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5-[Phenyl(pyridin-4-yl)amino]penta-2,4-diyn-1-ol

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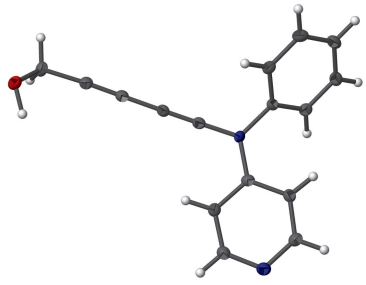
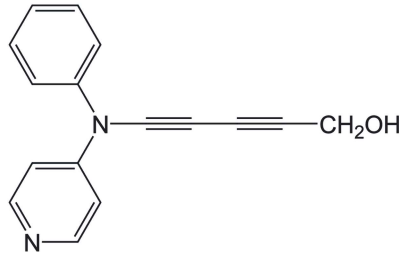
In the title diacetylene derivative, $C_{16}H_{12}N_2O$, the amino plane makes dihedral angles of 3.90 (4) and 60.53 (4)°, respectively, with the pyridyl and phenyl rings, indicating that an electron-deficient pyridyl ring makes better conjugation with a lone pair of the amino nitrogen atom. In the crystal, molecules form inversion dimers *via* pairs of hydrogen bonds between the hydroxy and pyridyl groups, with an O···N distance of 2.7765 (16) Å. The dimers stack along the *a* axis, but the title compound shows little solid-state polymerization reactivity.

3D view



Chemical scheme



Structure description

In diacetylene derivatives, a phenylpyridylamino group is connected to a terminal acetylene group. Solid-state polymerization of diacetylene derivatives (Wegner, 1969) affords polydiacetylenes whose one-dimensional π system has attracted attention from a materials science viewpoint. In order to improve their properties, several strategies for making novel polydiacetylenes have been examined, such as the introduction of hetero atoms directly to their π system. However, except for the case of iodine or nitrogen, these attempts have resulted in failure owing to limitations on molecular arrangement for solid-state polymerization of diacetylenes (Baughman, 1974), where the molecular arrangement is expressed in terms of stacking intervals and the inclination angle of the diacetylene unit to the stacking axis. Some heteroatom-substituted polydiacetylenes have been developed successfully (Galli *et al.*, 1988, 1989; Sarkar *et al.*, 1998; Okuno *et al.*, 2006; Tabata *et al.*, 2012, 2016; Tokutome *et al.*, 2012).

The title compound (Fig. 1) comprises two parts, *viz*. diacetylene and anilinopyridine units. The diacetylene unit curves slightly. The structure around the amino nitrogen atom is almost planar (r.m.s. deviation of C1/C6/C7/N1 plane = 0.0228 Å). The plane makes dihedral angles of 3.84 (7) and 60.64 (6)°, respectively, with the C1–C5/N2 pyridyl and C6–C11 phenyl rings, indicating that an electron-deficient pyridyl ring makes better conjugation with a lone pair of the amino nitrogen (Umezono & Okuno, 2015). The

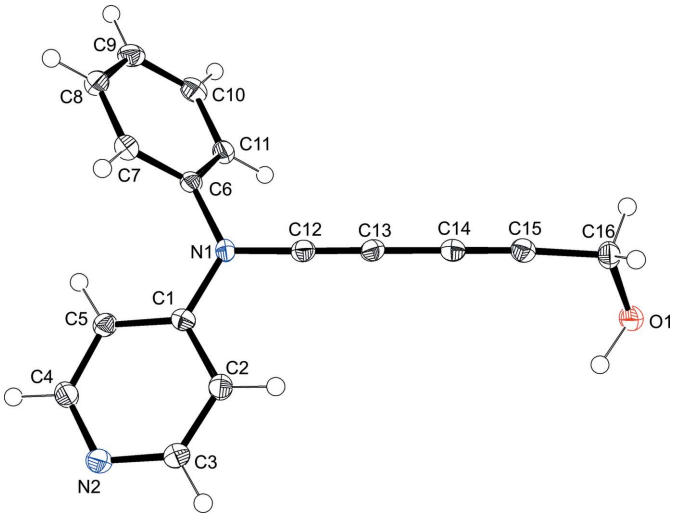


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radius.

structure of 5-(diphenylamino)-2,4-pentadiyne-1-ol (Tokutome *et al.*, 2012), where a pyridyl ring of the title compound is replaced by a phenyl ring, has an almost similar structure but here the phenyl rings make dihedral angles of 7.05 (9) and 82.5 (9)° with the amino plane. The large difference in the dihedral angle is thought to originate in intermolecular interactions.

In the crystal, molecules form centrosymmetric hydrogen-bonded dimers (Table 1, Fig. 2), where the O1···N2^{i} distance is 2.7765 (16) Å. These dimeric unit stacks along the *a* axis

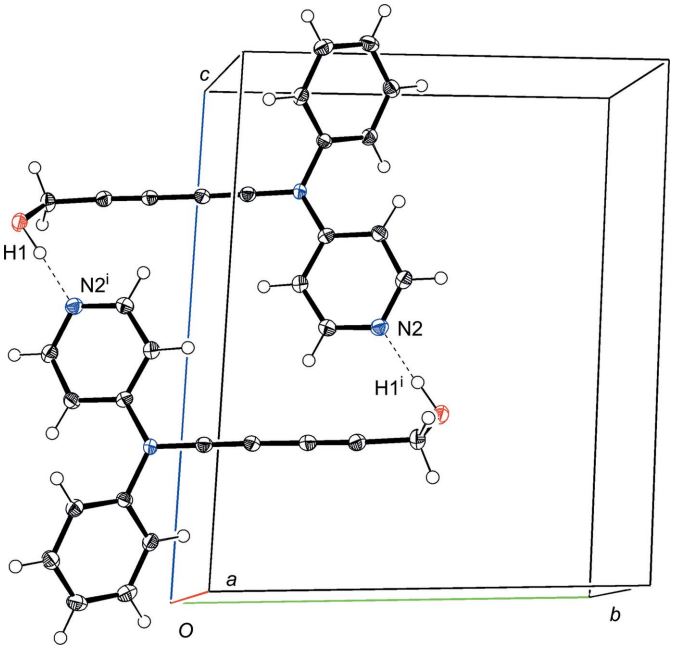


Figure 2
A view of the hydrogen-bonded dimer of the title compound [symmetry code: (i) -*x* + 1, -*y* + 1, -*z*].

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N2^{i}	1.01 (2)	1.77 (2)	2.7765 (16)	177 (2)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{12}N_2O$
M_r	248.28
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	93
a, *b*, *c* (Å)	4.0205 (11), 11.316 (3), 13.781 (4)
α, β, γ (°)	85.067 (9), 88.517 (9), 84.335 (6)
V (Å^3)	621.5 (3)
Z	2
Radiation type	Mo *K*α
μ (mm^{-1})	0.09
Crystal size (mm)	0.10 × 0.06 × 0.04
Data collection	
Diffractometer	Rigaku Saturn724+
Absorption correction	Numerical (*NUMABS*; Rigaku, 1999)
T_{min}, T_{max}	0.995, 0.997
No. of measured, independent and observed [$F^2 > 2.0\sigma(F^2)$] reflections	4294, 2156, 1773
R_{int}	0.019
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.038, 0.104, 1.06
No. of reflections	2156
No. of parameters	176
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.17

Computer programs: *CrystalClear* (Rigaku, 2008), *SIR92* (Altomare *et al.*, 1994), *SHELXL2013* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012) and *CrystalStructure* (Rigaku, 2014).

where the repeating intervals and inclination angle of the diacetylene unit to the stacking axis are 4.0205 (11) Å and *ca* 66°. These stacking parameters do not satisfy Baughman's limitation, and the title compound did not show any solid-state polymerization reactivity. In the case of 5-(diphenylamino)-2,4-pentadiyne-1-ol, polymeric hydrogen bonds are formed among the hydroxy groups and they play a crucial role in the molecular arrangement. However, in the case of the title compound, the hydroxy groups are used for making dimeric hydrogen bonds. This is the main reason for forming an inert structure regarding solid-state polymerization.

Synthesis and crystallization

Sodium hydride (0.28 g, 11.8 mmol) was added to a solution of *N*-phenylpyridin-4-amine (1.00 g, 5.88 mmol) in tetrahydrofuran. Trichloroethylene (1.06 ml, 11.8 mmol) was added to the solution and it was stirred for 24 h under an argon atmosphere. The solution was concentrated under reduced

pressure and extracted with chloroform. The chloroform solution was concentrated, and the residual oil was purified by column chromatography to give 0.55 g (36%) of (*E*)-*N*-(1,2-dichlorovinyl)-*N*-phenylpyridin-4-amine as a black oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.40 (*s*, 1H); 6.76 (*d*, *J* = 8.0 Hz, 2H); 7.33(m, 3H); 7.44 (*t*, *J* = 8.0 Hz, 2H); 8.38 (*d*, *J* = 8.0 Hz, 2H).

A solution of butyllithium in hexane (6.2 mmol) was added to a solution of (*E*)-*N*-(1,2-dichlorovinyl)-*N*-phenylpyridin-4-amine (0.55 g, 2.1 mmol) at 193 K, and the solution was stirred for 2 h. The reaction was quenched at 253 K, and the solution was concentrated by a rotary evaporator. The residue was extracted with chloroform, and the organic layer was washed with water and then brine. Removal of the solvent gave 0.35 g (86%) of *N*-ethynyl-*N*-phenylpyridin-4-amine as a black oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.97 (*s*, 1H); 7.02 (*dd*, *J* = 4.8,1.6 Hz, 2H); 7.30–7.43(*m*, 3H); 7.47(*t*, *J* = 8.0 Hz, 2H); 8.39 (*dd*, *J* = 4.8, 1.6 Hz, 2H).

Cu-TMEDA catalyst prepared from CuI (0.021 g, 0.21 mmol) and TMEDA (0.063 ml, 0.42 mmol) was added to a solution of *N*-ethynyl-*N*-phenylpyridin-4-amine (0.81 g, 4.2 mmol) and 2-propyn-1-ol (1.2 ml, 20.9 mmol) in acetone (30 ml). The solution was stirred for 1 d and concentrated under reduced pressure. The residue was extracted with dichloromethane, and the organic layer was washed with 5% ammonium hydroxide and water. It was concentrated by a rotary evaporator, and the residue was purified by column chromatography to afford the title compound (0.28 g, 28%) as a brown solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.07 (*s*, 1H); 4.41 (*s*, 2H); 7.03 (*dd*, *J* = 4.8,1.6 Hz, 2H); 7.38 (*m*, 3H); 7.49 (*t*, *J* = 8.1 Hz, 2H); 8.44 (*dd*, *J* = 4.8, 1.6 Hz, 2H).

Single colourless crystals of sufficient quality for X-ray crystallographic analysis were prepared by recrystallization from a dichloromethane solution.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2018). **3**, x180454 [https://doi.org/10.1107/S2414314618004546]

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Crystal data

$C_{16}H_{12}N_2O$
M_r = 248.28
Triclinic, $P\overline{1}$
a = 4.0205 (11) Å
b = 11.316 (3) Å
c = 13.781 (4) Å
α = 85.067 (9)°
β = 88.517 (9)°
γ = 84.335 (6)°
V = 621.5 (3) Å^3
Z = 2
$F(000)$ = 260.00
D_x = 1.327 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 2024 reflections
θ = 2.4–31.2°
μ = 0.09 mm^{-1}
T = 93 K
Block, colorless
0.10 × 0.06 × 0.04 mm

Data collection

Rigaku Saturn724+ diffractometer
Detector resolution: 7.111 pixels mm^{-1}
ω scans
Absorption correction: numerical (NUMABS; Rigaku, 1999)
T_{min} = 0.995, T_{max} = 0.997
4294 measured reflections
2156 independent reflections
1773 reflections with $F^2 > 2.0\sigma(F^2)$
R_{int} = 0.019
θ_{max} = 25.0°, θ_{min} = 3.4°
h = −4→4
k = −13→13
l = −14→16

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.104
S = 1.06
2156 reflections
176 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2 + 0.0857P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Special details

Refinement. Refinement was performed using all reflections. The weighted R-factor (wR) and goodness of fit (S) are based on F^2. R-factor (gt) are based on F. The threshold expression of $F^2 > 2.0$ sigma(F^2) is used only for calculating R-factor (gt).
The C-bound H atoms were placed at ideal positions and were refined as riding on their parent C atoms. U_{iso}(H) values of the H atoms were set at $1.2U_{eq}$(parent atom). The O-bound H atom was obtained from a difference Fourier map and was refined isotropically without any restrictions.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.1044 (3)	0.04808 (8)	0.19758 (7)	0.0212 (3)
N1	0.3981 (3)	0.65837 (10)	0.24201 (8)	0.0164 (3)
N2	0.6840 (3)	0.82820 (10)	−0.02810 (8)	0.0199 (3)
C1	0.4983 (3)	0.71528 (12)	0.15208 (10)	0.0158 (3)
C2	0.4407 (4)	0.66720 (13)	0.06486 (10)	0.0194 (3)
C3	0.5357 (4)	0.72613 (13)	−0.02170 (11)	0.0205 (3)
C4	0.7401 (4)	0.87159 (13)	0.05677 (11)	0.0200 (3)
C5	0.6560 (4)	0.81987 (12)	0.14734 (10)	0.0179 (3)
C6	0.4511 (4)	0.70306 (12)	0.33530 (10)	0.0162 (3)
C7	0.3302 (4)	0.81874 (13)	0.35304 (10)	0.0188 (3)
C8	0.3819 (4)	0.85885 (13)	0.44329 (10)	0.0209 (3)
C9	0.5475 (4)	0.78394 (13)	0.51558 (11)	0.0226 (3)
C10	0.6625 (4)	0.66780 (13)	0.49748 (11)	0.0227 (4)
C11	0.6158 (4)	0.62704 (13)	0.40721 (10)	0.0188 (3)
C12	0.2817 (4)	0.55039 (12)	0.24330 (10)	0.0174 (3)
C13	0.1761 (4)	0.45476 (12)	0.24541 (10)	0.0180 (3)
C14	0.0675 (4)	0.34287 (12)	0.24871 (10)	0.0175 (3)
C15	−0.0230 (4)	0.24424 (12)	0.25177 (10)	0.0185 (3)
C16	−0.1235 (4)	0.12318 (12)	0.25070 (11)	0.0207 (3)
H1	0.172 (6)	0.095 (2)	0.1360 (17)	0.064 (7)*
H2	0.33779	0.59513	0.06506	0.0233*
H3	0.49387	0.69255	−0.08052	0.0246*
H4	0.84561	0.94331	0.05448	0.0240*
H5	0.70444	0.85476	0.20501	0.0214*
H7	0.2138	0.86961	0.30405	0.0225*
H8	0.30342	0.93816	0.45577	0.0251*
H9	0.58203	0.81201	0.5772	0.0271*
H10	0.77347	0.61624	0.54709	0.0272*
H11	0.69548	0.54789	0.39459	0.0226*
H16A	−0.14132	0.08758	0.31859	0.0249*
H16B	−0.34725	0.12755	0.22151	0.0249*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0271 (6)	0.0156 (5)	0.0208 (6)	−0.0027 (4)	0.0032 (4)	−0.0008 (4)
N1	0.0215 (7)	0.0131 (6)	0.0153 (6)	−0.0046 (5)	0.0002 (5)	−0.0007 (5)

N2	0.0218 (7)	0.0204 (7)	0.0172 (7)	−0.0017 (5)	0.0009 (5)	−0.0007 (5)
C1	0.0141 (7)	0.0170 (7)	0.0154 (7)	0.0009 (5)	0.0005 (6)	0.0003 (5)
C2	0.0216 (8)	0.0174 (7)	0.0196 (8)	−0.0032 (6)	−0.0011 (6)	−0.0015 (6)
C3	0.0229 (8)	0.0214 (8)	0.0176 (8)	−0.0031 (6)	−0.0008 (6)	−0.0029 (6)
C4	0.0210 (8)	0.0179 (7)	0.0208 (8)	−0.0026 (6)	0.0013 (6)	0.0005 (6)
C5	0.0186 (8)	0.0185 (7)	0.0170 (7)	−0.0026 (6)	−0.0010 (6)	−0.0029 (6)
C6	0.0170 (7)	0.0185 (7)	0.0139 (7)	−0.0055 (6)	0.0006 (6)	−0.0019 (6)
C7	0.0182 (8)	0.0190 (7)	0.0190 (8)	−0.0028 (6)	−0.0004 (6)	0.0005 (6)
C8	0.0221 (8)	0.0201 (8)	0.0212 (8)	−0.0034 (6)	0.0015 (6)	−0.0043 (6)
C9	0.0233 (8)	0.0299 (9)	0.0160 (8)	−0.0081 (7)	0.0010 (6)	−0.0047 (6)
C10	0.0228 (8)	0.0265 (8)	0.0182 (8)	−0.0039 (6)	−0.0007 (6)	0.0035 (6)
C11	0.0207 (8)	0.0160 (7)	0.0195 (8)	−0.0022 (6)	0.0023 (6)	0.0001 (6)
C12	0.0197 (8)	0.0175 (8)	0.0145 (7)	0.0000 (6)	0.0002 (6)	−0.0015 (6)
C13	0.0221 (8)	0.0175 (8)	0.0144 (7)	−0.0016 (6)	0.0003 (6)	−0.0026 (6)
C14	0.0187 (8)	0.0191 (8)	0.0146 (7)	−0.0015 (6)	0.0001 (6)	−0.0011 (6)
C15	0.0190 (8)	0.0195 (8)	0.0168 (7)	−0.0022 (6)	0.0000 (6)	−0.0006 (6)
C16	0.0215 (8)	0.0166 (7)	0.0247 (8)	−0.0047 (6)	0.0018 (6)	−0.0026 (6)

Geometric parameters (Å, º)

O1—C16	1.4199 (18)	C12—C13	1.199 (2)
N1—C1	1.4146 (18)	C13—C14	1.376 (2)
N1—C6	1.4495 (19)	C14—C15	1.205 (2)
N1—C12	1.3496 (19)	C15—C16	1.467 (2)
N2—C3	1.346 (2)	O1—H1	1.01 (2)
N2—C4	1.339 (2)	C2—H2	0.950
C1—C2	1.395 (2)	C3—H3	0.950
C1—C5	1.393 (2)	C4—H4	0.950
C2—C3	1.380 (2)	C5—H5	0.950
C4—C5	1.381 (2)	C7—H7	0.950
C6—C7	1.390 (2)	C8—H8	0.950
C6—C11	1.3912 (19)	C9—H9	0.950
C7—C8	1.387 (2)	C10—H10	0.950
C8—C9	1.390 (2)	C11—H11	0.950
C9—C10	1.391 (2)	C16—H16A	0.990
C10—C11	1.387 (2)	C16—H16B	0.990
O1···C14	3.4566 (19)	C14···H9^{xiii}	3.1269
N2···C1	2.8111 (18)	C14···H10^{xiii}	2.9861
C1···C7	3.131 (2)	C14···H11^{v}	3.4092
C1···C13	3.470 (2)	C14···H16B^{iv}	3.2549
C2···C4	2.704 (2)	C15···H3^{x}	3.0559
C2···C12	2.780 (2)	C15···H3^{i}	3.2182
C2···C13	3.527 (2)	C15···H9^{xii}	3.2805
C3···C5	2.715 (2)	C15···H9^{xiii}	2.9730
C5···C6	2.941 (2)	C15···H10^{xiii}	3.5202
C5···C7	3.091 (2)	C15···H16B^{iv}	2.9371
C6···C9	2.770 (2)	C16···H1^{v}	3.33 (2)

C6···C13	3.455 (2)	C16···H3^{x}	3.2852
C7···C10	2.787 (2)	C16···H4^{ii}	3.5333
C7···C12	3.531 (2)	C16···H5^{ii}	3.2966
C8···C11	2.780 (2)	C16···H7^{iii}	3.0877
C11···C12	2.895 (2)	C16···H9^{xii}	3.0606
O1···N2^{i}	2.7765 (16)	C16···H9^{xiii}	3.4327
O1···C4^{ii}	3.360 (2)	C16···H16B^{iv}	3.1411
O1···C4^{i}	3.599 (2)	H1···N2^{i}	1.77 (2)
O1···C5^{ii}	3.4191 (19)	H1···C3^{i}	2.79 (2)
O1···C5^{iii}	3.3412 (18)	H1···C4^{ii}	3.47 (2)
O1···C7^{iii}	3.2907 (18)	H1···C4^{iii}	3.46 (2)
O1···C16^{iv}	3.412 (2)	H1···C4^{i}	2.67 (2)
N1···C5^{v}	3.5581 (19)	H1···C5^{iii}	3.50 (2)
N2···O1^{i}	2.7765 (16)	H1···C16^{iv}	3.33 (2)
N2···C2^{iv}	3.595 (2)	H1···H3^{x}	3.4742
N2···C3^{iv}	3.505 (2)	H1···H3^{i}	2.9075
N2···C15^{i}	3.468 (2)	H1···H4^{ii}	2.6024
N2···C16^{i}	3.529 (2)	H1···H4^{iii}	3.2841
C1···C4^{v}	3.590 (2)	H1···H4^{i}	2.7004
C1···C5^{v}	3.476 (2)	H1···H5^{ii}	3.5048
C2···N2^{v}	3.595 (2)	H1···H5^{iii}	3.3717
C2···C4^{v}	3.462 (2)	H1···H7^{iii}	3.2860
C3···N2^{v}	3.505 (2)	H1···H16B^{iv}	2.3651
C3···C14^{i}	3.586 (2)	H2···H2^{i}	3.0888
C4···O1^{vi}	3.360 (2)	H2···H3^{i}	3.2488
C4···O1^{i}	3.599 (2)	H3···N2^{v}	3.5445
C4···C1^{iv}	3.590 (2)	H3···C13^{i}	3.1243
C4···C2^{iv}	3.462 (2)	H3···C14^{x}	3.3519
C4···C4^{vii}	3.590 (2)	H3···C14^{i}	2.9072
C5···O1^{viii}	3.3412 (18)	H3···C15^{x}	3.0559
C5···O1^{vi}	3.4191 (19)	H3···C15^{i}	3.2182
C5···N1^{iv}	3.5581 (19)	H3···C16^{x}	3.2852
C5···C1^{iv}	3.476 (2)	H3···H1^{x}	3.4742
C7···O1^{viii}	3.2907 (18)	H3···H1^{i}	2.9075
C11···C12^{iv}	3.548 (2)	H3···H2^{i}	3.2488
C12···C11^{v}	3.548 (2)	H3···H16B^{x}	2.7220
C14···C3^{i}	3.586 (2)	H4···O1^{vi}	2.6700
C15···N2^{i}	3.468 (2)	H4···O1^{i}	3.4677
C16···O1^{v}	3.412 (2)	H4···N2^{vii}	3.1847
C16···N2^{i}	3.529 (2)	H4···N2^{ix}	3.3412
N1···H2	2.6234	H4···C4^{vii}	3.3081
N1···H5	2.6512	H4···C4^{ix}	3.0850
N1···H7	2.6388	H4···C16^{vi}	3.5333
N1···H11	2.6070	H4···H1^{viii}	3.2841
N2···H2	3.2545	H4···H1^{vi}	2.6024
N2···H5	3.2560	H4···H1^{i}	2.7004
C1···H3	3.2383	H4···H4^{vii}	3.2871
C1···H4	3.2303	H4···H4^{ix}	2.3084

C1···H7	2.9757	H4···H16B^{vi}	3.2650
C2···H5	3.2581	H5···O1^{viii}	3.0902
C3···H4	3.1322	H5···O1^{vi}	2.8336
C4···H3	3.1331	H5···N1^{iv}	3.4106
C5···H2	3.2563	H5···C1^{iv}	3.5062
C5···H7	2.8202	H5···C7^{iv}	3.2522
C6···H5	2.6323	H5···C16^{vi}	3.2966
C6···H8	3.2532	H5···H1^{viii}	3.3717
C6···H10	3.2581	H5···H1^{vi}	3.5048
C7···H5	2.5320	H5···H7^{iv}	2.5173
C7···H9	3.2680	H5···H16A^{vi}	3.2951
C7···H11	3.2760	H5···H16B^{vi}	3.1010
C8···H5	3.5015	H7···O1^{viii}	2.4028
C8···H10	3.2635	H7···C5^{v}	3.2741
C9···H7	3.2706	H7···C16^{viii}	3.0877
C9···H11	3.2665	H7···H1^{viii}	3.2860
C10···H8	3.2625	H7···H5^{v}	2.5173
C11···H7	3.2771	H7···H16A^{viii}	2.7455
C11···H9	3.2647	H8···C8^{xi}	3.1665
C12···H2	2.4740	H8···C9^{xi}	3.3170
C12···H11	2.6973	H8···H8^{xv}	2.9771
C13···H2	2.9264	H8···H8^{xi}	2.6026
C13···H11	3.2752	H8···H9^{xi}	2.9046
C14···H1	3.31 (2)	H8···H16A^{viii}	2.9425
C14···H16A	3.1522	H8···H16A^{vi}	3.3754
C14···H16B	3.1384	H8···H16A^{xii}	3.1552
C15···H1	2.48 (2)	H9···C14^{xiii}	3.1269
H1···H16A	2.7831	H9···C15^{xii}	3.2805
H1···H16B	2.3837	H9···C15^{xiii}	2.9730
H2···H3	2.3076	H9···C16^{xii}	3.0606
H4···H5	2.3072	H9···C16^{xiii}	3.4327
H5···H7	2.3700	H9···H8^{xi}	2.9046
H7···H8	2.3401	H9···H16A^{xii}	2.4989
H8···H9	2.3374	H9···H16A^{xiii}	3.0597
H9···H10	2.3404	H9···H16B^{xii}	3.0194
H10···H11	2.3398	H10···C11^{xiii}	3.3054
O1···H4^{ii}	2.6700	H10···C11^{xiv}	3.5289
O1···H4^{i}	3.4677	H10···C12^{xiii}	3.3258
O1···H5^{ii}	2.8336	H10···C13^{xiii}	2.9096
O1···H5^{iii}	3.0902	H10···C14^{xiii}	2.9861
O1···H7^{iii}	2.4028	H10···C15^{xiii}	3.5202
O1···H16A^{iv}	3.5826	H10···H10^{xiv}	3.3826
O1···H16B^{iv}	2.5017	H10···H11^{xiii}	2.8296
N1···H5^{v}	3.4106	H10···H11^{xiv}	2.7755
N2···H1^{i}	1.77 (2)	H11···C10^{xiii}	3.1938
N2···H3^{iv}	3.5445	H11···C11^{xiii}	3.5110
N2···H4^{vii}	3.1847	H11···C12^{iv}	3.1065
N2···H4^{ix}	3.3412	H11···C13^{iv}	2.9725

N2···H16B^{x}	3.0018	H11···C14^{iv}	3.4092
C1···H5^{v}	3.5062	H11···H10^{xiii}	2.8296
C3···H1^{i}	2.79 (2)	H11···H10^{xiv}	2.7755
C3···H16B^{x}	3.1581	H11···H11^{xiii}	3.4124
C4···H1^{viii}	3.46 (2)	H16A···O1^{v}	3.5826
C4···H1^{vi}	3.47 (2)	H16A···C7^{iii}	3.4255
C4···H1^{i}	2.67 (2)	H16A···C8^{iii}	3.5249
C4···H4^{vii}	3.3081	H16A···C8^{xii}	3.4832
C4···H4^{ix}	3.0850	H16A···C9^{xii}	3.1484
C5···H1^{viii}	3.50 (2)	H16A···H5^{ii}	3.2951
C5···H7^{iv}	3.2741	H16A···H7^{iii}	2.7455
C7···H5^{v}	3.2522	H16A···H8^{ii}	3.3754
C7···H16A^{viii}	3.4255	H16A···H8^{iii}	2.9425
C8···H8^{xi}	3.1665	H16A···H8^{xii}	3.1552
C8···H16A^{viii}	3.5249	H16A···H9^{xii}	2.4989
C8···H16A^{xii}	3.4832	H16A···H9^{xiii}	3.0597
C9···H8^{xi}	3.3170	H16A···H16B^{iv}	3.4852
C9···H16A^{xii}	3.1484	H16B···O1^{v}	2.5017
C10···H11^{xiii}	3.1938	H16B···N2^{x}	3.0018
C11···H10^{xiii}	3.3054	H16B···C3^{x}	3.1581
C11···H10^{xiv}	3.5289	H16B···C14^{v}	3.2549
C11···H11^{xiii}	3.5110	H16B···C15^{v}	2.9371
C12···H10^{xiii}	3.3258	H16B···C16^{v}	3.1411
C12···H11^{v}	3.1065	H16B···H1^{v}	2.3651
C13···H3^{i}	3.1243	H16B···H3^{x}	2.7220
C13···H10^{xiii}	2.9096	H16B···H4^{ii}	3.2650
C13···H11^{v}	2.9725	H16B···H5^{ii}	3.1010
C14···H3^{x}	3.3519	H16B···H9^{xii}	3.0194
C14···H3^{i}	2.9072	H16B···H16A^{v}	3.4852

C1—N1—C6	123.25 (12)	C16—O1—H1	108.4 (13)
C1—N1—C12	119.14 (12)	C1—C2—H2	120.577
C6—N1—C12	117.18 (11)	C3—C2—H2	120.570
C3—N2—C4	115.73 (12)	N2—C3—H3	117.923
N1—C1—C2	120.31 (13)	C2—C3—H3	117.912
N1—C1—C5	121.70 (13)	N2—C4—H4	117.566
C2—C1—C5	118.00 (13)	C5—C4—H4	117.556
C1—C2—C3	118.85 (14)	C1—C5—H5	120.814
N2—C3—C2	124.16 (14)	C4—C5—H5	120.826
N2—C4—C5	124.88 (14)	C6—C7—H7	120.459
C1—C5—C4	118.36 (13)	C8—C7—H7	120.463
N1—C6—C7	120.30 (12)	C7—C8—H8	119.721
N1—C6—C11	118.76 (12)	C9—C8—H8	119.737
C7—C6—C11	120.91 (13)	C8—C9—H9	120.083
C6—C7—C8	119.08 (13)	C10—C9—H9	120.079
C7—C8—C9	120.54 (14)	C9—C10—H10	119.904
C8—C9—C10	119.84 (14)	C11—C10—H10	119.914
C9—C10—C11	120.18 (13)	C6—C11—H11	120.284

C6—C11—C10	119.43 (13)	C10—C11—H11	120.285
N1—C12—C13	179.23 (15)	O1—C16—H16A	109.016
C12—C13—C14	177.71 (15)	O1—C16—H16B	109.017
C13—C14—C15	179.07 (15)	C15—C16—H16A	109.016
C14—C15—C16	177.00 (15)	C15—C16—H16B	109.011
O1—C16—C15	112.86 (12)	H16A—C16—H16B	107.794
C1—N1—C6—C7	55.63 (17)	C2—C1—C5—C4	1.36 (18)
C1—N1—C6—C11	−126.23 (13)	C5—C1—C2—C3	−1.25 (19)
C6—N1—C1—C2	179.77 (10)	C1—C2—C3—N2	0.3 (2)
C6—N1—C1—C5	−0.21 (18)	N2—C4—C5—C1	−0.6 (2)
C12—N1—C1—C2	7.53 (18)	N1—C6—C7—C8	179.43 (11)
C12—N1—C1—C5	−172.45 (11)	N1—C6—C11—C10	−178.76 (11)
C12—N1—C6—C7	−131.99 (12)	C7—C6—C11—C10	−0.6 (2)
C12—N1—C6—C11	46.15 (17)	C11—C6—C7—C8	1.3 (2)
C3—N2—C4—C5	−0.4 (2)	C6—C7—C8—C9	−1.0 (2)
C4—N2—C3—C2	0.5 (2)	C7—C8—C9—C10	0.0 (2)
N1—C1—C2—C3	178.77 (11)	C8—C9—C10—C11	0.7 (2)
N1—C1—C5—C4	−178.66 (10)	C9—C10—C11—C6	−0.4 (2)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) *x*−1, *y*−1, *z*; (iii) *x*, *y*−1, *z*; (iv) *x*+1, *y*, *z*; (v) *x*−1, *y*, *z*; (vi) *x*+1, *y*+1, *z*; (vii) −*x*+1, −*y*+2, −*z*; (viii) *x*, *y*+1, *z*; (ix) −*x*+2, −*y*+2, −*z*; (x) −*x*, −*y*+1, −*z*; (xi) −*x*+1, −*y*+2, −*z*+1; (xii) −*x*, −*y*+1, −*z*+1; (xiii) −*x*+1, −*y*+1, −*z*+1; (xiv) −*x*+2, −*y*+1, −*z*+1; (xv) −*x*, −*y*+2, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N2^{i}	1.01 (2)	1.77 (2)	2.7765 (16)	177 (2)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.