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Key indicators

Single-crystal X-ray study

T = 90 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.026

wR factor = 0.060

Data-to-parameter ratio = 28.0

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.(2,9-Dimethyl-4,7-diphenyl-1,10-phenanthroline)-
(tetrahydroborato)copper(I): a redetermination at 90 K

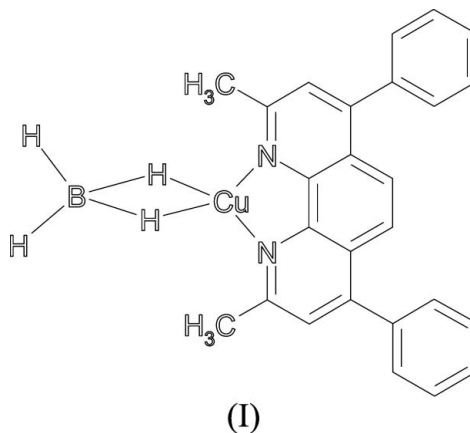
The structure of the title compound, $[\text{Cu}(\text{BH}_4)(\text{C}_{26}\text{H}_{20}\text{N}_2)]$, has been redetermined at 90 K. The geometric parameters of the present structure agree with those previously studied at room temperature [Green, Kennard, Smith, Elcombe, Moore, James & White (1984). *Inorg. Chim. Acta*, **83**, 177–189], but with significantly improved precision.

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Comment

The crystal structure of the title compound, (I), has been previously studied at room temperature (Green *et al.*, 1984) using single-crystal X-ray diffraction. In the present redetermination, data were collected at low temperature, using a diffractometer equipped with an APEX2 CCD area detector.



As shown in Fig. 1, the structure of (I) is composed of neutral $[\text{Cu}(\text{C}_{26}\text{H}_{20}\text{N}_2)(\text{BH}_4)]$ complex molecules. The results show that the coordination around Cu1 is distorted tetrahedral, with two N atoms of 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline [$\text{Cu1}-\text{N1} = 2.025(1) \text{ \AA}$ and $\text{Cu1}-\text{N2} = 2.038(1) \text{ \AA}$] and two H atoms of bidentate tetrahydroborate anion [$\text{Cu1}-\text{H1B} = 1.67(2) \text{ \AA}$ and $\text{Cu1}-\text{H2B} = 1.65(2) \text{ \AA}$]. The Cu atom assumes a distorted tetrahedral configuration as in $[\text{Cu}(2,9\text{-dimethyl-1,10-phenanthroline})(\text{BH}_4)]$ (Green *et al.*, 1980). On the other hand, the Cu atom is five-coordinate in $[\text{Cu}(\text{PPh}_3)(1,10\text{-phenanthroline})(\text{BH}_4)]$ (Green *et al.*, 1981, 1984) and $[\text{Cu}(\text{P}(\text{OEt})_3)(1,10\text{-phenanthroline})(\text{BH}_4)]$ (Makhaev *et al.*, 1993). The geometric parameters of the present structure agree well with those previously reported by Green *et al.* (1984), but with significantly improved precision. The precision of the Cu–N bond lengths (0.001 Å) and that of the mean C–C bond length (0.002 Å) obtained from the low-temperature study are significantly better than that obtained from room-temperature data (0.01 and 0.02,

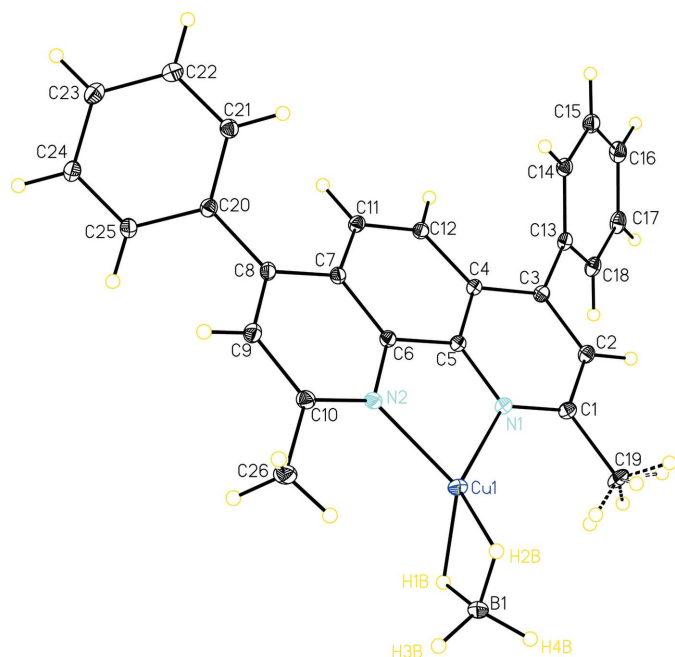


Figure 1
Perspective view of (I), with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Both components of the disordered atoms are shown.

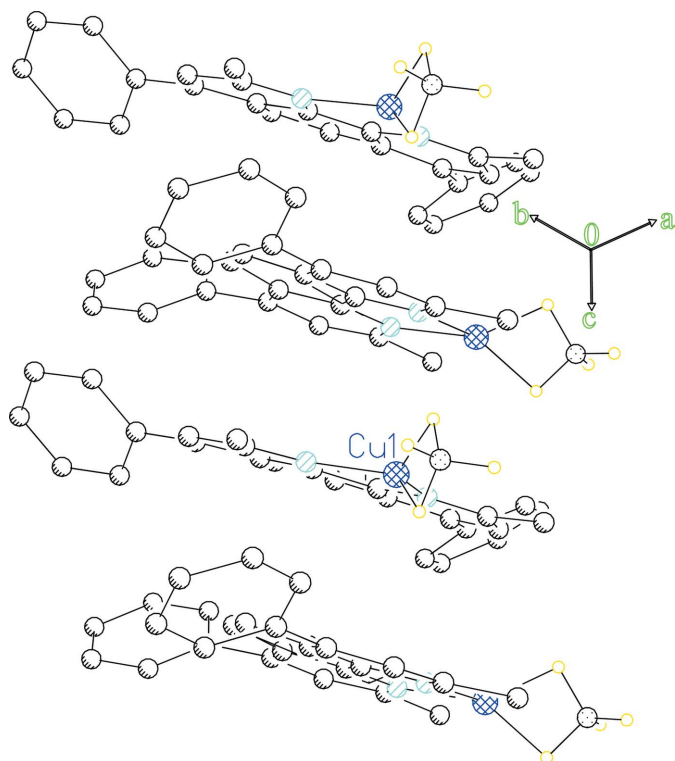


Figure 2
The π - π stacking interactions in the crystal structure of (I). C-bound H atoms have been omitted.

respectively). Similarly, the standard uncertainty for the N1—Cu1—N2 bond angle and mean C—C—C bond angles for the low-temperature data are 0.004 and 0.01°, respectively, whereas for the room-temperature data the standard deviation for the N1—Cu1—N2 bond angle is 0.005° and for the mean C—C—C bond angles is 1°.

There are π - π stacking interactions (Fig. 2) (Janiak, 2000) between symmetry-related adjacent phenanthroline rings [(x , $-y$, $z - \frac{1}{2}$) and (x , $-y$, $z + \frac{1}{2}$)] [distances between the phenanthroline rings are in the range 3.22–3.81 Å].

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Experimental

[Cu(PPh₃)₂(BH₄)] was prepared by the procedure reported by (Moncol *et al.*, 2005). [Cu(C₂₆H₂₀N₂)(BH₄)] was prepared by a modification of the procedure reported by (Green *et al.*, 1984), by adding 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (0.36 g, 1 mmol) dissolved in dichloromethane (5 ml) to a dichloromethane solution (10 ml) of [Cu(PPh₃)₂(BH₄)] (0.60 g, 1 mmol).

Crystal data

[Cu(BH₄)(C₂₆H₂₀N₂)]
 $M_r = 438.82$
 Monoclinic, Cc
 $a = 14.6291$ (5) Å
 $b = 19.9859$ (7) Å
 $c = 7.5743$ (3) Å
 $\beta = 105.529$ (1)°
 $V = 2133.70$ (13) Å³

$Z = 4$
 $D_x = 1.366$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 1.04$ mm⁻¹
 $T = 90$ (2) K
 Block, orange-red
 0.10 × 0.04 × 0.03 mm

Data collection

Bruker SMART APEX2
 diffractometer
 ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.903$, $T_{\max} = 0.970$

21394 measured reflections
 7938 independent reflections
 7219 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$
 $\theta_{\text{max}} = 33.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.060$
 $S = 1.01$
 7938 reflections
 284 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$w = 1/[\sigma^2(F_o^2) + (0.0227P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.64$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³
 Absolute structure: Flack (1983),
 3922 Friedel pairs
 Flack parameter: 0.027 (5)

Table 1

Selected geometric parameters (Å, °).

Cu1—N1	2.025 (1)	Cu1—H2B	1.65 (2)
Cu1—N2	2.038 (1)	Cu1—H1B	1.67 (2)
Cu1—B1	2.115 (1)		
N1—Cu1—N2	81.51 (4)	N2—Cu1—B1	138.86 (5)
N1—Cu1—B1	139.29 (5)		

BH₄⁻ H atoms were located in a difference map and their positions were refined with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{B})$ [B—H = 1.05 (1)–1.18 (1) Å]. The remaining H atoms were positioned geometrically with C—H = 0.95 and 0.98 Å for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl. The methyl group on atom C19 was constrained as an idealized disordered methyl group and occupancy factors for the H atoms were refined to values of 0.40 (2) and 0.60 (2).

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *SAINTE-Plus* (Bruker, 2004); program(s) used to solve structure: *XS* in *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *XL* in *SHELXTL*; molecular graphics: *XP* in *SHELXTL*; software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004).

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