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Synthesis and Characterisation of 4- Amino-3-Mercapto-5-Methyl-1, 2, 4 -Triazole And Its Mixed- Ligands Complex with Cd (II) Ions

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Abstract: *In the present investigation, the Schiff base (E)-4-(2-hydroxynaphthalen-1-yl) methylene) amino)-5-methyl-2,4-dihydro-3H-1,2,4-triazole-3-thione was prepared by the condensation reaction of 2-hydroxyl-1-naphthaldehyde with 4-amino-3-mercapto-5-methyl-1, 2, 4-triazole. The synthesized Schiff base (ligand, L_1) was used to prepare complex with cadmium (II) ion (Cd(II)) by the reaction of cadmium (II) chloride ($CdCl_2$) and the ligand under reflux. A mixed-ligands complex was prepared by treating the metal complex with Schiff base in solution with thiophene (the secondary ligand, L_2) in solution. The solid complex obtained with the secondary ligand was purified, dried and characterized on the basis of melting point, micro-analytical data, spectroscopic data (Fourier Transform Infrared, UV-visible, ^{1}H-NMR, powder X-ray diffraction). The assignment and the nature of the electronic transitions observed in the UV-visible spectra were analysed using the MultiWfn analyser.*

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Keywords: *1, 2, 4-triazole, Cd(II), complex, Schiff base, thiopene*

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1.0 Introduction

Triazole is a 5-membered heterocyclic compound of empirical formula $C_2H_3N_3$ (Mobinikhaledi *et al.*, 2010; Siddiqui *et al.*, 2010; Banerjee *et al.*, 2013). Triazole derivatives include amine and thione-substituted triazoles containing S=C-N-N structural units and similar to thiosemicarbazone. The presence of exocyclic thione groups in the heterocyclic moiety is much more interesting due to the combination of the two groups (amines and thiones) that produce species with excellent coordination potential. Bonding via amine and thione groups forms a stable metal complex with a highly preferred 5-membered ring system. The mercapto-triazole or amine thione-substituted triazole has the general formula $H_3N_3SC_2$ and exist in two tautomeric forms (Haddad *et al.*, 2013).When hydrogen binds to nitrogen, it becomes a thione type, and when it binds to sulfur, it becomes a thiol type.

Some compounds with 1,2,4, -triazole ring are well known in drug synthesis. These include 1, 2, 4 - triazoles containing amino groups. This is also important for obtaining various Schiff bases with known antibacterial properties. Many attempts have been made to synthesize various transition metal complexes using 1, 2, 4 - triazole as a ligand, and their structures and properties have been physically and chemically determined. 1, 2, 4 - triazoles are widely used in coordination chemistry because they can be used to synthesize transition metal coordination polymers with two adjacent nitrogen atoms (N_1 and N_2 or N_4 at the 4-position). This bridge can have different shapes depending on the donor atom of the ligand and the properties of the metal. Due to the position of the donor atom in the 5-membered ring, triazole can bond metal ions to each other. Therefore, triazole forms a bridge between metal ions. Triazoles and their derivatives have been extensively used in plant protection technology as pesticides (Jess *et al.*, 2014). A wide range of triazole herbicides have been synthesized to aid in selectively controlling the growth of weeds and they exhibit characteristics such as high level of activity, application flexibility, crop tolerance and minimal levels of toxicity to mammals (Selby, 2001).

Schiff bases are generally bidentate, tridentate, tetradentate or polydentate ligands that can form highly stable complexes with transition metals (Uwanta *et al.*, 2021; Ebong *et al.*, 2024; Anweting *et al.*, 2025). They function as coordination ligands only when they have a functional group (usually a hydroxyl) and are so close to the condensation site that they can form a 5- or 6-membered ring when reacted with a metal ion. In Schiff bases formation, the metal ion contributes effectively by creating stable complexes, hence producing a favourable overall free energy of reaction. Schiff bases have an efficient role as chelating ligands for most metal ions. Both syntheaize and properties of Schiff base complexes are very often intimately related to the associated metal ions. Therefore, this study focused on the synthesis, spectral characterization of Cd(II) metal mixed ligand complexes with 4-amino-3-mercapto-5-methyl-1,2,4-triazole as the primary ligand and thiophene as secondary ligand.

2.0 Materials and Methods

2.1 Materials

The pure chemical samples of thiocarbohydrazide (98.5%), 2-hydroxyl-I-naphthaldehyde (98.5%), thiophene (99%) were obtained from sigma Aldrich (United Kingdom), glacial acetic acid (98.5%), and metal salts of $CdCl_2$ were of analytical grade. All chemicals were used without further purification. The solvents used included ethanol, distilled water. The glassware used were 50ml, 100ml, 250ml conical flasks, 250ml beakers, 250ml quick-fit round bottom flask, 100ml measuring cylinder, stirring rod. Other apparatus used were: spatula, dropper, digital weighing balance, fume cupboard, heating mantle, retort-stand and clamp, quick fit Liebig condenser (reflux setup), funnels, Whatman filter papers, water bath, aluminium foil.

Instruments used were Gallen Kamp melting point apparatus, capillary tubes, electronic balance, The electronic absorption spectra were recorded (in water and ethanol as solvents) with a Perkin-Elmer UV-1700 series UV–visible spectrophotometer using a cell of 1 cm path length in the region 200 - 800 nm, GC-MS was recorded with Thermo Scientific LTQ XL Linear Ion Trap Mass Spectrometer equipped with an Electro Spray Ionization (ESI) probe, Ligand was analyzed in both positive and negative ion modes with a 10 $\mu L\ min^{-1}$ flow. The FTIR was recorded using Perkin Elmer FT-IR spectrophotometer (version 10.03.05) using KBr discs in the range 400 - 4000 `cm^{-1}. ^{1}H-NMR JEOL AL300 multinuclear FT-NMR

(300 MHz) spectrometer at room temperature using tetramethylsilane (TMS) as an internal standard, PXRD, Thermo Finnegan FLASH EA 1112 series.

2.2 Synthesis

2.2.1 Synthesis of 4-amino-3-mercapto-5-methyl-1, 2, 4-triazole (AMT)

A mixture of thiocarbohydrazide (5 g) and glacial acetic acid (15 ml) were put into a flask and heated under reflux in a fume chamber for 4 hours at 140 ^{0}C. The reaction was cooled to room temperature, and the excess glacial acetic acid was distilled off under reduced pressure. Solid products formed in the reaction mixture were filtered. The solid residue was crystallized from distilled water (75 ml) giving colourless shining shining flakes (4.8g; 78.28%) (Sun *et al*., 2008).

2.2.2 Synthesis of Schiff Base Ligand (E)-4-(2-hydroxynaphthalen-1-yl)methylene)amino)-5-methyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (L_1)

2-hydroxyl-1-naphthaldehyde (5mmol, 0.8 g) was weighed into a 250 ml round-bottom flask and 15ml glacial acetic acid was added to dissolve it. An equimolar amount of 4-amino-3-mercapto-5-methyl-1, 2, 4-triazole (L_1) (5mmol; 0.65g) was added and refluxed for 45 minutes at 120 ^{0}C. After cooling, the solid was recrystallized from anhydrous ethanol (75ml), filtered under suction and dried. A yellow powdery complex (1.4g; 98.59%) was obtained (Sun *et al*., 2008)

2.2.3 Synthesis of dichloro bis [4-(2-hydroxy-1-naphthyl) methyleneamino-3-mercapto-5-methyl-1, 2, 4-triazole] Cadmium (II) complex $(L_1)_2]Cl_2.2H_2O$

In a 250 ml round-bottom flask, cadmium chloride (0.5mmol, 0.183g) was dissolved in ethanol (10ml). An equimolar amount of 4-(2-hydroxy-1-naphthyl) methyleneamino-3-mercapto-5-methyl-1, 2, 4-triazole (L_1) (0.5mmol, 0.1g) was added and refluxed for 1 hour. After cooling, the solid was filtered yielding yellow powdery complex, (0.243g, 79.6%).

2.2.4 Synthesis of mixed [4-(2-hydroxy-1-naphthyl) methyleneamino-3-mercapto-5-methyl-1, 2, 4-triazole] (thiophene) Cd (II) complex $[Cd(L_1)(thiophene)(H_2O)_2]Cl$

In a 250ml flask, (1mmol, 0.284 g) of (E)-4-(2-hydroxynaphthalen-1-yl)methylene)amino)-5-methyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (L_1) dissolved in ethanol (10ml) and heated for 2 minutes, to completely dissolve and treated with 10ml ethanolic solution of $CdCl_2$ (1mmol; 0.183g) and the resulting solution was refluxed for 1hour. Then 1mmol, or 0.084g thiophene, dissolved in 10ml ethanol and refluxed for 1hour. A light-yellow residue obtained was filtered, washed with ethanol, recrystallized with hot methanol and dried over fused calcium chloride in a desiccator and the yield was (0.55g; 91.7 %).

2.3 Characterization

2.3.1 Melting point determination

The melting points of the ligand and complexes were determined by using Gallen Kamp capillary melting point apparatus. The solubility of the synthesized metal complexes was determined using ethanol and distilled water as solvents at room temperature and upon heating. The analyses of carbon, hydrogen, and nitrogen were determined microanalytically using Perkin Elmer Elemental analyzer 2400 Series ll (Waltham, MA, USA).The IR spectra were recorded on a Spectrum 2000 FT-IR spectrometer (Perkin Elmer) in the range 4000 to 400cm^{-1}. Electronic spectra were measured in the range 200-800 nm at room temperature with a Perkin-Elmer UV-visible spectrophotometer. Mass spectral analysis was carried out using Bruker micro TOF. ^{1}H-NMR Spectrum of the triazole Schiff base ligand was recorded in chloroform (CCl_4) solvent to determine the structural properties. High performance digital Avance III 600MHz.

Powder XRD patterns of all the complexes were recorded over the PANalytical X'pert Pro model $2\theta = 10 - 80^o$ range.

3.0 Results and Discussion

3.1 Physical properties

The physical appearance of the synthesized ligand (E)-4-(((2-hydroxynaphthalen-1-yl)methylene)amino)-5-methyl-2,4-dihydro-3H-1,2,4-triazole -3-thione, [$C_{14}H_{12}N_4OS$] and its metal (II) complexes was obtained by visual observation. They were all coloured and powdered solids. The colour and melting point are presented in Table 1.

Table 1: Analytical and Physical Data of Complexes

Compound Symbol	Molecular Formula	Physical State	Colour	M.P(^{o}C)
L_1	$C_{14}H_{12}N_4OS$	Powdery	Yellow	254
$Cd(L_1)Cl_2$	$[C_{28}H_{26}CdN_8O_4S_2]Cl_2$	√	Yellow	450
$[Cd(L_1)(C_4H_4S)Cl]Cl$	$(C_{18}H_{21}CdN_4O_3S_2)$	√	Deep yellow	280

The ligand melted at 254 ^{O}C, $[Cd(C_{14}H_{12}N_4OS)]Cl_2$ has a melting point of 450 ^{O}C and $[Cd(C_{14}H_{12}N_4OS)(C_4H_4S)]Cl$ melted at 280 ^{O}C. These high melting points of the complexes could be attributed to intermolecular and intramolecular hydrogen bonding interactions resulting in high lattice energy and also suggest that the complexes may be polymeric (Moghimi *et al.,* 2003; Khan *et al*., 2011). The poor solubility of the ligand and its metal complexes in the solvents tested could be attributed to the low solvation energy of the solvents and strong non-covalent interaction existing in the complexes.

3.2 Elemental analysis

Elemental analysis helps in the determination of the ratio of C, H and N in the synthesized compounds. The results of the elemental analysis obtained for the ligand and its metal complexes are presented in Table 2. Carbon, hydrogen and nitrogen percentage contents determined for the synthesized complexes are in agreement with theoretically calculated and elemental composition data obtained from the analysis for all the compounds.

Table 2: Microanalysis (Elemental Analysis) of the ligand and Cd^{2+} complexes

Compounds	%C	% H	%N
	Found /(calculated)	Found /(calculated)	Found /(calculated)
$C_{14}H_{12}N_4OS$	59.14(58.77)	425(4.09)	19.70(19.56)
$C_{28}H_{26}CdN_8O_4S_2$	47.03(42.11)	3.66(2.76)	15.72(9.33)
$C_{18}H_{21}CdN_4O_3S_2$	41.78(46.94)	4.06(3.33)	10.83(16.32)

3.3 Infrared Studies of the compounds

The significant infrared frequencies of the ligand and its complexes are explained as follows: in the IR spectrum of the free Schiff base the band at 1587 cm^{-1} was assigned to the azomethine C=N group, 1164 cm^{-1} was assigned to C=S and $_{v}$(S-H) was observed at 2749 cm$^{-1,}$ suggesting that the ligand exhibited the thione-thiol tautomerism in DMSO solution. The naphthalenic hydroxyl OH band was observed at 3068 cm^{-1}. These observations have also been made by other authors in the field (Singh *et al.,* 2011, Sumrra *et al.*, 2018, Joshi *et al*., 2019, Dash *et al*., 2011). The deprotonation of −SH group of triazole was indicated by the absence of bands in the spectra of metal complexes due to ν(S–H), ν(C=S), and ν(N–H). A new band appears ∼740 cm^{-1} in the spectra of the complexes, which is assigned to ν(C–S) and which indicates the complexation of ligands through S-atom with the metal atom. A sharp and strong band in the region of 1587 cm^{-1} for ν(N=CH) in the case of ligands was shifted about 17 cm^{-1} to lower wave numbers in the spectra of metal complexes, indicating the coordination of ligands through azomethine nitrogen to the metal atom. The metal nitrogen bond was further supported by the presence of a band at about ~538 cm^{-1} for ν(M–N), and the bands around ~571 cm^{-1} were assigned to ν (M–S) bond. A strong band in the region of 413 cm^{-1} was assigned to ν(M–Cl) (Kavitha & Reddy 2014; Mohamed *et al.*, 2005). The IR spectra of the ligand and metal complexes are presented in Figs 1-3.

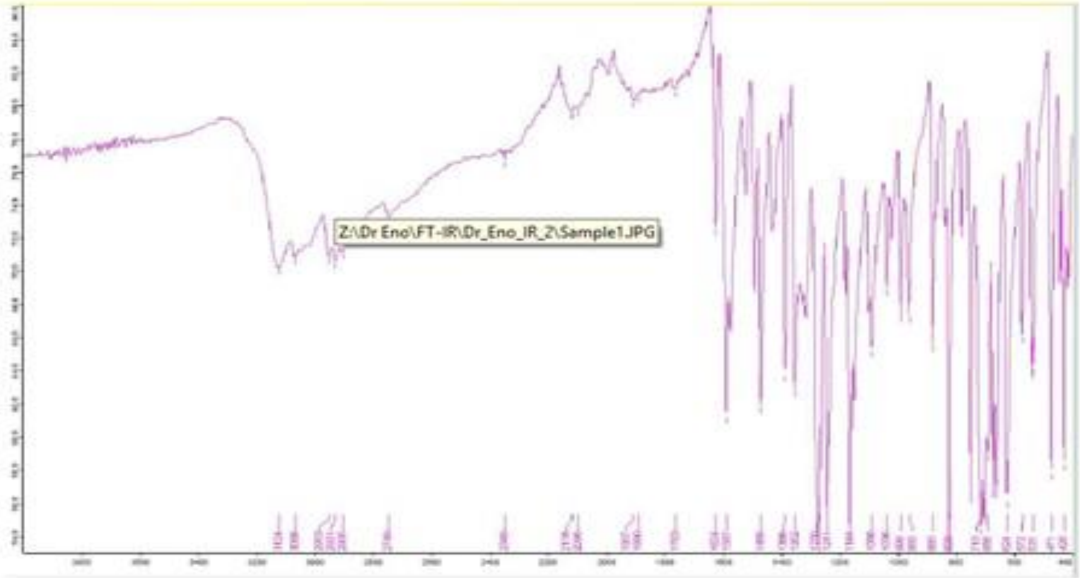

Fig. 1: IR Spectrum of (E)-4-(((2-hydroxynaphthalen-1-yl)methylene)amino)-5-methyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (L_1)

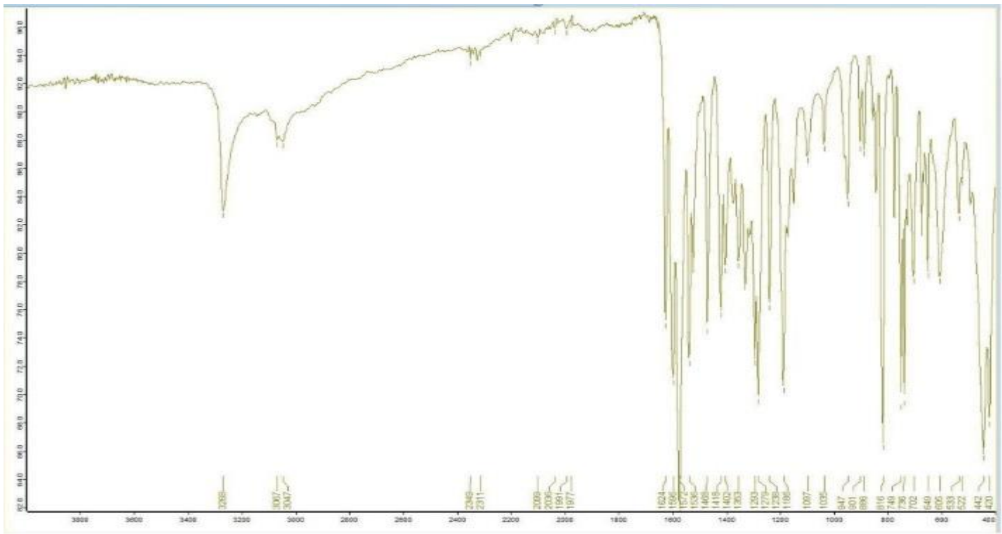

Fig. 2: IR Spectrum of Cd(L_1)Cl_2

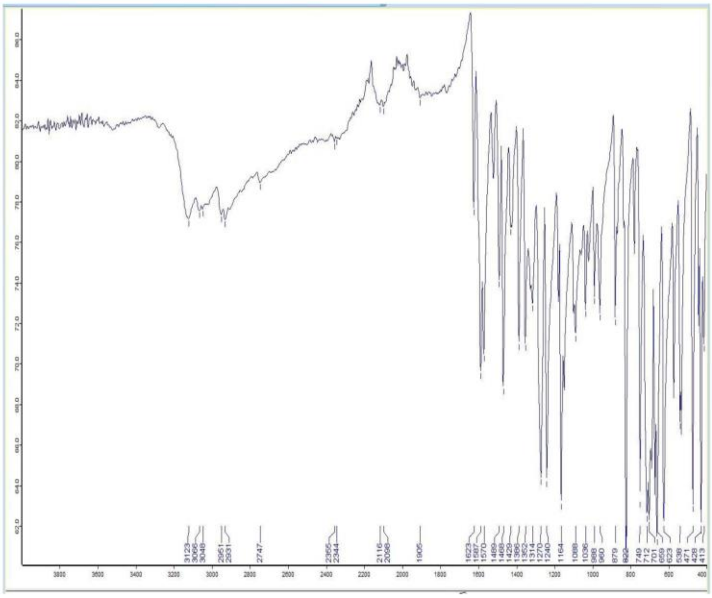

Fig. 3: IR Spectrum of [Cd(L₁)(C4H₄S)Cl]Cl

3.4 Electronic spectral studies

The electronic absorption studies were carried out in DMSO. The absorption spectrum of the free ligand exhibited three high intensity bands at around 222 , 225 nm and 232 nm, these bands observed in the high-energy region are assigned to the intraligand π–π* electronic transition of the conjugated system of Schiff base. The weak and broad absorption band observed at 323 nm, 354 nm, 372 nm and 391 nm can be attributed to the n–π* electronic transition resulting from the involved molecular orbitals of the azomethine (─HC═N─) chromophore, sulfur of the thione-thiol tautomeric system, Oxygen of the naphthalenic hydroxyl group and the conjugated π-bonds of the aromatic system. The UV spectrum of the synthesized Schiff base is shown in Fig. 4.

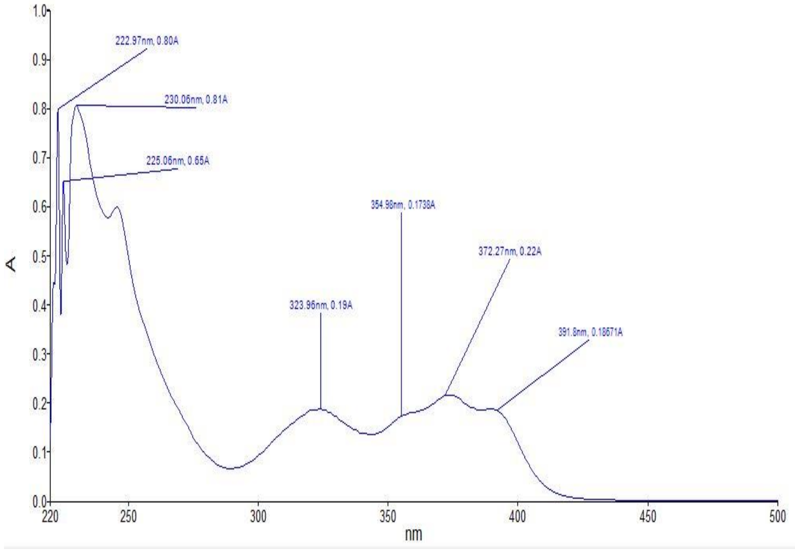

Fig. 4: UV spectrum for (E)-4-(((2-hydroxynaphthalen-1-yl)methylene)amino)-5-methyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (L₁)

3.5 Mass spectra

The mass spectra of the Schiff base showed a molecular ion peak at m/z 283(M^{+1}), which is equal to the molecular weight of the ligand. The mass spectrum of the ligand (L_1) exhibited molecular ion m/z bonds at 283.0643 atomic mass units, which is in accordance with the molecular weight proposed. The molecular weight of the ligand agreed with the elemental and multipack type of the mass spectrum, indicating the nature of the sequential degradation of base compound having peak's series identical to a variety of fractions. The intensity of peaks provided an insight about stability of fragments.

3.6 Proton-NMR

In the ^{1}H-NMR spectrum of ligand, six doublets observed at the range of 7.27-7.96 δ is due to naphthyl protons (Tyagi, *et al*., 2016, Sumrra, *et al*., 2020). The ring free ─OH proton of the ligand was found upfield at 11.45δ and the signals at 8.15δ and 11.56δ were due to ─CH=N (azomethine proton) and ─NH group. The singlet signals observed at the range 1.07-3.93δ were due to the aliphatic protons (Aouad *et al*., 2015, Warad *et al*., 2020, Gull & Hashmi *2015*). *The proton-NMR of the ligand is shown in* Fig. 5.

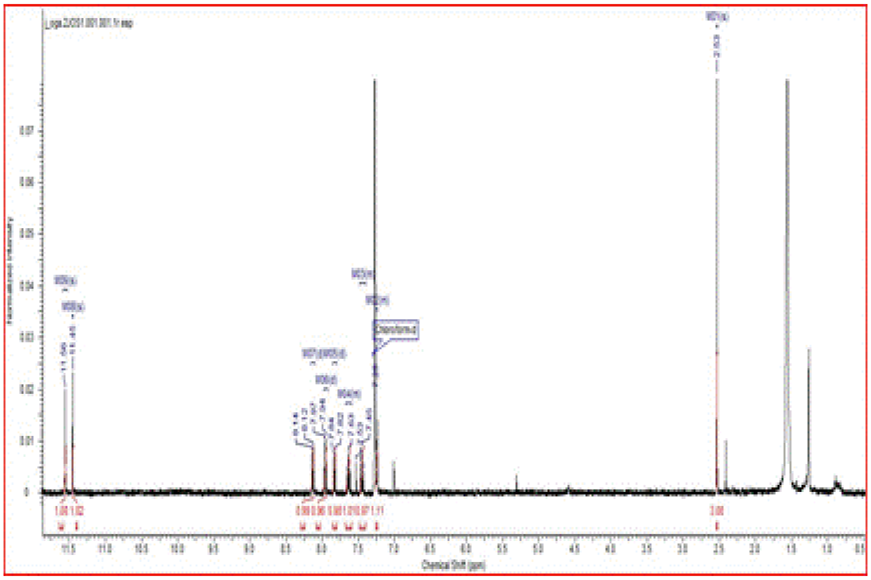

Fig. 5: ^{1}H-NMR-Spectrum of the (E)-4-(((2-hydroxynaphthalen-1-yl)methylene)amino)-5-methyl-2,4-dihydro-3H-1,2,4-triazole-3-thione ligand

3.7 Powder X-Ray diffraction

All the metal complexes displayed sharp crystalline peaks, indicating their crystalline nature. It is also one of the pieces of evidence to confirm the structure of the ligand- (E)-4-(((2-hydroxynaphthalen-1-yl) methylene)amino-5-methyl-2-4-dihydro-3H-1,2,4-triazole-3-thione (L_1) and its metal complexes. The PXRD patterns of the ligand and its metal complex are shown in Figs 6-7.

3.8 Proposed Structures

Based on the information obtained from characterization of the Schiff base and the metal complexes, the proposed structures of AMT, Schiff base, $Cd(L_1)Cl_2$ and $[Cd(L_1)(C_4H_4S)Cl]Cl$ are presented in Figs. 8, 9, 10 and 11, respectively.

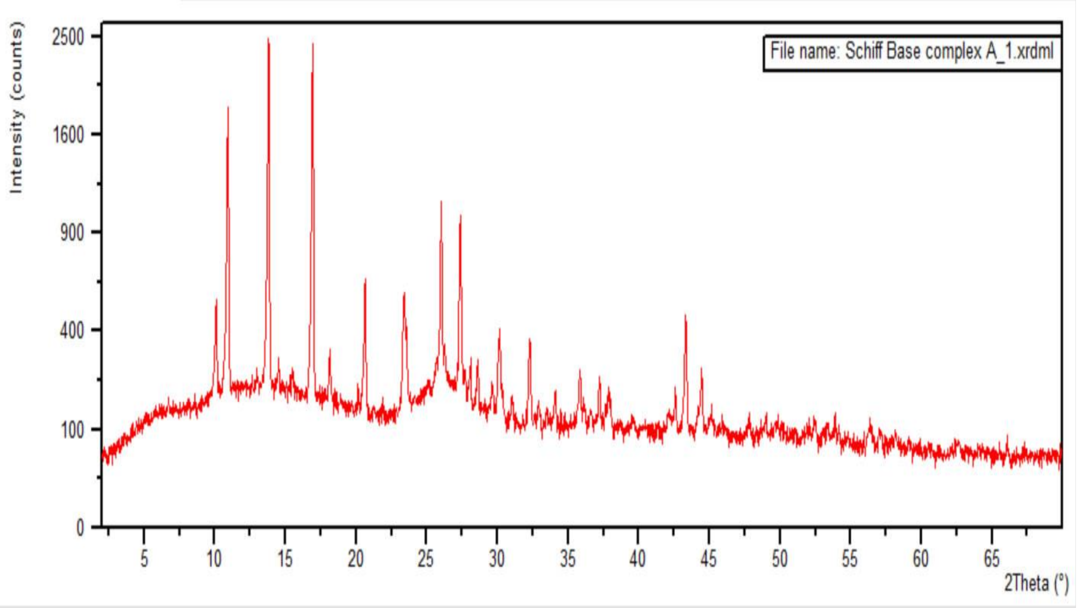

Fig. 6: PXRD for $Cd(L_1)Cl_2$

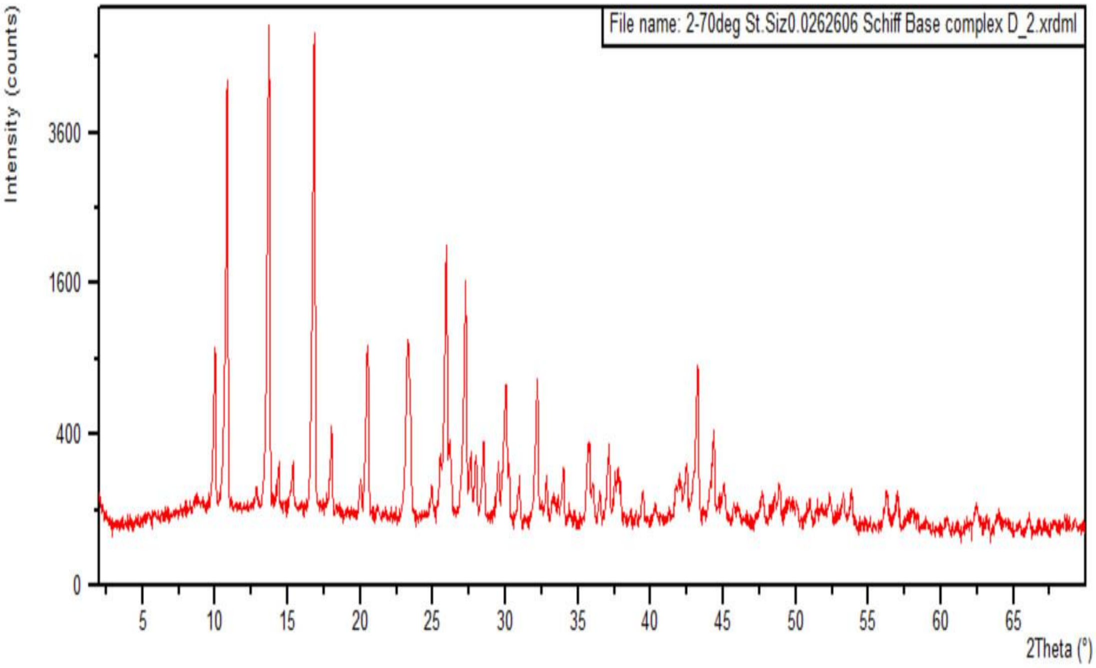

Fig. 7 : PXRD for $Cd(L_1)(C_4H_4S)Cl$

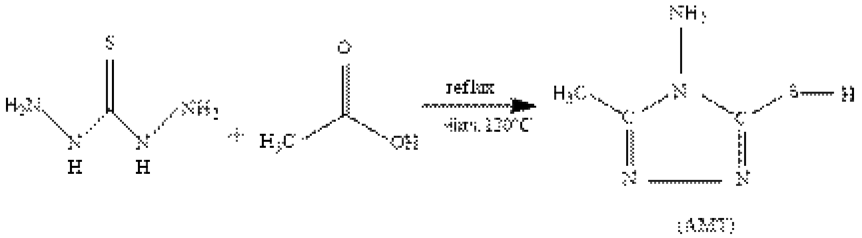

Fig. 8: Structure of 4-amino-3-mercapto-5- methyl-1, 2, 4-triazole (AMT)

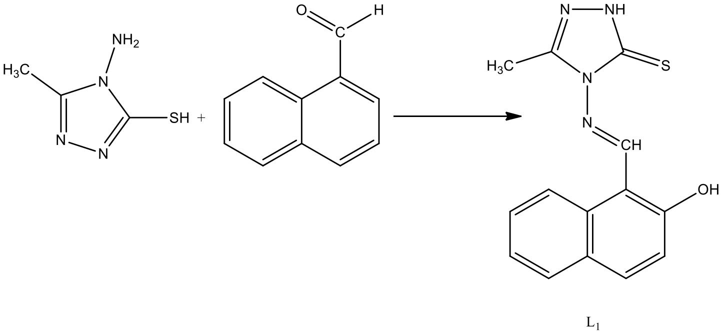

Fig. 9: Structure of Schiff Base Ligand (E)-4-(((2-hydroxynaphthalen-1-yl) methylene) amino)-5-methyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (L_1)

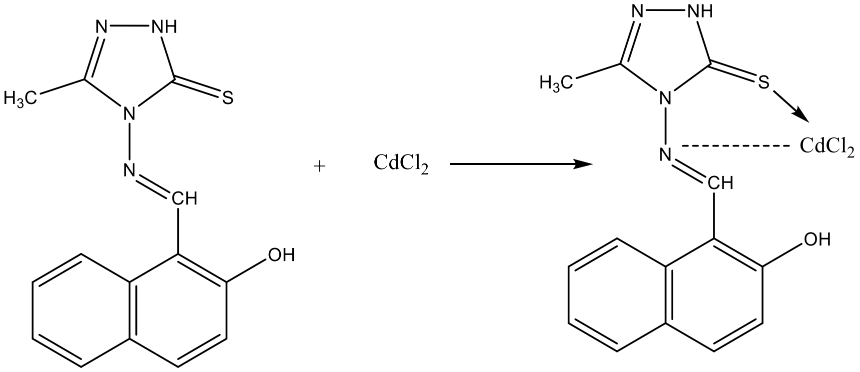

Fig. 10: Structure of dichloro bis [4-(2-hydroxy-1-naphthyl) methyleneamino-3-mercapto-5-methyl-1, 2, 4-triazole]cadmium (II) complex($(L_1)_2$]$Cl_2.2H_2O$

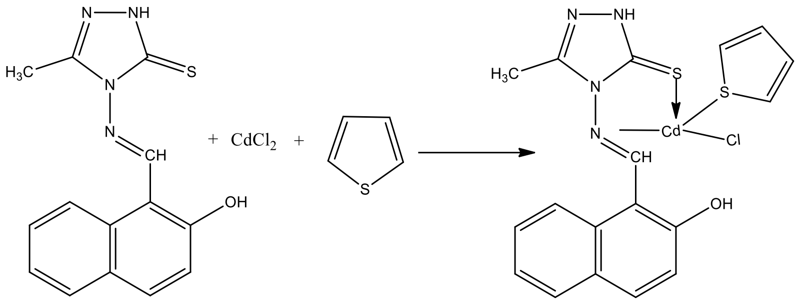

Fig. 11: Structure of mixed [4-(2-hydroxy-1-naphthyl) methyleneamino-3-mercapto-5-methyl-1, 2, 4-triazole] (thiophene) Cd (II) complex [Cd(L$_1$)(thiophene)(H$_2$O)$_2$]Cl

4.0 Conclusion

This research focused on the synthesis of a new Schiff base ligand by the reaction of 2-hydoxy-1-napthaldehyde with the 4-amino-3- mercapto -5-methyl-1, 2 4- traizole. The synthesized ligand was used to prepare complex with cadmium (II), from the reaction of MCl$_2$ by direct reaction under reflux. Mixed ligand complex was prepared from the metal complex with thiophene as a secondary ligand. The ligand and its metal (II) complex were characterized using various spectroscopic methods FT-IR, UV-vis, ^{1}H-NMR and single-crystal x-ray diffraction. The analytical data suggested the ligand coordinated in a bidentate mode through N- and S- S-donor atoms to the metal centers.

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Authors' Contribution

Nsikanabasi Ekerete Nkom designed the study, conducted the synthesis, and prepared the first draft. Ededet Akpan Eno supervised the experimental design and analysis. Offiong Efanga Offiong contributed to spectroscopic characterization and interpretation. Atim Sunday Johnson handled the literature review and manuscript refinement. Idongesit Bassey Anweting critically revised the manuscript.