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7-[4-(5,7-Dimethyl-1,8-naphthyridin-2-yloxy)phenoxy]-2,4-dimethyl-1,8-naphthyridine methanol disolvate

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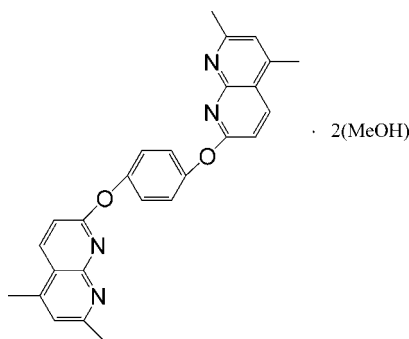
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.052; wR factor = 0.163; data-to-parameter ratio = 13.6.

The title compound, $\text{C}_{26}\text{H}_{22}\text{N}_4\text{O}_2 \cdot 2\text{CH}_3\text{OH}$, was synthesized and characterized by ^1H NMR spectroscopy and X-ray structure analysis. There is one half-molecule in the asymmetric unit with a centre of symmetry located at the centre of the benzene ring. The two bridged naphthyridine ring systems are in an antiparallel orientation. In the crystal structure, $\text{O}-\text{H} \cdots \text{N}$, $\text{C}-\text{H} \cdots \text{O}$ and $\text{C}-\text{H} \cdots \text{N}$ interactions define the packing.

Related literature

For related literature, see: Ferrarini *et al.* (2004); Goswami & Mukherjee (1997); Hooch *et al.* (1999); Jin, Liu & Chen (2007); Jin, Chen & Wang (2007); Nabanita *et al.* (2006); Nakatani *et al.* (2000); Nakataniz *et al.* (2001); Newkome *et al.* (1981); Stuk *et al.* (2003); Gavrilova & Bosnich (2004).



Experimental

Crystal data

$\text{C}_{26}\text{H}_{22}\text{N}_4\text{O}_2 \cdot 2\text{CH}_4\text{O}$
 $M_r = 486.56$
Triclinic, $P\bar{1}$
 $a = 7.009$ (3) Å
 $b = 9.244$ (3) Å

$c = 10.239$ (4) Å
 $\alpha = 78.679$ (6)°
 $\beta = 79.653$ (6)°
 $\gamma = 82.689$ (6)°
 $V = 637.0$ (4) Å³

$Z = 1$
Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹

$T = 298$ (2) K
 $0.27 \times 0.24 \times 0.19$ mm

Data collection

Bruker SMART APEX CCD
Diffractometer
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.977$, $T_{\max} = 0.984$

3379 measured reflections
2216 independent reflections
1236 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.163$
 $S = 1.03$
2216 reflections

163 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.27$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O2}-\text{H2} \cdots \text{N2}$	0.82	2.06	2.882 (3)	178
$\text{C6}-\text{H6} \cdots \text{O2}^i$	0.93	2.53	3.414 (4)	159
$\text{C10}-\text{H10A} \cdots \text{O2}^i$	0.96	2.54	3.436 (4)	156
$\text{C13}-\text{H13} \cdots \text{N2}^i$	0.93	2.61	3.450 (4)	151

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2143).

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7-[4-(5,7-Dimethyl-1,8-naphthyridin-2-yloxy)phenoxy]-2,4-dimethyl-1,8-naphthyridine methanol disolvate

S.-W. Jin, D.-Q. Wang and Y. Chen

Comment

Derivatives of 1,8-naphthyridine have been investigated over half a century because of their interesting complexation properties and medical uses. They can act as antimycobacterial and antimicrobial agents (Goswami *et al.*, 1997; Nakatani *et al.*, 2000; Ferrarini *et al.*, 2004; Stuk *et al.*, 2003) and as mono-nucleating and dinucleating ligands in coordination chemistry (Gavrilova & Bosnich, 2004). The derivatives of 1,8-naphthyridine have been widely utilized as molecular recognition receptors for urea, carboxylic acids and guanine (Goswami *et al.*, 1997; Nakatani *et al.*, 2000). Recently 1,8-naphthyridine derivatives have been reported to be excellent fluorescent markers of nucleic acids (Hooek *et al.*, 1999) and probe molecules (Nakataniz *et al.*, 2001). Many novel inorganic complexes have been synthesized using this kind of compounds as mono or bidentate ligands (Nabanita *et al.*, 2006; Jin, Liu & Chen, 2007; Jin, Chen & Wang, 2007). However, only a few mono and disubstituted 2,7-naphthyridine derivatives have been prepared. The potential multinucleating abilities of 1,8-naphthyridine derivatives as ligands in preparations of functional metalloorganic compounds stimulated us to explore bridged 1,8-naphthyridine compounds. In this paper, we report the synthesis and structure characterization of 7-(4-(5,7-dimethyl-1,8-naphthyridin-2-yloxy)phenoxy)-2,4-dimethyl-1,8-naphthyridine dimethanol solvate (I) (Fig. 1). The crystals of (I) were formed by slow evaporation of 7-(4-(5,7-dimethyl-1,8-naphthyridin-2-yloxy)phenoxy)-2,4-dimethyl-1,8-naphthyridine from methanol solution. An X-ray diffraction analysis of (I) is in agreement with the HNMR results. Bond lengths and angles are in the usual range. The bond lengths N(1)—C(8) and N(2)—C(2) are 1.303 (3) and 1.322 (3) Å, respectively, and display double-bond character. The bond lengths N(1)—C(1) and N(2)—C(1), both are 1.361 (3) Å and reveal a single-bond character. The conformations of the two naphthyridine rings towards the benzene ring is described by the torsion angle C(13)—C(12)—O(1)—C(8) (126.06 (2) °); they adopt (+)-anticlinal and (-)-anticlinal conformations. The torsion angle C(7), C(8), O(1), C(12) of 175.56 (2) ° defines the anti-parallel orientation of the two naphthyridine rings being in accord with C_i molecular symmetry. The closest contact between two adjacent naphthyridine carbons (C2...C4ⁱ, symmetry code: i) 1 - x, -y, 2 - z.) is 3.512 Å, which is in the range of π...π stacking interaction. The O—H...N, C—H...O and C—H...N interactions define the pcrystal packing (Table 1, Fig.2).

Experimental

Chemicals were obtained from commercial suppliers and used without further purification. 5,7-Dimethyl-2-chloro-1,8-naphthyridine was prepared according to (Newkome *et al.*, 1981). Reactions and product mixtures were routinely monitored by TLC on silica gel (precoated F254 Merck plates) with spot detection under UV light. NMR spectra were recorded on Bruker Avance-400 (400 MHz) spectrometer in deuterated chloroform. Chemical shifts (delta) are expressed in p.p.m. downfield to TMS at delta = 0 p.p.m. and coupling constants (J) are expressed in Hz.

A Schlenck tube was charged with 15 ml DMF and 5,7-dimethyl-2-chloro-1,8-naphthyridine, 0.77 g (4 mmol), sodium carbonate 0.33 g (2.4 mmol), *p*-hydroquinone 0.22 g (2 mmol). were added. The Schlenck tube was capped, evacuated, and back-filled with Ar three times. While still under Ar, it was immersed into a 413 K- hetaed oil bath. After stirring for 48 h,

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the mixture was cooled, filtered over celite, and evaporated *in vacuo*. The residue was washed with sodium hydroxide, then washed with water till the washing is neutral, filtered, dried in vacuum. The product 7-(4-(5,7-dimethyl-1,8-naphthyridin-2-yloxy)phenoxy)-2,4-dimethyl-1,8-naphthyridine precipitated was recrystallized from methanol. Yield: 0.42 g, 49.8%. Anal. Calcd. for (C₂₆H₂₂N₄O₂): C, 73.92%, H, 5.25%, N, 13.26%. Found: C, 73.78%, H, 5.25%, N, 13.45%. ¹H NMR (400 MHz, CDCl₃): δ = 2.65(s, 6H), 2.67(s, 6H), 7.09(s, 2H), 7.20(d, 2H, J = 9 Hz), 7.32(s, 4H), 8.30(d, 2H, J = 9 Hz).

Refinement

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93–0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Hydrogen atoms bound to methanol molecules were located in the Fourier difference map, and their distances were fixed and subject to an O—H = 0.85 Å with deviation of positive and negative 0.01 Å restraint.

Figures

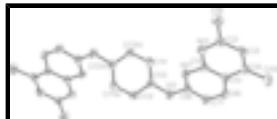


Fig. 1. The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Symmetry code to generate the molecule from an asymmetric unit ($-x, 1 - y, 1 - z$).

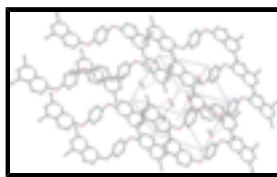


Fig. 2. The crystal packing of (I).

7-[4-(5,7-Dimethyl-1,8-naphthyridin-2-yloxy)phenoxy]-2,4-dimethyl-1,8-naphthyridine dimethanol solvate

Crystal data

C₂₆H₂₂N₄O₂·2CH₄O

$M_r = 486.56$

Triclinic, $P\bar{1}$

$a = 7.009$ (3) Å

$b = 9.244$ (3) Å

$c = 10.239$ (4) Å

$\alpha = 78.679$ (6)°

$\beta = 79.653$ (6)°

$\gamma = 82.689$ (6)°

$V = 637.0$ (4) Å³

$Z = 1$

$F_{000} = 258$

$D_x = 1.268$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 877 reflections

$\theta = 2.3$ – 24.7 °

$\mu = 0.09$ mm⁻¹

$T = 298$ (2) K

Block, colourless

$0.27 \times 0.24 \times 0.19$ mm

Data collection

Bruker SMART APEX CCD Diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

2216 independent reflections

1236 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.019$

$\theta_{\text{max}} = 25.0$ °

phi and ω scans $\theta_{\min} = 2.1^\circ$
 Absorption correction: multi-scan $h = -8 \rightarrow 5$
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.977$, $T_{\max} = 0.984$ $k = -10 \rightarrow 10$
 3379 measured reflections $l = -11 \rightarrow 12$

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
 Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites
 $R[F^2 > 2\sigma(F^2)] = 0.052$ H-atom parameters constrained
 $wR(F^2) = 0.163$ $w = 1/[\sigma^2(F_o^2) + (0.0658P)^2 + 0.2468P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.03$ $(\Delta/\sigma)_{\max} < 0.001$
 2216 reflections $\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$
 163 parameters $\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$
 Primary atom site location: structure-invariant direct methods Extinction correction: none

Special details

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.3076 (3)	0.1993 (2)	0.6758 (2)	0.0483 (6)
N2	0.5387 (3)	0.2103 (2)	0.8040 (2)	0.0496 (6)
O1	0.0813 (3)	0.19512 (19)	0.5388 (2)	0.0619 (6)
O2	0.3944 (3)	0.5192 (2)	0.7759 (3)	0.0967 (10)
H2	0.4376	0.4317	0.7831	0.145*
C1	0.4486 (4)	0.1239 (3)	0.7453 (3)	0.0430 (7)
C2	0.6819 (4)	0.1463 (3)	0.8699 (3)	0.0529 (8)
C3	0.7375 (4)	-0.0058 (3)	0.8838 (3)	0.0570 (8)
H3	0.8371	-0.0463	0.9328	0.068*
C4	0.6492 (4)	-0.0972 (3)	0.8273 (3)	0.0521 (8)
C5	0.4982 (4)	-0.0297 (3)	0.7537 (3)	0.0437 (7)
C6	0.3930 (4)	-0.1050 (3)	0.6864 (3)	0.0530 (8)
H6	0.4195	-0.2068	0.6903	0.064*

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C7	0.2544 (4)	-0.0296 (3)	0.6165 (3)	0.0557 (8)
H7	0.1844	-0.0778	0.5719	0.067*
C8	0.2194 (4)	0.1241 (3)	0.6135 (3)	0.0489 (7)
C9	0.7853 (5)	0.2444 (4)	0.9283 (4)	0.0748 (10)
H9A	0.7284	0.3448	0.9090	0.112*
H9B	0.9206	0.2389	0.8892	0.112*
H9C	0.7732	0.2123	1.0242	0.112*
C10	0.7114 (5)	-0.2600 (3)	0.8422 (4)	0.0768 (11)
H10A	0.6335	-0.3056	0.7965	0.115*
H10B	0.6944	-0.3037	0.9362	0.115*
H10C	0.8461	-0.2751	0.8035	0.115*
C11	0.1900 (4)	0.4419 (3)	0.4653 (3)	0.0532 (8)
H11	0.3177	0.4026	0.4418	0.064*
C12	0.0463 (4)	0.3504 (3)	0.5222 (3)	0.0470 (7)
C13	-0.1421 (4)	0.4064 (3)	0.5566 (3)	0.0498 (7)
H13	-0.2379	0.3428	0.5947	0.060*
C14	0.1928 (5)	0.5291 (4)	0.8034 (4)	0.0744 (10)
H14A	0.1519	0.4905	0.8970	0.112*
H14B	0.1452	0.4727	0.7488	0.112*
H14C	0.1418	0.6310	0.7834	0.112*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0538 (15)	0.0380 (13)	0.0556 (16)	-0.0017 (11)	-0.0224 (12)	-0.0039 (11)
N2	0.0545 (15)	0.0452 (13)	0.0511 (15)	-0.0090 (11)	-0.0184 (12)	-0.0019 (11)
O1	0.0721 (14)	0.0400 (11)	0.0834 (16)	0.0003 (10)	-0.0463 (12)	-0.0069 (10)
O2	0.0614 (16)	0.0542 (14)	0.176 (3)	-0.0057 (11)	-0.0178 (16)	-0.0239 (16)
C1	0.0442 (16)	0.0406 (15)	0.0437 (17)	-0.0039 (12)	-0.0123 (13)	-0.0015 (12)
C2	0.0496 (18)	0.0580 (19)	0.0515 (19)	-0.0095 (15)	-0.0141 (15)	-0.0026 (14)
C3	0.0475 (18)	0.065 (2)	0.056 (2)	0.0036 (15)	-0.0188 (15)	-0.0011 (15)
C4	0.0492 (18)	0.0499 (17)	0.0528 (19)	0.0061 (14)	-0.0097 (15)	-0.0040 (14)
C5	0.0427 (16)	0.0400 (15)	0.0458 (17)	-0.0005 (12)	-0.0083 (13)	-0.0024 (12)
C6	0.0608 (19)	0.0353 (15)	0.063 (2)	0.0006 (14)	-0.0152 (16)	-0.0075 (14)
C7	0.064 (2)	0.0426 (16)	0.067 (2)	-0.0048 (15)	-0.0239 (17)	-0.0114 (14)
C8	0.0507 (17)	0.0432 (16)	0.0535 (18)	-0.0017 (13)	-0.0204 (15)	-0.0014 (13)
C9	0.078 (2)	0.078 (2)	0.079 (3)	-0.0178 (19)	-0.038 (2)	-0.0080 (19)
C10	0.079 (2)	0.058 (2)	0.092 (3)	0.0232 (18)	-0.035 (2)	-0.0121 (18)
C11	0.0488 (18)	0.0544 (18)	0.055 (2)	0.0034 (14)	-0.0140 (15)	-0.0070 (14)
C12	0.0560 (19)	0.0397 (15)	0.0478 (18)	-0.0031 (14)	-0.0249 (15)	0.0005 (13)
C13	0.0481 (18)	0.0493 (17)	0.0497 (19)	-0.0110 (14)	-0.0121 (14)	0.0051 (13)
C14	0.070 (2)	0.074 (2)	0.084 (3)	-0.0051 (18)	-0.016 (2)	-0.0227 (19)

Geometric parameters (\AA , $^\circ$)

N1—C8	1.303 (3)	C7—C8	1.406 (4)
N1—C1	1.361 (3)	C7—H7	0.9300
N2—C2	1.322 (3)	C9—H9A	0.9600
N2—C1	1.361 (3)	C9—H9B	0.9600

O1—C8	1.364 (3)	C9—H9C	0.9600
O1—C12	1.406 (3)	C10—H10A	0.9600
O2—C14	1.385 (4)	C10—H10B	0.9600
O2—H2	0.8200	C10—H10C	0.9600
C1—C5	1.407 (4)	C11—C12	1.372 (4)
C2—C3	1.396 (4)	C11—C13 ⁱ	1.383 (4)
C2—C9	1.498 (4)	C11—H11	0.9300
C3—C4	1.373 (4)	C12—C13	1.367 (4)
C3—H3	0.9300	C13—C11 ⁱ	1.383 (4)
C4—C5	1.418 (4)	C13—H13	0.9300
C4—C10	1.499 (4)	C14—H14A	0.9600
C5—C6	1.416 (4)	C14—H14B	0.9600
C6—C7	1.350 (4)	C14—H14C	0.9600
C6—H6	0.9300		
C8—N1—C1	117.3 (2)	C2—C9—H9A	109.5
C2—N2—C1	117.9 (2)	C2—C9—H9B	109.5
C8—O1—C12	119.3 (2)	H9A—C9—H9B	109.5
C14—O2—H2	109.5	C2—C9—H9C	109.5
N2—C1—N1	114.1 (2)	H9A—C9—H9C	109.5
N2—C1—C5	123.2 (2)	H9B—C9—H9C	109.5
N1—C1—C5	122.7 (2)	C4—C10—H10A	109.5
N2—C2—C3	122.1 (3)	C4—C10—H10B	109.5
N2—C2—C9	117.1 (3)	H10A—C10—H10B	109.5
C3—C2—C9	120.8 (3)	C4—C10—H10C	109.5
C4—C3—C2	121.9 (3)	H10A—C10—H10C	109.5
C4—C3—H3	119.1	H10B—C10—H10C	109.5
C2—C3—H3	119.1	C12—C11—C13 ⁱ	119.0 (3)
C3—C4—C5	116.8 (3)	C12—C11—H11	120.5
C3—C4—C10	121.3 (3)	C13 ⁱ —C11—H11	120.5
C5—C4—C10	122.0 (3)	C13—C12—C11	121.2 (2)
C1—C5—C6	116.9 (2)	C13—C12—O1	116.5 (2)
C1—C5—C4	118.2 (3)	C11—C12—O1	122.1 (3)
C6—C5—C4	124.9 (2)	C12—C13—C11 ⁱ	119.8 (3)
C7—C6—C5	120.2 (3)	C12—C13—H13	120.1
C7—C6—H6	119.9	C11 ⁱ —C13—H13	120.1
C5—C6—H6	119.9	O2—C14—H14A	109.5
C6—C7—C8	117.9 (3)	O2—C14—H14B	109.5
C6—C7—H7	121.1	H14A—C14—H14B	109.5
C8—C7—H7	121.1	O2—C14—H14C	109.5
N1—C8—O1	119.7 (2)	H14A—C14—H14C	109.5
N1—C8—C7	124.9 (2)	H14B—C14—H14C	109.5
O1—C8—C7	115.3 (2)		
C2—N2—C1—N1	-177.8 (2)	C10—C4—C5—C6	-0.8 (5)
C2—N2—C1—C5	1.3 (4)	C1—C5—C6—C7	1.0 (4)
C8—N1—C1—N2	178.0 (2)	C4—C5—C6—C7	-178.5 (3)
C8—N1—C1—C5	-1.1 (4)	C5—C6—C7—C8	0.0 (5)
C1—N2—C2—C3	-2.0 (4)	C1—N1—C8—O1	-177.9 (2)

supplementary materials

C1—N2—C2—C9	177.3 (3)	C1—N1—C8—C7	2.2 (4)
N2—C2—C3—C4	1.5 (5)	C12—O1—C8—N1	4.6 (4)
C9—C2—C3—C4	-177.8 (3)	C12—O1—C8—C7	-175.5 (3)
C2—C3—C4—C5	0.0 (4)	C6—C7—C8—N1	-1.7 (5)
C2—C3—C4—C10	179.5 (3)	C6—C7—C8—O1	178.5 (3)
N2—C1—C5—C6	-179.4 (3)	C13 ⁱ —C11—C12—C13	0.3 (5)
N1—C1—C5—C6	-0.4 (4)	C13 ⁱ —C11—C12—O1	175.5 (2)
N2—C1—C5—C4	0.1 (4)	C8—O1—C12—C13	-126.0 (3)
N1—C1—C5—C4	179.1 (3)	C8—O1—C12—C11	58.6 (4)
C3—C4—C5—C1	-0.7 (4)	C11—C12—C13—C11 ⁱ	-0.3 (5)
C10—C4—C5—C1	179.7 (3)	O1—C12—C13—C11 ⁱ	-175.7 (2)
C3—C4—C5—C6	178.8 (3)		

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2 \cdots N2	0.82	2.06	2.882 (3)	178
C6—H6 \cdots O2 ⁱⁱ	0.93	2.53	3.414 (4)	159
C10—H10A \cdots O2 ⁱⁱ	0.96	2.54	3.436 (4)	156
C13—H13 \cdots N2 ⁱⁱⁱ	0.93	2.61	3.450 (4)	151

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

Fig. 1

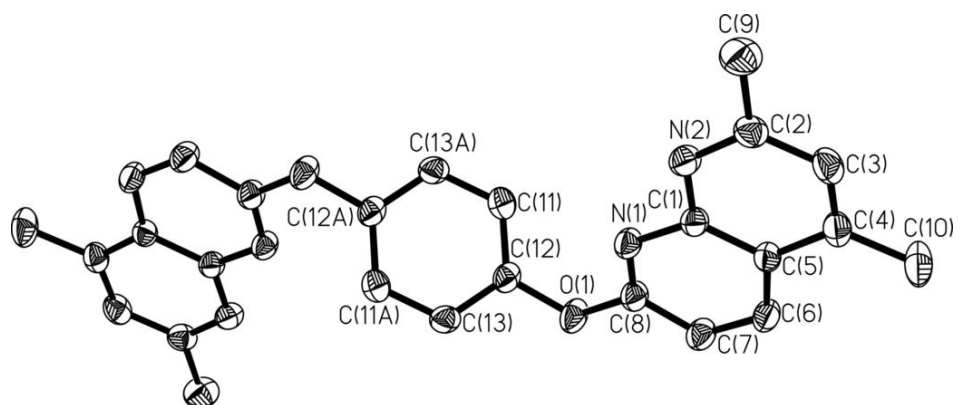


Fig. 2

