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2,2'-(1,10-Phenanthroline-2-ylimino)diethanol

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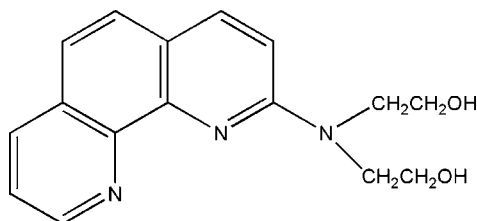
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.047; wR factor = 0.132; data-to-parameter ratio = 14.6.

In the title compound, $\text{C}_{16}\text{H}_{17}\text{N}_3\text{O}_2$, symmetry-related molecules are linked into one-dimensional chains along the a axis by a combination of intermolecular $\text{O}-\text{H}\cdots\text{N}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds and weak $\pi-\pi$ stacking interactions with a centroid-centroid distance of 3.5494 (12) Å.

Related literature

For recent crystal structure reports on the complexes formed with derivatives of 1,10-phenanthroline, see for example: Li *et al.* (2008) and Zhang *et al.* (2008).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{17}\text{N}_3\text{O}_2$
 $M_r = 283.33$
 Monoclinic, $P2_1/c$
 $a = 4.8480$ (10) Å
 $b = 14.854$ (3) Å
 $c = 18.858$ (4) Å
 $\beta = 93.523$ (3)°

$V = 1355.5$ (5) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 298$ K
 $0.54 \times 0.32 \times 0.20$ mm

Data collection

Bruker SMART APEX CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.951$, $T_{\max} = 0.982$

7520 measured reflections
 2765 independent reflections
 2149 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.132$
 $S = 1.03$
 2765 reflections

190 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O2}-\text{H5}\cdots\text{O1}^{\text{i}}$	0.91	1.91	2.8254 (17)	177
$\text{O1}-\text{H4}\cdots\text{N1}^{\text{ii}}$	0.83	2.08	2.8676 (18)	158

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

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2772).

References

- Bruker (1997). SMART and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Li, H., Hu, T. Q. & Zhang, S. G. (2008). *Acta Cryst.* E64, m771.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* A64, 112–122.
 Zhang, S. G., Hu, T. Q. & Li, H. (2008). *Acta Cryst.* E64, m769.

supplementary materials

Acta Cryst. (2009). E65, o541 [doi:10.1107/S1600536809004929]

2,2'-(1,10-Phenanthroline-2-ylimino)diethanol

X. Jin and J. M. Li

Comment

Derivatives of 1,10-phenanthroline play a vital role in a modern coordination chemistry and a number of complexes have been published with these types of compounds as ligands [see for example the recent publications by Li *et al.* (2008) and Zhang *et al.* (2008)]. An interest in designing new derivatives led to the synthesis the title compound, (I), and its crystal structure is reported herein.

The molecular structure of (I) is shown in Fig. 1. In the crystal structure, intermolecular O—H \cdots N and O—H \cdots O hydrogen bonds (see Table 1) connect symmetry related molecules to form 1-D chains along the a axis (see Fig. 2). In addition, within these chain there are weak π - π stacking interactions between symmetry related pyridyl rings, with the relevant distances being $Cg1\cdots Cg2^{iii} = 3.5494(12)$ Å, $Cg1\cdots Cg2^{iii}_{\text{perp}} = 3.464$ Å and $\alpha = 1.48^\circ$ [symmetry code (iii) $1+x, y, z$; $Cg1$ and $Cg2$ are the centroids of the C6C7C10-C12/N1 and C1—C5/N2 rings, respectively; $Cg1\cdots Cg2^{iii}_{\text{perp}}$ is the perpendicular distance from ring $Cg1$ to ring $Cg2^{iii}$; α is the dihedral angle between ring plane $Cg1$ and ring plane $Cg2^{iii}$].

Experimental

2-Diethanolamine-1,10-phenanthroline (0.0523 g, 0.185 mmol) was dissolved into 10 ml methanol and the yellow single crystals were obtained after the solution had been allowed to stand at room temperature for one week.

Refinement

The H atoms of hydroxyl groups were found in a difference Fourier map and were included in 'as found' positions with $d(\text{O—H}) = 0.83$ & 0.91 Å; all other H atoms were placed in calculated positions with $\text{C—H} = 0.93$ – 0.97 Å. All H atoms were refined as riding, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ for hydroxyl group and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for other groups.

Figures

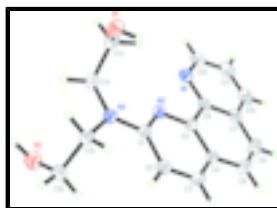


Fig. 1. The molecular structure of (I), showing the the atom numbering scheme with thermal ellipsoids drawn at the 30% probability level

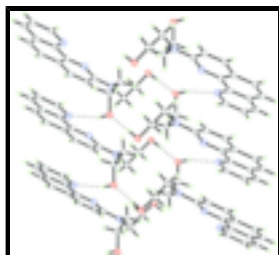


Fig. 2. Part of the crystal structure of (I) showing hydrogen bonds (dashed lines) between symmetry related molecules which form a one-dimensional chain along the a axis.

2,2'-(1,10-Phenanthroline-2-ylidino)diethanol

Crystal data

$C_{16}H_{17}N_3O_2$

$M_r = 283.33$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 4.8480$ (10) Å

$b = 14.854$ (3) Å

$c = 18.858$ (4) Å

$\beta = 93.523$ (3)°

$V = 1355.5$ (5) Å³

$Z = 4$

$F_{000} = 600$

$D_x = 1.388$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 2381 reflections

$\theta = 2.6$ – 27.3 °

$\mu = 0.09$ mm⁻¹

$T = 298$ K

Block, yellow

$0.54 \times 0.32 \times 0.20$ mm

Data collection

Bruker SMART APEX CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.951$, $T_{\max} = 0.982$

7520 measured reflections

2765 independent reflections

2149 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\max} = 26.5$ °

$\theta_{\min} = 1.8$ °

$h = -5 \rightarrow 6$

$k = -12 \rightarrow 18$

$l = -23 \rightarrow 23$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.047$

$wR(F^2) = 0.132$

$S = 1.03$

2765 reflections

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0762P)^2 + 0.108P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.008$

$\Delta\rho_{\max} = 0.24$ e Å⁻³

190 parameters

$$\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$$

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 $F^2 > \sigma(F^2)$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	-0.1483 (3)	0.89542 (10)	0.18581 (7)	0.0333 (3)
C2	-0.2322 (3)	0.81999 (11)	0.22608 (8)	0.0408 (4)
H2	-0.3697	0.7814	0.2077	0.049*
C3	-0.1090 (4)	0.80542 (11)	0.29126 (8)	0.0439 (4)
H3	-0.1645	0.7569	0.3181	0.053*
C4	0.1020 (3)	0.86265 (10)	0.31895 (8)	0.0391 (4)
C5	0.1770 (3)	0.93444 (10)	0.27517 (7)	0.0336 (3)
C6	0.3910 (3)	0.99566 (10)	0.30193 (7)	0.0356 (4)
C7	0.5185 (3)	0.98199 (12)	0.37060 (8)	0.0460 (4)
C8	0.4363 (4)	0.90765 (14)	0.41232 (9)	0.0597 (5)
H8	0.5227	0.8980	0.4571	0.072*
C9	0.2361 (4)	0.85156 (13)	0.38776 (9)	0.0553 (5)
H9	0.1833	0.8043	0.4163	0.066*
C10	0.7237 (4)	1.04302 (14)	0.39446 (10)	0.0593 (5)
H10	0.8122	1.0362	0.4393	0.071*
C11	0.7935 (4)	1.11242 (14)	0.35196 (11)	0.0598 (5)
H11	0.9289	1.1536	0.3673	0.072*
C12	0.6584 (4)	1.12040 (12)	0.28535 (10)	0.0494 (4)
H12	0.7079	1.1679	0.2567	0.059*
C13	-0.1736 (3)	0.99054 (11)	0.08028 (7)	0.0387 (4)
H13A	-0.2437	0.9841	0.0313	0.046*
H13B	0.0264	0.9875	0.0811	0.046*
C14	-0.2546 (3)	1.08165 (11)	0.10638 (8)	0.0402 (4)
H14A	-0.1674	1.0912	0.1535	0.048*
H14B	-0.1856	1.1273	0.0752	0.048*
C15	-0.4906 (3)	0.85897 (10)	0.08751 (8)	0.0392 (4)
H15A	-0.6007	0.8955	0.0538	0.047*
H15B	-0.6107	0.8389	0.1236	0.047*
C16	-0.3880 (4)	0.77747 (11)	0.04926 (8)	0.0465 (4)
H16A	-0.3010	0.7366	0.0840	0.056*

supplementary materials

H16B	-0.5452	0.7465	0.0262	0.056*
N1	0.4640 (3)	1.06489 (9)	0.25999 (7)	0.0415 (3)
N2	0.0550 (2)	0.94980 (8)	0.20978 (6)	0.0333 (3)
N3	-0.2733 (3)	0.91507 (8)	0.12111 (6)	0.0374 (3)
O1	-0.5458 (2)	1.09241 (8)	0.10935 (6)	0.0499 (3)
H4	-0.5826	1.0760	0.1499	0.075*
O2	-0.1971 (3)	0.79774 (9)	-0.00230 (6)	0.0586 (4)
H5	-0.2838	0.8313	-0.0377	0.088*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0325 (8)	0.0333 (8)	0.0345 (7)	0.0036 (6)	0.0044 (6)	-0.0001 (6)
C2	0.0433 (9)	0.0355 (8)	0.0436 (8)	-0.0043 (7)	0.0027 (7)	0.0017 (7)
C3	0.0524 (10)	0.0343 (9)	0.0457 (9)	0.0012 (7)	0.0090 (8)	0.0101 (7)
C4	0.0435 (9)	0.0375 (8)	0.0366 (8)	0.0056 (7)	0.0032 (7)	0.0059 (6)
C5	0.0338 (8)	0.0350 (8)	0.0322 (7)	0.0063 (6)	0.0043 (6)	0.0014 (6)
C6	0.0331 (9)	0.0370 (8)	0.0369 (8)	0.0080 (6)	0.0041 (6)	-0.0032 (6)
C7	0.0436 (10)	0.0529 (10)	0.0407 (8)	0.0079 (8)	-0.0035 (7)	-0.0069 (7)
C8	0.0682 (13)	0.0704 (13)	0.0385 (9)	0.0051 (10)	-0.0131 (8)	0.0081 (8)
C9	0.0676 (13)	0.0566 (11)	0.0411 (9)	0.0044 (9)	-0.0018 (8)	0.0161 (8)
C10	0.0505 (11)	0.0736 (14)	0.0522 (10)	0.0062 (10)	-0.0103 (8)	-0.0156 (9)
C11	0.0466 (11)	0.0627 (12)	0.0694 (12)	-0.0077 (9)	-0.0009 (9)	-0.0228 (10)
C12	0.0458 (10)	0.0443 (10)	0.0589 (10)	-0.0045 (8)	0.0096 (8)	-0.0127 (8)
C13	0.0360 (9)	0.0494 (9)	0.0306 (7)	-0.0033 (7)	0.0005 (6)	0.0062 (6)
C14	0.0358 (9)	0.0421 (9)	0.0423 (8)	-0.0042 (7)	-0.0003 (7)	0.0088 (6)
C15	0.0342 (9)	0.0430 (9)	0.0398 (8)	0.0002 (7)	-0.0022 (6)	0.0012 (6)
C16	0.0522 (11)	0.0417 (9)	0.0448 (9)	0.0025 (7)	-0.0047 (8)	-0.0014 (7)
N1	0.0383 (8)	0.0410 (7)	0.0455 (7)	-0.0010 (6)	0.0062 (6)	-0.0048 (6)
N2	0.0330 (7)	0.0344 (7)	0.0327 (6)	0.0026 (5)	0.0039 (5)	0.0027 (5)
N3	0.0404 (7)	0.0374 (7)	0.0337 (6)	-0.0034 (6)	-0.0025 (5)	0.0031 (5)
O1	0.0394 (7)	0.0602 (8)	0.0502 (7)	0.0085 (5)	0.0040 (5)	0.0163 (5)
O2	0.0552 (8)	0.0697 (9)	0.0514 (7)	0.0186 (6)	0.0069 (6)	-0.0034 (6)

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

C1—N2	1.3319 (19)	C11—C12	1.386 (3)
C1—N3	1.3603 (18)	C11—H11	0.9300
C1—C2	1.427 (2)	C12—N1	1.320 (2)
C2—C3	1.351 (2)	C12—H12	0.9300
C2—H2	0.9300	C13—N3	1.4590 (18)
C3—C4	1.406 (2)	C13—C14	1.501 (2)
C3—H3	0.9300	C13—H13A	0.9700
C4—C5	1.410 (2)	C13—H13B	0.9700
C4—C9	1.425 (2)	C14—O1	1.4255 (19)
C5—N2	1.3539 (18)	C14—H14A	0.9700
C5—C6	1.447 (2)	C14—H14B	0.9700
C6—N1	1.3573 (19)	C15—N3	1.4574 (19)
C6—C7	1.415 (2)	C15—C16	1.509 (2)

C7—C10	1.400 (3)	C15—H15A	0.9700
C7—C8	1.427 (3)	C15—H15B	0.9700
C8—C9	1.340 (3)	C16—O2	1.415 (2)
C8—H8	0.9300	C16—H16A	0.9700
C9—H9	0.9300	C16—H16B	0.9700
C10—C11	1.361 (3)	O1—H4	0.8330
C10—H10	0.9300	O2—H5	0.9147
N2—C1—N3	116.99 (13)	N1—C12—H12	117.9
N2—C1—C2	121.66 (13)	C11—C12—H12	117.9
N3—C1—C2	121.35 (14)	N3—C13—C14	114.71 (12)
C3—C2—C1	119.07 (14)	N3—C13—H13A	108.6
C3—C2—H2	120.5	C14—C13—H13A	108.6
C1—C2—H2	120.5	N3—C13—H13B	108.6
C2—C3—C4	120.81 (14)	C14—C13—H13B	108.6
C2—C3—H3	119.6	H13A—C13—H13B	107.6
C4—C3—H3	119.6	O1—C14—C13	113.19 (13)
C3—C4—C5	116.60 (14)	O1—C14—H14A	108.9
C3—C4—C9	123.31 (15)	C13—C14—H14A	108.9
C5—C4—C9	120.08 (15)	O1—C14—H14B	108.9
N2—C5—C4	123.15 (14)	C13—C14—H14B	108.9
N2—C5—C6	118.39 (13)	H14A—C14—H14B	107.8
C4—C5—C6	118.45 (13)	N3—C15—C16	114.57 (13)
N1—C6—C7	121.82 (14)	N3—C15—H15A	108.6
N1—C6—C5	118.68 (13)	C16—C15—H15A	108.6
C7—C6—C5	119.50 (14)	N3—C15—H15B	108.6
C10—C7—C6	117.59 (16)	C16—C15—H15B	108.6
C10—C7—C8	122.76 (16)	H15A—C15—H15B	107.6
C6—C7—C8	119.65 (15)	O2—C16—C15	113.97 (13)
C9—C8—C7	120.76 (15)	O2—C16—H16A	108.8
C9—C8—H8	119.6	C15—C16—H16A	108.8
C7—C8—H8	119.6	O2—C16—H16B	108.8
C8—C9—C4	121.54 (16)	C15—C16—H16B	108.8
C8—C9—H9	119.2	H16A—C16—H16B	107.7
C4—C9—H9	119.2	C12—N1—C6	117.92 (14)
C11—C10—C7	119.93 (17)	C1—N2—C5	118.67 (12)
C11—C10—H10	120.0	C1—N3—C15	122.52 (12)
C7—C10—H10	120.0	C1—N3—C13	119.64 (12)
C10—C11—C12	118.52 (17)	C15—N3—C13	117.64 (11)
C10—C11—H11	120.7	C14—O1—H4	105.8
C12—C11—H11	120.7	C16—O2—H5	109.1
N1—C12—C11	124.22 (18)		
N2—C1—C2—C3	2.5 (2)	C6—C7—C10—C11	0.1 (3)
N3—C1—C2—C3	-177.39 (14)	C8—C7—C10—C11	179.74 (17)
C1—C2—C3—C4	-1.0 (2)	C7—C10—C11—C12	-0.3 (3)
C2—C3—C4—C5	-0.4 (2)	C10—C11—C12—N1	0.2 (3)
C2—C3—C4—C9	178.88 (15)	N3—C13—C14—O1	-56.55 (17)
C3—C4—C5—N2	0.5 (2)	N3—C15—C16—O2	-54.91 (18)
C9—C4—C5—N2	-178.84 (14)	C11—C12—N1—C6	0.2 (2)

supplementary materials

C3—C4—C5—C6	179.25 (13)	C7—C6—N1—C12	-0.4 (2)
C9—C4—C5—C6	-0.1 (2)	C5—C6—N1—C12	179.63 (13)
N2—C5—C6—N1	-1.15 (19)	N3—C1—N2—C5	177.46 (12)
C4—C5—C6—N1	-179.99 (13)	C2—C1—N2—C5	-2.4 (2)
N2—C5—C6—C7	178.92 (13)	C4—C5—N2—C1	1.0 (2)
C4—C5—C6—C7	0.1 (2)	C6—C5—N2—C1	-177.83 (12)
N1—C6—C7—C10	0.3 (2)	N2—C1—N3—C15	177.43 (12)
C5—C6—C7—C10	-179.75 (14)	C2—C1—N3—C15	-2.7 (2)
N1—C6—C7—C8	-179.37 (14)	N2—C1—N3—C13	2.72 (19)
C5—C6—C7—C8	0.5 (2)	C2—C1—N3—C13	-177.39 (13)
C10—C7—C8—C9	179.05 (18)	C16—C15—N3—C1	-81.85 (17)
C6—C7—C8—C9	-1.3 (3)	C16—C15—N3—C13	92.96 (16)
C7—C8—C9—C4	1.3 (3)	C14—C13—N3—C1	-75.61 (17)
C3—C4—C9—C8	-179.92 (17)	C14—C13—N3—C15	109.43 (15)
C5—C4—C9—C8	-0.7 (3)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H5 \cdots O1 ⁱ	0.91	1.91	2.8254 (17)	177
O1—H4 \cdots N1 ⁱⁱ	0.83	2.08	2.8676 (18)	158

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

Fig. 1

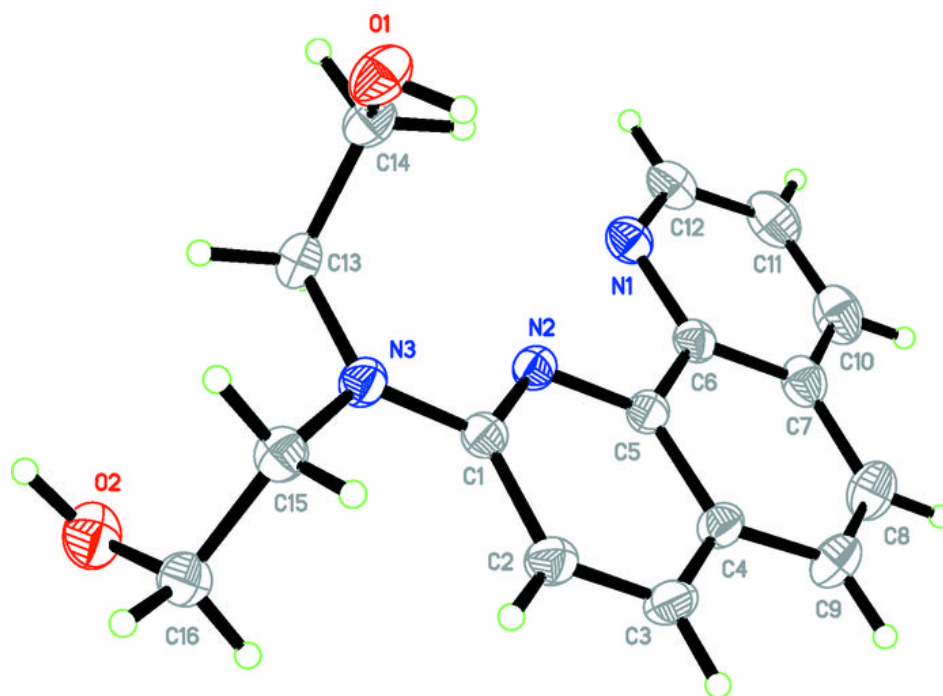


Fig. 2

