

Synthesis and Biological Activity and Thermal Analysis of Sulfaquinoxaline Mixed Metal Complexes

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ABSTRACT

New mixed sulfaquinoxaline metal complexes were synthesised by reacting sulfanamide ligand with transition metals which were as chlorides (Cr(III), Mn(II), Co(II), Cu(II), Hg(II), Fe(III), Zn(II), Cd(II), Ni(II)). The nine mixed complexes of sulfaquinoxaline is prepared by reflux method and determined by CHN analysis and IR. The results of IR spectral data showed that the binding of metals to the sulfonamidic nitrogen, oxygen of sulfone group and nitrogen of amino group. The geometries of all complexes are octahedral and that proved by magnetic measurements and spectral data. The thermogravimetric analysis (TGA) of mixed metal sulfaquinoxaline complexes and DTA that used to determine thermodynamic parameters of the thermal decomposition steps, E_a , ΔH^* and ΔS^* are carried out from ambient temperature to 700°C in inert nitrogen atmosphere.

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Introduction

Sulfonamides are the first antimicrobial agent used for eliminating a wide range of infections in human and other animal systems. Sulfaquinoxaline (Figure1) is one of sulfonamide family which used in veterinary medicine, meats, and in poultry industries [1-5]. Mixed metal complexes of sulfaquinoxaline is not reported in the literature and therefore, authors made and attempt to prepare mixed metal complexes of title compounds using routine reflux method and determined by spectroscopic and thermal analysis (DTA and TGA).

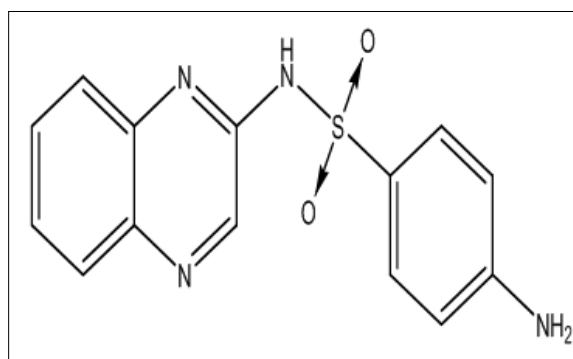


Figure 1: Sulfaquinoxaline (HL)

Experimental Procedure

Sulfaquinoxaline solution was applied to bidistilled water and the inorganic salts [Cr(III), Mn(II), Fe(III), Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II)] dissolved as chlorides in 40 mL ethanol getting nine mixed metal-ligand(sulfaquinoxaline)complexes. The molar quantity of metals chloride were mixed together as [Cu(II) and Co(II)], [Cu(II) and Zn(II)], [Cr(III) and Cd(II)], [Fe(III) and Mn(II)], [Hg(II) and Ni(II)], [Hg(II) and Fe(III)], [Cd(II) and Fe(III)], [Hg(II) and Zn(II)], each mixture of them was applied with constant stirring to the measured ligand quantity using specific mole ratios (M:M:L). Then the reaction mixture was refluxed for about 5 min then left over-night, the precipitates were formed, filtered and washed several times with a mixture of EtOH-H₂O and dried desiccator over anhydrous CaCl₂, the analytical results given in Tables (1) are determined by normal methods [6].

Table 1: Elemental Analysis, m.p., Formula, Stoichiometries and Color of Mixed Sulfaquinoxaline Complexes

| Complexes | Color | Calculated/(Found)% | | | | | |
|--|-------------|---------------------|----------------|------------------|----------------|------------------|------------------|
| | | C | H | N | S | M | Cl |
| [CrCd ₂ (HL) ₂ (H ₂ O) ₂ (Cl) ₅] | Green | 36.73 (36.95) | 3.08 (3.02) | 11.95 (11.90) | 6.84 (6.80) | 19.69 (19.52) | 12.61 (12.73) |
| [Mn Fe ₂ (HL) Cl ₄ (OH) ₂ (H ₂ O) ₆] | Brown | 22.39 (22.36) | 3.49 (3.52) | 7.46 (7.52) | 4.27 (4.32) | 22.19 (22.17) | 18.88 (18.82) |
| [FeHg(HL) ₂ Cl ₃ (H ₂ O) ₂] | Dark brown | 31.42 (31.47) | 2.64 (2.72) | 10.47 (10.41) | 5.99 (5.02) | 23.96 (23.87) | 16.56 (16.66) |
| [CoCu ₂ (HL)Cl ₄ (H ₂ O) ₈] | Green | 21.77 (21.72) | 3.65 (3.60) | 7.25 (7.15) | 4.15 (4.11) | 24.09 (24.05) | 18.36 (18.40) |
| [NiZn(HL)Cl ₄ (H ₂ O) ₃] | Green | 36.53 (36.41) | 3.28 (3.26) | 12.7 (12.12) | 6.97 (6.93) | 13.48 (13.38) | 15.40 (15.30) |
| [Cu Zn ₂ (HL) ₃ Cl ₄ (H ₂ O) ₄] | Green | 38.53 (38.51) | 3.39 (3.35) | 12.84 (12.81) | 7.35 (7.31) | 14.84 (14.89) | 10.83 (10.75) |
| [Zn Hg(HL) ₂ Cl ₄ (H ₂ O) ₃] | Pale orange | 31.65 (31.42) | 2.85 (2.81) | 10.55 (10.41) | 6.03 (6.05) | 25.03 (25.14) | 13.35 (13.23) |
| [Cd ₂ Fe (HL) ₃ Cl ₅ (H ₂ O) ₂] | Brown | 36.16 (36.10) | 2.89 (2.81) | 12.05 (12.02) | 6.89 (6.85) | 20.12 (20.03) | 12.71 (12.62) |
| [Hg Ni (HL) Cl ₄ (H ₂ O) ₄] | White | 21.74 (21.72) | 2.61 (2.55) | 7.24 (7.21) | 4.14 (4.15) | 33.52 (33.45) | 18.33 (18.29) |

All the Complexes have m.p > 300° C Measurements

The chloride content of complexes was determined by using the familiar Volhard method [7]. IR spectra of the sulfaquinoxaline and their mixed metal complexes were evaluated by Perkin Elmer spectrophotometer, Model 1430 with a range of 200-4000 cm⁻¹. Nujol mull spectra was used to determine spectra of sulfaquinoxaline and the solid mixed complexes by using Unicam UV/Vis spectrometer. Faraday's method were used to determine the magnetic measurements at room temperature (25 C). The reflection spectrometer operating was used to calculate ESR spectra at (9.1-9.8) GHZ in a cylindrical resonance cavity with modulation of 100 KHZ and by contrast with DPPH signal g values were determined. The differential thermal analysis (DTA) and thermogravimetric analysis (TGA) for sulfaquinoxaline and its mixed complexes were reported on Shimadzu DTA/TGA-60 thermal analyzer with heating rate 20°C/min under flow rate 20 ml/min of nitrogen atmosphere. The ligand was been applied by using Hyperchem. computer software using PM3 semi-empirical and Molecular Mechanics Force Field (MM+). Sulfaquinoxaline biological activity and its mixed metal complexes were examined against 5 microorganisms representing different categories of microbials.

Result and Discussion

Sulfaquinoxaline mixed metal complexes give broad bands in the 3315 – 3480cm⁻¹ region in all prepared complexes suggesting coordination with water which obtained from IR spectra of sulfaquinoxaline and its mixed complexes, Table (2). There was a metal-oxygen bands at 410 cm⁻¹ region [8] which indicate that there were coordinated water in these complexes. The bands appear between (3360-3437) and (3420-3576) cm⁻¹ are due to ν_{as}(NH₂) and ν_{sy}(NH₂) stretching vibrations of the amino (-NH₂) group for complexes but for sulfaquinoxaline the band appear between 3318 and 3356 cm⁻¹ due to ν_{as}(NH₂) and ν_{sy}(NH₂) stretching vibrations of the amino (-NH₂) group this shifting suggest that coordination occurred through nitrogen of amino group. The sharp band around 1350cm⁻¹ is assigned to SO₂ stretching vibration of sulfaquinoxaline at but for metal complexes is shifted to (1414-1416) cm- range that suggesting that ligand coordination with

metals through oxygen of sulfone group. The band of sulfonamidic NH group sulfaquinoxaline appears at 3192cm⁻¹ while for complexes appears at (3240-3361cm⁻¹) range for, this large shifting mean that there is metal ligand coordination occurred through nitrogen of secondary amino group. The narrow band appeared at 1308cm⁻¹ is assigned to C-N stretching vibration of spectrum of sulfaquinoxaline while for mixed complexes is correspond to (1307-1315cm⁻¹) that meaning that there is no shifting occurred in the band and so no metal ligand coordination occurred. The peak at 1637cm-corresponding to stretching vibration of C=N of sulfaquinoxaline but the complexes show this band at (1637-1642cm-) range so the shifting is not significant so there is no metal ligand coordination.. The sharp band appears at 2942cm-of CH stretching vibration of the spectrum of sulfaquinoxaline but for mixed complexes appears at (2920-2955) cm- range. The absorption band at 1598cm- is assigned to phenyl bending vibration of sulfaquinoxaline while for complexes appears at (1592-1594) cm⁻¹ range.

Electronic Spectral and Magnetic Studies

The pale green electronic absorption spectra of copper-cobalt complex [CoCu₂(HL)Cl₄(H₂O)₈] gave three bands at 302, 365 and 795 nm, Table (3). The nature of geometry is octahedral due to charge transfer of bands which are assigned to ²E_g → ²T_{2g} (D) transition and ⁴T_{1g}(F) → ⁴T_{2g}(P) transition. The octahedral geometry around the Cu (II) ions is determined by ESR data while the visible d-d electronic spectral band at 725 nm may probably be due to octahedral spatial configuration around Co (II). At room temperature the μ_{eff} value is of 6.51, that suggested the octahedral geometry of complex. The electronic pale green absorption spectra of copper-zinc complex [Cu Zn₂ (HL)₃Cl₄ (H₂O)₄] showed three bands at 285, 343 and 781 nm, Table(3). These values mean that the complex has octahedral geometry as result of 2E_g → 2T_{2g} (D) transition for copper and no d-d transition could be observed due to the d10- configuration of zinc so it,s geometry like that of copper. The bands obtained by electronic absorption spectra of iron-cadmium complex [Cd₂ Fe(HL)₃Cl₅(H₂O)₂] are of values 293, 338 and 359 nm, Table(3). Due to CT (t_{2g} → π*) and CT (π → e_g), Iron bands were appeared and no d-d transition could be observed due to the d10- configuration of cadmium (suggesting

that it,s geometry will similar to that of cadmium).The presence of octahedral geometry is suggested by μ_{eff} value that equal 4.56. Iron-manganese complex $[\text{Mn Fe}_2 (\text{HL}) \text{Cl}_4 (\text{OH})_2 (\text{H}_2\text{O})_6]$ showed three bands by electronic absorption spectra with values 293, 333 and 364 nm, Table(3). The appearance of octahedral geometry due to Due to CT ($t_{2g} \rightarrow \pi^*$) and CT ($\pi \rightarrow e_g$) of iron due to $\pi - \pi^*$ the first band appear while the second one is assigned to $6A_{1g} \rightarrow 4A_{1g}$, while the third is due to ${}^6A_{1g} \rightarrow {}^4T_{2g}$ transition[9-10]. with magnetic moment value equal to 4.8⁹ typified the existence of the complex in Oh geometry for complex. The electronic absorption spectra of iron-mercury complex $[\text{FeHg}(\text{HL})_2\text{Cl}_5(\text{H}_2\text{O})_2]$ appeared three bands at 280, 329 and 355 nm, Table(4-5), Figure(2,6).Iron bands appeared due to CT ($t_{2g} \rightarrow \pi^*$) and CT ($\pi \rightarrow e_g$) which assigned that the geometry is octahedral while mercury has no d-d transition could be observed due to the d_{10} - configuration so it,s geometry cannot be determined by ultraviolet-visible spectra and suggested that it similar to that of iron , So in general the geometry of complex is octahedral and with magnetic moment value equal to 4.32 B.M. The geometry of complex $[\text{Hg Ni} (\text{HL}) \text{Cl}_4(\text{H}_2\text{O})_4]$ octahedral because the three bands showed by electronic absorption spectra have values 288, 347 and 363 nm, Table (3). The geometry is assigned to ${}^4T_{1g}(\text{F}) \rightarrow {}^4T_{2g}(\text{P})$ transition of nickel but mercury cannot be determined by UV-VIS spectra as it has no d-d transition could be observed due to the d_{10} - configuration so expected to

be as nickel. Its room temperature μ_{eff} value of 2.44, typified the existence of Oh configuration. There are three bands obtained by electronic absorption spectra of zinc-mercury complex $[\text{ZnHg} (\text{HL})_2 \text{Cl}_4(\text{H}_2\text{O})_3]$ of 288, 314 and 347 nm, Table (3). Both of Zn(II) and Hg(II) has no d-d transition could be observed due to the d_{10} - configuration and cannot be determined by ultra-visible spectra and as a result the suggested geometry is octahedral like other complexes. The electronic absorption spectra of nickel-zinc complex $[\text{NiZn} (\text{HL})\text{Cl}_4 (\text{H}_2\text{O})_3]$ gave three bands at 291, 320 and 360 nm, Table (3). The geometry of complex is octahedral due to ${}^3T_1(\text{F}) \rightarrow {}^3T_1(\text{P})$ transition of nickel and zinc is suggested as nickel as it has no d-d transition could be observed due to the d_{10} - configuration (not determined by UV-VIS spectra). Its room temperature μ_{eff} value of 2.44, typified the existence of Oh configuration. The electronic absorption spectra of chromium-cadmium complex $[\text{CrCd}_2(\text{HL})_2(\text{H}_2\text{O})_2 (\text{Cl})_5]$ showed bands at 285, 309 and 479 nm which assigned to octahedral geometry as result of ${}^4A_{2g} \rightarrow {}^4T_{2g}(\text{F})$, ${}^4A_{2g} \rightarrow {}^4T_{1g}(\text{F})$ and ${}^4A_{2g} \rightarrow {}^4T_{1g}(\text{p})$ transitions for chromium but cadmium cannot be determined by spectra because it has no d-d transition could be observed due to the d_{10} - configuration so considered like chromium, Table(3). The octahedral geometry is suggested through Its room temperature μ_{eff} value of 6.40 B.M.

Table 2: Fundamental Infrared Bands (Cm⁻¹) of Sulfaquinoxaline and its Mixed Metal Complexes

| Compound | ν_{NH} 2 ^o amine | ν (O=S=O) | $\nu(\text{NH})$ 1 ^o amine sym | $\nu(\text{NH})$ 1 ^o amine asym | ν (C-N) | ν (C=N) | $\nu(\text{CH})$ stretch | $\nu(\text{NH})$ 1 ^o amine bend |
|---|---|------------------|---|--|-------------|-------------|-----------------------------|--|
| Sulfaquinoxaline | 3192 | 1350 | 3318 | 3356 | 1308 | 1637 | 2942 | 1598 |
| $[\text{CrCd}_2(\text{HL})_2(\text{H}_2\text{O})_2 (\text{Cl})_5]$ | 3240 | 1416 | 3361 | 3439 | 1313 | 1640 | 2905 | 1592 |
| $[\text{Mn Fe}_2 (\text{HL}) \text{Cl}_4 (\text{OH})_2 (\text{H}_2\text{O})_6]$ | 3240 | 1416 | 3360 | 3420 | 1313 | 1638 | 2920 | 1592 |
| $[\text{Cd}_2 \text{Fe} (\text{HL})_3\text{Cl}_5(\text{H}_2\text{O})_2]$ | 3075 | 1414 | 3360 | 3420 | 1310 | 1638 | 2920 | 1594 |
| $[\text{CoCu}_2(\text{HL})\text{Cl}_4(\text{H}_2\text{O})_8]$ | 3075 | 1416 | 3360 | 3440 | 1313 | 1639 | 2955 | 1592 |
| $[\text{Hg Ni} (\text{HL}) \text{Cl}_4(\text{H}_2\text{O})_4]$ | - | 1414 | 3361 | 3430 | 1311 | 1638 | 2937 | 1594 |
| $[\text{Cu Zn}_2 (\text{HL})_3\text{Cl}_4 (\text{H}_2\text{O})_4]$ | 3075 | 1412 | 3360 | 3440 | 1312 | 1639 | 2916 | 1594 |
| $[\text{Zn Hg}(\text{HL})_2 \text{Cl}_4(\text{H}_2\text{O})_3]$ | 3254 | 1412 | 3360 | 3439 | 1311 | 1639 | 2937 | 1593 |
| $[\text{NiZn}(\text{HL})\text{Cl}_4 (\text{H}_2\text{O})_3]$ | - | 1416 | 3360 | 3450 | 1313 | 1640 | 2920 | 1592 |
| $[\text{FeHg}(\text{HL})_2\text{Cl}_5(\text{H}_2\text{O})_2]$ | 3075 | 1414 | 3361 | 3436 | 1311 | 1637 | 2937 | 1594 |

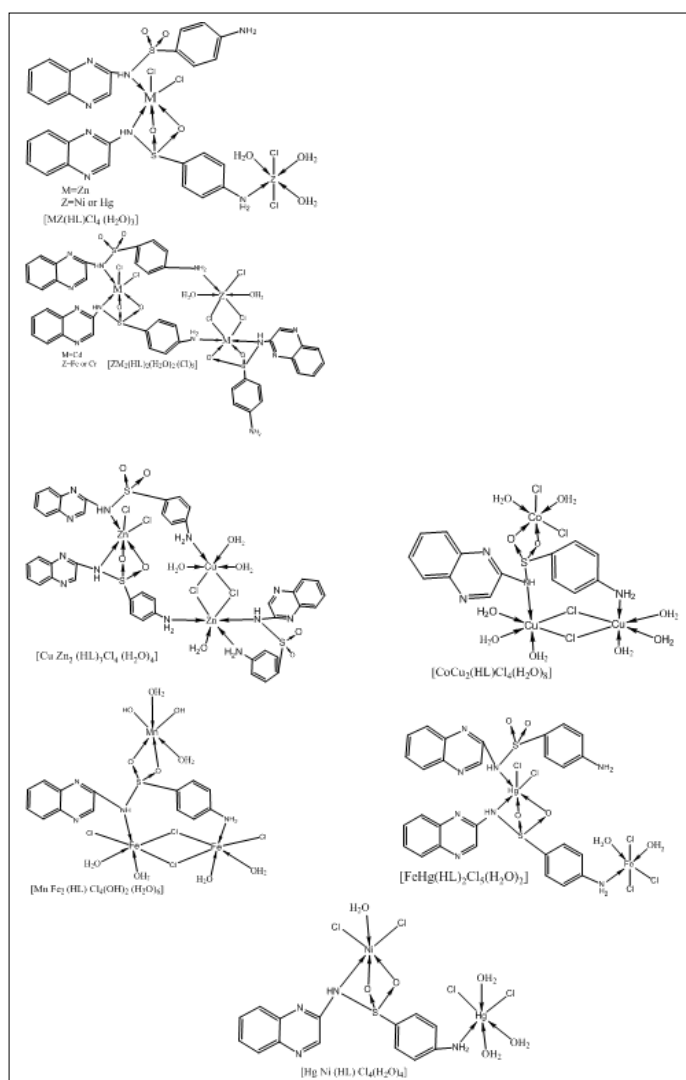


Figure 2: Proposed Structures of Sulfaquinoxaline Complexes

Table 3: Nujol Mull Electronic Absorption Spectra λ_{max} (nm), Room Temperature Effective Magnetic Moment Values (μ_{eff} at 298°K) and Geometries Of Sulfaquinoxaline Mixed Metal Complexes

| Complex | λ_{max} (nm) | μ_{eff} | Geometry |
|---|----------------------|-------------|----------------|
| [HgNi(HL)Cl ₄ (H ₂ O) ₄] | 288, 347, 363 | 2.44 | O _h |
| [CoCu ₂ (HL)Cl ₅ (H ₂ O) ₇] | 302, 365, 795 | 6.51 | O _h |
| [ZnHg(L) ₂ Cl ₂ (H ₂ O) ₂] | 288, 314, 347 | Diamagnetic | O _h |
| [CrCd ₂ (L) ₂ (H ₂ O) ₂ (Cl) ₃] | 285, 309, 492 | 6.40 | O _h |
| [NiZn(HL)Cl ₄ (H ₂ O) ₃] | 291, 320, 360 | 2.44 | O _h |
| [FeHg(HL) ₂ Cl ₅ (H ₂ O) ₂] | 280, 329, 355 | 4.32 | O _h |
| [MnFe ₂ (HL)Cl ₆ (H ₂ O) ₅] | 293, 333, 364 | 4.89 | O _h |
| [CdFe(HL) ₂ Cl ₅ (H ₂ O) ₂] | 293, 333, 364 | 4.56 | O _h |
| [CuZn ₂ (HL) ₃ Cl ₂ (H ₂ O) ₄] | 285, 343, 781 | 1.62 | O _h |

The groups that have the highest charges of sulfaquinoxaline are oxygen of sulfonyl group and nitrogen of both primary and secondary amine and so coordination occurred through those groups, charge density of sulfaquinoxaline atoms is calculated from hyper-chem program, Figure (3).

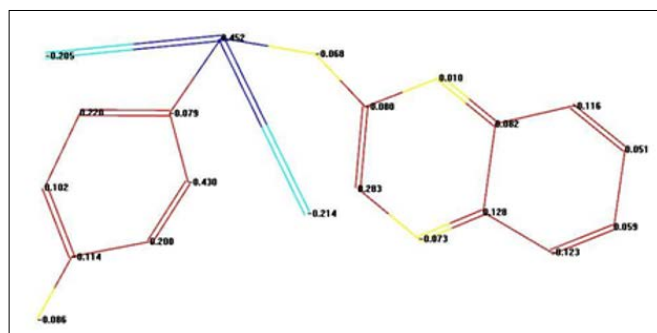


Figure 3: Charge density of sulfaquinoxaline

Electron Spin Resonance of Cobalt Copper Complex

The room temperature polycrystalline X-band ESR spectral pattern of [CoCu₂(HL)Cl₅(H₂O)₇] complex, figure (4) is isotropic nature with $g_s = 2.15$ and value of $A = 130$. The presence of ESR signals at $g < 4$ may assign the spin-spin interaction between the Cu and Co atoms show the diametric nature of complex.

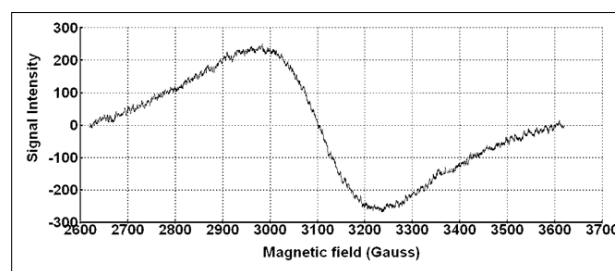


Figure 4: ESR of Cobalt Copper Sulfaquinoxaline Complex

Result and Discussion

Biological Activity

There were five microorganisms which involved {two Gram-positive (*Staphylococcus Aureas* ATCC6538P and *Bacillus subtilis* ATCC19659), two Gram negative (*Escherichia coli* ATCC8739 strain and *Pseudomonas aeruginosa* ATCC9027) bacteria and (*Candida albicans* as fungi) are used to study biological activity of sulfaquinoxaline and its complexes, in this study there are five complexes of different mixed metal ions are used and two different broadly antibiotics (Ciprofloxacin and Clotrimazole) are used as references (Table 4).

From the data we conclude that. All investigated compounds showed higher activity for *Staphylococcus aureus* except [MnFe₂(HL)Cl₆(OH)₂(H₂O)₆] which showed similar activity to it. The activities for all compounds are similar to *Candida albicans*, *Escherichia coli*, *Pseudomonas aeruginosa*, and *Bacillus subtilis* except [ZnHg(HL)₂Cl₄(H₂O)₃] that has higher activity to *Escherichia coli* and *Bacillus subtilis* which meant that presence of mercury affect on the activity of zinc in the complex. The activity of free ligands have lower activity than most of metal complexes, the increased activity of metal chelates could be discussed depending on the bases of overtone's concept and chelation theory [11-12]. As result of chelation, the polarity of metal ion is decreased to a larger extent due to the overlap of the ligand orbital and partial sharing of the positive charge of the metal ion with the donor groups which lead to raising the delocalization of p- and d-electrons over the whole chelate and enhances the lipophilicity of the complex. The cell is surrounded by the lipid membrane that its cell permeability prefers the passage of only lipid soluble materials depending on that liposolubility is an important factor that controls antimicrobial activity.

Table 4: The Antibacterial Activity of The Free Ligands and its Complexes Against Some Reference Strains Expressed in Absolute Activity (AU)

| complexes | Candida albicans | | Escherischia coli | | Pseudomonas aeruginosa | | Staphylococcus aureus | | Bacillus subtilis | |
|--|------------------|-----|-------------------|-----|------------------------|-----|-----------------------|-----|-------------------|-----|
| | Blank | Cpd | Blank | Cpd | Blank | Cpd | Blank | Cpd | Blank | Cpd |
| Sulfaquinoxaline | 9 | 9 | 9 | 9 | 9 | 9 | 9 | 9 | 9 | 9 |
| [Cu Zn ₂ (HL) ₃ Cl ₄ (H ₂ O) ₄] | 9 | 9 | 9 | 9 | 9 | 9 | 9 | 14 | 9 | 9 |
| [Zn Hg(HL) ₂ Cl ₄ (H ₂ O) ₃] | 9 | 9 | 16 | 9 | 9 | 9 | 9 | 23 | 9 | 17 |
| [[NiZn(HL)Cl ₄ (H ₂ O) ₃] | 9 | 9 | 9 | 9 | 9 | 9 | 9 | 16 | 9 | 9 |
| [Mn Fe ₂ (HL) Cl ₄ (OH) ₂ (H ₂ O) ₆] | 9 | 9 | 9 | 9 | 9 | 9 | 9 | 9 | 9 | 9 |
| [CoCu ₂ (HL)Cl ₄ (H ₂ O) ₈] | 9 | 9 | 9 | 9 | 9 | 9 | 9 | 25 | 9 | 9 |
| Ciprofloxacin (reference) | - | - | 9 | 30 | 9 | 30 | 9 | 30 | 10 | 30 |
| Clotrimazole (reference) | 9 | 18 | - | - | - | - | - | - | - | - |

Thermal Analysis

Masoud et al. has reported the thermal behavior of some biologically active compounds [13-19]. The thermograms (TGA) has been used to determine thermal decomposition of ligand and its investigated compounds, Decomposition occurred in two steps in the range of 50-700°C for sulfaquinoxaline. The first step of decomposition starts at 48°C and ends at 389.1°C with a corresponding weight loss 60.8%, DTA showed endothermic effect in this step. The last step of decomposition is observed at 389-684°C with a corresponding weight loss 17.90% that accompanied by exothermic effect from DTA curve.(Figure5,6), (Table 5,6)

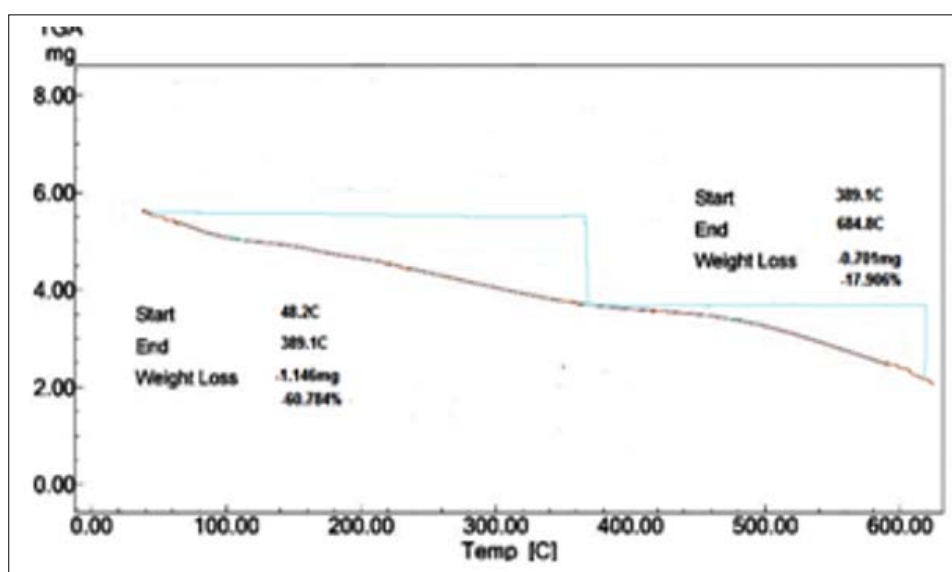
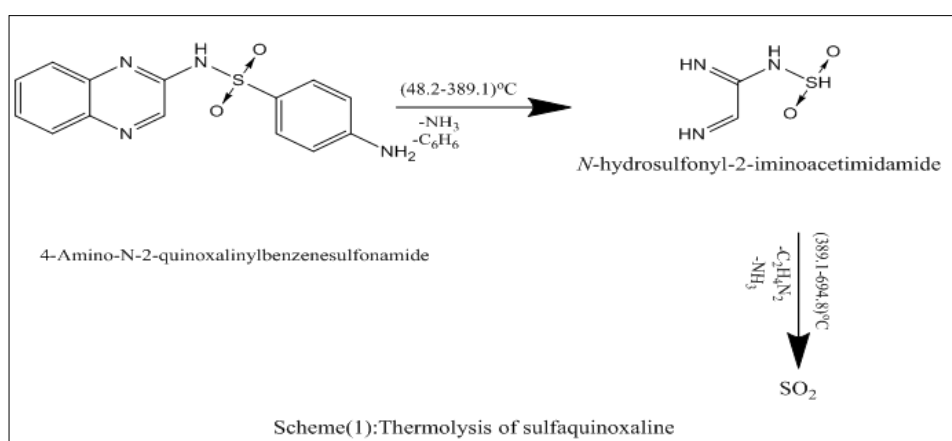


Figure (5):TGA of sulfaquinoxaline ligand

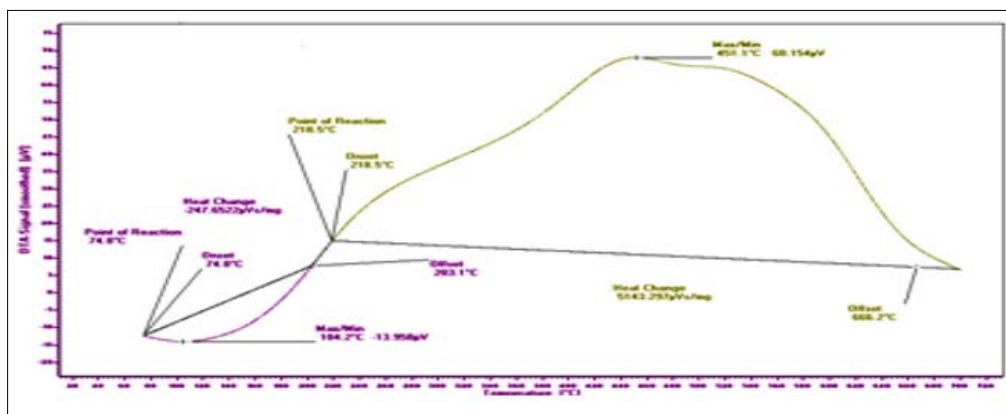


Figure 6: DTA of sulfaquinoxaline ligand

Table 5: DTA Analysis of Sulfaquinoxaline and its Mixed Complexes

| Complex | Type | Tm (°C) | Ea kJ mol ⁻¹ | n | am | DS# kJ K ⁻¹ mol ⁻¹ | DH# kJ mol ⁻¹ | 103 Z S ⁻¹ |
|---|------|---------|-------------------------|------|------|--|--------------------------|-----------------------|
| Sulfaquinoxaline | Endo | 104.2 | 3.38 | 1.99 | 0.50 | -0.30 | -31.41 | 0.004 |
| | Exo | 451.1 | 22.64 | 1.28 | 0.59 | -0.31 | -139.8 | 0.006 |
| [CoCu ₂ (HL)Cl ₃ (H ₂ O) ₇] | Endo | 90.5 | 2.86 | 1.87 | 0.51 | -0.30 | -27.19 | 0.004 |
| | Exo | 332 | 21.57 | 1.03 | 0.63 | -0.31 | -101.35 | 0.008 |
| | Exo | 555.8 | 27.38 | 1.22 | 0.59 | -0.31 | -173.34 | 0.006 |
| [Cd ₂ Fe(HL ₂) Cl ₅ (H ₂ O) ₂] | Endo | 82.6 | 11.43 | 1.99 | 0.50 | -0.29 | -23.74 | 0.017 |
| | Exo | 519.1 | 63.59 | 1.20 | 0.60 | -0.30 | -157.67 | 0.015 |
| [FeHg(HL) ₂ Cl ₅ (H ₂ O) ₂] | Endo | 92.1 | 12.98 | 1.66 | 0.54 | -0.29 | -26.54 | 0.017 |
| | Exo | 462.3 | 49.35 | 1.40 | 0.57 | -0.30 | -140.50 | 0.013 |
| [NiZn(HL)Cl ₄ (H ₂ O) ₃] | Endo | 133 | 6.12 | 1.99 | 0.50 | -0.30 | -39.97 | 0.006 |
| | Exo | 570 | 83.05 | 1.15 | 0.60 | -0.30 | -172.75 | 0.018 |

Table 6: TGA of Sulfaquinoxaline and its Mixed Complexes

| Complex | Temp. (°C) TGA | Wt. Loss % | | Assignment |
|---|------------------|------------|--------|---|
| | | Calc | Found | |
| Sulfaquinoxaline | 48.2-389.1°C | 59.60 | 60.87 | Elimination of NH ₃ and 2C ₆ H ₆ |
| | 389.1-684.6 °C | 17.20 | 17.71 | Elimination of C ₂ H ₄ N ₂ and NH ₃ |
| [CoCu ₂ (HL)Cl ₃ (H ₂ O) ₇] | 20.26 -87.13 °C | 18.89 | 18.91 | Elimination of 7H ₂ O |
| | 87.13-131.62 °C | 6.65 | 6.61 | Elimination of C ₆ H ₆ and 2HCl |
| | 131.62-232.07 °C | 10.02 | 9.96 | Elimination of C ₆ H ₇ N and C ₂ H ₄ N ₂ |
| | 232.07-321.04 °C | 2.89 | 2.86 | Elimination of NH ₃ and S |
| [FeHg(HL) ₂ Cl ₅ (H ₂ O) ₂] | 20.97-97.07 °C | 18.32 | 18.36 | Loss of 4H ₂ O , 8HCl and 2NH ₃ |
| | 97.07-149.14 °C | 3.18 | 3.17 | Loss of 4C ₆ H ₆ and 2HCl |
| | 149.14-230.68 °C | 3.49 | 3.55 | Loss of 4C ₆ H ₆ and 2NH ₃ |
| | 230.68-334.82 °C | 4.38 | 4.36 | Loss of 4C ₂ H ₄ N ₂ and 2NH ₃ |
| | 334.82-458.42 °C | 3.25 | 3.28 | Loss of 2NH ₃ ,3SO and S |
| [Cd ₂ Fe(HL ₂) Cl ₅ (H ₂ O) ₂] | 25.98-84.51°C | 25.93 | 25.90 | Elimination of 2H ₂ O,3C ₆ H ₆ and3HCl |
| | 84.51-230.27°C | 5.34 | 5.29 | Elimination of 4NH ₃ ,2C ₂ H ₄ N ₂ and2 HCl |
| | 230.27-377.37°C | 21.21 | 21.25 | Loss of 2NH ₃ , 3C ₆ H ₆ , 2S and SO |
| [NiZn(HL)Cl ₄ (H ₂ O) ₃] | 20.24-105.05 °C | 13.87 | 13.90 | Loss of 3H ₂ O and 2HCl |
| | 105.05-217.93 °C | 12.62 | 12.64 | Elimination of 2HCl and 2C ₆ H ₇ N |
| | 217.93-362.62 °C | 11.98 | 12.003 | Elimination of 2C ₈ H ₆ N ₂ and NH ₃ |
| | 362.62-475.50 °C | 2.65 | 2.61 | Loss of 2SO and NH ₃ |

In case of $[\text{NiZn}(\text{HL})\text{Cl}_4(\text{H}_2\text{O})_3]$ complex, (Table 5,6) (Figure7,8), there is no weight loss up to 20.24°C. The decomposition occurred in 4 steps, the first step of decomposition starts at 20.24°C and ends at 105.05°C with a corresponding weight loss 15.62%. The second step of decomposition occurred at (105.05-217.93)°C range with 12.63% weight loss. In case of third stage the decomposition starts at 217.93°C and ends at 362.62°C with a weight loss 12.003%. In the last step the weight loss was of 2.61% from decomposition at (362.62-475.50)°C range. The DTA data showed two peaks at 133 and 570°C first one is endothermic and second is with exothermic effect.

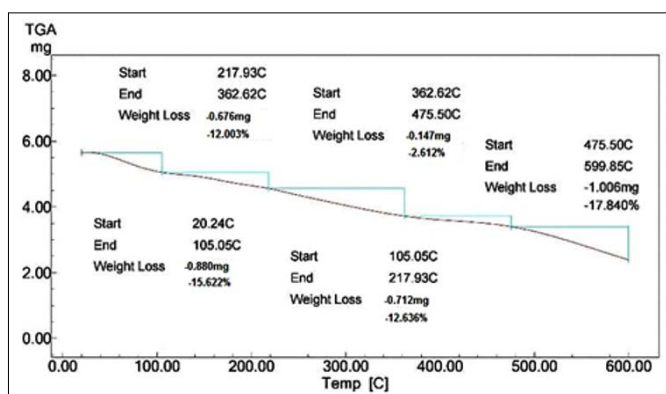


Figure 7: TGA of Ni-Zn Sulfaquinoxaline Complex

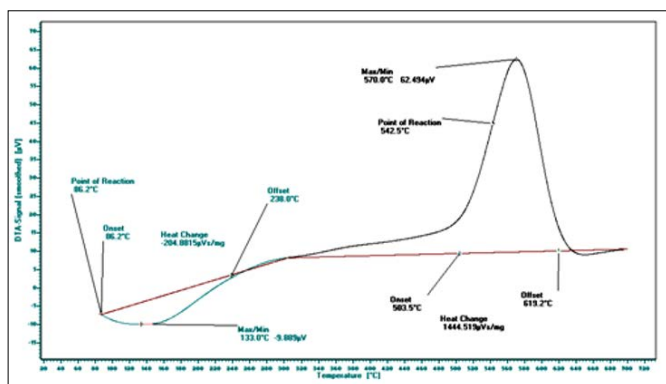


Figure 8: DTA of Ni-Zn Sulfaquinoxaline Complex

In case of $[\text{Cd}_2\text{Fe}(\text{HL})_3\text{Cl}_5(\text{H}_2\text{O})_2]$ complex, (Figure9,10), (Table5,6), there is no weight loss up to 25.98°C. TGA curve showed that the decomposition occurred 3 steps. In the first step the decomposition starts at 25.98°C and ends at 84.51°C with a corresponding weight loss 25.89%. The decomposition in second step include 5.48% weight loss and occurred at (84.51-230.27)°C. In case of last step the decomposition starts at 229.70°C and ends at 377.73°C with a corresponding weight loss 21.25. The DTA data gave two peaks at 82.6 and 519.1°C first one is endothermic and second is with exothermic effect.

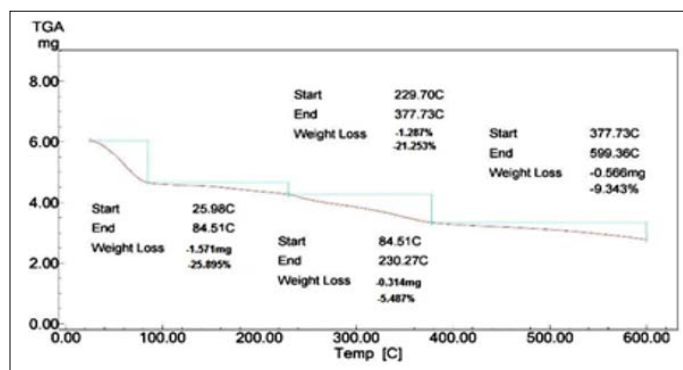
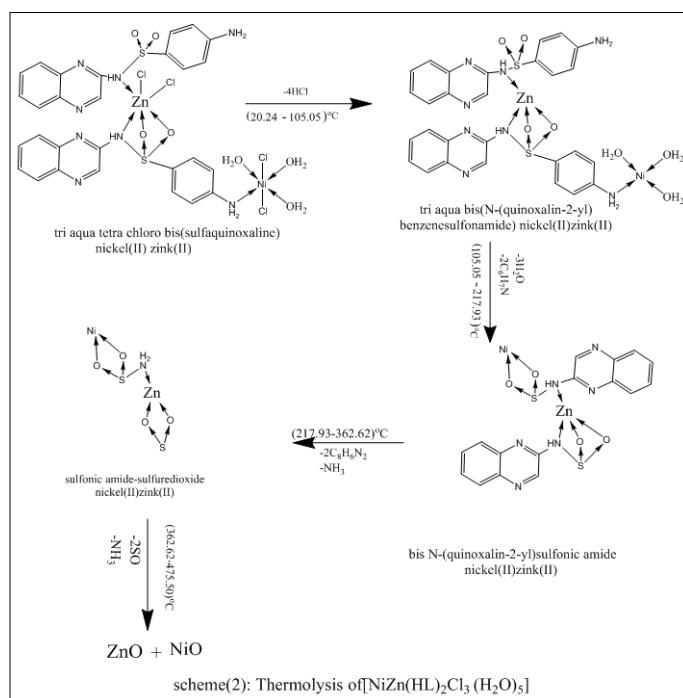


Figure 9: TGA of Fe-Cd Sulfaquinoxaline Complex

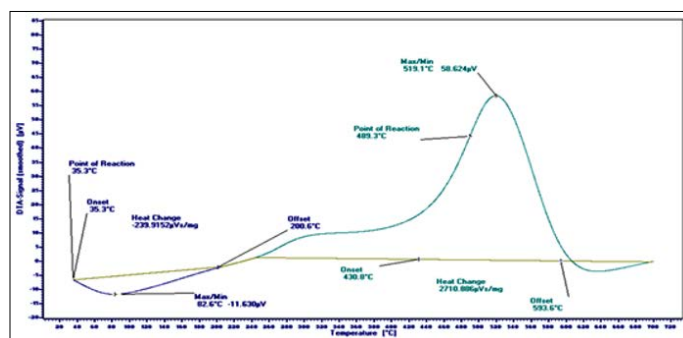


Figure 10: DTA of Fe-Cd Sulfaquinoxaline Complexes

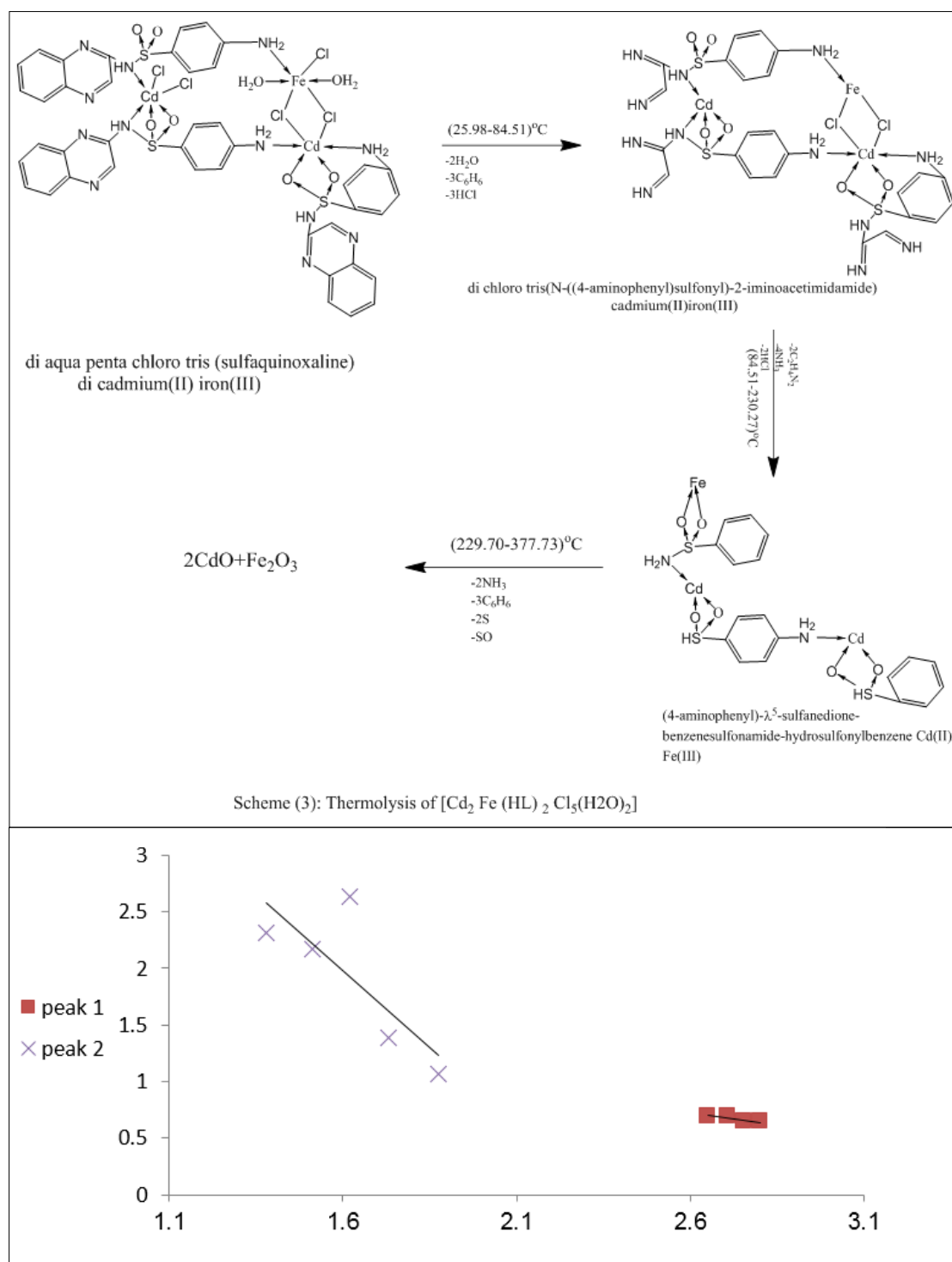


Figure 11: $\ln\Delta T$ against $103/T$ Relation for Sulfaquinoxaline Ligand

Conclusion

Different spectroscopic methods are used to analyze the prepared mixed metals complexes of sulfaquinoxaline and its Stoichiometries are different. Nujol and ESR spectra are used to confirm geometry which are Oh geometry for all mixed complexes have. Sulfaquinoxaline has different sites available for coordination which carries more electronegative charges and hyper chem. program discuss these results. The thermal decomposition of the complexes ended with the formation of metal oxides as a final product. The activity of sulfaquinoxaline complexes are higher than industrial sulfaquinoxaline for Staphylococcus aureus except $[Mn Fe_2 (HL) Cl_4(OH)_2 (H_2O)_6]$ complex. $[Zn Hg(HL)_2 C_{14}(H_2O)_3]$

has higher activity to Escherischia coli and Bacillus subtilis than sulfaquinoxaline while the other complexes has similar activity.

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