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4-{[5-Methyl-2-(propan-2-yl)phenoxy]methyl}-1-(4-nitrobenzyl)-1*H*-1,2,3-triazole

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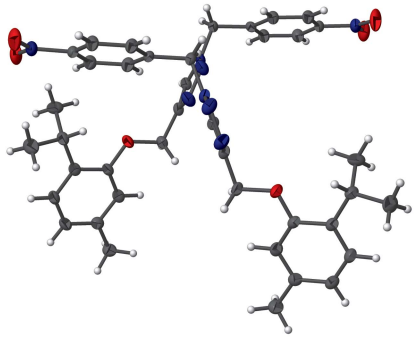
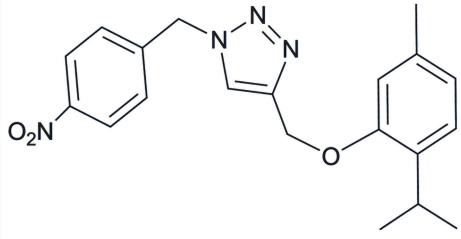
The asymmetric unit of the title compound, $C_{20}H_{22}N_4O_3$, comprises two independent molecules (*A* and *B*) with markedly different conformations as the C—H bonds of the imidazole rings point in opposite directions. In the crystal, C—H···N hydrogen bonds form chains of molecules extending along the *b*-axis direction, while C—H···O hydrogen bonds form zigzag chains along *a*. Additional intermolecular N···O···π(ring) contacts form parallel chains of like molecules along *a*. The structure was refined as a two-component twin.

3D view



Chemical scheme



Structure description

1,2,3-Triazole derivatives are often recognized as precursors in the synthesis of a variety of therapeutic agents. Indeed, heterocyclic compounds containing this motif exhibit anticancer activity (Stéphan *et al.*, 1995), and are known to be antineoplastic (Passannanti *et al.*,1998), antimicrobial (Sheremet *et al.*, 2004) and anti-HIV agents (Velázquez *et al.*, 1998). In this work we have synthesized 4-{[5-Methyl-2-(propan-2-yl)phenoxy]methyl}-1-(4-nitrobenzyl)-1*H*-1,2,3-triazole by a 1,3-dipolar cycloaddition reaction of 4-nitrobenzoyl azide with 1-isopropyl-4-methyl-2-(prop-2-yn-1-yloxy)benzene and report its structure here.

The asymmetric unit of the title compound comprises two independent molecules, *A* and *B*, with markedly different conformations (Fig. 1). The difference between the two unique molecules lies in the fact that the triazole ring is rotated by approximately 180° about the C7—N2 or C27—N6 bonds so that the C8—H8 and C28—H28 bonds point in diametrically opposite directions, as shown by the N2—C7—C4—C5 and N6—C27—C24—C25 torsion angles of 88.4 (3) and −89.4 (3)° respectively. Interestingly other features of the two molecules are quite similar, with the dihedral angles between the N2

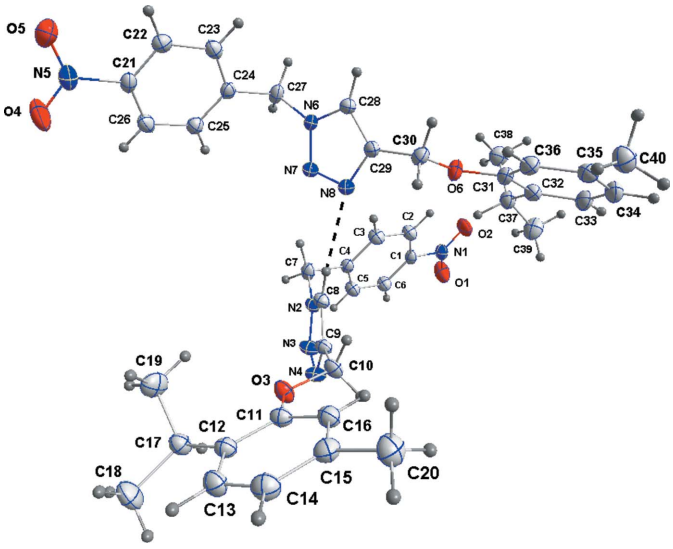


Figure 1
The asymmetric unit with labeling scheme and 50% probability ellipsoids. The C8—H8···N8 hydrogen bond is shown as a dashed line.

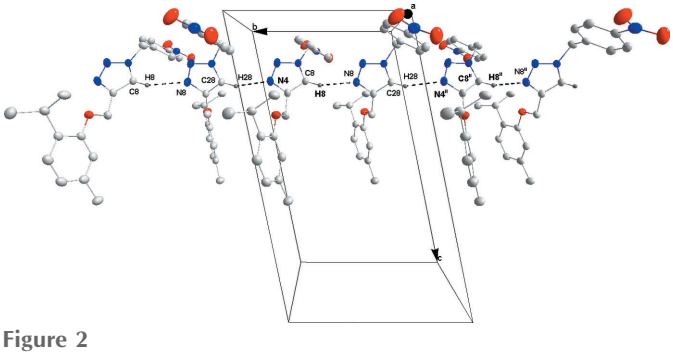


Figure 2
Packing viewed along the *a*-axis direction, showing the chain along the *b*-axis direction formed by the C—H···N hydrogen bonds (dashed lines) [symmetry codes: (i) $x, y+1, z$; (ii) $x, y-1, z$].

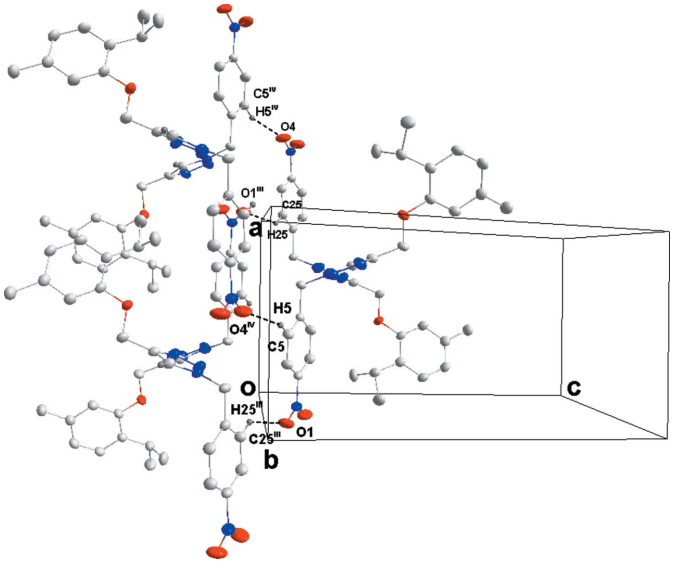


Figure 3
Packing viewed along the *b*-axis direction, showing the sheet parallel to the *ac* plane formed by C—H···O hydrogen bonds (dashed lines) [symmetry codes: (iii) $-x+1, -y+1, -z$; (iv) $-x+2, -y+1, -z$].

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···N8	0.95	2.43	3.300 (3)	151
C28—H28···N4^{i}	0.95	2.45	3.305 (3)	149
C5—H5···O4^{ii}	0.95	2.55	3.466 (3)	161
C25—H25···O1^{iii}	0.95	2.56	3.330 (3)	138

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{22}N_4O_3$
M_r	366.41
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, b, c (Å)	9.4118 (3), 10.8934 (4), 18.6996 (7)
α, β, γ (°)	101.629 (1), 91.752 (1), 90.351 (1)
V (Å^3)	1876.83 (12)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.73
Crystal size (mm)	0.22 × 0.13 × 0.11
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*TWINABS*; Sheldrick, 2009)
T_{min}, T_{max}	0.81, 0.92
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	22212, 11537, 9810
R_{int}	0.033
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.049, 0.197, 1.06
No. of reflections	11537
No. of parameters	495
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.34, −0.28

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXTL* and *CELL_NOW* (Sheldrick, 2008), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012), *SHELXTL* (Sheldrick, 2008).

and N6 imidazole rings and the corresponding nitrophenyl rings being 68.94 (9) and 67.37 (9)°, respectively, while those between the same imidazole rings and the C11–C16 and C21–C26 benzene rings are 65.85 (9) and 65.21 (9)°, respectively.

The two unique molecules are linked by a weak C8—H8···N8 hydrogen bond and these pairs of molecules are

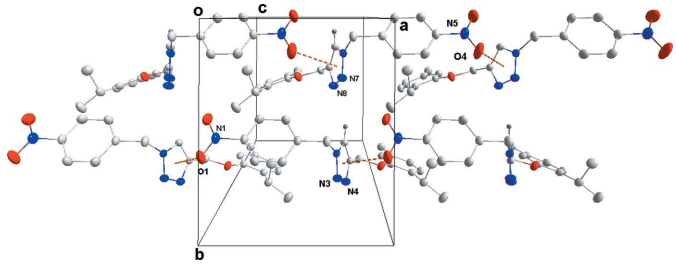


Figure 4
Chains of molecules formed along *a* by N═O···π(ring) contacts (shown as dashed lines).

further linked along the *b*-axis direction by C28—H28···N4^{i} hydrogen bonds (Table 1 and Fig. 2). A combination of C5—H5···O4^{ii} and C25—H25···O1^{iii} hydrogen bonds link alternate *A* and *B* molecules into zigzag chains along *a* (Table 1, Fig. 3). Additionally, the crystal packing is supplemented by N1⋯O1···π(ring)^{v} [O1···centroid of N2–N4/C8/C9 = 3.540 (2) Å; symmetry code: (v) $-1+x, y, z$]. These link type *A* molecules into chains along the *a*-axis direction. A second and parallel set of chains of *B* molecules form in an obverse fashion through N5⋯O4···π(ring)^{vi} [O4···centroid of N6–N8/C28/C29 = 3.550 (2) Å; symmetry code: (vi) $1+x, y, z$] interactions (Fig. 4).

Synthesis and crystallization

In a vial fitted with a screw cap, nitrobenzyl azide (100 mg, 0.56 mmol) and 1-isopropyl-4-methyl-2-(prop-2-yn-1-yloxy)benzene (110 mg, 0.58 mmol) were added to a mixture of copper(II) sulfate pentahydrate (7 mg, 0.028 mmol), sodium ascorbate (16.6 mg, 0.083 mmol), and β-cyclodextrin (15.9 mg, 0.014 mmol) dissolved in H_2O (1 ml) at room temperature. The reaction mixture was stirred for 15 min at room temperature. The resulting mixture was poured into CH_2Cl_2 (3 ml) and H_2O (3 ml), and the organic layer was separated. The aqueous layer was extracted with CH_2Cl_2 (3 ml) three times. The product was obtained as colorless crystals in a yield of 90%.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The structure was refined as a two-component twin.

Acknowledgements

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References

Brandenburg, K. & Putz, H. (2012). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2016). *APEX3*, *SAINT* and *SADABS*. Bruker AXS, Inc., Madison, Wisconsin, USA.
Passannanti, A., Diana, P., Barraja, P., Mingoia, F., Lauria, A. & Cirrincione, G. (1998). *Heterocycles*, **48**, 1229–1235.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2009). *TWINABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Sheremet, E. A., Tomanov, R. I., Trukhin, E. V. & Berestovitskaya, V. M. (2004). *Russ. J. Org. Chem.* **40**, 594–595.
Stéphan, F., Etienne, M. C., Wallays, C., Milano, G. & Clergue, F. (1995). *Am. J. Med.* **99**, 685–688.
Velázquez, S., Alvarez, R., Pérez, C., Gago, F., De Clercq, E., Balzarini, J. & Camarasa, M. J. (1998). *Antivir. Chem. Chemother.* **9**, 481–489.

full crystallographic data

IUCrData (2017). **2**, x170516 [https://doi.org/10.1107/S2414314617005168]

4-{[5-Methyl-2-(propan-2-yl)phenoxy]methyl}-1-(4-nitrobenzyl)-1*H*-1,2,3-triazole

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Crystal data

$C_{20}H_{22}N_4O_3$
M_r = 366.41
Triclinic, $P\overline{1}$
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b = 10.8934 (4) Å
c = 18.6996 (7) Å
α = 101.629 (1)°
β = 91.752 (1)°
γ = 90.351 (1)°
V = 1876.83 (12) Å^3
Z = 4
$F(000)$ = 776
D_x = 1.297 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9905 reflections
θ = 4.1–72.5°
μ = 0.73 mm^{-1}
T = 150 K
Column, colourless
0.22 × 0.13 × 0.11 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro-focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*TWINABS*; Sheldrick, 2009)
T_{min} = 0.81, T_{max} = 0.92
22212 measured reflections
11537 independent reflections
9810 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 72.4°, θ_{min} = 4.1°
h = −11→11
k = −13→13
l = 0→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.197
S = 1.06
11537 reflections
495 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
$w = 1/[\sigma^2(F_o^2) + (0.1284P)^2 + 0.5216P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015*b*), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0089 (12)

Special details

Experimental. Analysis of 2112 reflections having I/σ(I) > 12 and chosen from the full data set with *CELL_NOW* (Sheldrick, 2008) showed the crystal to belong to the triclinic system and to be twinned by a 180° rotation about the *c** axis. The raw data were processed using the multi-component version of *SAINT* under control of the two-component orientation file generated by *CELL_NOW*.

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. 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 $F^2 > 2\mathrm{sigma}(F^2)$ 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. Refined as a 2-component twin.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.0186 (2)	0.6403 (2)	0.05682 (12)	0.0451 (5)
O2	−0.0124 (2)	0.48631 (19)	0.11339 (12)	0.0416 (5)
O3	1.01236 (18)	0.83073 (17)	0.33021 (9)	0.0279 (4)
N1	0.0461 (2)	0.5660 (2)	0.08658 (12)	0.0289 (5)
N2	0.71275 (19)	0.67371 (17)	0.15685 (11)	0.0212 (4)
N3	0.7185 (2)	0.79683 (19)	0.15655 (12)	0.0295 (5)
N4	0.7727 (2)	0.85253 (19)	0.22071 (12)	0.0290 (5)
C1	0.2027 (2)	0.5720 (2)	0.08933 (12)	0.0220 (5)
C2	0.2757 (3)	0.4906 (2)	0.12426 (14)	0.0266 (5)
H2	0.2266	0.4331	0.1470	0.032*
C3	0.4234 (3)	0.4952 (2)	0.12530 (13)	0.0264 (5)
H3	0.4764	0.4401	0.1489	0.032*
C4	0.4939 (2)	0.5800 (2)	0.09194 (12)	0.0205 (5)
C5	0.4171 (2)	0.6616 (2)	0.05791 (13)	0.0257 (5)
H5	0.4657	0.7203	0.0358	0.031*
C6	0.2695 (3)	0.6577 (2)	0.05611 (13)	0.0262 (5)
H6	0.2159	0.7127	0.0326	0.031*
C7	0.6549 (2)	0.5839 (2)	0.09328 (13)	0.0254 (5)
H7A	0.6916	0.4994	0.0946	0.030*
H7B	0.6874	0.6078	0.0480	0.030*
C8	0.7630 (2)	0.6501 (2)	0.22044 (12)	0.0214 (5)
H8	0.7698	0.5711	0.2344	0.026*
C9	0.8023 (2)	0.7649 (2)	0.26084 (13)	0.0216 (5)
C10	0.8668 (2)	0.7957 (2)	0.33600 (13)	0.0253 (5)
H10A	0.8155	0.8658	0.3661	0.030*
H10B	0.8611	0.7221	0.3594	0.030*
C11	1.0938 (2)	0.8565 (2)	0.39394 (13)	0.0231 (5)
C12	1.2326 (2)	0.8995 (2)	0.38645 (13)	0.0239 (5)
C13	1.3178 (3)	0.9287 (2)	0.44992 (14)	0.0283 (5)
H13	1.4118	0.9591	0.4470	0.034*
C14	1.2696 (3)	0.9145 (2)	0.51735 (14)	0.0295 (5)

H14	1.3311	0.9352	0.5594	0.035*
C15	1.1329 (3)	0.8707 (2)	0.52406 (13)	0.0267 (5)
C16	1.0454 (3)	0.8421 (2)	0.46120 (14)	0.0266 (5)
H16	0.9513	0.8123	0.4645	0.032*
C17	1.2829 (3)	0.9113 (2)	0.31143 (13)	0.0268 (5)
H17	1.2032	0.9476	0.2861	0.032*
C18	1.4120 (3)	0.9978 (3)	0.31445 (17)	0.0409 (7)
H18A	1.4950	0.9597	0.3337	0.061*
H18B	1.3932	1.0787	0.3464	0.061*
H18C	1.4306	1.0107	0.2652	0.061*
C19	1.3141 (3)	0.7820 (3)	0.26560 (14)	0.0363 (6)
H19A	1.2280	0.7294	0.2604	0.055*
H19B	1.3900	0.7427	0.2898	0.055*
H19C	1.3442	0.7909	0.2172	0.055*
C20	1.0772 (3)	0.8530 (3)	0.59602 (15)	0.0331 (6)
H20A	1.0893	0.7655	0.6005	0.050*
H20B	0.9760	0.8738	0.5983	0.050*
H20C	1.1299	0.9081	0.6361	0.050*
O4	1.4652 (2)	0.1501 (2)	0.05767 (14)	0.0534 (6)
O5	1.4666 (2)	−0.0108 (2)	0.10798 (14)	0.0483 (6)
O6	0.46802 (17)	0.33027 (16)	0.33204 (9)	0.0274 (4)
N5	1.4050 (2)	0.0711 (2)	0.08389 (12)	0.0318 (5)
N6	0.7479 (2)	0.17490 (18)	0.15999 (11)	0.0220 (4)
N7	0.7423 (2)	0.29755 (19)	0.15935 (12)	0.0289 (5)
N8	0.6953 (2)	0.35348 (19)	0.22316 (12)	0.0295 (5)
C21	1.2489 (2)	0.0760 (2)	0.08802 (13)	0.0233 (5)
C22	1.1811 (3)	−0.0049 (2)	0.12393 (14)	0.0269 (5)
H22	1.2336	−0.0622	0.1461	0.032*
C23	1.0336 (3)	−0.0007 (2)	0.12694 (14)	0.0267 (5)
H23	0.9845	−0.0556	0.1515	0.032*
C24	0.9577 (2)	0.0835 (2)	0.09428 (12)	0.0217 (5)
C25	1.0299 (2)	0.1645 (2)	0.05917 (13)	0.0251 (5)
H25	0.9779	0.2226	0.0374	0.030*
C26	1.1767 (3)	0.1619 (2)	0.05538 (14)	0.0271 (5)
H26	1.2262	0.2172	0.0312	0.032*
C27	0.7972 (2)	0.0858 (2)	0.09618 (13)	0.0256 (5)
H27A	0.7584	0.1094	0.0511	0.031*
H27B	0.7608	0.0008	0.0975	0.031*
C28	0.7040 (2)	0.1506 (2)	0.22331 (13)	0.0221 (5)
H28	0.6978	0.0715	0.2371	0.027*
C29	0.6701 (2)	0.2653 (2)	0.26358 (13)	0.0223 (5)
C30	0.6138 (2)	0.2968 (2)	0.33840 (13)	0.0249 (5)
H30A	0.6218	0.2237	0.3623	0.030*
H30B	0.6687	0.3676	0.3684	0.030*
C31	0.3940 (2)	0.3557 (2)	0.39575 (13)	0.0238 (5)
C32	0.2544 (3)	0.3990 (2)	0.38793 (13)	0.0250 (5)
C33	0.1778 (3)	0.4283 (2)	0.45124 (14)	0.0286 (5)
H33	0.0840	0.4591	0.4483	0.034*

C34	0.2336 (3)	0.4141 (2)	0.51889 (14)	0.0296 (5)
H34	0.1775	0.4350	0.5609	0.036*
C35	0.3703 (3)	0.3697 (2)	0.52572 (13)	0.0271 (5)
C36	0.4499 (3)	0.3416 (2)	0.46302 (14)	0.0270 (5)
H36	0.5442	0.3122	0.4665	0.032*
C37	0.1960 (3)	0.4112 (2)	0.31303 (13)	0.0272 (5)
H37	0.2734	0.4478	0.2881	0.033*
C38	0.1579 (3)	0.2821 (3)	0.26654 (14)	0.0359 (6)
H38A	0.1249	0.2915	0.2177	0.054*
H38B	0.0823	0.2433	0.2897	0.054*
H38C	0.2420	0.2289	0.2624	0.054*
C39	0.0688 (3)	0.4979 (3)	0.31605 (16)	0.0397 (7)
H39A	0.0937	0.5795	0.3469	0.060*
H39B	−0.0118	0.4610	0.3365	0.060*
H39C	0.0428	0.5090	0.2666	0.060*
C40	0.4336 (3)	0.3527 (3)	0.59797 (14)	0.0337 (6)
H40A	0.4188	0.2658	0.6032	0.051*
H40B	0.3875	0.4094	0.6377	0.051*
H40C	0.5357	0.3718	0.6001	0.051*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0218 (9)	0.0650 (14)	0.0571 (13)	0.0067 (9)	−0.0009 (9)	0.0332 (11)
O2	0.0242 (9)	0.0456 (12)	0.0578 (13)	−0.0069 (8)	0.0084 (9)	0.0164 (10)
O3	0.0226 (8)	0.0359 (9)	0.0249 (9)	−0.0091 (7)	−0.0044 (7)	0.0069 (7)
N1	0.0197 (10)	0.0365 (12)	0.0301 (11)	−0.0008 (8)	0.0016 (8)	0.0058 (9)
N2	0.0165 (8)	0.0209 (9)	0.0264 (10)	−0.0024 (7)	−0.0017 (7)	0.0059 (7)
N3	0.0297 (11)	0.0221 (10)	0.0380 (12)	−0.0030 (8)	−0.0087 (9)	0.0108 (9)
N4	0.0301 (11)	0.0202 (10)	0.0362 (12)	−0.0030 (8)	−0.0091 (9)	0.0060 (8)
C1	0.0170 (10)	0.0266 (11)	0.0208 (11)	−0.0014 (8)	0.0005 (8)	0.0012 (9)
C2	0.0257 (12)	0.0275 (12)	0.0282 (12)	−0.0033 (9)	0.0024 (9)	0.0098 (10)
C3	0.0238 (11)	0.0288 (12)	0.0286 (12)	0.0006 (9)	−0.0024 (9)	0.0114 (10)
C4	0.0162 (10)	0.0232 (11)	0.0202 (11)	−0.0010 (8)	−0.0014 (8)	0.0001 (8)
C5	0.0221 (11)	0.0280 (12)	0.0283 (12)	−0.0036 (9)	−0.0003 (9)	0.0090 (10)
C6	0.0222 (11)	0.0288 (12)	0.0296 (12)	0.0018 (9)	−0.0009 (9)	0.0108 (10)
C7	0.0167 (11)	0.0319 (12)	0.0253 (12)	−0.0010 (9)	−0.0012 (8)	0.0010 (9)
C8	0.0201 (10)	0.0199 (11)	0.0253 (11)	−0.0014 (8)	−0.0018 (8)	0.0076 (9)
C9	0.0179 (10)	0.0188 (10)	0.0282 (12)	−0.0023 (8)	−0.0021 (9)	0.0053 (9)
C10	0.0197 (11)	0.0277 (12)	0.0281 (12)	−0.0067 (9)	−0.0028 (9)	0.0052 (9)
C11	0.0223 (11)	0.0223 (11)	0.0238 (12)	−0.0020 (9)	−0.0047 (9)	0.0034 (9)
C12	0.0230 (11)	0.0224 (11)	0.0257 (12)	−0.0020 (9)	−0.0023 (9)	0.0038 (9)
C13	0.0214 (11)	0.0312 (13)	0.0313 (13)	−0.0033 (9)	−0.0042 (9)	0.0045 (10)
C14	0.0283 (12)	0.0309 (13)	0.0279 (13)	−0.0016 (10)	−0.0063 (10)	0.0041 (10)
C15	0.0301 (12)	0.0245 (12)	0.0254 (12)	0.0016 (10)	−0.0006 (10)	0.0046 (9)
C16	0.0249 (12)	0.0246 (12)	0.0301 (13)	−0.0025 (9)	−0.0005 (9)	0.0050 (10)
C17	0.0269 (12)	0.0293 (12)	0.0238 (12)	−0.0050 (10)	−0.0026 (9)	0.0048 (9)
C18	0.0355 (14)	0.0487 (17)	0.0402 (16)	−0.0145 (13)	0.0024 (12)	0.0128 (13)

C19	0.0460 (16)	0.0378 (15)	0.0233 (13)	−0.0014 (12)	0.0020 (11)	0.0016 (11)
C20	0.0355 (14)	0.0377 (14)	0.0277 (13)	−0.0008 (11)	0.0004 (10)	0.0107 (11)
O4	0.0243 (10)	0.0775 (17)	0.0696 (15)	−0.0073 (10)	0.0044 (10)	0.0417 (13)
O5	0.0241 (9)	0.0473 (12)	0.0762 (16)	0.0072 (9)	−0.0005 (10)	0.0187 (11)
O6	0.0229 (8)	0.0355 (9)	0.0243 (9)	0.0086 (7)	0.0066 (7)	0.0064 (7)
N5	0.0211 (10)	0.0402 (12)	0.0333 (12)	−0.0020 (9)	0.0024 (8)	0.0052 (10)
N6	0.0188 (9)	0.0215 (9)	0.0261 (10)	0.0024 (7)	0.0057 (7)	0.0053 (8)
N7	0.0312 (11)	0.0226 (10)	0.0357 (12)	0.0042 (8)	0.0106 (9)	0.0106 (8)
N8	0.0330 (11)	0.0214 (10)	0.0357 (12)	0.0032 (8)	0.0120 (9)	0.0078 (8)
C21	0.0168 (11)	0.0270 (12)	0.0245 (11)	−0.0002 (9)	0.0020 (8)	0.0014 (9)
C22	0.0254 (12)	0.0258 (12)	0.0310 (13)	0.0032 (9)	0.0019 (9)	0.0091 (10)
C23	0.0237 (11)	0.0288 (12)	0.0297 (12)	−0.0015 (9)	0.0043 (9)	0.0100 (10)
C24	0.0204 (11)	0.0220 (11)	0.0213 (11)	0.0014 (8)	0.0035 (8)	0.0006 (8)
C25	0.0219 (11)	0.0261 (12)	0.0282 (12)	0.0022 (9)	0.0012 (9)	0.0072 (9)
C26	0.0247 (12)	0.0285 (12)	0.0294 (12)	−0.0030 (9)	0.0030 (9)	0.0089 (10)
C27	0.0176 (11)	0.0299 (12)	0.0264 (12)	0.0013 (9)	0.0027 (9)	−0.0013 (9)
C28	0.0217 (11)	0.0189 (11)	0.0270 (12)	0.0010 (8)	0.0033 (9)	0.0072 (9)
C29	0.0188 (10)	0.0207 (11)	0.0284 (12)	0.0006 (8)	0.0040 (9)	0.0067 (9)
C30	0.0219 (11)	0.0251 (12)	0.0277 (12)	0.0054 (9)	0.0045 (9)	0.0046 (9)
C31	0.0235 (11)	0.0220 (11)	0.0256 (12)	0.0017 (9)	0.0071 (9)	0.0030 (9)
C32	0.0253 (11)	0.0233 (11)	0.0274 (12)	−0.0005 (9)	0.0040 (9)	0.0067 (9)
C33	0.0228 (11)	0.0301 (13)	0.0323 (13)	0.0032 (9)	0.0067 (10)	0.0041 (10)
C34	0.0285 (12)	0.0329 (13)	0.0274 (13)	−0.0001 (10)	0.0087 (10)	0.0050 (10)
C35	0.0302 (12)	0.0242 (12)	0.0272 (13)	−0.0036 (10)	0.0032 (10)	0.0056 (9)
C36	0.0264 (12)	0.0251 (12)	0.0304 (13)	0.0033 (9)	0.0048 (9)	0.0071 (9)
C37	0.0239 (11)	0.0312 (13)	0.0275 (12)	0.0035 (10)	0.0041 (9)	0.0074 (10)
C38	0.0452 (16)	0.0371 (15)	0.0242 (13)	−0.0022 (12)	−0.0012 (11)	0.0041 (11)
C39	0.0363 (15)	0.0492 (17)	0.0354 (15)	0.0141 (13)	0.0006 (12)	0.0127 (13)
C40	0.0363 (14)	0.0387 (14)	0.0270 (13)	−0.0013 (11)	0.0016 (10)	0.0086 (11)

Geometric parameters (Å, º)

O1—N1	1.226 (3)	O4—N5	1.218 (3)
O2—N1	1.222 (3)	O5—N5	1.220 (3)
O3—C11	1.376 (3)	O6—C31	1.379 (3)
O3—C10	1.433 (3)	O6—C30	1.429 (3)
N1—C1	1.473 (3)	N5—C21	1.474 (3)
N2—C8	1.339 (3)	N6—N7	1.340 (3)
N2—N3	1.343 (3)	N6—C28	1.341 (3)
N2—C7	1.467 (3)	N6—C27	1.468 (3)
N3—N4	1.316 (3)	N7—N8	1.316 (3)
N4—C9	1.353 (3)	N8—C29	1.360 (3)
C1—C2	1.378 (3)	C21—C22	1.376 (3)
C1—C6	1.380 (3)	C21—C26	1.387 (3)
C2—C3	1.390 (3)	C22—C23	1.391 (3)
C2—H2	0.9500	C22—H22	0.9500
C3—C4	1.390 (3)	C23—C24	1.389 (3)
C3—H3	0.9500	C23—H23	0.9500

C4—C5	1.388 (3)	C24—C25	1.387 (3)
C4—C7	1.514 (3)	C24—C27	1.513 (3)
C5—C6	1.389 (3)	C25—C26	1.385 (3)
C5—H5	0.9500	C25—H25	0.9500
C6—H6	0.9500	C26—H26	0.9500
C7—H7A	0.9900	C27—H27A	0.9900
C7—H7B	0.9900	C27—H27B	0.9900
C8—C9	1.367 (3)	C28—C29	1.368 (3)
C8—H8	0.9500	C28—H28	0.9500
C9—C10	1.487 (3)	C29—C30	1.487 (3)
C10—H10A	0.9900	C30—H30A	0.9900
C10—H10B	0.9900	C30—H30B	0.9900
C11—C16	1.388 (4)	C31—C36	1.386 (3)
C11—C12	1.406 (3)	C31—C32	1.412 (3)
C12—C13	1.392 (3)	C32—C33	1.388 (3)
C12—C17	1.523 (3)	C32—C37	1.520 (3)
C13—C14	1.390 (4)	C33—C34	1.393 (4)
C13—H13	0.9500	C33—H33	0.9500
C14—C15	1.388 (4)	C34—C35	1.388 (4)
C14—H14	0.9500	C34—H34	0.9500
C15—C16	1.395 (4)	C35—C36	1.394 (3)
C15—C20	1.506 (4)	C35—C40	1.506 (4)
C16—H16	0.9500	C36—H36	0.9500
C17—C18	1.526 (3)	C37—C39	1.525 (4)
C17—C19	1.530 (4)	C37—C38	1.531 (4)
C17—H17	1.0000	C37—H37	1.0000
C18—H18A	0.9800	C38—H38A	0.9800
C18—H18B	0.9800	C38—H38B	0.9800
C18—H18C	0.9800	C38—H38C	0.9800
C19—H19A	0.9800	C39—H39A	0.9800
C19—H19B	0.9800	C39—H39B	0.9800
C19—H19C	0.9800	C39—H39C	0.9800
C20—H20A	0.9800	C40—H40A	0.9800
C20—H20B	0.9800	C40—H40B	0.9800
C20—H20C	0.9800	C40—H40C	0.9800

C11—O3—C10	116.88 (18)	C31—O6—C30	116.70 (18)
O2—N1—O1	123.4 (2)	O4—N5—O5	123.7 (2)
O2—N1—C1	118.3 (2)	O4—N5—C21	118.2 (2)
O1—N1—C1	118.2 (2)	O5—N5—C21	118.1 (2)
C8—N2—N3	111.29 (19)	N7—N6—C28	111.59 (19)
C8—N2—C7	127.9 (2)	N7—N6—C27	120.4 (2)
N3—N2—C7	120.77 (19)	C28—N6—C27	128.1 (2)
N4—N3—N2	106.60 (19)	N8—N7—N6	106.76 (19)
N3—N4—C9	109.07 (19)	N7—N8—C29	108.79 (19)
C2—C1—C6	123.0 (2)	C22—C21—C26	122.8 (2)
C2—C1—N1	118.4 (2)	C22—C21—N5	118.7 (2)
C6—C1—N1	118.6 (2)	C26—C21—N5	118.5 (2)

C1—C2—C3	118.0 (2)	C21—C22—C23	118.4 (2)
C1—C2—H2	121.0	C21—C22—H22	120.8
C3—C2—H2	121.0	C23—C22—H22	120.8
C2—C3—C4	120.4 (2)	C24—C23—C22	120.4 (2)
C2—C3—H3	119.8	C24—C23—H23	119.8
C4—C3—H3	119.8	C22—C23—H23	119.8
C5—C4—C3	120.1 (2)	C25—C24—C23	119.6 (2)
C5—C4—C7	119.9 (2)	C25—C24—C27	120.1 (2)
C3—C4—C7	119.9 (2)	C23—C24—C27	120.2 (2)
C4—C5—C6	120.1 (2)	C26—C25—C24	121.0 (2)
C4—C5—H5	119.9	C26—C25—H25	119.5
C6—C5—H5	119.9	C24—C25—H25	119.5
C1—C6—C5	118.3 (2)	C25—C26—C21	117.8 (2)
C1—C6—H6	120.8	C25—C26—H26	121.1
C5—C6—H6	120.8	C21—C26—H26	121.1
N2—C7—C4	111.59 (18)	N6—C27—C24	111.66 (18)
N2—C7—H7A	109.3	N6—C27—H27A	109.3
C4—C7—H7A	109.3	C24—C27—H27A	109.3
N2—C7—H7B	109.3	N6—C27—H27B	109.3
C4—C7—H7B	109.3	C24—C27—H27B	109.3
H7A—C7—H7B	108.0	H27A—C27—H27B	107.9
N2—C8—C9	104.7 (2)	N6—C28—C29	104.4 (2)
N2—C8—H8	127.7	N6—C28—H28	127.8
C9—C8—H8	127.7	C29—C28—H28	127.8
N4—C9—C8	108.4 (2)	N8—C29—C28	108.4 (2)
N4—C9—C10	123.1 (2)	N8—C29—C30	122.7 (2)
C8—C9—C10	128.5 (2)	C28—C29—C30	128.9 (2)
O3—C10—C9	107.65 (19)	O6—C30—C29	107.88 (19)
O3—C10—H10A	110.2	O6—C30—H30A	110.1
C9—C10—H10A	110.2	C29—C30—H30A	110.1
O3—C10—H10B	110.2	O6—C30—H30B	110.1
C9—C10—H10B	110.2	C29—C30—H30B	110.1
H10A—C10—H10B	108.5	H30A—C30—H30B	108.4
O3—C11—C16	123.7 (2)	O6—C31—C36	123.8 (2)
O3—C11—C12	114.8 (2)	O6—C31—C32	114.8 (2)
C16—C11—C12	121.4 (2)	C36—C31—C32	121.5 (2)
C13—C12—C11	116.6 (2)	C33—C32—C31	116.4 (2)
C13—C12—C17	123.5 (2)	C33—C32—C37	123.7 (2)
C11—C12—C17	119.9 (2)	C31—C32—C37	119.9 (2)
C14—C13—C12	122.1 (2)	C32—C33—C34	122.2 (2)
C14—C13—H13	119.0	C32—C33—H33	118.9
C12—C13—H13	119.0	C34—C33—H33	118.9
C15—C14—C13	120.9 (2)	C35—C34—C33	120.8 (2)
C15—C14—H14	119.6	C35—C34—H34	119.6
C13—C14—H14	119.6	C33—C34—H34	119.6
C14—C15—C16	117.9 (2)	C34—C35—C36	117.9 (2)
C14—C15—C20	122.5 (2)	C34—C35—C40	122.2 (2)
C16—C15—C20	119.6 (2)	C36—C35—C40	120.0 (2)

C11—C16—C15	121.1 (2)	C31—C36—C35	121.2 (2)
C11—C16—H16	119.5	C31—C36—H36	119.4
C15—C16—H16	119.5	C35—C36—H36	119.4
C12—C17—C18	113.6 (2)	C32—C37—C39	113.5 (2)
C12—C17—C19	110.4 (2)	C32—C37—C38	110.7 (2)
C18—C17—C19	110.1 (2)	C39—C37—C38	110.2 (2)
C12—C17—H17	107.5	C32—C37—H37	107.4
C18—C17—H17	107.5	C39—C37—H37	107.4
C19—C17—H17	107.5	C38—C37—H37	107.4
C17—C18—H18A	109.5	C37—C38—H38A	109.5
C17—C18—H18B	109.5	C37—C38—H38B	109.5
H18A—C18—H18B	109.5	H38A—C38—H38B	109.5
C17—C18—H18C	109.5	C37—C38—H38C	109.5
H18A—C18—H18C	109.5	H38A—C38—H38C	109.5
H18B—C18—H18C	109.5	H38B—C38—H38C	109.5
C17—C19—H19A	109.5	C37—C39—H39A	109.5
C17—C19—H19B	109.5	C37—C39—H39B	109.5
H19A—C19—H19B	109.5	H39A—C39—H39B	109.5
C17—C19—H19C	109.5	C37—C39—H39C	109.5
H19A—C19—H19C	109.5	H39A—C39—H39C	109.5
H19B—C19—H19C	109.5	H39B—C39—H39C	109.5
C15—C20—H20A	109.5	C35—C40—H40A	109.5
C15—C20—H20B	109.5	C35—C40—H40B	109.5
H20A—C20—H20B	109.5	H40A—C40—H40B	109.5
C15—C20—H20C	109.5	C35—C40—H40C	109.5
H20A—C20—H20C	109.5	H40A—C40—H40C	109.5
H20B—C20—H20C	109.5	H40B—C40—H40C	109.5

C8—N2—N3—N4	−0.2 (3)	C28—N6—N7—N8	0.5 (3)
C7—N2—N3—N4	179.00 (19)	C27—N6—N7—N8	−179.72 (19)
N2—N3—N4—C9	0.7 (3)	N6—N7—N8—C29	−0.6 (3)
O2—N1—C1—C2	−2.3 (3)	O4—N5—C21—C22	−173.7 (3)
O1—N1—C1—C2	178.5 (2)	O5—N5—C21—C22	5.2 (4)
O2—N1—C1—C6	176.9 (2)	O4—N5—C21—C26	6.4 (4)
O1—N1—C1—C6	−2.3 (3)	O5—N5—C21—C26	−174.7 (2)
C6—C1—C2—C3	−0.5 (4)	C26—C21—C22—C23	0.5 (4)
N1—C1—C2—C3	178.7 (2)	N5—C21—C22—C23	−179.3 (2)
C1—C2—C3—C4	0.1 (4)	C21—C22—C23—C24	0.0 (4)
C2—C3—C4—C5	0.6 (4)	C22—C23—C24—C25	−0.7 (4)
C2—C3—C4—C7	−179.9 (2)	C22—C23—C24—C27	178.6 (2)
C3—C4—C5—C6	−0.9 (4)	C23—C24—C25—C26	0.8 (4)
C7—C4—C5—C6	179.6 (2)	C27—C24—C25—C26	−178.5 (2)
C2—C1—C6—C5	0.2 (4)	C24—C25—C26—C21	−0.2 (4)
N1—C1—C6—C5	−179.0 (2)	C22—C21—C26—C25	−0.5 (4)
C4—C5—C6—C1	0.5 (4)	N5—C21—C26—C25	179.4 (2)
C8—N2—C7—C4	98.3 (3)	N7—N6—C27—C24	80.7 (3)
N3—N2—C7—C4	−80.7 (3)	C25—C24—C27—N6	−89.4 (3)
C5—C4—C7—N2	88.4 (3)	C23—C24—C27—N6	91.3 (3)

C3—C4—C7—N2	−91.0 (3)	N7—N6—C28—C29	−0.2 (3)
N3—N2—C8—C9	−0.4 (3)	C27—N6—C28—C29	−179.9 (2)
C7—N2—C8—C9	−179.5 (2)	N7—N8—C29—C28	0.5 (3)
N3—N4—C9—C8	−0.9 (3)	N7—N8—C29—C30	−179.0 (2)
N3—N4—C9—C10	179.1 (2)	N6—C28—C29—N8	−0.2 (3)
N2—C8—C9—N4	0.8 (3)	N6—C28—C29—C30	179.2 (2)
N2—C8—C9—C10	−179.2 (2)	C31—O6—C30—C29	176.28 (19)
C11—O3—C10—C9	−176.57 (19)	N8—C29—C30—O6	71.6 (3)
N4—C9—C10—O3	−71.7 (3)	C28—C29—C30—O6	−107.8 (3)
C8—C9—C10—O3	108.3 (3)	C30—O6—C31—C36	−4.9 (3)
C10—O3—C11—C16	4.6 (3)	C30—O6—C31—C32	174.7 (2)
C10—O3—C11—C12	−175.4 (2)	O6—C31—C32—C33	−178.6 (2)
O3—C11—C12—C13	179.0 (2)	C36—C31—C32—C33	1.0 (4)
C16—C11—C12—C13	−1.0 (4)	O6—C31—C32—C37	1.5 (3)
O3—C11—C12—C17	−1.4 (3)	C36—C31—C32—C37	−179.0 (2)
C16—C11—C12—C17	178.6 (2)	C31—C32—C33—C34	−1.1 (4)
C11—C12—C13—C14	0.9 (4)	C37—C32—C33—C34	178.9 (2)
C17—C12—C13—C14	−178.7 (2)	C32—C33—C34—C35	0.2 (4)
C12—C13—C14—C15	−0.2 (4)	C33—C34—C35—C36	0.8 (4)
C13—C14—C15—C16	−0.4 (4)	C33—C34—C35—C40	−179.7 (2)
C13—C14—C15—C20	179.4 (2)	O6—C31—C36—C35	179.5 (2)
O3—C11—C16—C15	−179.6 (2)	C32—C31—C36—C35	0.0 (4)
C12—C11—C16—C15	0.4 (4)	C34—C35—C36—C31	−0.9 (4)
C14—C15—C16—C11	0.3 (4)	C40—C35—C36—C31	179.6 (2)
C20—C15—C16—C11	−179.5 (2)	C33—C32—C37—C39	19.6 (3)
C13—C12—C17—C18	−19.9 (4)	C31—C32—C37—C39	−160.4 (2)
C11—C12—C17—C18	160.5 (2)	C33—C32—C37—C38	−104.8 (3)
C13—C12—C17—C19	104.3 (3)	C31—C32—C37—C38	75.1 (3)
C11—C12—C17—C19	−75.3 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···N8	0.95	2.43	3.300 (3)	151
C28—H28···N4^{i}	0.95	2.45	3.305 (3)	149
C5—H5···O4^{ii}	0.95	2.55	3.466 (3)	161
C25—H25···O1^{iii}	0.95	2.56	3.330 (3)	138

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