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## Structure Reports

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## 5,6-Dimethyl-1,10-phenanthroline

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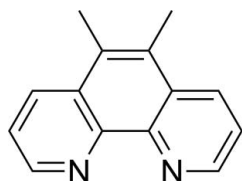
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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.035;  $wR$  factor = 0.100; data-to-parameter ratio = 10.8.

In the title compound,  $\text{C}_{14}\text{H}_{12}\text{N}_2$ , the  $\text{N}\cdots\text{N}$  distance is 2.719 (1) Å. The  $\text{N}-\text{C}-\text{C}-\text{N}$  torsion angle [ $0.9$  (1) $^\circ$ ] is close to the ideal value of  $0^\circ$  as expected. Bond lengths and angles are consistent with those observed for [1,10]phenanthroline and coordinated 5,6 dimethyl[1,10]phenanthroline. In the crystal,  $\text{C}-\text{H}\cdots\text{N}$  hydrogen bonds link the molecules into  $C(4)$  chains running parallel to the  $b$  axis. Weak  $\pi-\pi$  interactions between benzene and pyridine rings [centroid-centroid distance = 3.5337 (7) Å] and between benzene rings [centroid-centroid distances = 3.6627 (7) and 3.8391 (7) Å] also occur.

## Related literature

For [1,10]phenanthroline and 5,6-dimethyl[1,10]phenanthroline, see: Ton & Bolte (2005) and Gasque *et al.* (1999), respectively. For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



## Experimental

## Crystal data

$\text{C}_{14}\text{H}_{12}\text{N}_2$   
 $M_r = 208.26$   
Monoclinic,  $P2_1/c$   
 $a = 7.1932$  (7) Å

$b = 10.0572$  (10) Å  
 $c = 13.8729$  (13) Å  
 $\beta = 93.673$  (5) $^\circ$   
 $V = 1001.55$  (17) Å $^3$

$Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.08$  mm $^{-1}$

$T = 100$  K  
 $0.11 \times 0.10 \times 0.09$  mm

## Data collection

Bruker APEXII CCD diffractometer  
Absorption correction: multi-scan (SADABS; Bruker, 2009)  
 $T_{\min} = 0.991$ ,  $T_{\max} = 0.993$

16500 measured reflections  
1855 independent reflections  
1613 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.022$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$   
 $wR(F^2) = 0.100$   
 $S = 1.06$   
1855 reflections  
171 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.16$  e Å $^{-3}$   
 $\Delta\rho_{\text{min}} = -0.20$  e Å $^{-3}$

Table 1

Hydrogen-bond geometry (Å,  $^\circ$ ).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}2-\text{H}2\cdots\text{N}1^i$	0.945 (14)	2.439 (13)	3.3718 (15)	169.0 (10)

Symmetry code: (i)  $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$ .

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SIR97 (Altomare, *et al.* 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-32 (Farrugia, 2012); software used to prepare material for publication: WinGX (Farrugia, 2012).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2451).

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## supporting information

*Acta Cryst.* (2013). E69, o1560 [doi:10.1107/S1600536813025087]

## 5,6-Dimethyl-1,10-phenanthroline

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### S1. Comment

5,6-dimethyl-1,10-phenanthroline was obtained from Sigma-Aldrich. The distances and angles are consistent with those observed for [1,10]phenanthroline (Ton & Bolte, 2005) and coordinated 5,6-dimethyl-1,10-phenanthroline (Gasque, *et al.*, 1999). The crystal packing is stabilized by an intermolecular C—H···N hydrogen bond interaction which links the molecules into chains with graph-set notation  $C(4)$  (Bernstein, *et al.*, 1995) running parallel to the b axis. Weak intermolecular  $\pi$ - $\pi$  interactions, involving the benzene rings, and the pyridine rings, respectively with an average plane-to-plane separation of 3.5982 (7) Å further stabilize and reinforce the crystal structure.

### S2. Experimental

Crystals of the title compound, 5,6-dimethyl-1,10-phenanthroline, were obtained by sublimation at 160 °C under dynamic vacuum.

### S3. Refinement

All non-hydrogen atoms were refined anisotropically. Aromatic hydrogen atoms were located in the Fourier map and refined isotropically. Methyl hydrogen atoms were placed based on the expected geometry of the carbon atoms to which they were attached and refined using a riding model, with C—H distances 0.98 Å and  $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C})$ .

**Figure 1**

Top view of the structure of 5,6-dimethyl-1,10-phenanthroline, displacement ellipsoids drawn at the 50% probability level.

### 5,6-Dimethyl-1,10-phenanthroline

#### Crystal data

$C_{14}H_{12}N_2$   
 $M_r = 208.26$   
 Monoclinic,  $P2_1/c$   
 Hall symbol: -P 2ybc  
 $a = 7.1932$  (7) Å  
 $b = 10.0572$  (10) Å  
 $c = 13.8729$  (13) Å  
 $\beta = 93.673$  (5)°  
 $V = 1001.55$  (17) Å<sup>3</sup>  
 $Z = 4$

$F(000) = 440$   
 SHELXL-97  
 $D_x = 1.381$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å  
 Cell parameters from 9819 reflections  
 $\theta = 2.5$ – $25.4$ °  
 $\mu = 0.08$  mm<sup>-1</sup>  
 $T = 100$  K  
 Block, colourless  
 $0.11 \times 0.10 \times 0.09$  mm

#### Data collection

Bruker APEXII CCD  
 diffractometer  
 Radiation source: microfocus sealed tube  
 QUAZAR multilayer mirrors monochromator  
 Detector resolution: 8.366 pixels mm<sup>-1</sup>  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan  
 (SADABS; Bruker, 2009)  
 $T_{\min} = 0.991$ ,  $T_{\max} = 0.993$

16500 measured reflections  
 1855 independent reflections  
 1613 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.022$   
 $\theta_{\text{max}} = 25.5$ °,  $\theta_{\text{min}} = 2.5$ °  
 $h = -8 \rightarrow 8$   
 $k = -12 \rightarrow 12$   
 $l = -16 \rightarrow 16$

Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.035$   
 $wR(F^2) = 0.100$   
 $S = 1.06$   
 1855 reflections  
 171 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0576P)^2 + 0.2458P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$

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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.42701 (15)	0.02490 (12)	0.23673 (8)	0.0163 (3)
C2	0.42141 (15)	0.15376 (12)	0.19967 (8)	0.0169 (3)
C3	0.35250 (15)	0.17126 (12)	0.10622 (8)	0.0155 (3)
C4	0.29281 (14)	0.06112 (11)	0.04921 (8)	0.0133 (3)
C5	0.22138 (14)	0.07469 (11)	-0.05047 (8)	0.0140 (3)
C6	0.17602 (14)	-0.03576 (11)	-0.10406 (8)	0.0140 (3)
C7	0.19526 (14)	-0.16667 (11)	-0.06034 (8)	0.0136 (3)
C8	0.15222 (15)	-0.28403 (12)	-0.11278 (8)	0.0168 (3)
C9	0.17425 (16)	-0.40516 (12)	-0.06876 (8)	0.0192 (3)
C10	0.24086 (16)	-0.40937 (12)	0.02795 (9)	0.0193 (3)
C11	0.26123 (14)	-0.18229 (11)	0.03706 (8)	0.0132 (3)
C12	0.31061 (14)	-0.06488 (11)	0.09349 (8)	0.0128 (3)
C13	0.20306 (16)	0.21432 (12)	-0.08965 (8)	0.0182 (3)
H13A	0.3273	0.2519	-0.0961	0.027*
H13B	0.1353	0.2692	-0.0452	0.027*
H13C	0.1347	0.2125	-0.1530	0.027*
C14	0.10570 (15)	-0.03036 (12)	-0.20896 (8)	0.0188 (3)
H14A	-0.0264	-0.0549	-0.2148	0.028*
H14B	0.1771	-0.0926	-0.2464	0.028*
H14C	0.1207	0.0600	-0.2337	0.028*
N1	0.37466 (12)	-0.08243 (9)	0.18696 (7)	0.0150 (2)
N2	0.28440 (13)	-0.30292 (9)	0.08056 (7)	0.0163 (3)
H1	0.4752 (17)	0.0073 (13)	0.3036 (9)	0.019 (3)*
H2	0.4698 (17)	0.2240 (14)	0.2390 (9)	0.020 (3)*

H3	0.3500 (18)	0.2604 (15)	0.0801 (9)	0.023 (3)*
H8	0.1103 (18)	-0.2799 (14)	-0.1809 (10)	0.026 (3)*
H9	0.1465 (17)	-0.4866 (14)	-0.1012 (9)	0.022 (3)*
H10	0.2560 (18)	-0.4966 (14)	0.0606 (9)	0.024 (3)*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0132 (5)	0.0220 (7)	0.0137 (6)	-0.0005 (4)	0.0003 (4)	-0.0018 (5)
C2	0.0141 (6)	0.0184 (6)	0.0183 (6)	-0.0024 (4)	0.0026 (4)	-0.0061 (5)
C3	0.0146 (6)	0.0130 (6)	0.0194 (6)	0.0002 (4)	0.0043 (4)	0.0005 (5)
C4	0.0086 (5)	0.0157 (6)	0.0160 (6)	0.0004 (4)	0.0031 (4)	0.0004 (5)
C5	0.0095 (5)	0.0160 (6)	0.0167 (6)	0.0014 (4)	0.0030 (4)	0.0034 (5)
C6	0.0098 (5)	0.0184 (6)	0.0141 (6)	0.0011 (4)	0.0023 (4)	0.0007 (5)
C7	0.0093 (5)	0.0169 (6)	0.0149 (6)	0.0007 (4)	0.0020 (4)	-0.0005 (5)
C8	0.0154 (6)	0.0210 (7)	0.0142 (6)	-0.0015 (5)	0.0008 (4)	-0.0024 (5)
C9	0.0201 (6)	0.0157 (6)	0.0217 (6)	-0.0018 (5)	0.0014 (5)	-0.0045 (5)
C10	0.0209 (6)	0.0126 (6)	0.0241 (7)	0.0004 (5)	0.0001 (5)	0.0012 (5)
C11	0.0101 (5)	0.0145 (6)	0.0151 (6)	0.0005 (4)	0.0026 (4)	0.0012 (5)
C12	0.0091 (5)	0.0165 (6)	0.0130 (6)	0.0004 (4)	0.0019 (4)	0.0013 (4)
C13	0.0197 (6)	0.0169 (7)	0.0177 (6)	0.0004 (5)	0.0000 (5)	0.0029 (5)
C14	0.0192 (6)	0.0218 (7)	0.0150 (6)	0.0019 (5)	-0.0014 (4)	0.0023 (5)
N1	0.0138 (5)	0.0180 (5)	0.0131 (5)	0.0002 (4)	-0.0002 (4)	0.0002 (4)
N2	0.0172 (5)	0.0135 (5)	0.0178 (5)	-0.0001 (4)	-0.0003 (4)	0.0013 (4)

*Geometric parameters (Å, °)*

C1—N1	1.3230 (15)	C8—C9	1.3673 (17)
C1—C2	1.3939 (17)	C8—H8	0.975 (14)
C1—H1	0.985 (13)	C9—C10	1.3960 (17)
C2—C3	1.3695 (16)	C9—H9	0.950 (14)
C2—H2	0.945 (14)	C10—N2	1.3220 (15)
C3—C4	1.4118 (16)	C10—H10	0.990 (14)
C3—H3	0.967 (15)	C11—N2	1.3605 (14)
C4—C12	1.4105 (16)	C11—C12	1.4483 (15)
C4—C5	1.4505 (16)	C12—N1	1.3595 (14)
C5—C6	1.3645 (16)	C13—H13A	0.9800
C5—C13	1.5085 (15)	C13—H13B	0.9800
C6—C7	1.4525 (16)	C13—H13C	0.9800
C6—C14	1.5108 (15)	C14—H14A	0.9800
C7—C8	1.4104 (15)	C14—H14B	0.9800
C7—C11	1.4118 (15)	C14—H14C	0.9800
N1—C1—C2	124.44 (10)	C8—C9—H9	122.8 (8)
N1—C1—H1	114.6 (8)	C10—C9—H9	118.6 (8)
C2—C1—H1	121.0 (7)	N2—C10—C9	124.10 (11)
C3—C2—C1	117.96 (10)	N2—C10—H10	116.7 (8)
C3—C2—H2	123.3 (8)	C9—C10—H10	119.2 (8)

C1—C2—H2	118.7 (8)	N2—C11—C7	123.22 (10)
C2—C3—C4	120.54 (11)	N2—C11—C12	117.93 (10)
C2—C3—H3	118.1 (8)	C7—C11—C12	118.84 (10)
C4—C3—H3	121.3 (8)	N1—C12—C4	123.21 (10)
C12—C4—C3	116.43 (10)	N1—C12—C11	117.78 (10)
C12—C4—C5	121.10 (10)	C4—C12—C11	119.00 (10)
C3—C4—C5	122.46 (10)	C5—C13—H13A	109.5
C6—C5—C4	120.03 (10)	C5—C13—H13B	109.5
C6—C5—C13	123.31 (10)	H13A—C13—H13B	109.5
C4—C5—C13	116.65 (10)	C5—C13—H13C	109.5
C5—C6—C7	119.83 (10)	H13A—C13—H13C	109.5
C5—C6—C14	123.31 (10)	H13B—C13—H13C	109.5
C7—C6—C14	116.87 (10)	C6—C14—H14A	109.5
C8—C7—C11	116.73 (10)	C6—C14—H14B	109.5
C8—C7—C6	122.09 (10)	H14A—C14—H14B	109.5
C11—C7—C6	121.17 (10)	C6—C14—H14C	109.5
C9—C8—C7	119.99 (10)	H14A—C14—H14C	109.5
C9—C8—H8	119.4 (8)	H14B—C14—H14C	109.5
C7—C8—H8	120.6 (8)	C1—N1—C12	117.36 (9)
C8—C9—C10	118.62 (11)	C10—N2—C11	117.34 (10)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C2—H2 $\cdots$ N1 <sup>i</sup>	0.945 (14)	2.439 (13)	3.3718 (15)	169.0 (10)

Symmetry code: (i)  $-x+1, y+1/2, -z+1/2$ .