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**DOI**

[10.1515/znb-2017-0078](https://doi.org/10.1515/znb-2017-0078)

**Publication date**

2017

**Document Version**

Final published version

**Published in**

Zeitschrift für Naturforschung. B, A Journal of Chemical Sciences

**License**

Article 25fa Dutch Copyright Act

[Link to publication](#)

**Citation for published version (APA):**

Boom, D. H. A., Ehlers, A. W., Nieger, M., & Slootweg, J. C. (2017). Coordination of the ambiphilic phosphinoborane  $t\text{Bu}_2\text{PCH}_2\text{BPh}_2$  to  $\text{Cu}(\text{I})\text{Cl}$ . *Zeitschrift für Naturforschung. B, A Journal of Chemical Sciences*, *72*(11), 781-784. <https://doi.org/10.1515/znb-2017-0078>

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Devin H.A. Boom, Andreas W. Ehlers, Martin Nieger and J. Chris Sloatweg\*

# Coordination of the ambiphilic phosphinoborane $t\text{Bu}_2\text{PCH}_2\text{BPh}_2$ to $\text{Cu(I)Cl}$

<https://doi.org/10.1515/znb-2017-0078>

Received May 10, 2017; accepted June 2, 2017

**Abstract:** A chloro-bridged dimeric copper(I) complex with the ambiphilic phosphinoborane ligand  $t\text{Bu}_2\text{PCH}_2\text{BPh}_2$  is reported. The molecular structure was determined by single-crystal X-ray diffraction analysis, revealing a secondary  $\eta^1\text{-C}$  interaction of the ligand with the metal center. The complex exhibits green fluorescence when exposed to UV light at 366 nm.

**Keywords:** ambiphilic ligand; copper chloride; luminescence; MLCT; phosphinoborane.

**Dedicated to:** Professor Dietrich Gudat on the occasion of his 60<sup>th</sup> birthday.

## 1 Introduction

Ambiphilic ligands are donor acceptor ligands bearing a Lewis basic site capable of  $\sigma$  donation and a  $\sigma$ -accepting Lewis-acidic moiety, which provide a rich coordination chemistry with unique  $\text{M}\rightarrow\text{L}$  interactions [1, 2]. The variety of these Z-type ligands is dominated by phosphinoborane ligands, ranging from bidentate donor acceptors (DA) to tridentate ( $\text{D}_2\text{A}$ ) and tetradentate ( $\text{D}_3\text{A}$ ) systems [3]. The coordination chemistry of these ambiphilic ligands towards the coinage metals is well studied, in particular  $\text{Cu(I)Cl}$ , displaying a variety of Z-type interactions.

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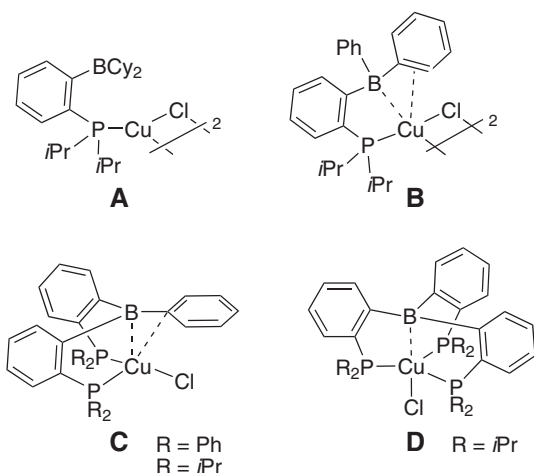
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Bouhadir, Bourissou and co-workers reported ligand **A** (Fig. 1) to form a chloro-bridged dimeric structure with  $\text{Cu(I)Cl}$ , which does not contain any Z-type interaction [4]. Interestingly, changing the cyclohexyl groups on boron for phenyl groups led to complex **B**, which shows  $\eta^3\text{-BCC}$  coordination of the  $\text{BPh}$  moiety to the copper center in the solid state. Switching to a tridentate, diphosphinoborane ligand, bearing phenyl and *iso*-propyl substituents on phosphorus, gave the monomeric  $\text{Cu(I)Cl}$  complexes **C** that display a unique  $\eta^2\text{-BC}$  coordination mode with a significantly shorter  $\text{Cu}\text{--}\text{B}$  distance [ $\text{Cu}\text{--}\text{B}$  2.396(5) ( $\text{R}=\text{Ph}$ ), 2.379(5) ( $\text{R}=\text{iPr}$ ) Å], indicating a stronger interaction compared to the one in complex **B** [ $\text{Cu}\text{--}\text{B}$  2.555(2) Å] [4]. Such a  $\text{Cu}\rightarrow\text{B}$  interaction was also observed for triphosphinoborane complex **D** [2.508(2) Å] in which the metal center is pentacoordinate and adopts a trigonal-bipyramidal geometry [5].

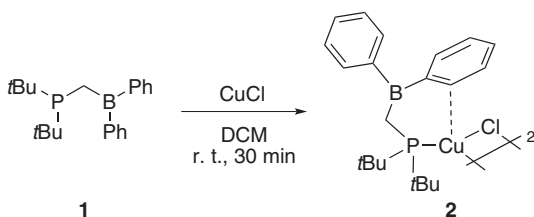
Recently, we developed the phosphinoborane  $t\text{Bu}_2\text{PCH}_2\text{BPh}_2$  (**1**), bearing a methylene linker between the donor and acceptor sites (Scheme 1), which displays frustrated Lewis pair reactivity [6–8] towards small molecules, such as  $\text{H}_2$ ,  $\text{CO}_2$ , and isocyanates [9]. The presence of a strongly donating bis(*tert*-butyl)phosphine moiety and a Lewis acidic borane akin to the ambiphilic ligands of Bourissou et al. prompted us to explore the coordination chemistry of **1** toward copper(I) chloride.

## 2 Results and discussion

Treatment of a suspension of  $\text{Cu(I)Cl}$  in dichloromethane (DCM) with 1 equiv. of  $t\text{Bu}_2\text{PCH}_2\text{BPh}_2$  (**1**;  $\delta^{31\text{P}\{^1\text{H}\}}=39.4$  ppm) [9] at room temperature (in the absence of light) gave after 30 min a yellow solution, which afforded after work-up complex **2** in 65% isolated yield ( $\delta^{31\text{P}\{^1\text{H}\}}=53.6$  ppm; Scheme 1). The  $^{11\text{B}\{^1\text{H}\}}$  NMR chemical shift of **2** at 70.7 ppm is diagnostic for tricoordinate diarylalkylboranes [4], and is only shifted 1.6 ppm from the free ligand (72.3 ppm) [9], indicating the absence of any  $\text{Cu}\rightarrow\text{B}$  interaction. At room temperature, only three aromatic signals for the  $\text{BPh}_2$  group were observed in the  $^1\text{H}$  NMR spectrum of **2** and the  $^{13\text{C}}$  NMR signal for the *ortho*-carbons ( $\delta^{13\text{C}\{^1\text{H}\}}(\text{CD}_2\text{Cl}_2)=136.2$  ppm) was observed in the same range as for the free ligand ( $\delta^{13\text{C}\{^1\text{H}\}}(\text{C}_6\text{D}_6)=136.8$  ppm) [9], indicating that both phenyl



**Fig. 1:** Examples of  $\text{Cu}(\text{I})\text{Cl}$  complexes with phosphinoborane-based amphiphilic ligands.

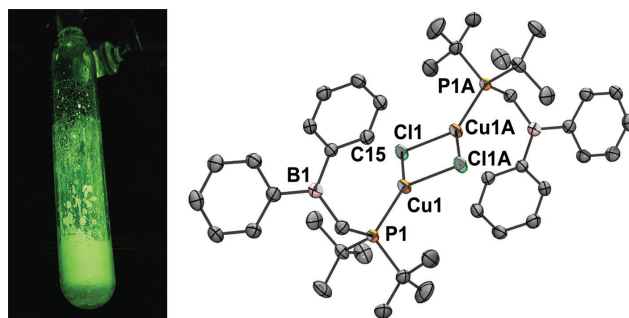


**Scheme 1:** Synthesis of dimeric copper complex **2**.

groups rapidly exchange in solution at the NMR time scale, akin to what was reported for complex **B** (Fig. 1) [4].

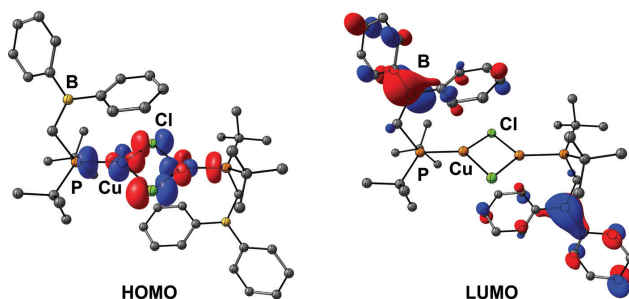
The structure of **2** in the solid state was established by a single-crystal X-ray diffraction analysis of green crystals that were obtained by vapor diffusion of *n*-pentane into a solution of **2** in DCM at room temperature. The molecular structure of **2** revealed a chloro-bridged dimeric complex (Fig. 2, right) with crystallographic  $C_i$  symmetry and a Cu–P distance of 2.1887(5) Å, which is comparable to the Cu–P distances of complexes **A** and **B** (2.173(2) and 2.215(1) Å, respectively; Fig. 1). The Cu–B distance of 3.828(2) Å just exceeds the sum of the van der Waals radii (3.80 Å) [10] making any Cu→B interaction negligible, which is in agreement with the planar environment of the boron center ( $\Sigma \text{C–B–C} = 359.9^\circ$ ). Interestingly, the distance between the copper center Cu1 and the *ortho*-phenyl carbon C15 is 2.876(2) Å, suggesting an  $\eta^1\text{-C}$  interaction of the *ortho*-phenyl carbon with the copper center in the solid state.

Interestingly, complex **2** shows green luminescent properties in the solid phase upon UV irradiation at 366 nm (Fig. 2, left). This photophysical behavior is known for  $\text{Cu}(\text{I})\text{Cl}$  complexes bearing arylphosphine ligands



**Fig. 2:** (Left) Photograph of the green fluorescence emission under UV irradiation at 366 nm. (Right) Molecular structure of **2** in the crystal (ellipsoids are set at 50% probability). Selected bond lengths (Å) and angles (deg): Cu1–Cu1A 3.0281(5), Cu1–Cl1 2.2906(5), Cu1–Cl1A 2.3337(5), Cu1–P1 2.1887(5), Cu1–B1 3.828(2), Cu1–C15 2.876(2); P1–Cu1–Cl1 131.86(2), P1–Cu1–Cl1A 128.34(2), Cu1–Cl1–Cu1A 81.81(2), Cl1–Cu1–Cl1A 98.19(2). Symmetry code: (A)  $-x+1, -y, -z+1$ .

[11–13], but to our knowledge has never been reported for  $\text{Cu}(\text{I})\text{Cl}$  complexes with amphiphilic ligands. For the reported arylphosphine  $\text{Cu}(\text{I})\text{X}$  complexes, emission of light occurs after metal-to-ligand charge transfer (MLCT) mixed with halide-to-ligand charge transfer (XLCT), where a metal *d* electron or halide *p* electron is excited into a delocalized  $\pi^*$  orbital of the arylphosphine ligand. To gain more insight in the luminescent properties of alkylphosphine complex **2**, we resorted to DFT calculations at the  $\omega\text{B97X-D}/6\text{-31G}^*$  (Def2-QZVP for Cu) level of theory [14]. These calculations have revealed that also for **2** the HOMO (–6.79 eV) is located at the dimeric copper(I)chloride core (Fig. 3, left), similar to the luminescent dimeric  $\text{Cu}(\text{I})$  P-aryl systems [11–13]. The LUMO (0.20 eV) in complex **2**, however, is a localized empty *p* orbital on boron (Fig. 3, right), whereas the B-aryl  $\pi^*$  orbitals correspond to the higher lying LUMO –2 and LUMO –3 (1.74 eV and 1.75 eV, respectively). These HOMO and LUMO calculations indicate that the lowest excited state of **2** is to be attributed to the transition of an electron from the dimeric  $\text{Cu}(\text{I})\text{Cl}$  core



**Fig. 3:** Plots of the HOMO (left) and LUMO (right) of **2** calculated at the  $\omega\text{B97X-D}/6\text{-31G}^*$  (Def2-QZVP for Cu) level of theory. Hydrogens are omitted for clarity.

to the empty  $p$  orbital on boron, which is different from the MLCT transition found in the arylphosphine  $\text{Cu}(\text{I})\text{Cl}$  complexes where an empty antibonding  $\pi^*$  orbital of the ligand takes part [11–13].

### 3 Conclusion

We have shown that the methylene-bridged phosphinoborane **1** coordinates to  $\text{Cu}(\text{I})\text{Cl}$ , forming a dimeric species with an  $\eta^1\text{-C}$  interaction of the *ortho*-phenyl carbon with the copper center, which possesses luminescent properties in the solid state. These results emphasize the potential of **1** as an ambiphilic ligand for the synthesis of unique coordination complexes with photophysical properties, which we are currently exploring in our laboratories.

## 4 Experimental section

### 4.1 General considerations

All manipulations were carried out under an atmosphere of dry nitrogen, using standard Schlenk and drybox techniques, and were performed in the dark. Solvents were purified, dried and degassed according to standard procedures.

$^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  NMR spectra were recorded on a Bruker Avance 400 and internally referenced to the residual solvent resonances ( $\text{CD}_2\text{Cl}_2$ :  $^1\text{H}$   $\delta=5.32$  ppm,  $^{13}\text{C}\{^1\text{H}\}$   $\delta=53.8$  ppm).  $^{31}\text{P}\{^1\text{H}\}$  and  $^{11}\text{B}\{^1\text{H}\}$  NMR spectra were recorded on a Bruker Avance 400 and externally referenced (85%  $\text{H}_3\text{PO}_4$ ,  $\text{BF}_3 \cdot \text{OEt}_2$ , respectively). The melting point was measured on a sample in a sealed capillary and is uncorrected.

Mass spectra were collected on an AccuTOF GC v 4g, JMS-T100GCV Mass spectrometer (JEOL, Japan). FD Emitter, Carbotec or Linden (Germany), FD 10  $\mu\text{m}$ . Current rate 51.2 mA  $\text{min}^{-1}$  over 1.2 min. Typical measurement conditions are: Counter electrode  $-10$  kV, Ion source 37 V.

$t\text{Bu}_2\text{PCH}_2\text{BPh}_2$  (**1**) was prepared following a literature procedure [9] and  $\text{Cu}(\text{I})\text{Cl}$   $\geq 99\%$  was purchased from Sigma-Aldrich and used without any further purification.

### 4.2 Preparation of complex 2

A solution of  $t\text{Bu}_2\text{PCH}_2\text{BPh}_2$  (**1**; 0.193 g, 0.60 mmol, 1.0 equiv.) in DCM (4 mL) was added to a suspension of

$\text{Cu}(\text{I})\text{Cl}$  (0.059 g, 0.60 mmol, 1.0 equiv.) in DCM (6 mL) at room temperature. The reaction mixture was stirred for 30 min at room temperature after which a yellow solution was obtained. Next, the solution was filtered and the solvent was removed *in vacuo* to yield a pale yellow-green solid, which was washed with pentane ( $3 \times 5$  mL) and dried *in vacuo* to yield a pale yellow-green powder (0.164 g, 65%). X-ray quality crystals were grown at room temperature by vapor diffusion of *n*-pentane into a solution of **2** in DCM. Melting point (nitrogen, sealed capillary): 117°C (decomp.). –  $^1\text{H}$  NMR (400.1 MHz,  $\text{CD}_2\text{Cl}_2$ , 297 K):  $\delta=7.75$  (d,  $^3J_{\text{H-H}}=6.9$  Hz, 4H; *o*-PhH), 7.59 (t,  $^3J_{\text{H-H}}=7.3$  Hz, 2H; *p*-PhH), 7.52 (t,  $^3J_{\text{H-H}}=7.2$  Hz, 4H; *m*-PhH), 2.36 (d,  $^2J_{\text{H-P}}=13.9$  Hz, 2H;  $\text{PCH}_2\text{B}$ ), 1.31 (d,  $^3J_{\text{H-P}}=14.2$  Hz, 18H;  $\text{PC}(\text{CH}_3)_3$ ). –  $^{13}\text{C}\{^1\text{H}\}$  NMR (100.6 MHz,  $\text{CD}_2\text{Cl}_2$ , 296 K):  $\delta=141.4$  (only observed in the HMBC spectrum,  $^2J_{\text{C-H}}$  coupling with *o*-PhH,  $^3J_{\text{C-H}}$  coupling with *m*-PhH and  $\text{PCH}_2\text{B}$ ; *ipso*-PhC), 136.2 (s; *o*-PhC), 132.6 (s; *p*-PhC), 129.1 (s; *m*-PhC), 34.2 (d,  $^1J_{\text{C-P}}=17.3$  Hz;  $\text{PC}(\text{CH}_3)_3$ ), 29.9 (d,  $^2J_{\text{C-P}}=7.3$  Hz;  $\text{PC}(\text{CH}_3)_3$ ), 16.6 (d,  $^1J_{\text{C-P}} \approx -9$  Hz;  $\text{PCH}_2\text{B}$ ; observed in the HSQC spectrum). –  $^{31}\text{P}\{^1\text{H}\}$  NMR (162.0 MHz,  $\text{CD}_2\text{Cl}_2$ , 297 K):  $\delta=53.6$  (br. s). –  $^{11}\text{B}\{^1\text{H}\}$  NMR (128.4 MHz,  $\text{CD}_2\text{Cl}_2$ , 297 K):  $\delta=70.7$  (br. s). – HRMS (FD):  $m/z=844.2319$  (calcd. 844.2325 for  $\text{C}_{42}\text{H}_{60}\text{B}_2\text{P}_2\text{Cl}_2\text{Cu}_2$ ,  $[\text{M}]^+$ ), 809.2682 (calcd. 809.2637 for  $\text{C}_{42}\text{H}_{60}\text{B}_2\text{P}_2\text{Cl}_1\text{Cu}_2$ ,  $[\text{M}-\text{Cl}]^+$ ).

**Table 1:** Crystal structure data for **2**.

Formula	$\text{C}_{42}\text{H}_{60}\text{B}_2\text{Cl}_2\text{Cu}_2\text{P}_2$
$M_r$	846.44
Cryst. size, mm <sup>3</sup>	0.32 × 0.24 × 0.16
Crystal system	Triclinic
Space group	$P\bar{1}$ (no. 2)
$a$ , Å	9.0624(7)
$b$ , Å	9.1088(8)
$c$ , Å	14.4241(13)
$\alpha$ , deg	79.065(7)
$\beta$ , deg	75.929(7)
$\gamma$ , deg	66.417(7)
$V$ , Å <sup>3</sup>	1052.75(17)
$Z$	2
$D_{\text{calcd}}$ , g cm <sup>-3</sup>	1.34
$\mu(\text{MoK}\alpha)$ , mm <sup>-1</sup>	1.2
$F(000)$ , e	444
$hkl$ range	$-7 \leq h \leq +12$ $-12 \leq k \leq +12$ $-20 \leq l \leq +19$
$2\theta_{\text{max}}$ , deg	60
Refl. measured/unique/ $R_{\text{int}}$	8522/5244/0.026
Param. refined	226
$R1$ (for 4551 $I > 2 \sigma(I)$ )	0.033
$wR(I)$ (all refl.)	0.085
GoF ( $I$ )	1.05
$\Delta\rho_{\text{fin}}$ (max/min), e Å <sup>-3</sup>	0.35/−0.38

### 4.3 X-ray structure determination of 2

Data were collected on an Agilent Super Nova diffractometer with EOS CCD-detector using graphite-monochromatized  $\text{MoK}\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ) at  $T = -100^\circ\text{C}$ . The structure was solved by Direct Methods and refined by full-matrix least-squares on  $F^2$  [15, 16]. A semi-empirical absorption correction was applied. All non-hydrogen atoms were refined anisotropically, and hydrogen atoms were located from difference Fourier maps and refined at idealized positions using a riding model. For details, see Table 1.

CCDC 1548516 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

## 5 Supporting information

1D and 2D NMR spectra as well as the Cartesian coordinates for complex 2 are given as Supporting Information available online (DOI: 10.1515/znb-2017-0078).

**Acknowledgment:** This work was supported by the Council for Chemical Sciences of The Netherlands Organization for Scientific Research (NWO/CW) by a VIDI grant (J.C.S.) and benefitted from interactions within the COST Action CM1205 CARISMA (Catalytic Routines for Small Molecule Activation). We thank Ed Zuidinga for the exact mass determination and Dr. Ferial Rekhroukh for proofreading this manuscript.

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**Supplemental Material:** The online version of this article offers supplementary material (<https://doi.org/10.1515/znb-2017-0078>).



## Graphical synopsis

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Z. Naturforsch. 2017; x(x)b: xxx–xxx

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