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Bis[4-(2-pyridylmethylamino)phenyl]-methane dihydrate

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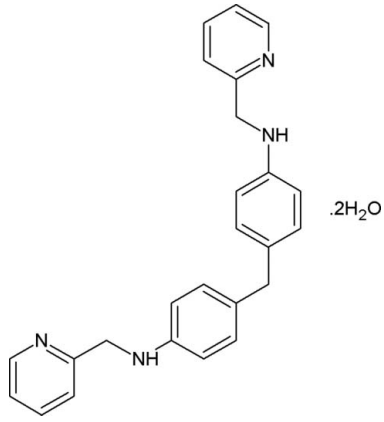
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.042; wR factor = 0.132; data-to-parameter ratio = 11.0.

The pyridyl and benzene rings in the title compound, $C_{25}H_{26}N_4\cdot 2H_2O$, show significant deviation from coplanarity due to a twist in the molecule, and this is reflected in the C—N(H)—C—C torsion angles of −82.8 (3) and 70.8 (3)°. The supramolecular architecture is effected by O—H···O quadrilaterals, one-dimensional N—H···O chains, two-dimensional networks of O—H···O, N—H···O and O—H···N interactions, face-to-face C—H···π and π–π interactions [with a ring-centroid separation of 3.904 (2) Å] and intra- and intermolecular van der Waals interactions. The overall structure of the title compound is maze-like, consisting of interleaved molecules stabilized by π–π interactions and by a hydrogen-bonding network involving water molecules, amino groups and pyridyl N atoms.

Related literature

For a detailed account of the synthetic methodology of the anhydrous form, *L*2, of the title compound, *L*2·2H$_2$O, see Keegan *et al.* (2001), and for its metal complexes, see: Yoshida & Ichikawa (1997); Hannon *et al.* (1997); He *et al.* (2000); Keegan *et al.* (2002). For related literature, see: Bernstein *et al.* (1995); Desiraju (1989); Desiraju & Steiner (1999); Etter (1990); Jodry & Lacour (2000); Kruger *et al.* (2001).



Experimental

Crystal data

$C_{25}H_{26}N_4\cdot 2H_2O$
M_r = 416.51
Monoclinic, $P2_1/n$
a = 13.8250 (13) Å
b = 6.5843 (6) Å
c = 25.291 (2) Å
β = 96.461 (2)°
V = 2287.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
0.45 × 0.41 × 0.27 mm

Data collection

Bruker SMART 1000 diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1998)
T_{min} = 0.966, T_{max} = 0.979
15266 measured reflections
3292 independent reflections
2173 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 23.3°

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.132
S = 1.02
3292 reflections
298 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

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

*Cg*2, *Cg*3 and *Cg*4 are the centroids of the C39/N40/C41–C44, C11–C16 and C31–C36 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
N17—H17···O1W^{i}	0.88 (3)	2.08 (3)	2.951 (3)	170 (3)
N37—H37···O2W	0.87 (3)	2.05 (3)	2.916 (3)	171 (3)
O1W—H1W1···O2W^{ii}	0.91 (3)	1.86 (3)	2.751 (3)	166 (3)
O1W—H1W2···N20^{iii}	0.91 (3)	1.92 (3)	2.829 (3)	174 (3)
O2W—H2W1···O1W	0.90 (3)	1.87 (3)	2.763 (3)	176 (3)
O2W—H2W2···N40^{iv}	1.02 (3)	1.79 (3)	2.804 (3)	177 (2)
C1—H1A···*Cg*4^{v}	0.97	3.03	3.777	135
C1—H1B···*Cg*2^{vi}	0.97	3.24	4.106	150
C23—H23···*Cg*3^{vii}	0.93	3.10	3.959	154

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SIR97* (Altomare *et al.* 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics:

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PLATON (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2420).

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supplementary materials

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Bis[4-(2-pyridylmethylamino)phenyl]methane dihydrate

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Comment

The self assembly of highly ordered molecular structures from small components is of much current interest with applications in areas such as fabrication of nanoscale devices and molecular machinery. An example of such self assembly is the formation of double- or triple-helical architectures (Jodry & Lacour, 2000) from multidentate organic ligands and their metal complexes (Yoshida & Ichikawa, 1997, Hannon *et al.*, 1997, He *et al.*, 2000, Keegan *et al.*, 2002). The design of these structures usually involves suitably compartmentalized ligands, which are capable of forming weak π···π interactions between their aromatic rings (Kruger *et al.*, 2001). This paper reports the molecular and supramolecular architecture of the title compound which displays several of these and other interactions.

The molecular structure of the title compound, L2.2H_2O, is shown in Fig. 1 and selected geometric parameters are given in Table 1. The N17—C18 & N37—C38 distances are consistent with C—N single bonds whereas the C14—N17 & C34—N37 bond distances are shorter than those expected for C—N single bonds. However, the C14—N17—C18 & C34—N37—C38 bond angles are considerably larger than expected for sp^3 hybridized nitrogen atoms, which may suggest a possible interaction between the lone pair on nitrogen and the aromatic cloud of the attached phenyl ring, thereby suggesting considerable sp^2 character for these nitrogen atoms. This is further confirmed by the sum of the bond angles around the two aliphatic nitrogen atoms, N17 & N37 [359.5 (2)].

Any two adjacent pairs of rings are non-coplanar as confirmed by their interplanar angles, cg1 & cg3, 80.55 (8)°, cg3 & cg4, 78.54 (7)° and cg2 & cg4, 83.44 (9)°. The two pyridyl rings are also relatively non-coplanar as shown by their interplanar angle (cg1 & cg2, 80.6 (1)°). The interplanar angles between cg1 & cg4 is 3.2 (1)° and cg2 & cg3 is 31.7 (8)°. The pyridyl and phenyl rings (cg1 & cg3 and cg2 & cg4) show significant deviation from coplanarity due to a twist in the molecule as reflected in the torsion angles, C14—N17—C18—C19 and C34—N37—C38—C39. cg1, cg2, cg3 and cg4 refer to C19/N20/C21/C22/C23/C24, C39/N40/C41/C42/C43/C44, C11—C16 and C31—C36 rings, respectively.

The crystal structure of L2.2H_2O is stabilized by the interplay of O—H···O, N—H···O, O—H···N, C—H···π (Table 2), π···π and van der Waals interactions. The hydrogen bond distances are similar to those reported in the literature (Desiraju & Steiner, 1999; Desiraju, 1989). The N—H···O interactions generate a motif of graph set $R^2_2(7)$ (Bernstein *et al.*, 1995 & Etter, 1990) and form infinite one-dimensional chains along [010] (Fig. 2). Directed 4-membered cooperative O—H···O quadrilaterals of graph set $R^2_2(8)$ contribute to the supramolecular aggregation of the title compound. The N—H···O, O—H···N and O—H···O interactions together generate an infinite two-dimensional network along the base vectors [010] & [101] and through the plane (10–1). There are three significant intermolecular C—H···π interactions (Table 2) in the title compound. The C—H···π interactions in the title compound can best be classified as face-to-face interactions in contrast to the presence of edge-to-face C—H···π and N—H···π interactions in L2 (Keegan *et al.*, 2001). There is a significant face-to-face π···π interaction between cg1 and cg4 ($-x$, $2-y$, $-z$) at 3.904 Å. Within and between asymmetric units, there are two significant vdW interactions, O1W & O2W, 2.763Å and O2W & O1W ($0.5-x$, $-1/2+y$, $0.5-z$), 2.751 Å, respectively.

The overall structure of L2.2H_2O is maze-like consisting of interleaved molecules stabilized by π···π interactions and by a H-bonding network involving water molecules, amino groups and pyridyl nitrogen atoms.

Experimental

The anhydrous analogue of the title compound, L2, was prepared by reported methods (Keegan *et al.*, 2001). An attempted synthesis of a Pt(II) complex of L2 resulted in the title compound, L2.2H_2O, whose details are given below. Potassium tetrachloroplatinate(II) (0.5 g, 1.2 mmol) dissolved in 6 ml of deionized water by gentle heating was added dropwise to 10 ml solution of L2 (0.23 g, 0.6 mmol) in THF dropwise under a N_2 atmosphere. The orange-red suspension formed was stirred under a N_2 atmosphere and then filtered. The filtrate was allowed to evaporate slowly at room temperature to yield yellow crystals of the title compound.

Refinement

The methylene and phenyl H atoms were included in calculated positions with C—H distances at 0.97 & 0.93 Å, respectively, and refined by a riding model. The hydrogen atoms of two water molecules were located from the difference map and refined. The thermal parameters of all H-atoms were obtained as 1.2U_{eq} of the respective carrier atoms.

Figures

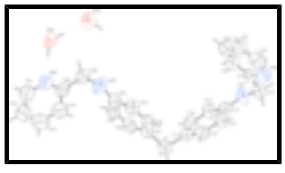

Fig. 1. The asymmetric unit of L2.2H_2O, with atom labels and 50% probability displacement ellipsoids for non-H atoms. H-atoms are depicted as spheres of arbitrary radius.

Fig. 2. View along *y*-axis showing O—H···O quadrilaterals and N—H···O networks. Hydrogen bonds are shown as dashed lines.

Bis[4-(2-pyridylmethylamino)phenyl]methane dihydrate

Crystal data

$C_{25}H_{26}N_4·2H_2O$	F_{000} = 888
M_r = 416.51	D_x = 1.209 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 3590 reflections
a = 13.8250 (13) Å	θ = 1.6–23.3°
b = 6.5843 (6) Å	μ = 0.08 mm^{-1}
c = 25.291 (2) Å	T = 293 (2) K
β = 96.461 (2)°	Prism, yellow
V = 2287.6 (4) Å^3	0.45 × 0.41 × 0.27 mm

$Z = 4$

Data collection

Bruker SMART 1000 diffractometer	3292 independent reflections
Radiation source: fine-focus sealed tube	2173 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.030$
$T = 293(2)$ K	$\theta_{max} = 23.3°$
ω scans	$\theta_{min} = 1.6°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1998)	$h = -15 \rightarrow 15$
$T_{min} = 0.966$, $T_{max} = 0.979$	$k = -7 \rightarrow 7$
15266 measured reflections	$l = -29 \rightarrow 29$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.132$	$w = 1/[\sigma^2(F_o^2) + (0.0471P)^2 + 0.9837P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
3292 reflections	$\Delta\rho_{max} = 0.38$ e Å^{-3}
298 parameters	$\Delta\rho_{min} = -0.12$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. Elemental analysis for $C_{25}H_{28}N_4O_2$ (%) calculated (found) C 72.1 (72.6), H 6.7 (6.6), N 13.5 (13.7). Mass spectrometry (HL^{2+}) 381 ^{1}H NMR ($CDCl_3$, p.p.m.): 8.60 (s, 2H), 7.70 (td, 2H), 7.39 (d, 2H), 7.24 (t, 2H), 7.00 (d, 4H), 6.61 (d, 4H), 4.49 (s, 4H), 3.77 (s, 2H)

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > 2$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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}

C1	0.87779 (18)	0.5628 (5)	0.42784 (10)	0.0827 (8)
H1A	0.9162	0.4405	0.4254	0.099*
H1B	0.9076	0.6702	0.4090	0.099*
C11	0.77551 (18)	0.5258 (4)	0.40167 (10)	0.0677 (7)
C12	0.7160 (2)	0.6854 (4)	0.38258 (10)	0.0748 (7)
H12	0.7415	0.8162	0.3838	0.090*
C13	0.62098 (19)	0.6572 (4)	0.36204 (10)	0.0718 (7)
H13	0.5838	0.7682	0.3493	0.086*
C14	0.57952 (18)	0.4653 (4)	0.36000 (9)	0.0635 (6)
C15	0.63801 (19)	0.3049 (4)	0.37885 (10)	0.0727 (7)
H15	0.6125	0.1742	0.3781	0.087*
C16	0.73429 (19)	0.3368 (4)	0.39883 (10)	0.0734 (7)
H16	0.7722	0.2258	0.4107	0.088*
N17	0.48350 (17)	0.4426 (4)	0.34013 (10)	0.0833 (7)
H17	0.449 (2)	0.547 (4)	0.3274 (11)	0.100*
C18	0.43134 (19)	0.2539 (4)	0.34144 (10)	0.0774 (8)
H18A	0.4692	0.1480	0.3268	0.093*
H18B	0.3704	0.2661	0.3186	0.093*
C19	0.40925 (17)	0.1884 (5)	0.39610 (10)	0.0714 (7)
N20	0.38042 (16)	−0.0038 (4)	0.39905 (9)	0.0821 (7)
C21	0.3573 (2)	−0.0687 (6)	0.44635 (15)	0.1067 (11)
H21	0.3371	−0.2026	0.4492	0.128*
C22	0.3620 (2)	0.0530 (9)	0.49074 (14)	0.1204 (14)
H22	0.3457	0.0021	0.5229	0.144*
C23	0.3909 (3)	0.2485 (8)	0.48680 (15)	0.1195 (13)
H23	0.3941	0.3344	0.5161	0.143*
C24	0.4152 (2)	0.3174 (5)	0.43919 (13)	0.0948 (9)
H24	0.4358	0.4508	0.4359	0.114*
C31	0.87835 (16)	0.6217 (4)	0.48559 (10)	0.0678 (7)
C32	0.85431 (18)	0.4802 (4)	0.52260 (11)	0.0748 (7)
H32	0.8413	0.3472	0.5117	0.090*
C33	0.84924 (19)	0.5308 (4)	0.57473 (11)	0.0726 (7)
H33	0.8331	0.4317	0.5984	0.087*
C34	0.86786 (18)	0.7282 (4)	0.59285 (10)	0.0661 (7)
C35	0.89461 (18)	0.8687 (4)	0.55661 (11)	0.0735 (7)
H35	0.9099	1.0008	0.5676	0.088*
C36	0.89886 (17)	0.8149 (4)	0.50424 (11)	0.0731 (7)
H36	0.9163	0.9131	0.4806	0.088*
N37	0.8591 (2)	0.7726 (4)	0.64518 (10)	0.0911 (8)
H37	0.842 (2)	0.674 (5)	0.6653 (12)	0.109*
C38	0.8702 (2)	0.9706 (4)	0.66792 (11)	0.0796 (8)
H38A	0.8344	0.9760	0.6988	0.096*
H38B	0.8407	1.0681	0.6422	0.096*
C39	0.9734 (2)	1.0348 (4)	0.68468 (10)	0.0721 (7)
N40	0.98277 (19)	1.2202 (4)	0.70503 (9)	0.0897 (7)
C41	1.0730 (3)	1.2855 (7)	0.72215 (14)	0.1294 (15)
H41	1.0806	1.4148	0.7368	0.155*
C42	1.1531 (3)	1.1699 (11)	0.71890 (16)	0.158 (2)
H42	1.2143	1.2200	0.7314	0.190*

C43	1.1442 (3)	0.9828 (10)	0.69761 (15)	0.1420 (18)
H43	1.1988	0.9030	0.6948	0.170*
C44	1.0528 (3)	0.9122 (6)	0.68019 (12)	0.1015 (10)
H44	1.0445	0.7830	0.6655	0.122*
O1W	0.65233 (15)	0.2278 (3)	0.69672 (8)	0.0831 (6)
H1W1	0.651 (2)	0.140 (4)	0.7242 (11)	0.100*
H1W2	0.646 (2)	0.153 (4)	0.6664 (11)	0.100*
O2W	0.81936 (17)	0.4559 (3)	0.72040 (7)	0.0841 (6)
H2W1	0.764 (2)	0.386 (4)	0.7116 (11)	0.101*
H2W2	0.879 (2)	0.370 (5)	0.7162 (11)	0.101*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0612 (17)	0.108 (2)	0.0804 (18)	−0.0081 (15)	0.0146 (13)	−0.0091 (16)
C11	0.0611 (15)	0.0806 (19)	0.0626 (15)	−0.0043 (14)	0.0131 (12)	−0.0071 (14)
C12	0.0813 (19)	0.0669 (17)	0.0761 (17)	−0.0181 (15)	0.0092 (14)	−0.0013 (14)
C13	0.0803 (19)	0.0594 (16)	0.0734 (17)	−0.0030 (14)	−0.0016 (14)	0.0038 (13)
C14	0.0663 (16)	0.0631 (16)	0.0595 (14)	−0.0014 (13)	0.0007 (12)	−0.0008 (12)
C15	0.0754 (18)	0.0564 (16)	0.0839 (18)	−0.0034 (14)	−0.0015 (14)	−0.0021 (14)
C16	0.0690 (17)	0.0724 (18)	0.0774 (17)	0.0098 (14)	0.0015 (13)	−0.0031 (14)
N17	0.0748 (16)	0.0674 (15)	0.1012 (18)	−0.0049 (12)	−0.0184 (13)	0.0072 (13)
C18	0.0681 (17)	0.0843 (19)	0.0767 (18)	−0.0089 (14)	−0.0052 (13)	0.0045 (15)
C19	0.0516 (14)	0.089 (2)	0.0717 (17)	−0.0012 (14)	−0.0025 (12)	−0.0064 (16)
N20	0.0722 (15)	0.0931 (18)	0.0804 (16)	−0.0068 (13)	0.0053 (12)	0.0068 (14)
C21	0.087 (2)	0.125 (3)	0.108 (3)	−0.011 (2)	0.012 (2)	0.025 (2)
C22	0.082 (2)	0.203 (5)	0.078 (2)	−0.010 (3)	0.0149 (18)	0.009 (3)
C23	0.084 (2)	0.187 (4)	0.088 (3)	−0.010 (3)	0.0137 (19)	−0.033 (3)
C24	0.084 (2)	0.117 (3)	0.083 (2)	−0.0076 (18)	0.0075 (16)	−0.024 (2)
C31	0.0490 (14)	0.0813 (19)	0.0725 (17)	−0.0074 (13)	0.0041 (12)	−0.0014 (15)
C32	0.0761 (18)	0.0635 (16)	0.0830 (19)	−0.0072 (14)	0.0013 (14)	−0.0039 (15)
C33	0.0828 (18)	0.0609 (16)	0.0734 (18)	−0.0088 (14)	0.0054 (14)	0.0060 (14)
C34	0.0690 (16)	0.0594 (15)	0.0692 (17)	−0.0057 (13)	0.0044 (12)	0.0047 (13)
C35	0.0797 (18)	0.0607 (16)	0.0790 (18)	−0.0120 (13)	0.0043 (14)	0.0031 (14)
C36	0.0627 (16)	0.0785 (19)	0.0773 (18)	−0.0138 (14)	0.0040 (13)	0.0133 (15)
N37	0.136 (2)	0.0649 (16)	0.0749 (17)	−0.0216 (15)	0.0226 (15)	−0.0046 (12)
C38	0.090 (2)	0.0688 (18)	0.0803 (18)	−0.0060 (15)	0.0122 (15)	−0.0059 (15)
C39	0.0812 (19)	0.0817 (19)	0.0538 (15)	0.0013 (16)	0.0098 (13)	0.0048 (14)
N40	0.0999 (19)	0.0971 (19)	0.0734 (15)	−0.0213 (15)	0.0159 (13)	−0.0147 (14)
C41	0.126 (3)	0.171 (4)	0.094 (3)	−0.056 (3)	0.026 (2)	−0.041 (3)
C42	0.101 (3)	0.290 (7)	0.083 (3)	−0.039 (4)	0.012 (2)	−0.046 (4)
C43	0.089 (3)	0.258 (6)	0.077 (2)	0.040 (3)	−0.001 (2)	−0.005 (3)
C44	0.096 (2)	0.129 (3)	0.078 (2)	0.027 (2)	0.0021 (18)	0.0007 (19)
O1W	0.0961 (14)	0.0633 (12)	0.0872 (14)	−0.0003 (10)	−0.0018 (12)	0.0047 (10)
O2W	0.1074 (16)	0.0667 (12)	0.0776 (12)	−0.0125 (11)	0.0077 (11)	−0.0065 (10)

Geometric parameters (Å, °)

C1—C31	1.510 (3)	C31—C36	1.375 (3)

C1—C11	1.512 (3)	C31—C32	1.387 (3)
C1—H1A	0.9700	C32—C33	1.369 (3)
C1—H1B	0.9700	C32—H32	0.9300
C11—C16	1.367 (3)	C33—C34	1.392 (3)
C11—C12	1.387 (4)	C33—H33	0.9300
C12—C13	1.369 (3)	C34—N37	1.374 (3)
C12—H12	0.9300	C34—C35	1.382 (3)
C13—C14	1.386 (3)	C35—C36	1.379 (3)
C13—H13	0.9300	C35—H35	0.9300
C14—N17	1.374 (3)	C36—H36	0.9300
C14—C15	1.382 (3)	N37—C38	1.426 (3)
C15—C16	1.386 (3)	N37—H37	0.87 (3)
C15—H15	0.9300	C38—C39	1.502 (4)
C16—H16	0.9300	C38—H38A	0.9700
N17—C18	1.439 (3)	C38—H38B	0.9700
N17—H17	0.88 (3)	C39—N40	1.326 (3)
C18—C19	1.512 (4)	C39—C44	1.377 (4)
C18—H18A	0.9700	N40—C41	1.344 (4)
C18—H18B	0.9700	C41—C42	1.354 (6)
C19—N20	1.332 (3)	C41—H41	0.9300
C19—C24	1.377 (4)	C42—C43	1.345 (6)
N20—C21	1.342 (4)	C42—H42	0.9300
C21—C22	1.375 (5)	C43—C44	1.372 (5)
C21—H21	0.9300	C43—H43	0.9300
C22—C23	1.355 (5)	C44—H44	0.9300
C22—H22	0.9300	O1W—H1W1	0.91 (3)
C23—C24	1.364 (5)	O1W—H1W2	0.91 (3)
C23—H23	0.9300	O2W—H2W1	0.90 (3)
C24—H24	0.9300	O2W—H2W2	1.02 (3)
C31—C1—C11	111.6 (2)	C23—C24—H24	120.2
C31—C1—H1A	109.3	C19—C24—H24	120.2
C11—C1—H1A	109.3	C36—C31—C32	116.5 (2)
C31—C1—H1B	109.3	C36—C31—C1	123.2 (3)
C11—C1—H1B	109.3	C32—C31—C1	120.3 (3)
H1A—C1—H1B	108.0	C33—C32—C31	121.9 (2)
C16—C11—C12	116.3 (2)	C33—C32—H32	119.1
C16—C11—C1	122.2 (3)	C31—C32—H32	119.1
C12—C11—C1	121.3 (3)	C32—C33—C34	121.1 (2)
C13—C12—C11	122.4 (2)	C32—C33—H33	119.5
C13—C12—H12	118.8	C34—C33—H33	119.5
C11—C12—H12	118.8	N37—C34—C35	123.7 (2)
C12—C13—C14	120.8 (2)	N37—C34—C33	118.9 (2)
C12—C13—H13	119.6	C35—C34—C33	117.4 (2)
C14—C13—H13	119.6	C36—C35—C34	120.6 (2)
N17—C14—C15	123.2 (2)	C36—C35—H35	119.7
N17—C14—C13	119.4 (2)	C34—C35—H35	119.7
C15—C14—C13	117.4 (2)	C31—C36—C35	122.5 (2)
C14—C15—C16	120.7 (2)	C31—C36—H36	118.7
C14—C15—H15	119.6	C35—C36—H36	118.7

C16—C15—H15	119.6	C34—N37—C38	124.5 (2)
C11—C16—C15	122.3 (2)	C34—N37—H37	117 (2)
C11—C16—H16	118.8	C38—N37—H37	118 (2)
C15—C16—H16	118.8	N37—C38—C39	115.2 (2)
C14—N17—C18	123.5 (2)	N37—C38—H38A	108.5
C14—N17—H17	120 (2)	C39—C38—H38A	108.5
C18—N17—H17	115.9 (19)	N37—C38—H38B	108.5
N17—C18—C19	114.9 (2)	C39—C38—H38B	108.5
N17—C18—H18A	108.6	H38A—C38—H38B	107.5
C19—C18—H18A	108.5	N40—C39—C44	121.8 (3)
N17—C18—H18B	108.6	N40—C39—C38	114.6 (3)
C19—C18—H18B	108.5	C44—C39—C38	123.5 (3)
H18A—C18—H18B	107.5	C39—N40—C41	117.8 (3)
N20—C19—C24	122.3 (3)	N40—C41—C42	122.5 (4)
N20—C19—C18	114.5 (2)	N40—C41—H41	118.8
C24—C19—C18	123.2 (3)	C42—C41—H41	118.8
C19—N20—C21	117.1 (3)	C43—C42—C41	120.0 (4)
N20—C21—C22	123.2 (4)	C43—C42—H42	120.0
N20—C21—H21	118.4	C41—C42—H42	120.0
C22—C21—H21	118.4	C42—C43—C44	118.6 (4)
C23—C22—C21	118.8 (4)	C42—C43—H43	120.7
C23—C22—H22	120.6	C44—C43—H43	120.7
C21—C22—H22	120.6	C43—C44—C39	119.2 (4)
C22—C23—C24	119.0 (4)	C43—C44—H44	120.4
C22—C23—H23	120.5	C39—C44—H44	120.4
C24—C23—H23	120.5	H1W1—O1W—H1W2	107 (3)
C23—C24—C19	119.7 (3)	H2W1—O2W—H2W2	112 (3)
C31—C1—C11—C16	−93.3 (3)	C11—C1—C31—C36	−108.3 (3)
C31—C1—C11—C12	82.0 (3)	C11—C1—C31—C32	69.5 (3)
C16—C11—C12—C13	0.1 (4)	C36—C31—C32—C33	1.3 (4)
C1—C11—C12—C13	−175.4 (2)	C1—C31—C32—C33	−176.6 (2)
C11—C12—C13—C14	0.7 (4)	C31—C32—C33—C34	0.2 (4)
C12—C13—C14—N17	178.7 (2)	C32—C33—C34—N37	178.2 (2)
C12—C13—C14—C15	−0.7 (4)	C32—C33—C34—C35	−2.0 (4)
N17—C14—C15—C16	−179.5 (2)	N37—C34—C35—C36	−177.9 (3)
C13—C14—C15—C16	−0.1 (4)	C33—C34—C35—C36	2.3 (4)
C12—C11—C16—C15	−1.0 (4)	C32—C31—C36—C35	−1.0 (4)
C1—C11—C16—C15	174.5 (2)	C1—C31—C36—C35	176.9 (2)
C14—C15—C16—C11	1.0 (4)	C34—C35—C36—C31	−0.8 (4)
C15—C14—N17—C18	6.1 (4)	C35—C34—N37—C38	4.3 (4)
C13—C14—N17—C18	−173.3 (2)	C33—C34—N37—C38	−175.9 (3)
C14—N17—C18—C19	70.8 (3)	C34—N37—C38—C39	−82.8 (3)
N17—C18—C19—N20	−166.2 (2)	N37—C38—C39—N40	179.0 (2)
N17—C18—C19—C24	15.8 (4)	N37—C38—C39—C44	−1.7 (4)
C24—C19—N20—C21	−0.2 (4)	C44—C39—N40—C41	−1.0 (4)
C18—C19—N20—C21	−178.3 (2)	C38—C39—N40—C41	178.3 (3)
C19—N20—C21—C22	0.1 (5)	C39—N40—C41—C42	0.5 (5)
N20—C21—C22—C23	0.3 (6)	N40—C41—C42—C43	0.4 (7)
C21—C22—C23—C24	−0.7 (6)	C41—C42—C43—C44	−0.9 (7)

C22—C23—C24—C19	0.6 (5)	C42—C43—C44—C39	0.4 (6)
N20—C19—C24—C23	−0.1 (4)	N40—C39—C44—C43	0.5 (4)
C18—C19—C24—C23	177.8 (3)	C38—C39—C44—C43	−178.7 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N17—H17···O1W^{i}	0.88 (3)	2.08 (3)	2.951 (3)	170 (3)
N37—H37···O2W	0.87 (3)	2.05 (3)	2.916 (3)	171 (3)
O1W—H1W1···O2W^{ii}	0.91 (3)	1.86 (3)	2.751 (3)	166 (3)
O1W—H1W2···N20^{iii}	0.91 (3)	1.92 (3)	2.829 (3)	174 (3)
O2W—H2W1···O1W	0.90 (3)	1.87 (3)	2.763 (3)	176 (3)
O2W—H2W2···N40^{iv}	1.02 (3)	1.79 (3)	2.804 (3)	177 (2)
C1—H1A···Cg4^{v}	0.97	3.03	3.777	135
C1—H1B···Cg2^{vi}	0.97	3.24	4.106	150
C23—H23···Cg3^{vii}	0.93	3.10	3.959	154

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

Fig. 1

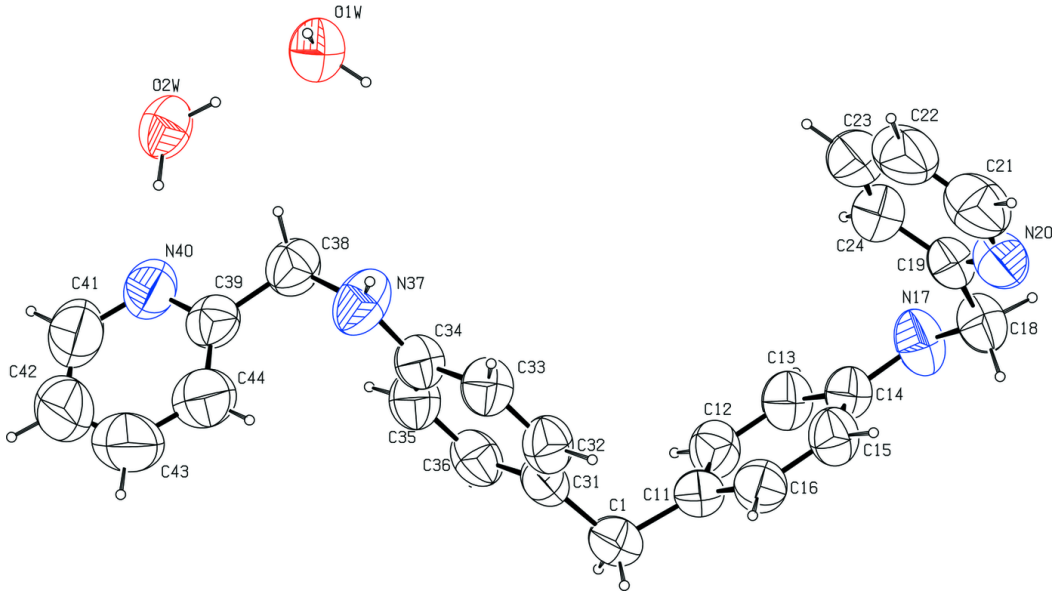
O1W
O2W
C23
C22
C21
N20
C24
C19
N17
C18
C13
C14
C12
C15
C16
C11
C1
C31
C32
C33
C34
C35
C36
N37
C38
C39
N40
C41
C42
C43
C44

Fig. 2