

4,4'-Bipyridylum di- $\mu$ -chlorotetrachlorodocuprate(II):  
a redeterminationChristian Näther,<sup>a</sup> Inke Jeß<sup>a</sup> and  
Michael Bolte<sup>b\*</sup><sup>a</sup>Institut für Anorganische Chemie, der Universität Kiel, Olshausener Straße 40, 24098 Kiel, Germany, and <sup>b</sup>Institut für Organische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, GermanyCorrespondence e-mail:  
bolte@chemie.uni-frankfurt.de

## Key indicators

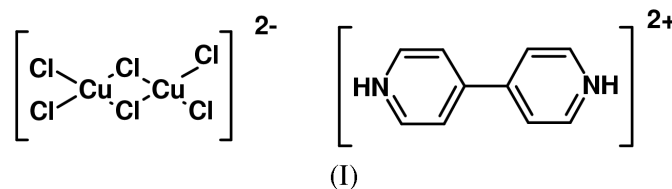
Single-crystal X-ray study  
 $T = 293\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$   
 $R$  factor = 0.028  
 $wR$  factor = 0.068  
Data-to-parameter ratio = 24.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The structure of the title compound,  $(\text{C}_{10}\text{H}_{10}\text{N}_2)[\text{Cu}_2\text{Cl}_6]$ , previously determined by Bukowska-Strzyzewska & Tosik [*Pol. J. Chem.* (1979), **53**, 2423–2428], has been reinvestigated. Our structure determination is of a significantly higher precision and, in contrast to the previous investigation, H atoms could be clearly found. Thus, it can be shown that the chains of alternating bipyridylum cations and  $\text{Cu}_2\text{Cl}_6^{2-}$  anions are stabilized by bifurcated hydrogen bonds. The cation and anion both lie on inversion centres.

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## Comment

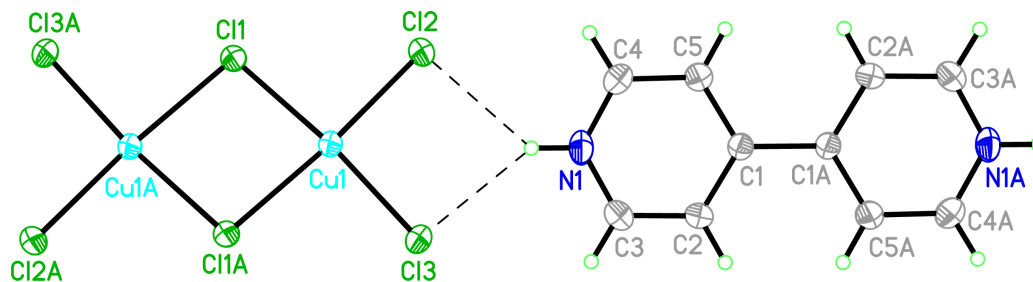
The structure determination of the title compound, (I), was undertaken within a project on the preparation and structural investigation of new coordination polymers based on copper(I) halides and aromatic bases. Our results are of a significantly higher precision than the previously determined structure (Bukowska-Strzyzewska & Tosik, 1979) and we have been able to locate all H atoms.



The title compound crystallizes in parallel sheets which are composed of rows of alternating bipyridyl cations and  $\text{Cu}_2\text{Cl}_6^{2-}$  anions. These rows run approximately along the  $[\bar{1}45]$  vector and are stabilized by bifurcated  $\text{N}-\text{H}\cdots\text{Cl}$  hydrogen bonds (Fig. 1). The cation and anion are located on crystallographic centres of inversion and, as a consequence of that, the pyridyl rings are coplanar. Bond lengths and angles are in the usual ranges compared with structures retrieved from the Cambridge Structural Database (Version 5.20 of October 2000; Allen & Kennard, 1993). The bonds between the terminal Cl atoms and the Cu atoms are significantly shorter than the bridging Cu–Cl bonds.

## Experimental

The title compound was prepared by the reaction of  $\text{Cu}_2\text{I}_2$ –4,4'-bipyridine [freshly prepared by the reaction of 4,4'-bipyridine and copper(I) iodide] with an excess of diluted hydrochloric acid. After one day standing at room temperature, light orange needles had formed in 60% yield. The homogeneity was checked by X-ray powder diffraction.



**Figure 1**

A perspective view of the title compound with the atom-numbering scheme. Atoms labelled *A* are centrosymmetrically related to the others. Displacement ellipsoids are at the 50% probability level.

#### Crystal data

(C<sub>10</sub>H<sub>10</sub>N<sub>2</sub>)[Cu<sub>2</sub>Cl<sub>6</sub>]  
*M<sub>r</sub>* = 497.98  
 Triclinic, *P* $\bar{1}$   
*a* = 3.9158 (8) Å  
*b* = 10.021 (2) Å  
*c* = 10.698 (2) Å  
 $\alpha$  = 113.92 (3)°  
 $\beta$  = 95.16 (3)°  
 $\gamma$  = 96.06 (3)°  
*V* = 377.51 (13) Å<sup>3</sup>

*Z* = 1  
*D<sub>x</sub>* = 2.190 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 70 reflections  
 $\theta$  = 11–19°  
 $\mu$  = 3.86 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Needle, light orange  
 0.15 × 0.07 × 0.07 mm

#### Data collection

Phillips PW-1100 four-circle diffractometer  
 $\omega/\theta$  scans  
 Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 1998)  
 $T_{\min}$  = 0.549,  $T_{\max}$  = 0.599  
 2500 measured reflections  
 2205 independent reflections  
 1716 reflections with  $I > 2\sigma(I)$

$R_{\text{int}}$  = 0.018  
 $\theta_{\text{max}}$  = 30.0°  
 $h = 0 \rightarrow 5$   
 $k = -14 \rightarrow 14$   
 $l = -14 \rightarrow 14$   
 4 standard reflections  
 frequency: 120 min  
 intensity decay: none

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.028$   
 $wR(F^2) = 0.068$   
 $S = 1.03$   
 2205 reflections  
 92 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.023P)^2 + 0.415P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.53 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.46 \text{ e \AA}^{-3}$   
 Extinction correction: *SHELXL97*  
 Extinction coefficient: 0.0084 (12)

**Table 1**

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1N···Cl2 <sup>i</sup>	0.86	2.54	3.236 (2)	138
N1—H1N···Cl3 <sup>i</sup>	0.86	2.49	3.198 (2)	140

Symmetry code: (i) 1 + *x*, *y*, *z*.

All H atoms were located by difference Fourier synthesis and refined with fixed individual displacement parameters [ $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $U(\text{H}) = 1.2U_{\text{eq}}(\text{N})$ ] using a riding model with aromatic C—H = 0.93 Å or N—H = 0.86 Å.

Data collection and cell refinement: *DIF4* (Stoe & Cie, 1992); data reduction: *REDU4* (Stoe & Cie, 1992); structure solution: *SHELXS97* (Sheldrick, 1990); structure refinement: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *CIFTAB* in *SHELXL97*.

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