

# The Crystal Structure of Tetrapyridine Copper(I) Perchlorate and Tetrapyridine Silver(I) Perchlorate at 260 K

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The crystal structures of  $[M(C_5H_5N)_4]ClO_4$ , where  $M = Cu$  or  $Ag$ , have been determined from X-ray intensity data collected at 260 K with a CAD-4 diffractometer. The compounds are isostructural. The space group is  $I\bar{4}$  with  $Z=2$ ;  $a=12.471(3)$ ,  $c=6.894(2)$  for the Cu and  $a=12.874(1)$ ,  $c=6.748(4)$  Å for the Ag compound. The refinements converged to  $R=0.040$  (Cu) and  $R=0.026$  (Ag). The structure is composed of discrete ions  $[M(C_5H_5N)_4]^+$  and  $ClO_4^-$ . The coordination geometry is close to tetrahedral with metal–nitrogen distances 2.046(4) (Cu) and 2.322(3) (Ag). The difference between the distances Ag–N and Cu–N is larger than that found between the distances Ag–P and Cu–P in complexes formed by ligands coordinating *via* phosphorous. This indicates that the difference in covalency is larger between the bonds Ag–P and Cu–P than between the bonds Ag–N and Cu–N.

The crystal structure determination of  $[Cu(C_5H_5N)_4]ClO_4$  and  $[Ag(C_5H_5N)_4]ClO_4$  was undertaken as part of a research programme that deals with complexes formed in pyridine solution. It is of interest to find correlations between thermodynamic parameters, such as the enthalpy and entropy of complex formation in solution, and structural properties such as coordination geometries and steric effects. The latter can be established in the solid state by diffraction methods.

Since the silver compound slowly loses pyridine at room temperature the data collection was made at 260 K. Tetrapyridine copper(I) perchlorate has previously been investigated at room temperature.<sup>1</sup> We have redetermined the structure at 260 K.

## EXPERIMENTAL

**Preparation of crystals.**  $[Cu(C_5H_5N)_4]ClO_4$  was prepared by treating  $[Cu(CH_3CN)_4]ClO_4$  with pyridine.<sup>2</sup> The needle-shaped crystals were light yellow with a slight green tint. Copper was determined by EDTA titration (found 13.2 %, calc. 13.3 %).  $[Ag(C_5H_5N)_4]ClO_4$  was prepared by dissolving silver(I) perchlorate in pyridine to a saturated solution. On cooling, colourless crystals were formed. Silver was determined by Volhard titration (found 20.6 %, calc. 20.6 %).

**X-Ray data collection.** Weissenberg photographs for the Ag-compound taken at 260 K revealed the Laue class  $4/m$  and the systematic extinctions  $hkl$ ,  $h+k+l \neq 2n$  consistent with the space groups  $I\bar{4}$ ,  $I\bar{4}$  and  $I4/m$ . For both compounds, cell dimensions were obtained at 260 K by least squares calculations from  $\theta$ -values determined as  $\theta_{hkl} = (\omega_{hkl} - \omega_{\bar{h}\bar{k}\bar{l}})/2$ , (Table 1). A CAD-4 diffractometer in the bisecting mode, measuring  $\omega_{\bar{h}\bar{k}\bar{l}}$  at negative  $\theta$  angle, was used. The cooling device is described previously.<sup>3</sup>

The intensity data sets were collected at 260 K with the same diffractometer. Zr-filtered  $MoK\alpha$  radiation was used and reflexions  $-17 \leq h \leq 17$ ,  $-17 \leq k \leq 17$ ,  $0 \leq l \leq 8$  were measured for both compounds. During the data collections three standard reflexions were measured at regular intervals. No systematic variation in their intensities or in the orientation of the crystals was found. Information concerning the collection and reduction of the data sets and the refinements of the crystal structures is given in Table 1. The values of  $I$  and  $\sigma_c(I)$  were corrected for Lorentz, polarization and absorption effects, the latter by numerical integration. The values of  $\sigma_c(I)$  are based on counting statistics. Reflexions with  $I \leq 2.5 \sigma_c(I)$  were considered insignificantly different from the background and excluded from all subsequent calculations. The

Table 1. Crystal data, collection and reduction of intensity data and least-squares refinement.

	[Cu(C <sub>5</sub> H <sub>5</sub> N) <sub>4</sub> ]ClO <sub>4</sub>	[Ag(C <sub>5</sub> H <sub>5</sub> N) <sub>4</sub> ]ClO <sub>4</sub>
Crystal size (mm)	0.16 × 0.19 × 0.25	0.64 × 0.36 × 0.25
<i>a</i> (Å)	12.471(3)	12.874(1)
<i>c</i> (Å)	6.894(2)	6.748(4)
<i>V</i> (Å <sup>3</sup> )	1072.2	1118.4
<i>D<sub>x</sub></i> (g cm <sup>-3</sup> )	1.48	1.55
<i>Z</i>		2
$\lambda$ (Å)		0.7093
$\mu$ (cm <sup>-1</sup> )	12.2	10.3
Range of transmission factor	0.749–0.844	0.779–0.799
$\theta$ interval (°)		3–29
$\omega$ –2 $\theta$ scan width, $\Delta\omega$ (°)		1.0+
		0.5 tan $\theta$
$\sigma_c(I)/I$ requested in a scan		0.030
Maximum recording time (s)		120
Number of reflexions measured	1898	2987
Number of reflexions with $I > 2.5\sigma(I)$	1249	1848
Number of independent reflexions with $I > 2.5\sigma(I), m$		539
Number of parameters refined, <i>n</i>	75	75
$R = \Sigma( F_o  -  F_c )/\Sigma F_o $	0.0396	0.0255
$R_w = [\Sigma w( F_o  -  F_c )^2 / \Sigma w F_o ^2]^{1/2}$	0.0469	0.0285
$S = [\Sigma w( F_o  -  F_c )^2 / (m - n)]^{1/2}$	2.8	1.5
<i>C</i> (weighting function)	0.010	0.015

intensities of symmetry dependent reflexions were then averaged.

## STRUCTURE DETERMINATION AND REFINEMENTS

Of the possible space groups only  $I\bar{4}$  gives a reasonable coordination geometry for the metal. The positions of Ag and Cl were deduced from a vector map. The positions of the other non-hydrogen atoms in the silver compound were located in a difference map. The positional and anisotropic thermal parameters were refined by fullmatrix least-squares, minimizing  $\Sigma w(|F_o| - |F_c|)^2$  with weights  $w = [(\sigma_c^2/4|F_o|^2) + (C|F_o|)^2]^{-1}$ . The constant *C* was adjusted so that constant values of  $\langle w(|F_o| - |F_c|)^2 \rangle$  were obtained in different  $|F_o|$  and  $\sin \theta$  intervals. The hydrogen atoms were included in the final cycles of refinement. They were placed geometrically (*C*–*H* = 1.0 Å) and only the isotropic temperature factor was refined for each H-atom. Scattering factors, with corrections for anomalous dispersion, were taken from the International Tables.<sup>4</sup>

The structure of the Cu-compound was refined using the parameters of the Ag-compound as a starting set. This model for the Cu-compound is essentially the same as the one reported by Lewin *et al.*<sup>1</sup>

Details of the refinements are given in Table 1. The final positional and thermal parameters are given in Tables 2 and 3 and Table 4 gives selected interatomic distances and angles. A list of structure factors may be obtained through the authors.

## DESCRIPTION OF THE STRUCTURES

The compounds investigated are isostructural, consisting of discrete  $[M(C_5H_5N)_4]^+$  and  $ClO_4^-$  ions. The crystal packing is shown in Fig. 1. The complexes are stacked in columns along *c* and the shortest metal–metal distance is the vector *c* [*Cu*⋯*Cu* = 6.894(2) and *Ag*⋯*Ag* = 6.748(4) Å]. In this direction the larger silver(I) ion thus gives a more efficient packing of the complexes than the smaller copper(I) ion. The cation, as well as the

Table 2. Atomic coordinates ( $\times 10^4$ ) and thermal parameters  $\beta \times 10^4$  and  $B_{\text{iso}}$  with e.s.d.'s for  $[\text{Cu}(\text{C}_5\text{H}_5\text{N})_4]\text{ClO}_4$ . The form of the temperature factor is  $\exp(-\beta_{11}h^2 - 2\beta_{12}hk - \dots)$ .

	x	y	z	$\beta_{11}, B_{\text{iso}}$	$\beta_{22}$	$\beta_{33}$	$\beta_{12}$	$\beta_{13}$	$\beta_{23}$
Cu	5000	5000	5000	58.5(7)	58.5(7)		230(4)		
N	4258(3)	3892(3)	3271(7)	57(3)	52(3)	207(11)	-2(2)	-4(5)	1(5)
Cl	0	5000	2500	58(1)	58(1)	210(7)			
C(1)	3489(5)	4187(4)	2026(9)	92(5)	58(4)	226(15)	-6(3)	-51(7)	23(6)
C(2)	3004(6)	3485(6)	719(10)	104(6)	100(6)	246(16)	-24(5)	-57(8)	22(9)
C(3)	3339(6)	2431(5)	688(9)	105(6)	85(5)	245(17)	-39(4)	-5(8)	15(7)
C(4)	4109(5)	2120(5)	1974(11)	86(5)	55(4)	316(19)	-9(3)	12(8)	-23(7)
C(5)	4540(4)	2853(4)	3239(9)	56(3)	59(4)	277(15)	3(3)	-10(7)	-1(7)
O	154(7)	4115(6)	1381(13)	195(8)	194(8)	777(35)	7(7)	22(15)	-266(15)
H(1)	3252	4955	2030	2030	6(1)				
H(2)	2431	3753	-171	10(2)					
H(3)	3022	1914	-257	6(2)					
H(4)	4359	1355	1999	10(2)					
H(5)	5089	2598	4206	3(1)					

Table 3. Atomic coordinates ( $\times 10^4$ ) and thermal parameters  $\beta \times 10^4$  and  $B_{\text{iso}}$  with e.s.d.'s for  $[\text{Ag}(\text{C}_5\text{H}_5\text{N})_4]\text{ClO}_4$ . The form of the temperature factor is  $\exp(-\beta_{11}h^2 - 2\beta_{12}hk - \dots)$ .

	x	y	z	$\beta_{11}, B_{\text{iso}}$	$\beta_{22}$	$\beta_{33}$	$\beta_{12}$	$\beta_{13}$	$\beta_{23}$
Ag	5000	5000	5000	75.5(3)	75.5(3)	329(2)			
N	4180(3)	3747(2)	3083(6)	70(2)	61(2)	300(10)	4(2)	-15(4)	2(4)
Cl	0	5000	2500	73(1)	73(1)	232(5)			
C(1)	3460(4)	3990(3)	1760(9)	94(3)	75(3)	403(16)	1(2)	-72(6)	36(6)
C(2)	3094(4)	3288(4)	428(8)	111(4)	117(4)	327(21)	-22(3)	-69(7)	37(7)
C(3)	3479(4)	2285(4)	409(10)	109(3)	103(3)	355(25)	-41(3)	11(7)	-24(7)
C(4)	4188(4)	2036(3)	1799(8)	95(3)	65(3)	375(15)	-7(2)	6(7)	-2(6)
C(5)	4521(3)	2765(3)	3104(7)	73(2)	67(2)	325(13)	3(2)	-23(5)	10(5)
O	230(4)	4144(4)	1311(4)	151(4)	157(4)	599(18)	20(3)	1(8)	-160(8)
H(1)	3130	4700	1910	7(1)					
H(2)	2470	3480	-450	14(3)					
H(3)	3100	1770	-330	9(2)					
H(4)	4480	1310	1830	12(2)					
H(5)	5000	2650	4250	5(1)					

anion, have the symmetry  $\bar{4}$ . The coordination polyhedra around the metal atoms are tetragonal disphenoids but the deviation from a tetrahedron is small for both compounds as shown by the N-M-N angles (Table 4).

The atoms of the pyridine molecule are designated in Fig. 2. The pyridine molecules are almost planar (Table 4) and the metal ions are situated less than 0.4 Å from this plane. Distance and angles within the pyridine molecule are much the same in both compounds and they agree well

with those observed in other pyridine complexes.<sup>5,6</sup> The geometry of the pyridine molecule is not perceptibly influenced by the coordination to a metal ion as judged from a microwave study of pyridine,<sup>7</sup> where the following distances and angles were found: N-C=1.340(1) Å, C-C=1.394(1) and 1.395(1) Å, N-C-C=123.9°, C-N-C=116.8°, C-C-C=118.5 and 118.3°. The smaller C-C distances observed in the solid state are most probably artifacts due to the libration motion of the pyridine molecule.

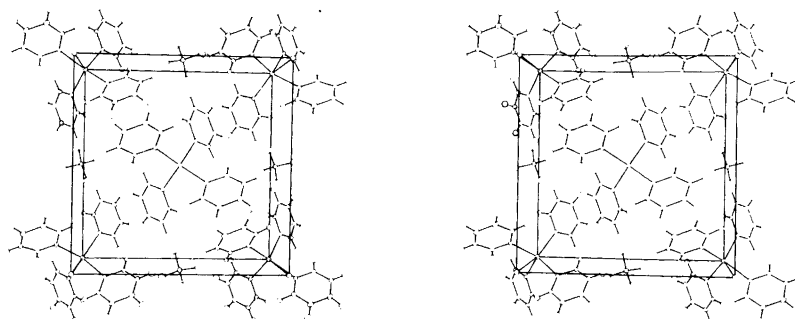


Fig. 1. Stereoscopic view of the unit-cell content.

The perchlorate ion is ordered in both compounds and the geometry is close to tetrahedral (Table 4). The difference observed between the Cl—O distance in the two compounds, 0.034(10) Å, is hardly significant.

## DISCUSSION

The Cu—N and Ag—N distances in  $[M-(C_5H_5N)_4]ClO_4$  are somewhat longer than found in other solvates containing N-donors in a tetrahedral arrangement. In the acetonitrile solvate,<sup>8</sup>  $[Cu(CH_3CN)_4]ClO_4$ , the average Cu—N = 1.99(1) Å. In the polymeric compounds Cu(NC-

Table 4. Selected interatomic distances (Å) and angles (°) and *r.m.s.* deviation (Å) from the least-squares planes through the pyridine rings.

The superscripts (i)–(iv) denote the following transformation applied to *x, y, z* values given in Tables 2 and 3:

(i)  $1-x, 1-y, z$

(ii)  $y, 1-x, 1-z$

(iii)  $1-x, 1-y, z$

(iv)  $1/2-y, 1/2+x, 1/2-z$

	Ag	Cu		Ag	Cu
(a) The coordination polyhedron					
M—N	2.322(3)	2.046(4)	N—M—N <sup>i</sup>	112.3(2)	108.8(3)
N...N <sup>i</sup>	3.856(4)	3.326(5)	N—M—N <sup>ii</sup>	108.1(1)	109.8(1)
N...N <sup>ii</sup>	3.759(4)	3.349(5)			
(b) The pyridine molecule					
N—C(1)	1.324(6)	1.339(8)	N—C(1)—C(2)	122.1(4)	123.5(5)
C(1)—C(2)	1.359(8)	1.395(9)	C(1)—C(2)—C(3)	120.2(5)	118.5(6)
C(2)—C(3)	1.384(8)	1.379(10)	C(2)—C(3)—C(4)	117.3(5)	118.3(6)
C(3)—C(4)	1.348(8)	1.363(10)	C(3)—C(4)—C(5)	120.1(4)	120.0(6)
C(4)—C(5)	1.356(7)	1.373(9)	C(4)—C(5)—N	122.9(4)	123.4(5)
C(5)—N	1.339(5)	1.342(7)	C(5)—N—C(1)	117.4(4)	116.3(5)
<i>R.m.s. dev</i>	0.0093	0.0085			
(c) The perchlorate ion					
Cl—O	1.394(5)	1.360(8)	O—Cl—O <sup>iii</sup>	109.8(5)	110.9(7)
			O—Cl—O <sup>iv</sup>	109.3(2)	108.8(4)

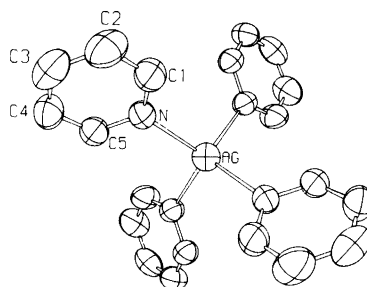
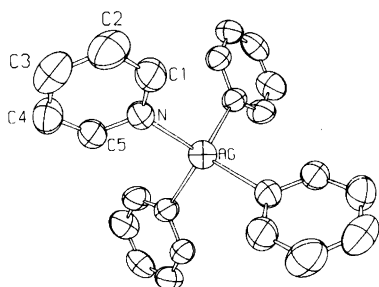


Fig. 2. Stereoscopic view of the  $[\text{Ag}(\text{C}_5\text{H}_5\text{N})_4]^+$  ion.

$(\text{CH}_2)_4\text{CN})_2\text{NO}_3$ <sup>9</sup> and  $\text{Ag}(\text{NC}(\text{CH}_2)_4\text{CN})_2\text{ClO}_4$ <sup>10</sup>  $\text{M}-\text{N}=1.98$  and  $2.28$  Å, respectively (Table 5). The difference between the distances  $\text{Cu}-\text{N}$  and  $\text{Ag}-\text{N}$  is almost the same as in the pyridine solvates, however.

For a comparison between  $\text{Cu}-\text{N}$  and  $\text{Ag}-\text{N}$  distances, complexes other than mononuclear tetrahedral ones are also of interest. The cluster compounds  $[\text{MIC}_5\text{H}_{11}\text{N}]_4$ ,  $\text{M}=\text{Cu}$ <sup>11</sup> and  $\text{Ag}$ ,<sup>12</sup> contain an  $\text{M}_4\text{I}_4$  core. Each metal atom is also coordinated to one nitrogen atom, resulting in a tetrahedral arrangement around M. Again, the difference between the distances  $\text{Cu}-\text{N}$  and  $\text{Ag}-\text{N}$  is almost the same as for the pyridine solvates. Even if the ligands and the coordination geometries are fairly different this difference is evidently close to  $0.28$  Å (Table 5).

In compounds where  $\text{Cu}(\text{I})$  and  $\text{Ag}(\text{I})$  are coordinated to phosphors the difference between the

distances  $\text{Cu}-\text{P}$  and  $\text{Ag}-\text{P}$  is considerably smaller. Thus, in cluster compounds with  $\text{M}_4\text{X}_4$  core, analogous to those discussed above, the difference is only  $\approx 0.20$  Å (Table 5). The same difference is found for the pair<sup>13</sup>  $\text{MCIPP}$ ,  $\text{M}=\text{Cu}$  or  $\text{Ag}$ .

The distance between the metal atom, M, and the ligand atom, X, may be regarded as composed of four terms  $d_{\text{M}-\text{X}}=r_{\text{M}}+r_{\text{X}}+\sigma_{\text{rep},\text{X}-\text{X}}-\sigma_{\text{cov},\text{M}-\text{X}}$ . Here  $r_{\text{M}}$  and  $r_{\text{X}}$  are the radii for given coordination numbers observed in ionic compounds,  $\sigma_{\text{rep},\text{X}-\text{X}}$  represents repulsive forces between the ligands<sup>14</sup> and  $\sigma_{\text{cov},\text{M}-\text{X}}$  is a function of the covalency of the  $\text{M}-\text{X}$  bond.<sup>15</sup> If two compounds containing the bonds  $\text{M}_1-\text{X}$  and  $\text{M}_2-\text{X}$  are isostructural, the differences for the two ligands  $\text{X}_1$  and  $\text{X}_2$  are:

$$\Delta_1 = d_{\text{M}_1-\text{X}_1} - d_{\text{M}_2-\text{X}_1} = r_{\text{M}_1} - r_{\text{M}_2} - \sigma_{\text{cov},\text{M}_1-\text{X}_1} + \sigma_{\text{cov},\text{M}_2-\text{X}_1}$$

Table 5. Survey of structure containing  $\text{M}-\text{N}$  and  $\text{M}-\text{P}$  bonds (Å).

	$d_{\text{Cu}-\text{N}}$	$d_{\text{Ag}-\text{N}}$	$d_{\text{Ag}-\text{N}} - d_{\text{Cu}-\text{N}}$	Ref.
$[\text{M}(\text{C}_5\text{H}_5\text{N})_4]\text{ClO}_4$	2.046(4)	2.322(3)	0.276(6)	This work
$\text{M}(\text{NC}(\text{CH}_2)_4\text{CN})_2\text{A}^a$	1.98(1)	2.28(1)	0.30(1)	9 and 10
$[\text{MIC}_5\text{H}_{11}\text{N}]_4$	2.052(7)	2.329(15)	0.277(17)	11 and 12
Average			0.278(5)	
	$d_{\text{Cu}-\text{P}}$	$d_{\text{Ag}-\text{P}}$	$d_{\text{Ag}-\text{P}} - d_{\text{Cu}-\text{P}}$	
$[\text{PEt}_3\text{MCl}]_4$	2.176(2)	2.390(2)	0.214(3)	16 and 17
$[\text{PEt}_3\text{MBr}]_4$	2.199(2)	2.402(5)	0.203(6)	16 and 17
$[\text{PEt}_3\text{MI}]_4$	2.254(3)	2.438(2)	0.184(4)	19 and 20
$\text{MCIPP}^b$	2.258(2)	2.458(3)	0.200(4)	13
Average	2.217(4)	2.411(3)	0.194(5)	
			0.201(2)	

<sup>a</sup>  $\text{A}=\text{ClO}_4$  (Ag) and  $\text{A}=\text{NO}_3$  (Cu). <sup>b</sup> PP = 2,11-bis(diphenylphosphinomethyl)benzo [c] phenanthrene.

$$\Delta_2 = d_{M_1-X_2} - d_{M_2-X_2} = r_{M_1} - r_{M_2} - \sigma_{\text{cov}, M_1-X_2} + \sigma_{\text{cov}, M_2-X_2},$$

if the difference between the terms  $\sigma_{\text{rep}, X-X}$  can be neglected.

The difference  $\Delta = \Delta_1 - \Delta_2$  is given by:

$$\Delta = (\sigma_{\text{cov}, M_2-X_1} - \sigma_{\text{cov}, M_1-X_1}) - (\sigma_{\text{cov}, M_2-X_2} - \sigma_{\text{cov}, M_1-X_2}).$$

The size of  $\Delta$  thus reflects the difference in covalency between the  $M_2-X_1$  and  $M_1-X_1$  bonds as compared to the  $M_2-X_2$  and  $M_1-X_2$  bonds.

The M-N distances obtained in this study may be compared to the M-P distances observed in the cluster compounds  $[\text{PET}_3\text{MCl}]_4$  and  $[\text{PET}_3\text{MBr}]_4$ .<sup>16,17</sup> In all compounds compared, the closest distance of approach between atoms in the coordination spheres is longer than their van der Waals contact distances. Therefore, it is reasonable to neglect the repulsive forces between the ligands. The simplified expression derived above should thus be valid, i.e.:

$$\Delta = (\sigma_{\text{cov}, \text{Ag-P}} - \sigma_{\text{cov}, \text{Cu-P}}) - (\sigma_{\text{cov}, \text{Ag-N}} - \sigma_{\text{cov}, \text{Cu-N}})$$

with  $\Delta$  equal to 0.062(7) and 0.073(9) Å for the chloride and bromide compounds, respectively.

Thermodynamic measurements indicate that among the bonds discussed, the covalent interaction is weakest for the Cu-N-bond,<sup>18</sup> i.e.:  $\sigma_{\text{cov}, \text{Ag-N}} > \sigma_{\text{cov}, \text{Cu-N}}$ . Then, since  $\Delta > 0$ , it follows that  $\sigma_{\text{cov}, \text{Ag-P}} > \sigma_{\text{cov}, \text{Cu-P}}$ . This also conforms with the behaviour of these donor-acceptor atoms in solution, where the largest covalent interaction is expected between Ag and P.<sup>18</sup>

As  $\Delta > 0$ , it may further be concluded that the difference in covalency is larger between the bonds Ag-P and Cu-P than between the bonds Ag-N and Cu-N.

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