colourless crystals of the title compound.

Spectroscopic data: ¹H NMR (DMSO, 400 MHz): δ = 7.62 (*t*, 2H, Ph), 7.36 (*d*, 2H, Ph), 7.22 (*d*, 2H, Ph), 7.12 (*t*, 2H, Ph), 4.13 (*s*, 4H, CH₂), 3.71 (*s*, 6H, OCH₃), 1.66 (*s*, 4H, CH₂). ¹³C NMR (125 MHz, DMSO): 156.56, 152.18, 133.10, 131.20, 120.80, 112.26, 111.91, 55.50, 46.63, 25.57 ppm.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were included in calculated positions using a riding model, with C—H = 0.95–1.00 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for all others. Two reflections (100 and 110) were omitted because of truncation by the beamstop.

Funding information

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Table 2

Experimental details.

Crystal data	
Chemical formula	C ₂₀ H ₂₂ N ₈ O ₂
<i>M_r</i>	406.45
Crystal system, space group	Monoclinic, <i>P</i> ₂ ₁ / <i>c</i>
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	13.2904 (2), 10.2785 (2), 14.4968 (3)
β (°)	100.2538 (9)
<i>V</i> (Å ³)	1948.71 (6)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.10
Crystal size (mm)	0.1 × 0.1 × 0.08
Data collection	
Diffractionmeter	Bruker APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T</i> _{min} , <i>T</i> _{max}	0.706, 0.745
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	26494, 4008, 3516
<i>R</i> _{int}	0.021
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.627
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.037, 0.098, 1.05
No. of reflections	4008
No. of parameters	273
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.25, -0.37

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXS* (Sheldrick, 2008), *SHELXL* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

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supporting information

Acta Cryst. (2019). E75, 1844-1847 [https://doi.org/10.1107/S2056989019014877]

Crystal structure of 1,4-bis[5-(2-methoxyphenyl)-2H-tetrazol-2-yl]butane

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Computing details

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: *SHELXS* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

1,4-Bis[5-(2-methoxyphenyl)-2H-tetrazol-2-yl]butane

Crystal data

$C_{20}H_{22}N_8O_2$

$M_r = 406.45$

Monoclinic, $P2_1/c$

$a = 13.2904$ (2) Å

$b = 10.2785$ (2) Å

$c = 14.4968$ (3) Å

$\beta = 100.2538$ (9)°

$V = 1948.71$ (6) Å³

$Z = 4$

$F(000) = 856$

$D_x = 1.385$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9933 reflections

$\theta = 2.5$ – 26.4 °

$\mu = 0.10$ mm⁻¹

$T = 100$ K

Block, colorless

$0.1 \times 0.1 \times 0.08$ mm

Data collection

Bruker APEXII CCD
diffractometer

φ and ω scans

Absorption correction: multi-scan
(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.706$, $T_{\max} = 0.745$

26494 measured reflections

4008 independent reflections

3516 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\max} = 26.5$ °, $\theta_{\min} = 2.4$ °

$h = -16$ → 16

$k = -12$ → 12

$l = -18$ → 18

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.098$

$S = 1.05$

4008 reflections

273 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

$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2 + 0.7236P]$

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

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

$\Delta\rho_{\max} = 0.25$ e Å⁻³

$\Delta\rho_{\min} = -0.37$ 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.

Refinement. 1. Fixed Uiso At 1.2 times of: All C(H) groups, All C(H,H) groups At 1.5 times of: All C(H,H,H) groups 2.a Secondary CH2 refined with riding coordinates: C18(H18A,H18B), C19(H19A,H19B), C17(H17A,H17B), C20(H20A,H20B) 2.b Aromatic/amide H refined with riding coordinates: C7(H7), C13(H13), C15(H15), C4(H4), C6(H6), C12(H12), C5(H5), C14(H14) 2.c Idealised Me refined as rotating group: C8(H8A,H8B,H8C), C16(H16A,H16B,H16C)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.95431 (7)	0.24652 (8)	0.11811 (6)	0.0258 (2)
N1	0.86468 (7)	0.47789 (10)	0.06134 (7)	0.0217 (2)
N2	0.81869 (8)	0.58734 (10)	0.02753 (7)	0.0217 (2)
N5	0.60298 (7)	0.58496 (10)	-0.44500 (7)	0.0226 (2)
N6	0.68182 (8)	0.51604 (10)	-0.40215 (7)	0.0247 (2)
N4	0.77518 (8)	0.57102 (11)	0.15994 (7)	0.0269 (2)
C3	0.92713 (9)	0.26068 (12)	0.20399 (8)	0.0220 (3)
C1	0.83621 (8)	0.46963 (11)	0.14486 (8)	0.0198 (2)
N3	0.76475 (8)	0.64445 (11)	0.08397 (7)	0.0278 (2)
C9	0.62616 (9)	0.59998 (12)	-0.52995 (8)	0.0220 (2)
O2	0.46627 (8)	0.77149 (12)	-0.51135 (6)	0.0463 (3)
C10	0.56607 (9)	0.67077 (12)	-0.60922 (8)	0.0215 (2)
N8	0.71619 (9)	0.54247 (13)	-0.53723 (8)	0.0348 (3)
C7	0.83412 (9)	0.38359 (12)	0.30302 (8)	0.0238 (3)
H7	0.7911	0.4547	0.3117	0.029*
C2	0.86647 (9)	0.36911 (12)	0.21675 (8)	0.0207 (2)
C13	0.46290 (10)	0.80750 (13)	-0.76430 (9)	0.0284 (3)
H13	0.4280	0.8544	-0.8169	0.034*
C18	0.74738 (9)	0.55296 (12)	-0.13870 (8)	0.0232 (3)
H18A	0.6763	0.5728	-0.1314	0.028*
H18B	0.7596	0.4593	-0.1251	0.028*
C19	0.75877 (9)	0.57977 (12)	-0.23988 (8)	0.0242 (3)
H19A	0.7355	0.6692	-0.2580	0.029*
H19B	0.8314	0.5718	-0.2464	0.029*
C15	0.59024 (9)	0.65380 (12)	-0.69829 (8)	0.0237 (3)
H15	0.6431	0.5947	-0.7061	0.028*
C4	0.95664 (9)	0.17429 (12)	0.27783 (9)	0.0268 (3)
H4	0.9983	0.1016	0.2696	0.032*
C6	0.86318 (10)	0.29699 (13)	0.37594 (9)	0.0282 (3)
H6	0.8405	0.3085	0.4339	0.034*
N7	0.75068 (9)	0.48940 (13)	-0.45441 (8)	0.0366 (3)
C17	0.82070 (9)	0.63252 (12)	-0.06795 (8)	0.0240 (3)
H17A	0.8909	0.6243	-0.0814	0.029*
H17B	0.8010	0.7255	-0.0736	0.029*

C12	0.43688 (10)	0.82663 (13)	-0.67683 (9)	0.0299 (3)
H12	0.3844	0.8866	-0.6698	0.036*
C11	0.48755 (10)	0.75822 (13)	-0.59921 (8)	0.0276 (3)
C8	1.00848 (10)	0.13020 (13)	0.10279 (10)	0.0302 (3)
H8A	1.0215	0.1294	0.0383	0.045*
H8B	0.9673	0.0541	0.1129	0.045*
H8C	1.0737	0.1275	0.1467	0.045*
C5	0.92583 (10)	0.19321 (13)	0.36327 (9)	0.0289 (3)
H5	0.9478	0.1346	0.4135	0.035*
C20	0.69416 (10)	0.48127 (13)	-0.30300 (8)	0.0259 (3)
H20A	0.7268	0.3946	-0.2934	0.031*
H20B	0.6259	0.4753	-0.2849	0.031*
C14	0.53931 (10)	0.72068 (13)	-0.77559 (8)	0.0265 (3)
H14	0.5566	0.7071	-0.8357	0.032*
C16	0.38052 (17)	0.8497 (2)	-0.50095 (12)	0.0780 (8)
H16A	0.3195	0.8174	-0.5428	0.117*
H16B	0.3934	0.9400	-0.5170	0.117*
H16C	0.3697	0.8453	-0.4359	0.117*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0290 (5)	0.0251 (4)	0.0246 (4)	0.0063 (4)	0.0083 (4)	0.0012 (3)
N1	0.0245 (5)	0.0214 (5)	0.0190 (5)	0.0009 (4)	0.0038 (4)	0.0011 (4)
N2	0.0242 (5)	0.0221 (5)	0.0186 (5)	0.0009 (4)	0.0035 (4)	0.0003 (4)
N5	0.0233 (5)	0.0256 (5)	0.0184 (5)	0.0025 (4)	0.0024 (4)	0.0011 (4)
N6	0.0260 (5)	0.0293 (6)	0.0184 (5)	0.0058 (4)	0.0030 (4)	0.0014 (4)
N4	0.0316 (6)	0.0290 (6)	0.0208 (5)	0.0073 (4)	0.0070 (4)	0.0029 (4)
C3	0.0189 (5)	0.0251 (6)	0.0217 (6)	-0.0028 (4)	0.0029 (4)	0.0003 (5)
C1	0.0183 (5)	0.0222 (6)	0.0186 (5)	-0.0020 (4)	0.0024 (4)	-0.0023 (4)
N3	0.0332 (6)	0.0288 (6)	0.0227 (5)	0.0071 (5)	0.0082 (4)	0.0018 (4)
C9	0.0234 (6)	0.0241 (6)	0.0185 (6)	0.0016 (5)	0.0037 (4)	-0.0030 (5)
O2	0.0503 (6)	0.0703 (8)	0.0190 (5)	0.0395 (6)	0.0080 (4)	0.0032 (5)
C10	0.0227 (6)	0.0234 (6)	0.0181 (6)	-0.0002 (5)	0.0024 (4)	-0.0010 (5)
N8	0.0351 (6)	0.0489 (7)	0.0212 (5)	0.0174 (5)	0.0067 (5)	0.0042 (5)
C7	0.0229 (6)	0.0275 (6)	0.0208 (6)	-0.0020 (5)	0.0032 (5)	-0.0014 (5)
C2	0.0192 (5)	0.0233 (6)	0.0188 (6)	-0.0028 (4)	0.0015 (4)	0.0004 (5)
C13	0.0335 (7)	0.0285 (6)	0.0214 (6)	-0.0001 (5)	0.0000 (5)	0.0046 (5)
C18	0.0253 (6)	0.0254 (6)	0.0188 (6)	0.0001 (5)	0.0040 (5)	0.0036 (5)
C19	0.0268 (6)	0.0267 (6)	0.0193 (6)	0.0018 (5)	0.0047 (5)	0.0038 (5)
C15	0.0265 (6)	0.0242 (6)	0.0213 (6)	-0.0004 (5)	0.0067 (5)	-0.0012 (5)
C4	0.0221 (6)	0.0264 (6)	0.0313 (7)	0.0007 (5)	0.0030 (5)	0.0052 (5)
C6	0.0283 (6)	0.0363 (7)	0.0200 (6)	-0.0044 (5)	0.0047 (5)	0.0025 (5)
N7	0.0370 (6)	0.0523 (8)	0.0214 (5)	0.0202 (6)	0.0075 (5)	0.0051 (5)
C17	0.0292 (6)	0.0245 (6)	0.0189 (6)	-0.0004 (5)	0.0062 (5)	0.0041 (5)
C12	0.0311 (7)	0.0320 (7)	0.0254 (6)	0.0100 (5)	0.0019 (5)	0.0018 (5)
C11	0.0293 (6)	0.0345 (7)	0.0187 (6)	0.0067 (5)	0.0035 (5)	-0.0008 (5)
C8	0.0318 (7)	0.0250 (6)	0.0356 (7)	0.0057 (5)	0.0107 (6)	-0.0010 (5)

C5	0.0259 (6)	0.0338 (7)	0.0256 (6)	-0.0028 (5)	0.0008 (5)	0.0098 (5)
C20	0.0304 (6)	0.0292 (7)	0.0174 (6)	0.0015 (5)	0.0026 (5)	0.0046 (5)
C14	0.0335 (7)	0.0288 (6)	0.0177 (6)	-0.0033 (5)	0.0060 (5)	0.0005 (5)
C16	0.0869 (14)	0.1223 (19)	0.0290 (8)	0.0798 (14)	0.0218 (9)	0.0134 (10)

Geometric parameters (Å, °)

O1—C3	1.3644 (14)	C18—H18A	0.9900
O1—C8	1.4331 (15)	C18—H18B	0.9900
N1—N2	1.3315 (14)	C18—C19	1.5262 (16)
N1—C1	1.3339 (15)	C18—C17	1.5214 (17)
N2—N3	1.3179 (14)	C19—H19A	0.9900
N2—C17	1.4647 (14)	C19—H19B	0.9900
N5—N6	1.3243 (14)	C19—C20	1.5232 (17)
N5—C9	1.3306 (15)	C15—H15	0.9500
N6—N7	1.3166 (15)	C15—C14	1.3843 (17)
N6—C20	1.4618 (15)	C4—H4	0.9500
N4—C1	1.3619 (15)	C4—C5	1.3858 (18)
N4—N3	1.3218 (15)	C6—H6	0.9500
C3—C2	1.4069 (17)	C6—C5	1.3854 (19)
C3—C4	1.3924 (17)	C17—H17A	0.9900
C1—C2	1.4719 (16)	C17—H17B	0.9900
C9—C10	1.4700 (16)	C12—H12	0.9500
C9—N8	1.3555 (16)	C12—C11	1.3945 (18)
O2—C11	1.3597 (15)	C8—H8A	0.9800
O2—C16	1.4242 (18)	C8—H8B	0.9800
C10—C15	1.3959 (16)	C8—H8C	0.9800
C10—C11	1.4043 (17)	C5—H5	0.9500
N8—N7	1.3240 (16)	C20—H20A	0.9900
C7—H7	0.9500	C20—H20B	0.9900
C7—C2	1.4009 (16)	C14—H14	0.9500
C7—C6	1.3833 (18)	C16—H16A	0.9800
C13—H13	0.9500	C16—H16B	0.9800
C13—C12	1.3864 (18)	C16—H16C	0.9800
C13—C14	1.3834 (18)		
C3—O1—C8	116.89 (10)	C10—C15—H15	119.1
N2—N1—C1	101.66 (9)	C14—C15—C10	121.79 (11)
N1—N2—C17	122.10 (10)	C14—C15—H15	119.1
N3—N2—N1	114.36 (9)	C3—C4—H4	119.7
N3—N2—C17	123.30 (10)	C5—C4—C3	120.67 (12)
N6—N5—C9	101.65 (9)	C5—C4—H4	119.7
N5—N6—C20	122.20 (10)	C7—C6—H6	120.4
N7—N6—N5	114.48 (10)	C7—C6—C5	119.12 (11)
N7—N6—C20	123.17 (10)	C5—C6—H6	120.4
N3—N4—C1	106.28 (10)	N6—N7—N8	105.79 (10)
O1—C3—C2	117.10 (10)	N2—C17—C18	110.45 (9)
O1—C3—C4	123.32 (11)	N2—C17—H17A	109.6

C4—C3—C2	119.58 (11)	N2—C17—H17B	109.6
N1—C1—N4	111.73 (10)	C18—C17—H17A	109.6
N1—C1—C2	126.98 (10)	C18—C17—H17B	109.6
N4—C1—C2	121.26 (10)	H17A—C17—H17B	108.1
N2—N3—N4	105.96 (10)	C13—C12—H12	119.9
N5—C9—C10	126.72 (10)	C13—C12—C11	120.22 (12)
N5—C9—N8	111.99 (10)	C11—C12—H12	119.9
N8—C9—C10	121.28 (10)	O2—C11—C10	116.32 (11)
C11—O2—C16	117.36 (11)	O2—C11—C12	123.66 (11)
C15—C10—C9	118.61 (11)	C12—C11—C10	120.02 (11)
C15—C10—C11	118.25 (11)	O1—C8—H8A	109.5
C11—C10—C9	123.11 (10)	O1—C8—H8B	109.5
N7—N8—C9	106.08 (10)	O1—C8—H8C	109.5
C2—C7—H7	119.1	H8A—C8—H8B	109.5
C6—C7—H7	119.1	H8A—C8—H8C	109.5
C6—C7—C2	121.70 (12)	H8B—C8—H8C	109.5
C3—C2—C1	123.62 (10)	C4—C5—H5	119.8
C7—C2—C3	118.41 (11)	C6—C5—C4	120.45 (12)
C7—C2—C1	117.97 (11)	C6—C5—H5	119.8
C12—C13—H13	119.7	N6—C20—C19	112.38 (10)
C14—C13—H13	119.7	N6—C20—H20A	109.1
C14—C13—C12	120.50 (12)	N6—C20—H20B	109.1
H18A—C18—H18B	107.8	C19—C20—H20A	109.1
C19—C18—H18A	109.0	C19—C20—H20B	109.1
C19—C18—H18B	109.0	H20A—C20—H20B	107.9
C17—C18—H18A	109.0	C13—C14—C15	119.21 (11)
C17—C18—H18B	109.0	C13—C14—H14	120.4
C17—C18—C19	112.93 (10)	C15—C14—H14	120.4
C18—C19—H19A	110.0	O2—C16—H16A	109.5
C18—C19—H19B	110.0	O2—C16—H16B	109.5
H19A—C19—H19B	108.4	O2—C16—H16C	109.5
C20—C19—C18	108.39 (10)	H16A—C16—H16B	109.5
C20—C19—H19A	110.0	H16A—C16—H16C	109.5
C20—C19—H19B	110.0	H16B—C16—H16C	109.5

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C7—H7 \cdots N4	0.95	2.48	2.8371 (16)	102
C15—H15 \cdots N8	0.95	2.53	2.8586 (17)	101
C17—H17A \cdots O1 ⁱ	0.99	2.58	3.4337 (15)	144

Symmetry code: (i) $-x+2, -y+1, -z$.