

SYNTHESIS AND MOLECULAR STRUCTURE OF 1,1'-(ETHYLENE-1,2-DIYL)DIPYRIDINIUM TETRACHLORIDOCUPRATE(II)

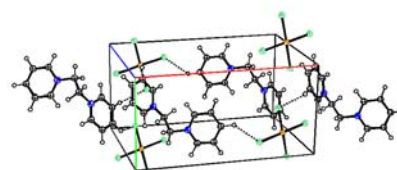
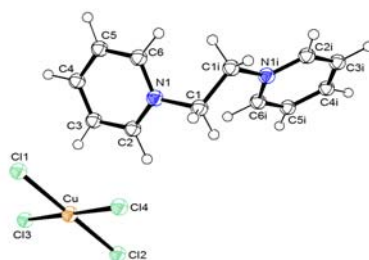
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To explore the propensity of the 1,1'-(ethylene-1,2-diyl)dipyridinium dication of forming organic-inorganic hybrid salts by treatment with suitable metal halide, the organic-inorganic ionic compound $[\text{C}_{12}\text{H}_{14}\text{N}_2][\text{CuCl}_4]$ is prepared by reacting of $[\text{C}_{12}\text{H}_{14}\text{N}_2]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$ with CuCl_2 . The obtained hybrid salt is fully characterized by multinuclear NMR, FTIR and UV-vis spectroscopy, elemental analyses and powder X-ray diffraction techniques.



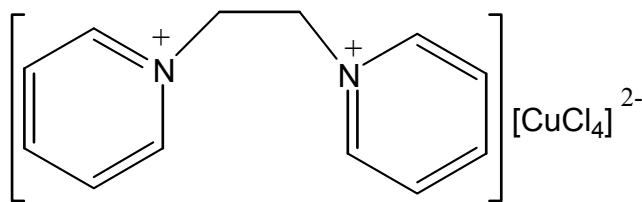
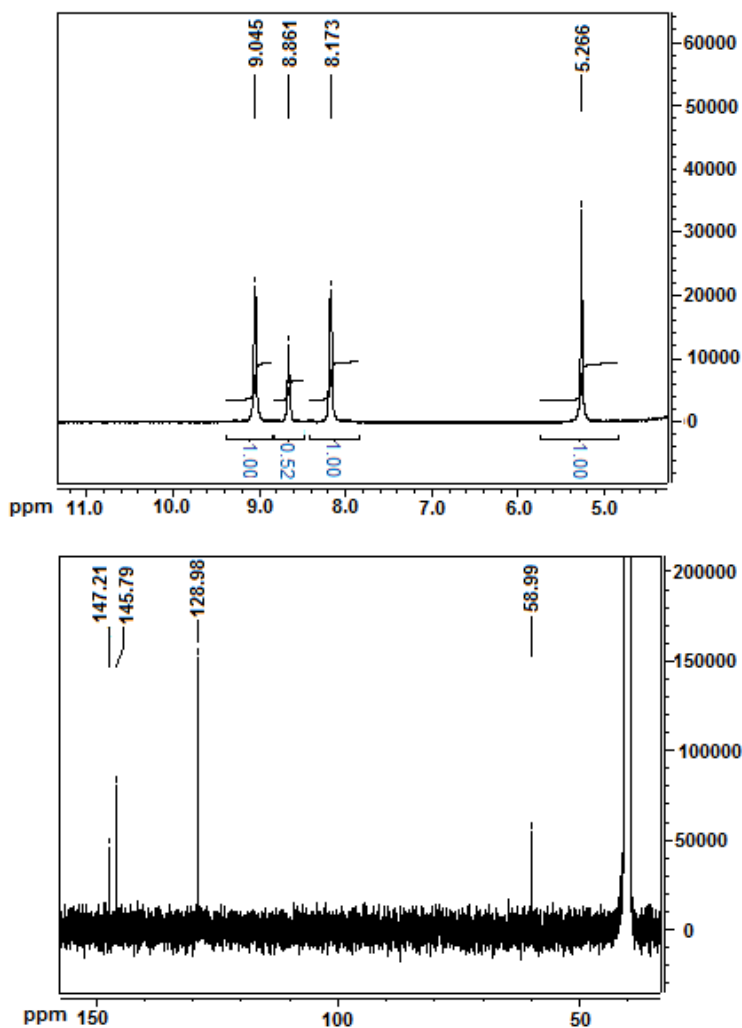
INTRODUCTION

The continually request of new materials is always a big task facing workers in various research fields.¹ Designing of hybrid organic-inorganic ionic compounds by combination of organic and inorganic parts gives these types of salts some desired properties, which are probably unobtainable in the pure organic or inorganic parts. Recently, organic-inorganic hybrid salts are found to have potential applications in diverse domains particularly in crystal engineering, supramolecular chemistry, materials science,²⁻⁵ and optical semiconductor materials.^{6,7} In the late years, the potential ability of the dication 1,1'-methylenedipyridinium $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2]^{2+}$ to form organic-inorganic ionic compounds was utilized by us to synthesis many organic-inorganic hybrid salts

such as: $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2][\text{MCl}_4]$ ($\text{M} = \text{Zn},^8 \text{Cd},^8 \text{Cu},^9 \text{Pt}^{10}$). In addition $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2][\text{AuCl}_4]_2,$ ⁹ $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2][\text{PtCl}_6],$ ¹⁰ $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2][\text{Cu}(\text{NO}_4)_4]$ ¹¹ and $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2][\text{Fe}(\text{CN})_5\text{NO}]$ ¹² are also synthesized and characterized. Furthermore, the optical nonlinearity of $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2][\text{Fe}(\text{CN})_5\text{NO}],$ ¹² $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2][\text{CuCl}_4]$ ¹³ and $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2][\text{AuCl}_4]_2$ ¹⁴ were also investigated, suggesting them to have potential applications in optical domain.

Previously, few hybrid salts of 1,1'-methylenedipyridinium with mixed halo-osmium(IV) anions of the type $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2][\text{OsF}_n\text{Cl}_{6-n}]$ ($n = 0-6$) were prepared. The two isomers cis- and trans- $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2][\text{OsCl}_4\text{F}_2],$ ¹⁵ the isomeric pair fac- and mer- $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2][\text{OsCl}_3\text{F}_3],$ ¹⁶ the trans- $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2][\text{OsF}_4\text{Cl}_2] \cdot \text{H}_2\text{O},$ ¹⁷ cis- $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2][\text{OsBr}_2\text{F}_4]$ ¹⁸ and $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2][\text{Cu}(\text{NCS})_4]$ ¹⁹ were also described.

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Fig. 1 – Molecular structure of $[C_{12}H_{14}N_2][CuCl_4]$.Fig. 2 – 1H (above) and $^{13}C\{^1H\}$ NMR (below) spectra of $[C_{12}H_{14}N_2][CuCl_4]$ ($DMSO-d_6$, 25°C).

This motivated us to prepare 1,1'-(ethylene-1,2-diyl)dipyridinium dication $[C_{12}H_{14}N_2]^{2+}$ for use in the syntheses of new organic-inorganic hybrid ionic complexes.²⁰ Recently, the synthesis, characterization and third-order optical non-linear polarizability of the 1,1'-(ethylene-1,2-diyl)dipyridinium tetranitratocuprate(II) were reported.²¹ In this article, we present the preparation, the spectroscopic characterization and the molecular structure investigations of $[C_{12}H_{14}N_2][CuCl_4]$ by powder X-ray diffraction studies.

RESULTS AND DISCUSSION

Treatment of $[C_{12}H_{14}N_2]Cl_2 \cdot 2H_2O$ with one equivalent of $CuCl_2$ in an aqueous solution at ambient temperature gave the organic-inorganic ionic compound $[C_{12}H_{14}N_2CH_2][CuCl_4]$. The hybrid salt was obtained as air stable yellowish orange powdery product; highly insoluble in common organic solvents such as pyridine or ethanol, but it is water soluble and it has a good solubility in DMSO. The obtained organic-inorganic hybrid salt was isolated as

pure product and fully characterized by multi-nuclear NMR, FTIR and UV-Vis spectroscopy, elemental analyses, and its molecular structure (Figure 1) was identified by powder X-ray diffraction investigation.

The FTIR spectrum of $[\text{C}_{12}\text{H}_{14}\text{N}_2][\text{CuCl}_4]$ is depicted in Figure 3. It is noticeable the presence of the characteristic absorption bands at 1191.79 cm^{-1} (C–N), 1629.55 cm^{-1} (C=N) and 1488.78 cm^{-1} (CH_2). The UV-Vis spectrum of $[\text{C}_{12}\text{H}_{14}\text{N}_2][\text{CuCl}_4]$ is shown in Figure 4. The absorption band at $\lambda_{\text{max}} = 270.8\text{ nm}$ is due to the combination of $\pi - \pi^*$ and/or intraligand charge-transfer (ILCT) transitions. The very broad spectral feature in range 300–400 nm, which can be distinguished in the spectrum is more likely to be associated with the ($d \rightarrow d^*$) transition and/or ligand-to-metal charge-transfer (LMCT) in the inorganic part $[\text{CuCl}_4]^{2-}$ of the complex.

The ^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra for $[\text{C}_{12}\text{H}_{14}\text{N}_2][\text{CuCl}_4]$ (Figure 2) have similar features to their corresponding ones of the parent salt $[\text{C}_{12}\text{H}_{14}\text{N}_2]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$ ²⁰ and the structurally related hybrid salt $[\text{C}_{12}\text{H}_{14}\text{N}_2][\text{Cu}(\text{NO}_3)_4]$.²¹ The observed ^1H NMR spectrum shows four resonances (broadening peaks) corresponding to four different proton environments. The obtained peaks are in their expected chemical shifts and relative intensity ratio. Broadening peaks are also observed in ^1H NMR spectra of the $[(\text{C}_5\text{H}_5\text{N})_2\text{CH}_2][\text{CuX}_4]$ ($\text{X} = \text{Cl}$ or NO_3),^{9,11} which most probably due to the electric quadruple effects of the paramagnetic Cu(II) centers in the complex. The $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum also gave the predicted four resonances consistent with four environmentally different C centers.

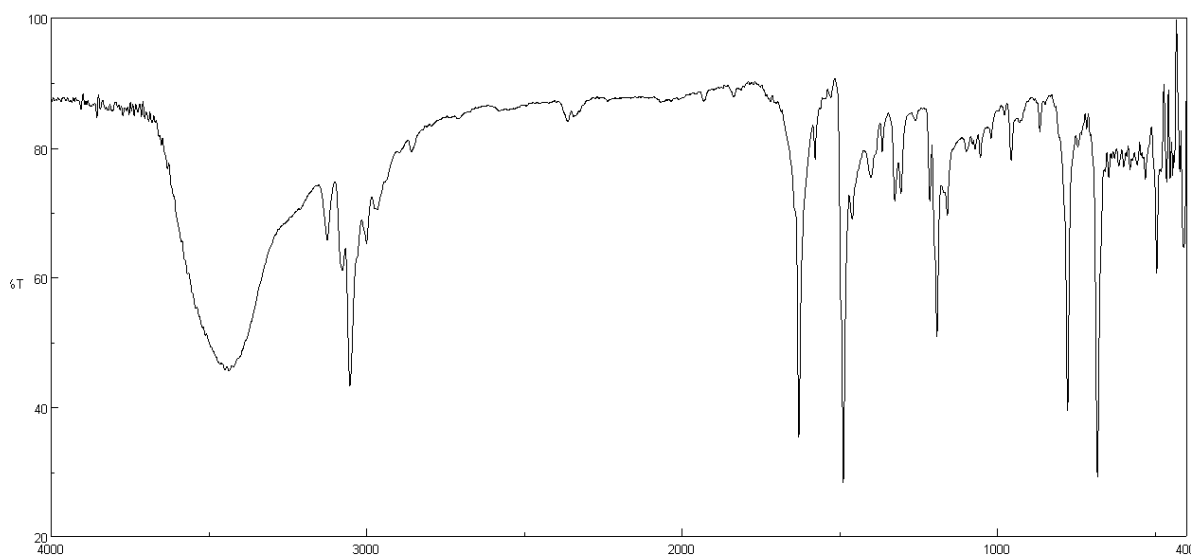


Fig. 3 – FTIR spectrum of $[\text{C}_{12}\text{H}_{14}\text{N}_2][\text{CuCl}_4]$ (KBr disc).

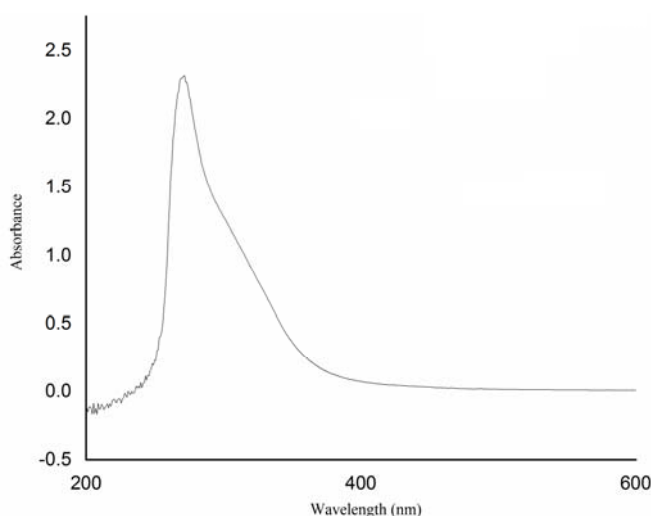


Fig. 4 – UV-Vis spectrum of $[\text{C}_{12}\text{H}_{14}\text{N}_2][\text{CuCl}_4]$ in DMSO at concentration of $6 \times 10^{-4}\text{ M}$.

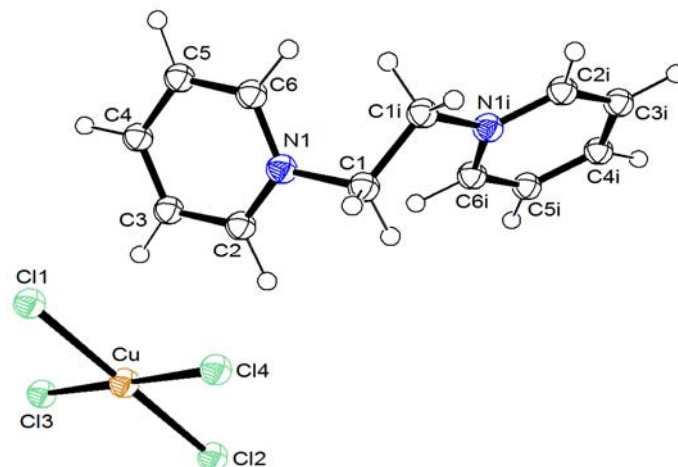


Fig. 5 – The asymmetric unit of crystal structure of $[C_{12}H_{14}N_2][CuCl_4]$, showing the atomic numbering scheme.

Table 1

Selected bond lengths (Å) and angles (°) for $[C_{12}H_{14}N_2][CuCl_4]$

Bond	Bond length (Å)	Bonds angle (°)	angle (°)
Cu—C11	2.221(6)	Cl1—Cu—Cl2	179.63
Cu—Cl2	2.220(4)	Cl1—Cu—Cl3	90.33
Cu—Cl3	2.262(3)	Cl1—Cu—Cl4	89.72
Cu—Cl4	2.261(1)	Cl2—Cu—Cl3	89.55
C1—N1	1.490(2)	Cl2—Cu—Cl4	90.39
Cl1i—N1i	1.491(7)	Cl3—Cu—Cl4	179.44
C2—N1	1.347(8)	C2—N1—C6	120.67
C2—C3	1.384(9)	C2—N1—C1	119.96
C3—C4	1.387(1)	C6—N1—C1	119.37
C4—C5	1.404(9)	N1—C1—C1i	110.51

The molecular structure of $[C_{12}H_{14}N_2][CuCl_4]$ was confirmed by powder X-ray diffraction studies, because of no suitable single-crystal available for this salt. The crystal and molecular structures show discrete 1,1'-(ethylene-1,2-diyl)dipyridinium dication and tetrachlorocuprate (II) anion. A view of the molecular with atomic labeling is shown in Figure 5. The copper atom is situated in a square planar environment with almost equal Cu—Cl bond distances [2.222(5) – 2.261(4) Å] (Table 1), which lie within the normal range and very close to its corresponding average value in the hybrid salt $[C_{11}H_{12}N_2][CuCl_4]$.⁹ The organic dication has an inversion centre located at the mid-point of the C1—C1i bond [symmetry code: (i) $-x + 1, -y + 1, -z + 1$]. The bond lengths and angles are comparable to normal values (Tables 1).²² No significant changes were noted to the intra-dication C—C and C—N bond lengths compared with the corresponding bond distances in the parent 1,1'-(ethylene-1,2-diyl)dipyridinium dichloridedihydrate.²⁰

For simplification, in the crystal packing of $[C_{12}H_{14}N_2][CuCl_4]$, only those hydrogen bonds C—H \cdots Cl (Table 2) lying within the sum of the van der Waals radii for C and Cl atoms (3.45 Å)²³ were considered as non-standard hydrogen bonds. The crystal packing (Figure 6) is achieved by C—H \cdots Cl short contacts, where each individual dication is involved with two Cl centres of two different surrounding $[CuCl_4]^{2-}$ anions. One Cl center of particular $[CuCl_4]$ unit is linked to aromatic H of one dication (para position of the pyridinium), while the second Cl center of the same $[CuCl_4]$ unit interacts with aromatic H (meta) of a second dication to form infinite chains with dication \cdots Cl \cdots dication backbones. The two Cl atoms of each individual $[CuCl_4]$ anion involved in the short contacts are *cis*- to each other. These non-standard hydrogen bondings are most likely effective in the stabilization of the crystal packing of the hybrid salt.

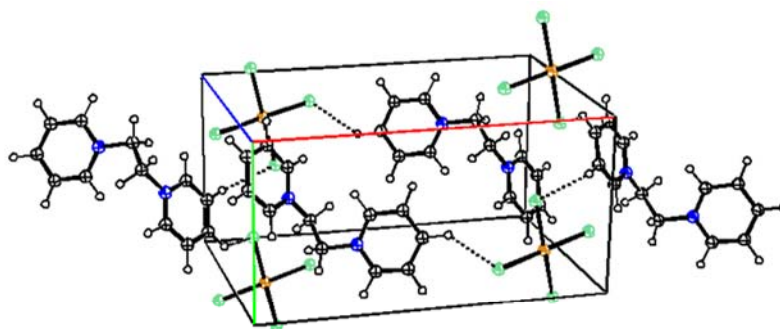


Fig. 6 – Crystal packing of $[C_{12}H_{14}N_2][CuCl_4]$. Short contacts are shown as dashed lines.

Table 2

Hydrogen bond distances (Å) and bond angles (°) for $[C_{12}H_{14}N_2][CuCl_4]$.

D–H...A	d(D–H)	d(H...A)	d(D...A)	Angle(D–H...A)
C4i–H4i...Cl2 ⁱ	1.07	2.574	3.419	135.36
C5–H5 ...Cl4 ⁱ	1.06	2.577	3.445	138.08

Symmetry code (i): $-x + 1, -y + 1, -z + 2$

EXPERIMENTAL

Materials

All reactions and manipulations were carried out in air with reagent grade solvents. $[C_{12}H_{14}N_2]Cl_2 \cdot 2H_2O$ was prepared according to the reported literature method.²⁰ $CuCl_2$ (BDH, Germany) was a commercial sample and used as received. 1H and $^{13}C\{^1H\}$ NMR spectra were recorded on a Bruker Bio spin 400 spectrometer (Switzerland). Fourier transform Infrared (FTIR) spectrum was recorded on Thermo-Nicolet 6700 FTIR Spectrometer, taken as KBr disc with a resolution of 4 cm^{-1} . The Ultra-Violet-Visible (UV-Vis) absorption spectrum was recorded in the wavelength range 190–1100 nm using UV-1601 PC Shimadzu Spectrophotometer. Microanalysis was performed using EURO EA (Italy). Powder X-ray diffraction was performed by Stoe Transmission diffractometer (Stadi P) (Stoe & CIE GmbH, Germany).

Synthesis of 1,1'-(ethylene-1,2-diyl)dipyridinium tetrachloridocuprate(II)

A solution of $[C_{12}H_{14}N_2]Cl_2 \cdot 2H_2O$ (0.240 g, 0.82 mmol) in H_2O (4 mL) was added to a solution of $CuCl_2$ (0.114 g, 0.82 mmol) in H_2O (3 mL) at room temperature and stirred overnight. The solvent was then removed in vacuum to afford $[C_{12}H_{14}N_2][CuCl_4]$, which was washed with EtOH to afford a yellowish orange powder.

Characterization and NMR, FTIR and UV-vis spectroscopic data of $[C_{12}H_{14}N_2][CuCl_4]$

Yellowish orange powder, m.p. 201°C (dec.), Yield: 80%; Anal. Calcd for $C_{12}H_{14}Cl_4N_2Cu$ (391.61): C, 36.80; H, 3.60; N, 7.15 Found: C, 36.40; H, 3.55; N, 7.38; 1H NMR (400.1 MHz, DMSO- d_6): δ_H 5.26 (4H, b, CH_2), 8.17 (4H, b, py), 8.86 (2H, m, py), 9.04 (4H, b, py); $^{13}C\{^1H\}$ NMR (100.6 MHz, DMSO- d_6): δ_C 58.99 (s, CH_2), 128.98 (s, py), 145.79 (s, py), 147.21 (s, py). IR (KBr) $\nu\text{ cm}^{-1}$: 1191.79 (C–N), 1629.55

(C=N), 1488.78 (CH_2). UV (DMSO): λ_{max} (ϵ) = 270.8 nm (2.3).

Crystal structure determination for $[C_{12}H_{14}N_2][CuCl_4]$ from powder X-ray diffraction

Compound $[C_{12}H_{14}N_2][CuCl_4]$ has a tendency to crystallize in the form of a very fine yellowish orange powder. Since no single-crystal of sufficient thickness and quality could be obtained, a crystal structure determination by powder X-ray diffraction was attempted. Therefore, the powder X-ray diffraction data of the compound was collected using the transmission diffractometer (STADI-P STOE, Darmstadt, Germany), with copper K_α radiation ($\lambda = 1.54060\text{ \AA}$) and Ge monochromator. The scanning angle starts at 5° and ends at 100° with a scanning rate of 1° per minute.

The first attempt to find the crystal structure is performed with the help of the program *DicVol6.0*,²⁴ which is implemented in the *EXPO2014* software.²⁵ We used the first 22 lines of our powder pattern to find the unit cell. The best unit cell with sufficiently acceptable figures of merit [$M(22) = 29.5$, $F(22) = 70.5(0.0065, 48)$] is found to be triclinic and the best-estimated space groups is $P\bar{1}$. The initial structure of $[C_{12}H_{14}N_2][CuCl_4]$ is determined by *ab initio* and direct methods using the *EXPO2014* package.²⁵ The found model by these programs is introduced as an input for the program *GSAS II*,²⁶ where the structure further refined using Rietveld method that is implemented in the same software. A triclinic cell and $P\bar{1}$ space group with improved figure of merit [$M(20) = 47.12$] are found for the material. The crystal parameters are: $a = 14.7342(10)\text{ \AA}$; $b = 7.51743(26)\text{ \AA}$; $c = 7.4106(4)\text{ \AA}$; $\alpha = 101.2893(9)^\circ$; $\beta = 101.7513(20)^\circ$; $\gamma = 92.350(4)^\circ$. The final values of the crystallographic data and refinement goodness factors are shown in Table 3, where the weighted profile R-factor (Rwp) is equal to 0.07. While the observed and computed structure factors, FO,hkl and FC,hkl , respectively are identical i.e $R(F^2) = 0.0$. The final Rietveld plots of the X-ray diffraction patterns are given in Figure 7. The atomic fractional coordinates in the unit cell are shown in appendix (A).

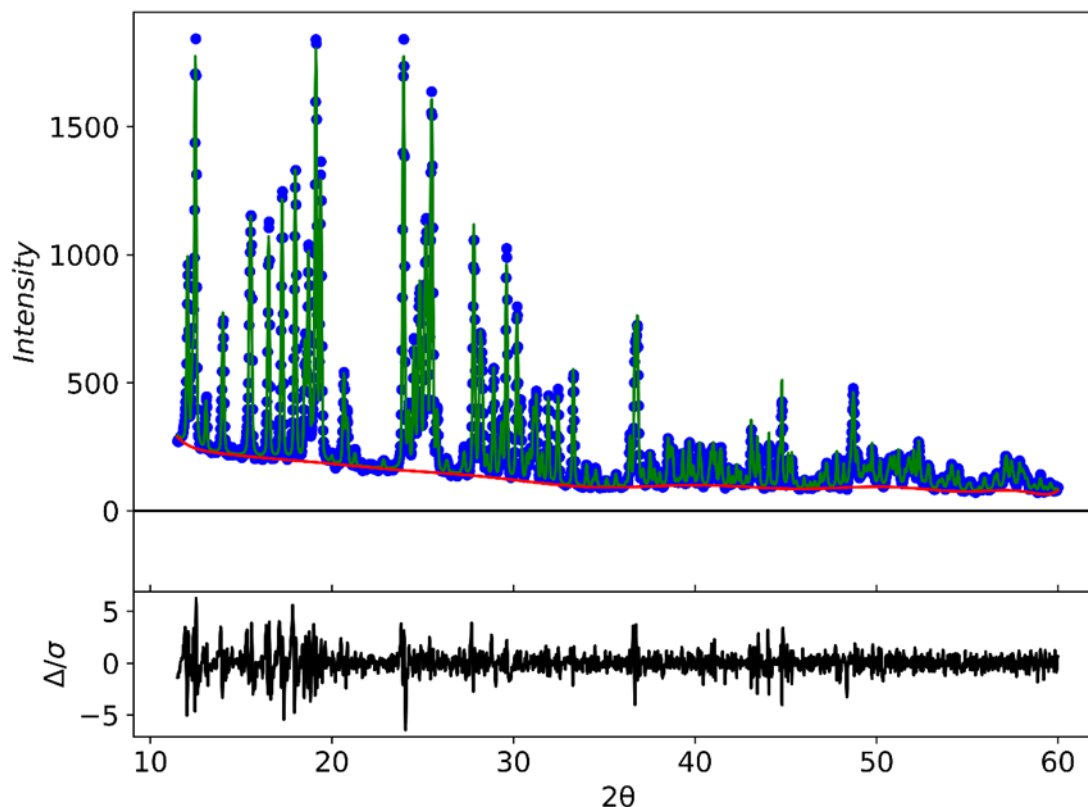


Fig. 7 – The final Rietveld pattern for $[\text{C}_{12}\text{H}_{14}\text{N}_2][\text{CuCl}_4]$. The (•) is the measured values and (–) are the fitted ones. (–) is the difference between the measured and the fitted values. Red line is the background.

Table 3

Crystallographic data and refinement summary for $[\text{C}_{12}\text{H}_{14}\text{N}_2][\text{CuCl}_4]$

Formula	$\text{C}_{12}\text{H}_{14}\text{N}_2 \cdot \text{CuCl}_4$
Temperature (K)	298
Formula weight	391.62
Crystal system	Triclinic
Space group	P -1
a (Å)	14.7342(10)
b (Å)	7.5174(3)
c (Å)	7.4106(4)
α (°)	101.2894(9)
β (°)	101.751(2)
γ (°)	92.350(4)
V (Å ³)	785.27(2)
Z	2
D_x (Mg m ⁻³)	1.656
$2\theta_{\min}$ (°)	4.98°
$2\theta_{\max}$ (°)	89.96
Increment in 2θ (°)	0.02
Cu $K\alpha_1$ radiation, Wavelength (Å)	1.54060
F_{000}	0.0
Specimen shape, Dimensions	powder
Particle morphology, Colour	yellowish orange powder
R_p	0.053
R_{wp}	0.070
R_{exp}	0.065
$R(F^2)$	0.00000
χ^2	1.164

Table 3 (continued)

Number of data points	4250
Background function	"chebyshev-1" function with 15 terms: 135.3(5), -87.0(8), 32.5(7), -11.1(7), -1.0(7), -3.1(6), 7.4(6), -3.8(6), 1.9(6), -0.3(6), 2.7(6), -2.4(6), 1.0(6), 3.0(5), 3.5(5)
Profile function	Finger-Cox-Jephcoat function parameters U, V, W, X, Y, SH/L: peak variance(Gauss) = Utan(Th) ² +Vtan(Th)+W; peak HW(Lorentz) = X/cos(Th)+Ytan(Th); SH/L = S/L+H/L U, V, W in (centideg) ² , X & Y in centideg -624.865, 248.581, -2.495, 205.484, -48.885, 0.002, Crystallite size in microns with "isotropic" model: parameters: Size, G/L mix 1.000, 1.000, Microstrain, "isotropic" model (106 * delta Q/Q) parameters: Mustrain, G/L mix 1000.000, 1.000,
Preferred orientation correction	March-Dollase correction coef. = 1.000 axis = [0, 0, 1]

CONCLUSION

The organic-inorganic ionic compound [C₁₂H₁₄N₂][CuCl₄] has been synthesized, isolated as pure salt and characterized by spectroscopic methods, and X-ray diffraction technique. The molecular structure shows discrete 1,1'-(ethylene-1,2-diyl)dipyridinium dication and tetrachloridocuprate(II) anion. The crystal structure of [C₁₂H₁₄N₂][CuCl₄] is determined by *ab initio* and direct methods and further refined using Rietveld method. A triclinic cell and $P\bar{1}$ space

group with figure of merit [M (20) =47.12] are found.

SUPPLEMENTARY INFORMATION

CCDC 1996398 contains the supplementary crystallographic data for this paper. This data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033).

Appendix (A)

The atomic fractional coordinates in the unit cell

	x	y	z
C2	0.17330	0.36580	0.50710
C3	0.10840	0.21650	0.45930
C4	0.03080	0.21080	0.31570
C5	0.01970	0.35610	0.22150
C6	0.08570	0.50090	0.27520
N1	0.16110	0.50540	0.41730
H2	-0.02010	0.09690	0.27650
H3	0.23440	0.37360	0.61480
H4	0.11950	0.10990	0.53550
H5	-0.03870	0.35510	0.10930
H6	0.08090	0.61380	0.20710
C6i	0.36630	0.56460	0.26800
C5i	0.44760	0.52650	0.20720
C4i	0.48580	0.64640	0.11690
C3i	0.44150	0.80380	0.08940
C2i	0.36090	0.83430	0.15120
N1i	0.32380	0.71500	0.23810
H6i	0.54870	0.61960	0.06910
H5i	0.33560	0.47720	0.34030
H4i	0.47850	0.40490	0.23060
H3i	0.46950	0.89940	0.02180
H2i	0.32500	0.95250	0.13470
Cl1	0.01494	0.28312	0.82806

Cl2	0.31354	0.18426	0.97559
Cu	0.16430	0.23410	0.90280
C1	0.23090	0.66630	0.47280
H1A	0.21120	0.76280	0.58270
H1B	0.29710	0.62100	0.52990
Cl _i	0.23540	0.75220	0.30170
H1iA	0.17860	0.69780	0.18440
H1iB	0.23270	0.89730	0.33760
Cl3	0.12530	-0.06670	0.86880
Cl4	0.20348	0.53424	0.93387

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