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## SYNTHESIS & CHARACTERIZATION OF Cu(II) COMPLEXES DERIVED FROM ACYL PYRAZOLONE & 2-AMINO PHENOL

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### ABSTRACT

A series of tridentate pyrazolone-based Schiff bases were synthesized by the interaction of 4-acyl/aroyl pyrazolones with 2-Amino Phenol in an ethanolic medium. All of these ligands were characterized on the basis of elemental analysis, infrared (IR) and <sup>1</sup>H-NMR data. Nuclear magnetic resonance (NMR) suggests the amine-one form of ligand in solution at room temperature. Copper Schiff-base complexes [Cu<sub>2</sub>(L)<sub>2</sub>], have been prepared by the interaction of the aqueous solution of copper acetate monohydrate with hot ethanolic solution of the appropriate ligand. The resulting complexes have been characterized by elemental analysis, metal content determination, molar conductance, fast atom bombardment mass spectra, magnetic measurements, thermogravimetric analysis (TGA), IR and electronic spectral studies. Suitable square planar structure is proposed for these complexes.

**Key words:** Acyl pyrazolones, Copper complexes, Schiff bases, Spectral studies

### INTRODUCTION

The chemistry of pyrazolone derivatives has attracted much attention because of their interesting structural properties and applications in diverse areas [1-4]. They are useful reagents for the extraction and separation of various metal ions [5, 6]. They can also be used in laser materials, as <sup>1</sup>H NMR shift reagents, in chromatographic study and in the petrochemical industry [7, 8]. Many of these ligands exhibit keto enol tautomerism and because of this they show interesting structural and spectroscopic properties which have been the subject matter of many reports [9]. β-diketones have played and continue to play a key role in coordination compounds that have found wide application in several fields, from new materials to catalysts [10], as precursors for CVD in the microelectronic industry and as potential antitumourals [11]. Even the simplest pyrazolone-5 derivatives for instance, antipyrine and amidopyrine are well-known analgesics and widely used in medicine. Pyrazolone derivatives are also used as starting materials for the synthesis of biologically active compounds and for the construction of condensed heterocyclic systems [12]. Among these ligands, acyl pyrazolones have been studied extensively owing to their effective properties with respect to extracting metal ions [13]. On the other hand pyrazolone-based Schiff base chemistry is less extensive.

Pyrazolyl and pyrazolyl-derived ligands can form relevant coordination compounds with different metal ions. Copper complexes have been extensively studied, especially dinuclear and polynuclear species, as mimics of the proteins hemocyanin and tyrosinase or as compounds with interesting catalytic and magnetic properties [14]. In most of these complexes, copper ions are linked by N, N'-bridging pyrazolato anions, or coordinated by one nitrogen atom of the pyrazole ring. However, recently an unusual coordination mode of this type of ligand was described, where nitrogen atoms of the pyrazole rings are not bound to copper, but involved in strong intramolecular hydrogen bonds.

Recently, some Schiff bases from 3-methyl-1-(4'-methylphenyl)-2-pyrazoline-5-one and aromatic amines were

prepared and their molecular structures were determined. These ligands can exist in three tautomeric forms: keto-imine, imine-ol and keto-amine, although the ketoamine form is predominant in the solid state. Their corresponding mononuclear copper(II) complexes were also prepared and characterized by spectroscopic techniques, indicating a tetragonal geometry around the copper(II) ion, N,O coordinated [15].

We were interested to synthesis a new series of pyrazolone-based Schiff base ligands, which can form stable neutral complexes with Cu(II) metal ions and may find some application in solvent extraction chemistry. With this view we have synthesized a series of ligands by condensation of acyl pyrazolones with 2-amino phenol. The reaction of these ligands in alcoholic medium with copper acetate resulted the novel square planar complexes, which were then characterized by various spectral techniques.

### MATERIALS AND METHODS

All the chemicals used in the present study were of best quality. Dioxane was obtained from Sisco Chem. Pvt Ltd, Mumbai and used after purification. Calcium hydroxide and acetyl chlorides were supplied by Suvindhinath Labs, Baroda. 2-aminophenol was obtained from LOBA Chem. Pvt. Ltd., Mumbai. Ethanol was obtained from Baroda Chemicals Industry Ltd., Baroda and Benzoyl chloride was obtained from Gayatri Mineral and Chemicals, Baroda. In preparation of metal complexes of Cu(II), copper acetate was used.

### Physical measurements

Elemental analyses (C, H, and N) were performed on a model 2400 Perkin-Elmer elemental analyzer. Infrared (IR) spectra were recorded on a Perkin Elmer FT-IR spectrum RX 1 spectrometer as KBr pellets. NMR was recorded on a model 400 Bruker FT NMR. The electronic spectra were recorded on a model Perkin Elmer Lambda 35 UV-VIS spectrometer. A simultaneous TG/DTA was recorded on EXSTAR6000 TG/DTA6300 model. A FAB mass spectrum was recorded on a Jeol SX 102/Da-600 mass spectrometer at room temperature using m-nitro benzyl alcohol as a matrix and Argon/Xenon as the FAB gas. Specific conductivity of the complexes was

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measured on a model Elico CM 180 conductivity meter. Magnetic moments were measured on Gouy balance.

### Synthesis of ligands

The ligands used in the present study were prepared in two steps as follows:

1. Preparation of the 4-acetyl/benzoyl-2-pyrazolin-5-ones
2. Preparation of Schiff-bases

#### Preparation of the 4-acetyl/benzoyl-2-pyrazoline-5-ones

##### Preparation of 4-acetylc-3-methyl-1-phenyl-2-pyrazoline-5-one

3-Methyl-1-phenyl-2-pyrazoline-5-one (17.4g, 0.1mol) was dissolved in hot dioxane (80cm<sup>3</sup>) in a flask equipped with a stirrer, separating funnel and reflux condenser. Calcium hydroxide (14.81g, 0.2mol) was added to this solution, followed by acetyl chloride (10ml) added drop wise with precaution, as this reaction was exothermic. During this addition the whole mass was converted into a thick paste. After the complete addition, the reaction mixture was refluxed for two hour and then it was poured into cold dilute hydrochloric acid (200cm<sup>3</sup>, 2M). The coloured crystals thus obtained were separated by filtration, washed with water and dried. Yield 60%, M.P.63<sup>0</sup>C.

##### Preparation of 4-acetylc-3-methyl-1-(3'-chloro phenyl)-2-pyrazoline-5-one

It was prepared analogously from 3-methyl-1-(3-chloro phenyl)-2-pyrazoline-5-one (20.85 g, 0.1 mol). Yellow crystals were obtained. Yield 89%, M.P. 72<sup>0</sup>C.

##### Preparation of 4-acetylc-3-methyl-1-(4'-methyl phenyl) -2-pyrazoline -5-one

It was prepared analogously from 3-methyl-1-(4'-methylphenyl)-2-pyrazoline-5-one (20.1g, 0.1mol). Yellow crystals were obtained. Yield 74.75%, M.P.91<sup>0</sup>C.

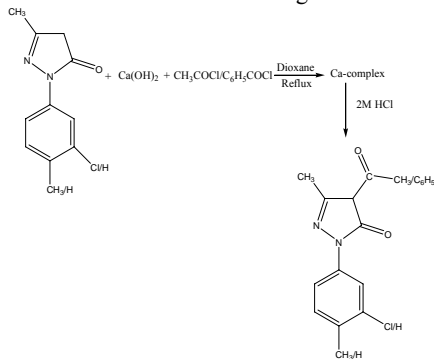
##### Preparation of 4-benzoyl-3-methyl-1-(4'-methylphenyl)-2-pyrazoline-5-one

It was prepared analogously from 3-methyl-1-(4'-methyl phenyl)-2-pyrazoline-5-one (20.1g, 0.1mol) and instead of acetyl chloride, benzoyl chloride (10ml) was taken. Yellow crystals were obtained. Yield 88%, M.P. 106<sup>0</sup>C.

##### Preparation of 4-benzoyl-3-methyl-1-phenyl-2-pyrazoline-5-one

It was prepared analogously from 3-methyl-1-phenyl-2-pyrazoline-5-one (17.4g, 0.1mol) and instead of acetyl chloride, benzoyl chloride (10ml) was taken. Yellow crystals were obtained. Yield 86%, M.P. 93<sup>0</sup>C.

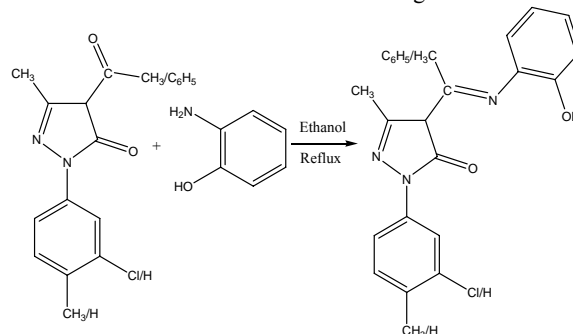
These can be summarized in a following reaction scheme:



### Preparation of Schiff bases

An identical procedure has been adopted in the preparation of all the Schiff bases used in the present study. The Schiff bases were prepared by refluxing 5-ones and 2-aminophenol in ethanol for two hours. The Schiff bases thus obtained were filtered and dried. The physical properties of ligands are given in the Table-1.

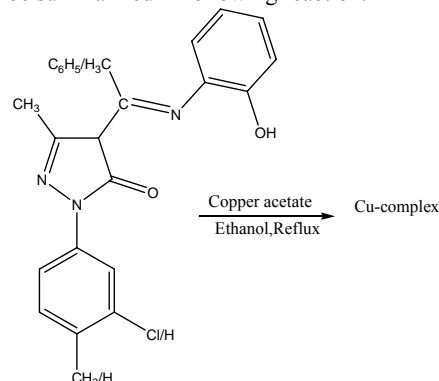
The reaction can be summarized in following scheme:



### Synthesis of complexes

The appropriate ligand, dissolved in hot ethanol was added drop wise into an aqueous solution of copper acetate monohydrate with stirring. Ligand: Metal ratio was 1:1. After the complete addition, the reaction mixture was refluxed for two hour. Then cool it to room temperature. During this period, a light greenish microcrystalline solid was separated, which was isolated by filtration, washed with hot water with several times till colourless filtrate comes followed by one little wash of ethanol and dried.

This can be summarized in following reaction:



## RESULTS AND DISCUSSION

### Characterization of ligands

#### Analytical data

Analytical data of ligands are listed in Table - 1. All the ligands gave satisfactory elemental analysis, which are in close agreement with the empirical formula of the respective ligand.

**Table - 1** Analytical data of ligands

No.	Ligands	M.P.( <sup>0</sup> C)	%C	%H	%N	Yield(%)
1.	AP-AP	224	70.28 (70.34)	5.54 (5.57)	13.63 (13.67)	75.86
2.	AMC-AP	195	62.02 (63.24)	4.57 (4.72)	12.13 (12.29)	82.22
3.	APT-AP	189	70.01 (71.01)	5.75 (5.95)	13.00 (13.07)	63.78
4.	BPT-AP	233	73.45 (75.18)	5.28 (5.52)	10.48 (10.96)	74.42
5.	BP-AP	280	74.11 (74.78)	4.96 (5.18)	11.38 (11.37)	56.99

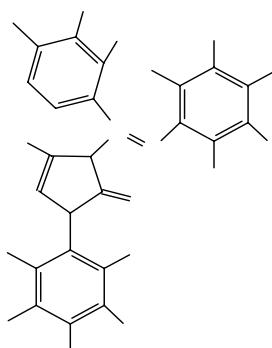
### Infrared spectra

All Ligands show sharp band at  $\sim 1600\text{ cm}^{-1}$ . This band is due to  $\nu(\text{C}=\text{O})$  coordinated +  $\nu(\text{C}=\text{O})$ . There is some contribution from aromatic vibrations and also azomethine  $\nu(\text{C}=\text{N})$  at  $\sim 1620\text{ cm}^{-1}$ . The ligands also show band at  $\sim 3000\text{ cm}^{-1}$  which may be due to  $\nu(\text{O}-\text{H})$ . The IR data of ligands are presented in Table - 2.

**Table - 2** Relevant IR frequencies ( $\text{cm}^{-1}$ ) for the ligands

No.	Ligands	$\nu_{\text{OH}}$	$\nu_{\text{C}=\text{O}(\text{coord.})+\nu_{\text{C}=\text{O}}}$	$\nu_{\text{C}=\text{N}}$ (cyclic)
1.	AP-AP	3060	1621	1581
2.	AMC-AP	3060	1625	1580
3.	APT-AP	3028	1611	1583
4.	BPT-AP	3164	1618	1588
5.	BP-AP	3232	1614	1585

### The $^1\text{H}$ NMR spectra



**Proton numbering scheme for  $^1\text{H}$  NMR**

The important NMR data of the ligands are listed in Table 3 and Table 4. The  $^1\text{H}$  NMR spectra of these three ligands were recorded in  $\text{CDCl}_3$  at room temperature. The signals are due to methyl a proton (two  $\text{CH}_3$  for AMC-AP and three  $\text{CH}_3$  for APT-AP, BPT-AP) appears as singlet. The singlet corresponds to highest  $\delta$  value  $2.343\text{ ppm}$  is assigned to the toluoyl protons while the other singlet in the region  $\delta 1.522\text{--}2.327\text{ ppm}$  is due to other methyl groups. In the aromatic region, a few doublets and in a few cases some overlapping doublets/multiplets are observed. The appearance of multiplet is mainly because of the different substituent at meta and para positions of the one of the two benzene rings [15]. Here,  $\text{H}_h$  proton appears at highest  $\delta$  value in case of BPT-AP while in other two cases  $\text{H}_e$  proton resonates at highest  $\delta$  value. The all signals due to aromatic protons of ligands are assigned and are depicted in Table 4. Another singlet in much low field region corresponding to one proton for all compounds is observed in the range of  $\delta 12.368\text{--}12.716\text{ ppm}$ . This signal disappeared when a  $\text{D}_2\text{O}$  exchange experiment was carried out. It can be assigned either to OH or NH, in either case it is strongly deshielded because of hydrogen bonding with other atom (N/O). It may be noted that the integration of this signal perfectly matches with one proton and there is no other fragment(s) of this signal, which suggests that only one tautomeric form of the ligands exists in solution under the experimental conditions. We have not done any temperature dependent experiments comparing with the solid state study. We prefer to assign this signal to NH; however, assignment of this peak to OH cannot be ruled out provided solid state structural evidence is not considered [15].

**Table - 3**  $^1\text{H}$  NMR spectral data for the aliphatic protons

No.	Ligands	Methyl protons ( $\delta$ ppm)			Aryl protons ( $\delta$ ppm)	-OH/-NH ( $\delta$ ppm)
1.	AMC-AP	2.327 singlet 3H	2.098 singlet 3H		[6.907-8.058] multiplet 8H	12.368 single 1H
2.	APT-AP	2.382 singlet 3H	2.343 Singlet 3H	2.186 singlet 3H	[6.914-7.843] multiplet 8H	12.513 singlet 1H
3.	BPT-AP	2.335 singlet 3H		1.522 singlet 3H	[6.223-7.917] multiplet 13H	12.716 singlet 1H

**Table - 4**  $^1\text{H}$  NMR spectral data for the aromatic protons

Ligands	$\text{H}_a$	$\text{H}_b$ $\text{H}_d$	$\text{H}_c$	$\text{H}_e$	$\text{H}_f$	$\text{H}_g$	$\text{H}_h$	$\text{H}_i$	$\text{H}_j$
AMC-AP	6.928 d $J=7.5$	7.020 t $J=6.5$	7.110 d $J=7.5$	8.058 s	7.925 d $J=7.5$	7.687 s	–	–	–
APT-AP	7.058 d $J=7.5$	6.941 M	7.203 m		7.835 d $J=8$	7.203 m	–	–	–
BPT-AP	6.223 s	6.395 d $J=8.5$	6.686 s		7.357 s	7.188 d $J=7.5$	7.766 d $J=7.5$	7.587 m	7.1 s

### Characterization of complexes

#### Analytical data

**Table - 5** Analytical data and physical properties of complexes

No.	Complexes	Colour	Yield (%)	M.P. ( $^{\circ}\text{C}$ )	Cu Estimation (%)	$\Lambda_{\text{M}}^{\text{p}}$ ( $\Omega^{-1}\text{ cm}^2\text{ mol}^{-1}$ )	$\mu_{\text{eff}}$ (BM)
1.	$[\text{Cu}_2(\text{AP-AP})_2]$	Majestic Mountain	67.67	>260	15.35 (15.99)	16.0	1.3
2.	$[\text{Cu}_2(\text{AMC-AP})_2]$	Autumn Gold	75.63	>260	14.99 (15.04)	3.0	1.3
3.	$[\text{Cu}_2(\text{APT-AP})_2]$	Green Gold	57.68	>260	14.56 (15.81)	2.0	1.4
4.	$[\text{Cu}_2(\text{BPT-AP})_2]$	Ming Jade	74.52	>260	11.04 (13.69)	2.0	1.3
5.	$[\text{Cu}_2(\text{BP-AP})_2]$	Green	59.33	>260	12.84 (14.12)	1.0	1.4

The conductance of complexes in DMF show values from 1 to  $16\ \Omega^{-1}\text{ cm}^2\text{ mol}^{-1}$  indicating non-electrolytic nature of complexes [16].

#### Infrared spectra

The important IR frequencies of the complexes are summarized in table 6. The strong band at  $1610\text{--}1625\text{ cm}^{-1}$ , for  $\nu(\text{C}=\text{N})$  azomethine of the ligand is shifted to lower frequency  $1583\text{--}1591\text{ cm}^{-1}$ , suggesting coordination of the azomethine nitrogen to the metal ion. The new band appeared in the spectra of complexes in the range  $562\text{--}573\text{ cm}^{-1}$  is probably due to the formation of M-N bond. The presence of coordinating water molecule was also supported by thermo gravimetric study.

**Table - 6** Relevant IR frequencies (cm<sup>-1</sup>) for the complexes

Complexes	$\nu_{C=N(\text{coord.})} + \nu_{C=O}$	$\nu_{C=N}$ (cyclic)	$\nu_{M-N}$
[Cu <sub>2</sub> (AP-AP) <sub>2</sub> ]	1591(s,s)	1566(s,s)	568(s,s)
[Cu <sub>2</sub> (AMC-AP) <sub>2</sub> ]	1589(s,s)	1564(s,s)	567(s,s)
[Cu <sub>2</sub> (APT-AP) <sub>2</sub> ]	1591(s,s)	1565(s,s)	562(s,s)
[Cu <sub>2</sub> (BPT-AP) <sub>2</sub> ]	1588(s,s)	1556(s,s)	565(s,s)
[Cu <sub>2</sub> (BP-AP) <sub>2</sub> ]	1583(s,s)	1560(s,s)	573(s,s)

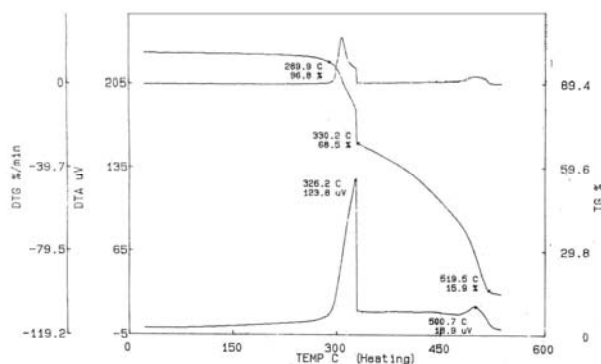
Abbreviations: br = broad, s = strong, m = medium

#### Magnetic Measurement

The complexes of Cu(II) exhibit subnormal magnetic measurement and it can be attributed to the magnetic interaction between the metals present in a dimeric structure [17]. Magnetic measurements are shown in Table 5.

#### Thermal analysis

The TG-DTA-DTG curve of one of the complexes is shown in Fig. 1. The TG curve follows the decrease in sample mass with increase in temperature. In the present investigation, heating rates were suitably controlled at 10°Cmin<sup>-1</sup> and mass loss followed up to 50-600°C. From the TG curve the mass loss for the complex was calculated. The mass loss occurred at 290°C corresponds to the loss of 3.7% (calc. 3.2%) for two methyl groups. This process is accompanied by exothermic process at 326°C in the DTA curve of the complex. The mass loss occurred at 519°C corresponds to the loss of 84.5% (calc. 84.1%) for two ligand molecules. This process is accompanied by small exothermic process at 500°C in the DTA curve of the complex.

**Fig.1** The TG curve of the complex Cu(AMC-AP)<sub>2</sub>

#### Electronic spectral studies

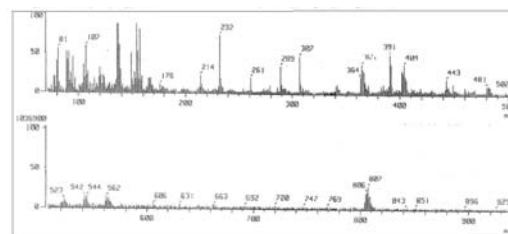
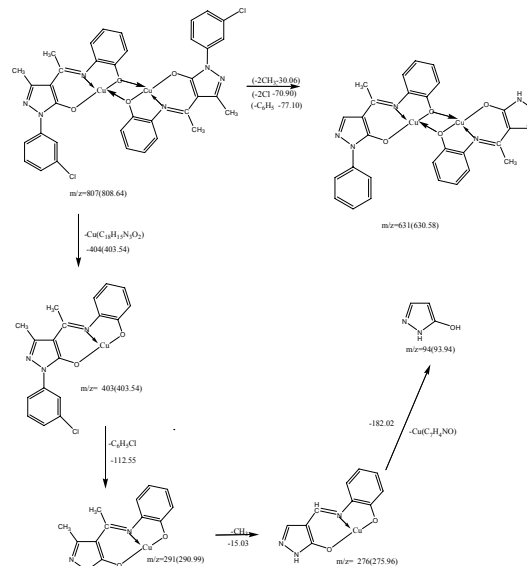
The electronic spectra of all the complexes were recorded in DMF and the data are presented in Table 7. For square planar copper (II) complexes, the expected transitions are <sup>2</sup>B<sub>1g</sub> → <sup>2</sup>A<sub>1g</sub> and <sup>2</sup>B<sub>1g</sub> → <sup>2</sup>E<sub>g</sub> with the respective absorption at 505-520 and 665-650 nm. In general, due to J-T distortions, square planar Cu(II) complexes give a broad absorption band between 600-700 nm and the peak at 505-520 nm merges with the broad band [18]. The  $\lambda_{\text{max}}$  value of 602 nm for the present Cu(II) complex indicates square planar geometry.

**Table - 7** Electronic spectral data of complexes

Complexes	d-d maxima (nm)	d-d maxima (cm <sup>-1</sup> )	$\epsilon$ (molar absorptivity)
[Cu <sub>2</sub> (AP-AP) <sub>2</sub> ]	519	19268	30
[Cu <sub>2</sub> (AMC-AP) <sub>2</sub> ]	520	18519	32
[Cu <sub>2</sub> (APT-AP) <sub>2</sub> ]	515	19417	52
[Cu <sub>2</sub> (BPT-AP) <sub>2</sub> ]	665	15038	37
[Cu <sub>2</sub> (BP-AP) <sub>2</sub> ]	505	19802	27

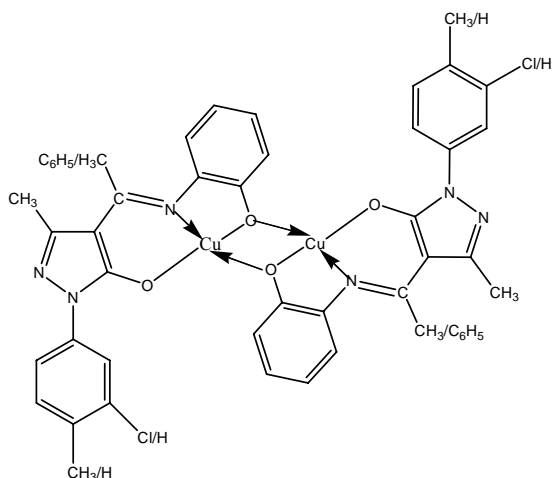
#### Mass Spectra

The FAB Mass spectrum of the complex [Cu<sub>2</sub>(AMC-AP)<sub>2</sub>] was recorded in m-nitro benzyl alcohol as a matrix. The isotropic peak can be observed at m/z =807, which is corresponding to the complex. The FAB mass spectrum of the complex [Cu<sub>2</sub>(AMC-AP)<sub>2</sub>] is shown in Fig. 2 and the fragmentation pattern for complex [Cu<sub>2</sub>(AMC-AP)<sub>2</sub>] is shown below:

**Fig.2** The FAB-Mass spectra of the complex Cu<sub>2</sub>(AMC-AP)Fragmentation pattern for complex [Cu<sub>2</sub>(AMC-AP)<sub>2</sub>]

## CONCLUSION

IR data suggests the O, N, O coordination nature of the ligands. In the NMR spectra of ligands, the integral intensities of each signal are found to agree with the number of different types of protons present. The observed molar conductances in DMF solution of these complexes are consistent with non-electrolytic nature of the complexes. The elemental analysis of ligands as well as of complexes supports the proposed structure which is depicted below.



Common suggested structure for the Copper(II) complexes

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**Review process:** Submitted papers are peer-reviewed by two to three independent reviewers after approval by the Editorial Board. Authors are encouraged to suggest three names of expert reviewers with their e-mail IDs, but selection remains the prerogative of the Editorial Board.

**Articles of the following categories are also considered for publication in PRAJNA:**

**Short Communications** are limited to a maximum of two figures and one table. They should present a complete study that is more limited in scope than is found in full-length papers. The items of manuscript preparation listed above apply to Short Communications with the following differences: (1) Abstracts are limited to 100 words; (2) instead of a separate Materials and Methods section, experimental procedures may be incorporated into Figure Legends and Table footnotes; (3) Results and Discussion should be combined into a single section.

**Review Articles** intended to provide concise in-depth reviews of both established and new areas and summarize recent insights in specific research areas within the scope of PRAJNA are solicited by the Editorial Board from leading researchers. The manuscript of this category should be limited to 5,000 words with an abstract of no more than 250 words, a maximum of 5 tables and figures (total), and up to 50 references. Word count includes only the main body of text (i.e., not tables, figures, abstracts or references).

**Commentaries** call attention to papers of particular note and are written at the invitation of the Editorial Board.

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**Also announcement of forthcoming Seminars / Conferences / Symposia / Workshops etc. will be considered for publication in PRAJNA.**

**File format for soft copies:**

Texts (should be of Times New Roman with 9 point for Abstract and 11 point for other matter) and Tables, if any, must be saved in \*.doc (Word) or \*.rtf (rich text) format, graphs in Excel and for illustrations (diagrams, maps, drawings, etc.), the TIF format (300 dpi minimal resolution) is the most appropriate (\*.TIF or \*.JPEG extension).

**Instructions for preparation of manuscripts:**

1. The paper should be written in English and neatly typed with double spacing.
2. The title of the paper and the name(s) of the author(s) be in capital letters. The name of the institution be given in small letters below the name (s) of the author(s).
3. The 'Abstract of the paper, in not more than 150 words, should be provided on a separate page along with 4-6 keywords.
4. The sub-titles, e.g. INTRODUCTION, should be written in capital letters.

5. Displayed formulae, mathematical equations and expressions should be numbered serially. Table should be with a title in addition to a serial number for it.
6. Photographs / Figures should be original with good contrast so as to be in a form suitable for direct reproduction / scanning.
7. Footnotes are not normally allowed, except to identify the author for correspondence.
8. All figures must be numbered serially as they appear in the text, and their legends / captions should necessarily be provided.
9. References should be numbered in brackets [ ] in the order of appearance in the text. All the references in the bibliographic list must correspond to in-text references and vice versa. Abbreviated periodical titles should follow standard subject Abstracts. Names which are not listed by any standard subject indexing organizations should be spelled out in full.
10. All references should be clear and follow the examples below:

**Periodical articles**

- [2] Sadqui, M., Fushman, D. and Munoz, V. (2006) Atom – by – atom analysis of global downhill protein folding. *Nature*, **442**: 317 – 321.

**Books**

- [16] Stebbins, G. L. (1974) *Flowering plants: Evolution above the species level*, Arnold Press, London, pp. 1 – 399.

**Chapters from a book**

- [19] Schafer, H. and Muyzer, G. (2001) Denaturing gradient gel electrophoresis in marine microbial ecology. In *Methods in Microbiology* (Ed. Paul, J. H.), Academic Press, London, Vol. 30, pp. 425 – 468.

**Thesis or other diplomas**

- [21] Nayaka, S. (2004) *The visionary studies on the lichen genus Lecanora sensu lato in India*. Ph. D. Thesis, Dr. R. M. L. Avadh University, Faizabad, India.

**Conference proceedings**

- [4] Mohapatra, G. C. (1981) Environment and culture of early man in the valley of rivers Chenab and Ravi, western sub-Himalayas. In *Proceedings X Congress of IUPPS*, Mexico, pp. 90 – 123.

**Online documentation**

- [9] Koning, R. E. (1994). Home Page for Ross Koning. Retrieved 26-6-2009 from *Plant Physiology Information Website*: <http://plantphys.info/index.html>.

**Note:**

**Manuscripts prepared faithfully in accordance with the instructions will accelerate their processing towards publication; otherwise it would be delayed in view of their expected re-submission.**

**For and on behalf of Editorial Board, PRAJNA**

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