Crystal structure of N,N′-dibenzyl-3,3′-dimethoxybenzidine

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The title compound, (systematic name: N,N′-dibenzyl-3,3′-dimethoxy-1,1′-biphenyl-4,4′-diamine), $C_{28}H_{28}N_2O_2$, was synthesized by the reduction of a Schiff base prepared via a condensation reaction between o-dianisidine and benzaldehyde under acidic conditions. The molecule lies on a crystallographic inversion centre so that the asymmetric unit contains one half-molecule. The biphenyl moiety compound is essentially planar. Two intramolecular N—H···O hydrogen bonds occur. The dihedral angle between the terminal phenyl and phenylene rings of a benzidine unit is 48.68 (6)°. The methylene C atom of the benzyl group is disordered over two sets of sites, with occupancy ratio 0.779 (18):0.221 (18). In the crystal, molecules are connected by hydrogen bonding between o-dianisidine O atoms and H atoms of the terminal benzyl groups, forming a one-dimensional ladder-like structure. In the data from DFT calculations, the central biphenyl showed a twisted conformation.

1. Chemical context

Benzidine derivatives have received increasing attention in recent years beacuse of their applications in a wide variety of domains, for instance as building blocks in the construction of functionalized organic/organometallic materials and as sensor materials (Hmadeh et al., 2008; Satapathi, 2015; Nagaraja et al., 2017). The chemical and physical properties of benzidine-based compounds have enabled their use in cell biology as staining reagents (Liu et al., 2004). Benzidine derivatives are also relevant examples of simple redox systems, which could find applications as OLEDs (Zhang et al., 2004) or electro-active organic polymeric compounds (D'Eramo et al., 1994). Recently, we have reported copper(I) coordination polymers based on pyromellitic diimide derivatives, and shown that photoluminescence emission peaks are shifted depending on the solvent (Kang et al., 2015). In an extension of previous research, we have synthesized a benzidine derivative as a di-amine intermediate, in which a benzidine moiety was used instead of a pyromellitic diimide spacer unit, and report its crystal structure here.

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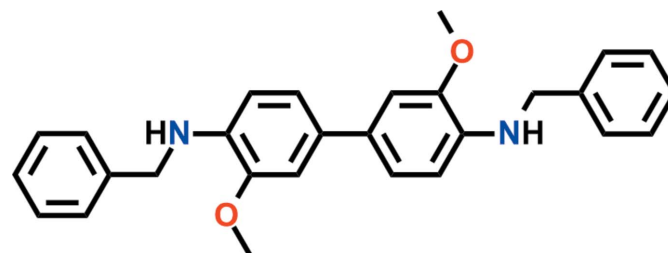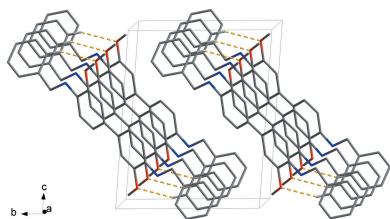
Table 1
Hydrogen-bond geometry (Å, °).

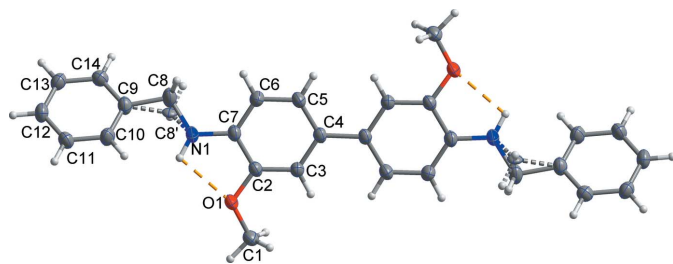| D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C10—H10⋯O1[i]	0.95	2.66	3.400 (2)	135
N1—H1⋯O1	0.88	2.33	2.6464 (19)	101

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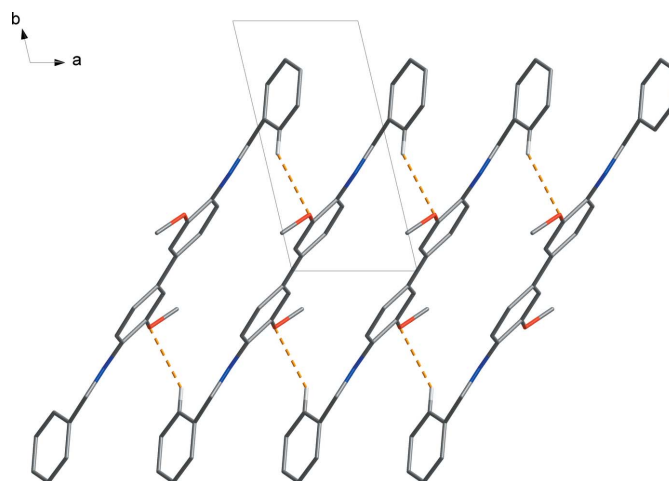
2. Structural commentary

The molecular structure of the title compound consists of a central dimethoxybenzidine unit and two terminal benzyl groups (Fig. 1). The molecule lies about a crystallographic inversion centre at the midpoint of the C4—C4($-x$, $-y$, $-z + 1$) bond, thus the asymmetric unit contains one half-molecule. The dihedral angle between the terminal phenyl and phenylene rings of a benzidine unit is 48.68 (6)°. Disorder was modelled for the methylene C atom of the benzyl group over two sets of sites with an occupancy ratio of 0.779 (18):0.221 (18). The biphenyl moiety is strictly planar [dihedral angle between rings = 0°; maximum deviation of 0.015 (2) Å for atom C3]. There is no pronounced anisotropy in the aryl anisotropic displacement parameters, indicating that there is no disorder or dynamic twisting process to accommodate the steric crowding of the *ortho* H atoms of the biphenyl moiety (El-Shafei *et al.*, 2003). The molecular conformation is in part influenced by the formation of weak intramolecular N1—H1⋯O1 hydrogen bonds that enclose S(5) rings (Fig. 1, Table 1).

3. Supramolecular features

In the crystal, neighbouring molecules are linked by C10—H10⋯O1 hydrogen bonds (Table 1; yellow dashed lines in Fig. 2) that generate $R_2^2(24)$ rings. These contacts stack adjacent molecules, forming a one-dimensional ladder-like structure (Fig. 2). Neighbouring stacks of molecules in these ladders are not connected but lie parallel to the (01$\bar{2}$) plane (Fig. 3).

Figure 2
C—H⋯O hydrogen bonds (orange dashed lines) link adjacent molecules. H atoms not involved in intermolecular interactions have been omitted for clarity.

4. Database survey

The Cambridge Database (Version 5.27, last update February 2017; Groom *et al.*, 2016) reveals polymorphs of related biphenyl derivatives that have both twisted and planar biphenyl conformations (Hoser *et al.*, 2012). However, in the biphenyl compounds 4,4′-diamino-2,2′,6,6′-tetramethylbiphenyl (Batsanov *et al.*, 2006), 2,2′-dichloro-5,5′-dipropoxybenzidine and 2,2′-dimethyl-5,5′-dipropoxybenzidine (El-Shafei *et al.*, 2004), in which atoms other than hydrogen are substituted in the *ortho* positions of the biphenyl unit, adopt twisted biphenyl conformations due to steric repulsion between substituted atoms. Hybrid inorganic–organic complexes with benzidine dications display structures with either twisted or planar conformations for the benzidine unit and, in some case, even both conformations (Dobrzycki & Woźniak, 2009). Related structures with an essentially planar
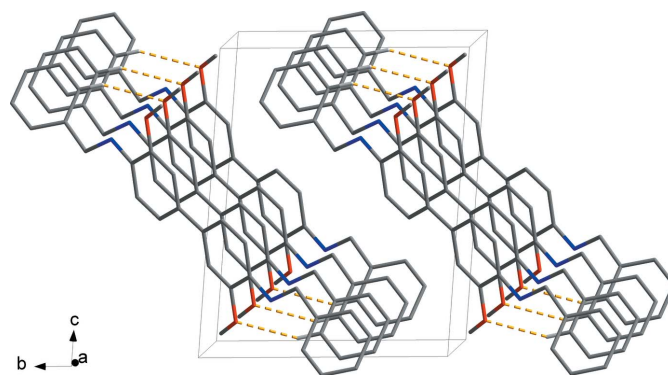

Figure 1
The asymmetric unit of the title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as small spheres of arbitrary radius and yellow dashed lines represent the intramolecular N—H⋯O hydrogen bonds. Unlabelled atoms are generated by the symmetry operation ($-x$, $-y$, $-z + 1$).

Figure 3
Overall packing diagram of title compound, showing the one-dimensional ladder structure (hydrogen bonds drawn as orange dashed lines). H atoms not involved in intermolecular interactions have been omitted for clarity.

Table 2
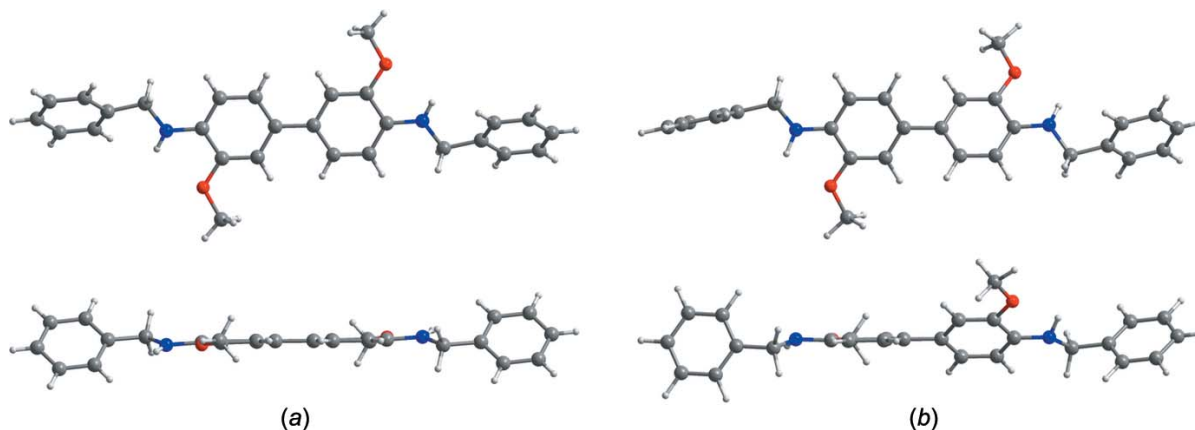Experimental and calculated bond lengths (Å).

Bond	X-ray	B3LYP (6–311G*)
O1—C1	1.425 (2)	1.4208
O1—C2	1.374 (3)	1.3744
N1—C7	1.394 (2)	1.3872
N1—C8	1.438 (5)	1.4567
C2—C3	1.378 (2)	1.3859
C3—C4	1.399 (2)	1.4104
C4—C5	1.389 (2)	1.3951
C5—C6	1.386 (2)	1.3964
C6—C7	1.385 (2)	1.3972
C2—C7	1.408 (2)	1.4189
C8—C9	1.498 (6)	1.5139
C9—C10	1.389 (3)	1.400)
C10—C11	1.379 (3)	1.3921
C11—C12	1.380 (2)	1.3966
C12—C13	1.377 (3)	1.3923
C13—C14	1.383 (3)	1.3965
C9—C14	1.382 (2)	1.3976
C4—C4^i	1.491 (2)	1.4823

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

benzidine conformation include 3,3′-dipropoxybenzidine (El-Shafei *et al.*, 2003), *N,N*-bis(diphenylphosphino)benzidine (Kayan *et al.*, 2012) and *N,N*′-bis(4-chlorobenzylidene)-3,3′-dimethoxybiphenyl-4,4′-diamine (Subashini *et al.*, 2011).

5. Theoretical calculations

DFT calculations have been performed to support the experimental values on the basis of the diffraction study using the *GAUSSIAN09* software package (Frisch *et al.*, 2009). Full geometry optimizations were performed using B3LYP levels of theory with a 6-311G* basis set. The bond lengths of the optimized parameter are in excellent agreement with the experimental crystallographic data (Table 2). Interestingly, however, while the central biphenyl conformation from the crystal structure is found to be planar, that from the DFT calculations shows an angle of 37.67° between the two aromatic rings, Fig. 4. Furthermore, the dihedral angle between the terminal phenyl and phenylene rings of the title compound is 48.68 (6)° from the crystallographic data but 76.69° from the DFT calculation. Similarly, as a result of the

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{28}H_{28}N_2O_2$
M_r	424.52
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	173
a, b, c (Å)	4.7089 (2), 9.6760 (4), 12.1952 (5)
α, β, γ (°)	93.387 (3), 92.165 (2), 103.180 (2)
V (Å^3)	539.32 (4)
Z	1
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	$0.31 \times 0.18 \times 0.06$
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.659, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6528, 1888, 1683
R_{int}	0.019
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.594
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.048, 0.145, 1.10
No. of reflections	1888
No. of parameters	156
No. of restraints	6
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.37, −0.60

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXS97* and *SHELXTL* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *DIAMOND* (Brandenburg, 2010) and *publCIF* (Westrip, 2010).

twisted conformation found in the DFT calculations, the lengths of the intramolecular N—H···O hydrogen bonds from the X-ray and DFT calculation data are also slightly different, at 2.33 and 2.21 Å, respectively.

6. Synthesis and crystallization

A mixture of *o*-dianisidine (4.88 g, 20 mmol), benzaldehyde (4.71 g, 40 mmol) and acetic acid (2.47 g, 40 mmol) in 30 mL of toluene and 7 mL of ethanol was heated at refluxed for 6 h. Sodium borohydride (1.62 g, 40 mmol) was added and the

Figure 4
The central biphenyl conformation from the crystallographic data is planar (*a*), while that from the DFT calculations is twisted (*b*).

mixture was refluxed for two h. After cooling to room temperature, water was added to the reaction mixture. The organic layer was collected and the water layer was extracted with dichloromethane. The combined organic layer was dried with anhydrous sodium sulfate then evaporated to give a solid. Column chromatography (silica gel, ethyl acetate/hexane = 30/70 (v/v) gave the pure product. Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of an ethyl acetate/n-hexane solution (v/v = 30/70) of the title compound. ^{1}H NMR (300 MHz, DMSO): δ = 8.31 (s, 2H, CHCO), 7.28 (m, 10H, phenyl), 6.64 (d, 2H, CCHC), 6.41 (d, 2H, CHCN), 5.52 (t, 2H, NH), 4.33 (d, 4H, CH$_2$), 3.88 (s, 6H, CH$_3$).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were positioned geometrically and refined using a riding model: C—H = 0.95–0.99 Å with U_{iso}(H) = 1.2U_{eq}(C). The methylene C8 atom of the benzyl group is disordered over two sets of sites. Their occupancies refined to 0.779 (18) and 0.221 (18).

References

Batsanov, A. S., Low, P. J. & Paterson, M. A. J. (2006). *Acta Cryst.* E**62**, o2973–o2975.

Brandenburg, K. (2010). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.

Bruker (2014). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

D'Eramo, F., Arévalo, A. H., Silber, J. J. & Sereno, L. (1994). *J. Braz. Chem. Soc.* **5**, 213–218.

Dobrzycki, L. & Woźniak, K. (2009). *J. Mol. Struct.* **921**, 18–33.

El-Shafei, A., Boyle, P. D., Hinks, D. & Freeman, H. S. (2003). *Acta Cryst.* C**59**, o71–o73.

El-Shafei, A., Hinks, D., Boyle, P. D. & Freeman, H. S. (2004). *Acta Cryst.* C**60**, o569–o571.

Frisch, M. J., Trucks, G. W., Schlegel, H. B., Scuseria, G. E., Robb, M. A., Cheeseman, J. R., Scalmani, G., Barone, V., Mennucci, B., Petersson, G. A., Nakatsuji, H., Caricato, M., Li, X., Hratchian, H. P., Izmaylov, A. F., Bloino, J., Zheng, G., Sonnenberg, J. L., Hada, M., Ehara, M., Toyota, K., Fukuda, R., Hasegawa, J., Ishida, M., Nakajima, T., Honda, Y., Kitao, O., Nakai, H., Vreven, T., Montgomery, J. J. A., Peralta, J. E., Ogliaro, F., Bearpark, M., Heyd, J. J., Brothers, E., Kudin, K. N., Staroverov, V. N., Kobayashi, R., Normand, J., Raghavachari, K., Rendell, A., Burant, J. C., Iyengar, S. S., Tomasi, J., Cossi, M., Rega, N., Millam, J. M., Klene, M., Knox, J. E., Cross, J. B., Bakken, V., Adamo, C., Jaramillo, J., Gomperts, R., Stratmann, R. E., Yazyev, O., Austin, A. J., Cammi, R., Pomelli, C., Ochterski, J. W., Martin, R. L., Morokuma, K., Zakrzewski, V. G., Voth, G. A., Salvador, P., Dannenberg, J. J., Dapprich, S., Daniels, A. D., Farkas, O., Foresman, J. B., Ortiz, J. V., Cioslowski, J. & Fox, D. J. (2009). *Gaussian09*. Gaussian Inc, Wallingford, Connecticut, USA.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Hmadeh, M., Traboulsi, H., Elhabiri, M., Braunstein, P., Albrecht-Gary, A. & Siri, O. (2008). *Tetrahedron*, **64**, 6522–6529.

Hoser, A. A., Jarzembska, K. N., Dobrzycki, Ł., Łukasz, , Gutmann, M. J. & Woźniak, K. (2012). *Cryst. Growth Des.* **12**, 3526–3539.

Kang, G., Jeon, Y., Lee, K. Y., Kim, J. & Kim, T. H. (2015). *Cryst. Growth Des.* **15**, 5183–5187.

Kayan, C., Biricik, N., Aydemir, M. & Scopelliti, R. (2012). *Inorg. Chim. Acta*, **385**, 164–169.

Liu, L. F., Yulan, Z. & Qian, X. H. (2004). *Dyes Pigments*, **60**, 17–21.

Nagaraja, V., Kumar, M. K. & Giddappa, N. (2017). *Spectrochim. Acta A Mol. Biomol. Spectrosc.* **173**, 407–417.

Satapathi, S. (2015). *Inorg. Chem. Commun.* **56**, 22–34.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Subashini, A., Ramamurthi, K. & Stoeckli-Evans, H. (2011). *Acta Cryst.* E**67**, o1296–o1297.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

Zhang, S. T., Wang, Z. J., Zhao, J. M., Zhan, Y. Q., Wu, Y., Zhou, Y. C., Ding, X. M. & Hou, X. Y. (2004). *Appl. Phys. Lett.* **84**, 2916–2918.

supporting information

Crystal structure of *N*,*N′*-dibenzyl-3,3′-dimethoxybenzidine

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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: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg, 2010); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

N,N′-dibenzyl-3,3′-dimethoxy-1,1′-biphenyl-4,4′-diamine

Crystal data

$C_{28}H_{28}N_2O_2$	$Z = 1$
$M_r = 424.52$	$F(000) = 226$
Triclinic, $P\bar{1}$	$D_x = 1.307$ Mg m^{-3}
$a = 4.7089$ (2) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 9.6760$ (4) Å	Cell parameters from 5519 reflections
$c = 12.1952$ (5) Å	$\theta = 2.6$–28.0°
$\alpha = 93.387$ (3)°	$\mu = 0.08$ mm^{-1}
$\beta = 92.165$ (2)°	$T = 173$ K
$\gamma = 103.180$ (2)°	Plate, yellow
$V = 539.32$ (4) Å^3	$0.31 \times 0.18 \times 0.06$ mm

Data collection

Bruker APEXII CCD diffractometer	1888 independent reflections
φ and ω scans	1683 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2014)	$R_{int} = 0.019$
	$\theta_{max} = 25.0°$, $\theta_{min} = 1.7°$
$T_{min} = 0.659$, $T_{max} = 0.746$	$h = -5 \rightarrow 5$
6528 measured reflections	$k = -11 \rightarrow 11$
	$l = -13 \rightarrow 14$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.048$	$w = 1/[\sigma^2(F_o^2) + (0.0797P)^2 + 0.2226P]$
$wR(F^2) = 0.145$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{max} < 0.001$
1888 reflections	$\Delta\rho_{max} = 0.37$ e Å^{-3}
156 parameters	$\Delta\rho_{min} = -0.60$ e Å^{-3}
6 restraints	

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.2453 (3)	0.22554 (13)	0.81729 (9)	0.0350 (4)	
N1	0.6872 (3)	0.38961 (16)	0.72486 (12)	0.0367 (4)	
H1	0.6890	0.3862	0.7968	0.044*	
C1	−0.0076 (4)	0.15213 (19)	0.86818 (14)	0.0355 (4)	
H1A	−0.1827	0.1701	0.8312	0.053*	
H1B	0.0024	0.1860	0.9459	0.053*	
H1C	−0.0163	0.0498	0.8625	0.053*	
C2	0.2594 (3)	0.19646 (17)	0.70622 (13)	0.0275 (4)	
C3	0.0682 (3)	0.08840 (16)	0.64431 (13)	0.0270 (4)	
H3	−0.0911	0.0316	0.6786	0.032*	
C4	0.1022 (3)	0.06000 (16)	0.53238 (13)	0.0261 (4)	
C5	0.3347 (4)	0.14802 (18)	0.48559 (14)	0.0328 (4)	
H5	0.3646	0.1318	0.4098	0.039*	
C6	0.5243 (4)	0.25895 (18)	0.54660 (14)	0.0329 (4)	
H6	0.6790	0.3178	0.5114	0.039*	
C7	0.4931 (3)	0.28575 (17)	0.65775 (14)	0.0284 (4)	
C8	0.8843 (16)	0.5024 (3)	0.6758 (5)	0.0414 (13)	0.779 (18)
H8A	0.7710	0.5531	0.6291	0.050*	0.779 (18)
H8B	1.0144	0.4613	0.6282	0.050*	0.779 (18)
C8′	0.791 (3)	0.4999 (12)	0.7085 (11)	0.023 (3)	0.221 (18)
H8′1	0.6326	0.5513	0.7158	0.028*	0.221 (18)
H8′2	0.8268	0.4949	0.6291	0.028*	0.221 (18)
C9	1.0656 (4)	0.60586 (18)	0.76161 (15)	0.0336 (4)	
C10	1.1686 (4)	0.56526 (18)	0.86005 (15)	0.0373 (5)	
H10	1.1067	0.4695	0.8787	0.045*	
C11	1.3596 (4)	0.66246 (19)	0.93095 (14)	0.0368 (4)	
H11	1.4302	0.6334	0.9978	0.044*	
C12	1.4480 (4)	0.80198 (18)	0.90472 (15)	0.0370 (4)	
H12	1.5781	0.8693	0.9538	0.044*	
C13	1.3474 (4)	0.84355 (18)	0.80734 (15)	0.0357 (4)	
H13	1.4098	0.9394	0.7889	0.043*	
C14	1.1558 (4)	0.74595 (18)	0.73633 (14)	0.0339 (4)	
H14	1.0855	0.7754	0.6696	0.041*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0359 (7)	0.0396 (7)	0.0222 (6)	−0.0042 (5)	0.0004 (5)	−0.0061 (5)
N1	0.0347 (8)	0.0398 (9)	0.0262 (8)	−0.0081 (7)	0.0009 (6)	−0.0086 (6)

C1	0.0359 (9)	0.0416 (10)	0.0252 (9)	0.0021 (7)	0.0029 (7)	−0.0022 (7)
C2	0.0303 (8)	0.0285 (8)	0.0224 (8)	0.0056 (6)	−0.0033 (6)	−0.0015 (6)
C3	0.0277 (8)	0.0256 (8)	0.0253 (8)	0.0017 (6)	−0.0015 (6)	0.0008 (6)
C4	0.0270 (8)	0.0258 (8)	0.0244 (8)	0.0050 (7)	−0.0047 (7)	−0.0001 (6)
C5	0.0333 (9)	0.0375 (9)	0.0228 (8)	−0.0001 (7)	−0.0019 (7)	−0.0028 (7)
C6	0.0307 (9)	0.0339 (9)	0.0286 (9)	−0.0033 (7)	0.0007 (7)	0.0001 (7)
C7	0.0265 (8)	0.0285 (8)	0.0273 (9)	0.0025 (6)	−0.0043 (6)	−0.0023 (6)
C8	0.048 (2)	0.0322 (14)	0.037 (3)	−0.0026 (15)	−0.014 (2)	−0.0002 (13)
C8′	0.022 (4)	0.029 (4)	0.018 (4)	0.005 (3)	0.011 (3)	0.003 (3)
C9	0.0356 (9)	0.0290 (9)	0.0328 (9)	0.0032 (7)	−0.0054 (7)	−0.0041 (7)
C10	0.0454 (10)	0.0255 (8)	0.0366 (10)	0.0003 (7)	−0.0055 (8)	0.0017 (7)
C11	0.0429 (10)	0.0362 (10)	0.0272 (9)	0.0023 (8)	−0.0065 (8)	0.0008 (7)
C12	0.0346 (9)	0.0331 (9)	0.0367 (10)	−0.0027 (7)	−0.0055 (8)	−0.0056 (7)
C13	0.0351 (9)	0.0276 (9)	0.0405 (10)	−0.0009 (7)	0.0001 (8)	0.0033 (7)
C14	0.0368 (9)	0.0337 (9)	0.0298 (9)	0.0055 (7)	−0.0010 (7)	0.0033 (7)

Geometric parameters (Å, º)

O1—C2	1.374 (2)	C6—H6	0.9500
O1—C1	1.425 (2)	C8—C9	1.498 (3)
N1—C8′	1.100 (12)	C8—H8A	0.9900
N1—C7	1.394 (2)	C8—H8B	0.9900
N1—C8	1.438 (7)	C8′—C9	1.549 (11)
N1—H1	0.8800	C8′—H8′1	0.9900
C1—H1A	0.9800	C8′—H8′2	0.9900
C1—H1B	0.9800	C9—C14	1.382 (2)
C1—H1C	0.9800	C9—C10	1.389 (3)
C2—C3	1.378 (2)	C10—C11	1.379 (2)
C2—C7	1.408 (2)	C10—H10	0.9500
C3—C4	1.399 (2)	C11—C12	1.379 (2)
C3—H3	0.9500	C11—H11	0.9500
C4—C5	1.389 (2)	C12—C13	1.377 (3)
C4—C4ⁱ	1.491 (3)	C12—H12	0.9500
C5—C6	1.386 (2)	C13—C14	1.383 (2)
C5—H5	0.9500	C13—H13	0.9500
C6—C7	1.385 (2)	C14—H14	0.9500
C2—O1—C1	117.19 (12)	C9—C8—H8A	109.3
C8′—N1—C7	129.4 (6)	N1—C8—H8B	109.3
C7—N1—C8	119.6 (2)	C9—C8—H8B	109.3
C7—N1—H1	120.2	H8A—C8—H8B	108.0
C8—N1—H1	120.2	N1—C8′—C9	131.8 (9)
O1—C1—H1A	109.5	N1—C8′—H8′1	104.3
O1—C1—H1B	109.5	C9—C8′—H8′1	104.3
H1A—C1—H1B	109.5	N1—C8′—H8′2	104.3
O1—C1—H1C	109.5	C9—C8′—H8′2	104.3
H1A—C1—H1C	109.5	H8′1—C8′—H8′2	105.6
H1B—C1—H1C	109.5	C14—C9—C10	118.83 (15)

O1—C2—C3	124.56 (15)	C14—C9—C8	117.8 (2)
O1—C2—C7	114.59 (14)	C10—C9—C8	123.1 (2)
C3—C2—C7	120.84 (15)	C14—C9—C8′	124.5 (4)
C2—C3—C4	121.84 (15)	C10—C9—C8′	114.0 (5)
C2—C3—H3	119.1	C11—C10—C9	120.71 (16)
C4—C3—H3	119.1	C11—C10—H10	119.6
C5—C4—C3	116.84 (15)	C9—C10—H10	119.6
C5—C4—C4ⁱ	122.18 (18)	C10—C11—C12	119.89 (16)
C3—C4—C4ⁱ	120.98 (18)	C10—C11—H11	120.1
C6—C5—C4	121.75 (16)	C12—C11—H11	120.1
C6—C5—H5	119.1	C13—C12—C11	119.95 (16)
C4—C5—H5	119.1	C13—C12—H12	120.0
C7—C6—C5	121.41 (16)	C11—C12—H12	120.0
C7—C6—H6	119.3	C12—C13—C14	120.10 (16)
C5—C6—H6	119.3	C12—C13—H13	120.0
C6—C7—N1	124.04 (15)	C14—C13—H13	120.0
C6—C7—C2	117.31 (15)	C9—C14—C13	120.52 (16)
N1—C7—C2	118.54 (15)	C9—C14—H14	119.7
N1—C8—C9	111.4 (4)	C13—C14—H14	119.7
N1—C8—H8A	109.3		

C1—O1—C2—C3	−9.3 (2)	C3—C2—C7—N1	−177.38 (14)
C1—O1—C2—C7	171.68 (14)	C7—N1—C8—C9	177.7 (3)
O1—C2—C3—C4	−176.98 (14)	C7—N1—C8′—C9	−160.1 (9)
C7—C2—C3—C4	1.9 (2)	N1—C8—C9—C14	−149.9 (3)
C2—C3—C4—C5	−1.4 (2)	N1—C8—C9—C10	36.3 (6)
C2—C3—C4—C4ⁱ	178.26 (16)	N1—C8′—C9—C14	−175.1 (12)
C3—C4—C5—C6	−0.1 (3)	N1—C8′—C9—C10	−13.8 (19)
C4ⁱ—C4—C5—C6	−179.73 (17)	C14—C9—C10—C11	−0.6 (3)
C4—C5—C6—C7	1.0 (3)	C8—C9—C10—C11	173.1 (4)
C5—C6—C7—N1	175.71 (16)	C8′—C9—C10—C11	−163.1 (6)
C5—C6—C7—C2	−0.5 (3)	C9—C10—C11—C12	0.6 (3)
C8′—N1—C7—C6	43.5 (11)	C10—C11—C12—C13	−0.6 (3)
C8—N1—C7—C6	18.3 (4)	C11—C12—C13—C14	0.6 (3)
C8′—N1—C7—C2	−140.3 (11)	C10—C9—C14—C13	0.6 (3)
C8—N1—C7—C2	−165.6 (3)	C8—C9—C14—C13	−173.5 (4)
O1—C2—C7—C6	178.08 (14)	C8′—C9—C14—C13	161.1 (7)
C3—C2—C7—C6	−0.9 (2)	C12—C13—C14—C9	−0.6 (3)
O1—C2—C7—N1	1.6 (2)		

Symmetry code: (i) −x, −y, −z+1.

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C10—H10⋯O1ⁱⁱ	0.95	2.66	3.400 (2)	135
N1—H1⋯O1	0.88	2.33	2.6464 (19)	101

Symmetry code: (ii) x+1, y, z.