

Jan Moncol,\* Milan Gembicky  
and Philip CoppensDepartment of Chemistry, State University of  
New York at Buffalo, NY 14260-3000, USA

Correspondence e-mail: moncol@buffalo.edu

## Key indicators

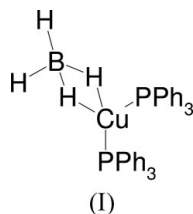
Single-crystal X-ray study  
 $T = 90$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
 $R$  factor = 0.023  
 $wR$  factor = 0.064  
Data-to-parameter ratio = 22.9For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.(Tetrahydroborato)bis(triphenylphosphine)copper(I):  
a redetermination at 90 K

The structure of the title compound,  $[\text{Cu}(\text{PPh}_3)_2(\text{BH}_4)]$ , has been redetermined at 90 K. The geometric parameters of the present structure agree with those previously studied at room temperature [Lippard & Melmed (1967). *Inorg. Chem.* **6**, 2223–2228; Li *et al.* (2000). *Chin. J. Struct. Chem.* **19**, 91–94], but with significantly improved precision. The molecule has twofold rotation symmetry.

Received 4 November 2004  
Accepted 22 November 2004  
Online 8 January 2005

## Comment

The crystal structure of the title compound, (I), has been previously studied at room temperature (Lippard & Melmed, 1967; Li *et al.*, 2000). In the present redetermination, data were collected at low temperature, using a diffractometer equipped with a new APEX2 CCD area detector. As shown in Fig. 1, the structure of (I) is composed of neutral  $[\text{Cu}(\text{PPh}_3)_2(\text{BH}_4)]$  complex molecules with crystallographic twofold rotation symmetry. The results show that the coordination around  $\text{Cu}^{\text{I}}$  is distorted tetrahedral, with two triphenylphosphine P atoms [ $\text{Cu}-\text{P1} = 2.2780$  (3) Å] and two H atoms of the bidentate tetrahydroborate anion [ $\text{Cu}-\text{H1B} = 1.79$  (2) Å], as in the pyridine solvate  $[\text{Cu}(\text{PPh}_3)_2(\text{BH}_4)]\cdot\text{pyridine}$  (Engelhardt *et al.*, 1985). The geometric parameters of the present structure agree with those previously reported by Lippard & Melmed (1967) and Li *et al.* (2000), but with significantly improved precision. The precision of the  $\text{Cu}-\text{P1}$  bond length (0.0003 Å), the  $\text{P}-\text{C}$  bond lengths (mean 0.001 Å) and the  $\text{C}-\text{C}$  bond lengths (mean 0.0016 Å) for the low-temperature study are better than the room-temperature results reported by Lippard & Melmed (0.001, 0.005 and 0.008 Å, respectively) and Li *et al.* (0.001, 0.004 and 0.006 Å, respectively). Similarly, the standard uncertainty for the  $\text{P1}-\text{Cu}-\text{P1}$  bond angle for the low-temperature data is  $0.001^\circ$ , whereas for both the room-temperature structures it is  $0.006^\circ$ .



## Experimental

$[\text{Cu}(\text{PPh}_3)_2(\text{BH}_4)]$  was prepared by a modification of the procedure reported by Fleet & Harding (1979). Finely powdered copper(I) chloride (0.40 g, 4 mmol) was added to a stirred solution of tri-

phenylphosphine (2.16 g, 8.2 mmol) in chloroform (10 ml) over a period of five minutes. The reaction mixture was stirred until the copper chloride had dissolved (30 min), treated with a solution of sodium tetrahydroborate (0.15 g, 4 mmol) in ethanol (3 ml), stirred for one hour, and then added to water (3 ml). The chloroform layer was then washed with water (3 ml), dried (magnesium sulfate) and filtrated. Crystals were grown by slowly diffusing diethyl ether vapor into a solution of  $[\text{Cu}(\text{PPh}_3)_2(\text{BH}_4)]$  in chloroform.

#### Crystal data

$[\text{Cu}(\text{P}(\text{C}_6\text{H}_5)_3)_2(\text{BH}_4)]$   
 $M_r = 602.92$   
 Monoclinic,  $C2/c$   
 $a = 24.260$  (1) Å  
 $b = 9.0818$  (5) Å  
 $c = 15.0164$  (8) Å  
 $\beta = 115.516$  (1)°  
 $V = 2985.8$  (3) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.341$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 6488 reflections  
 $\theta = 2.4$ – $30.0^\circ$   
 $\mu = 0.86$  mm<sup>-1</sup>  
 $T = 90$  (1) K  
 Needle, colorless  
 $0.25 \times 0.05 \times 0.04$  mm

#### Data collection

Bruker SMART APEX2 diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.813$ ,  $T_{\max} = 0.966$   
 25 523 measured reflections

4344 independent reflections  
 4037 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.021$   
 $\theta_{\text{max}} = 30.0^\circ$   
 $h = -34 \rightarrow 34$   
 $k = -12 \rightarrow 12$   
 $l = -21 \rightarrow 21$

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.023$   
 $wR(F^2) = 0.064$   
 $S = 1.02$   
 4344 reflections  
 190 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0376P)^2 + 2.2722P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.002$   
 $\Delta\rho_{\text{max}} = 0.47$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.27$  e Å<sup>-3</sup>

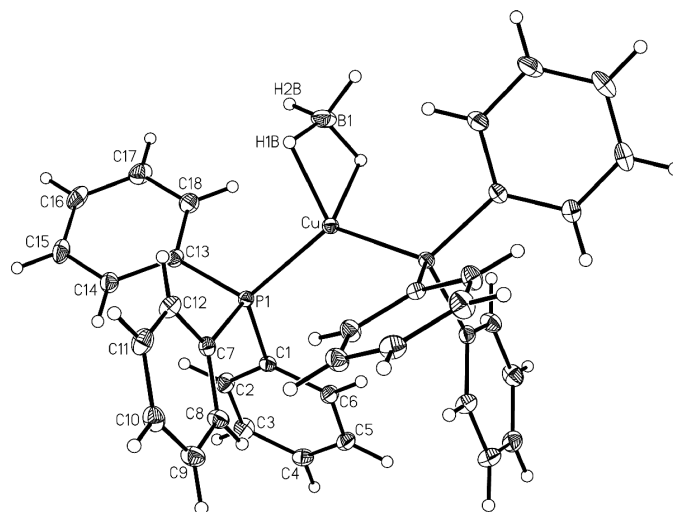
**Table 1**

Selected geometric parameters (Å, °).

Cu–P1	2.2780 (3)	Cu–H1B	1.79 (2)
P1–Cu–P1 <sup>i</sup>	122.99 (1)	P1 <sup>i</sup> –Cu–H1B	122.5 (6)
P1–Cu–H1B	106.0 (6)		

Symmetry code: (i)  $2 - x, y, \frac{3}{2} - z$ .

H atoms of the tetrahydroborate anion were located in a difference Fourier map and refined isotropically. The H atoms of aromatic rings were placed in calculated positions and were treated as riding on their parent atoms, with C–H = 0.95 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{iso}}(\text{parent atom})$ .



**Figure 1**

The structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. Unlabeled atoms are related to labeled atoms by the symmetry operation  $(2 - x, y, \frac{3}{2} - z)$ .

Data collection: APEX2 (Bruker, 2004); cell refinement: APEX2; data reduction: SAINT-Plus (Bruker, 2004); program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: XL in SHELXTL (Sheldrick, 2000); molecular graphics: XP in SHELXTL; software used to prepare material for publication: enCIFer (Allen *et al.*, 2004).

Financial support of this work by the National Science Foundation (CHE9981864 and CHE0236317) is gratefully acknowledged.

#### References

- Allen, F. H., Johnson, O., Shields, G. P., Smith, B. R. & Towler, M. (2004). *J. Appl. Cryst.* **37**, 335–338.
- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Bruker (2004). APEX2 and SAINT-Plus. Bruker AXS Inc., Madison, Wisconsin, USA.
- Engelhardt, L. M., Pakawatchai, C., White, A. H. & Healy, P. C. (1985). *J. Chem. Soc. Dalton Trans.* pp. 125–133.
- Fleet, G. W. J. & Harding, P. J. C. (1979). *Tetrahedron Lett.* pp. 975–978.
- Li, C.-Y., Zhang, Y.-G., Zhou, Y. & Zhang, L.-F. (2000). *Chin. J. Struct. Chem.* **19**, 91–94.
- Lippard, S. J. & Melmed, K. M. (1967). *Inorg. Chem.* **6**, 2223–2228.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (2000). SHELXTL. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.