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Synthesis and characterization of metal complexes of 4-((furan-2-ylmethylene) amino) benzene sulfonamide

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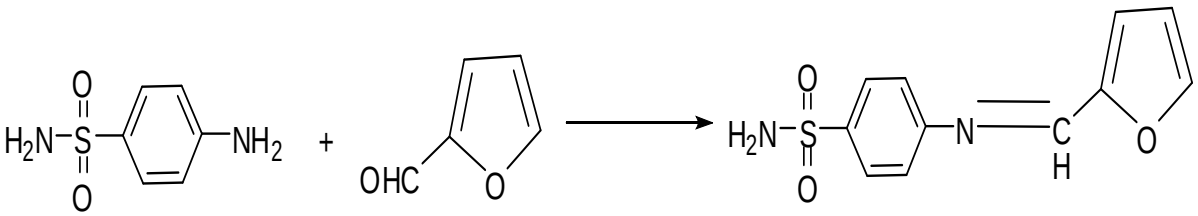
Abstract: *The Fe(III), Ru(III), Co(II), Ni(II), Cu(II), Pd(II), Zn(II), Cd(II) and Hg(II) complexes of a Schiff base derived from 4-aminobenzene sulfonamide and furan-2-carbaldehyde (FMABS, Scheme:1) have been synthesized and structurally characterized by various physico-chemical data. The ligand acts as a neutral, bidentate one towards the metal ions coordinating through azomethine nitrogen and furan oxygen. The geometry and the bonding characteristics associated with the complexes have been deduced from relevant spectral data. All the complexes are coloured and stable towards air and moisture. Fe, Ru, Co and Cu complexes are paramagnetic while the remaining are diamagnetic. Consolidating all the data obtained, the Fe, Ru and Cu complexes have been assigned an octahedral geometry, Ni and Pd complexes, a square planar geometry and the Zn, Cd and Hg complexes, a tetrahedral geometry.*
Further, the ligand and the metal complexes have been screened for their antimicrobial activities against two gram positive bacterial strains Basillus Subtillus, Staphylococcus Aurus and two gram negative bacterial strains Escherichia coli, Salmonella typhi, and two fungal strains Aspergillus niger and Penicillium rubrum by agar plate technique and the results are presented. Both the antibacterial and antifungal activities of the synthesized metal complexes have been found to be more as compared to those of the ligand.
Keywords: *Sulfonamide, furan-2-cabaldehyde, Schiff base, metal complexes, Synthesis, Characterization.*

I. Introduction

Sulfa drugs are used for the treatment of bacterial infections, such as eye infections, influenza, meningitis, actinomices infections, and urinary tract infections. They can also be used as model compounds for mechanistic investigation of the action of drugs[1,2]. Sulfanilamide itself, a potent antibiotic, never gained wide-spread use due to its greater human toxicity versus its various derivatives. In the long history of drug discovery, an interesting phenomenon has been noted that compounds with the same structural feature show diverse biological activities. For instance, sulfonamides, with different substituted groups, display a wide variety of pharmacological activities such as antibacterial, insulin-releasing antidiabetic, carbonic anhydrase inhibitory, high-ceiling diuretic, and antithyroid[3]. Recently many papers have reported several kinds of antitumor agents possessing the structural features of sulfonamide[4,5]. The present paper is oriented towards the synthesis of sulfonamide based Schiff base by condensing 4-aminobenzenesulfonamide with furan-2-cabaldehyde.



II. Expermental

All the chemicals used were of AR or BDH grade. The ligand FMABS was prepared by refluxing an equimolar mixture of furan-2-carbaldehyde and 4-aminobenzenesulfonamide in methanol in presence of a few drops of acetic acid for about 3 hrs. The solid that separated was filtered, washed with water and recrystallized from methanol. The colour, yield (%), m.p.(^{0}C) and C, H, N analysis (%) of FMABS are respectively, black, 62, 130 and 51.54, 3.88, 10.52.

The Fe(III), Ru(III), Co(II), Cu(II), Pd(II), and Hg(II) complexes of the ligand were prepared taking metal chlorides, and Ni(II), Zn(II) and Cd(II) complexes taking respective metal acetates. In the preparation metal complexes, the metal and the ligand were combined in 1:2 mole ratio in the case of Fe(III), Ru(III) and Cu(II) and in 1:1 ratio in the case of Co(II), Ni(II), Pd(II), Zn(II), Cd(II) and Hg(II) using required quantities of methanol so as effect the solubility of the metal salts and ligand. The contents were refluxed on a hot water bath for 2-3 hrs and the solid that separated was filtered , washed with water, hot methanol and ether and was vacuum dried over fused $CaCl_2$.

The elemental analysis (C,H,N) for the ligand and the complexes were carried out at CDRI, Lucknow. Conductance measurements on the complexes were made in DMF at 10^{-3} M concentration on a Digisun digital conductivity meter. Gouy balance calibrated with $Hg[Co(NCS)_4]$ was used to measure the magnetic susceptibility of metal complexes at room temperature. The IR spectra of the ligand and its metal complexes in KBr were recorded in the range 4000-450 cm^{-1} using Perkin Elmer FT-IR spectrophotometer. The electronic spectra of the metal complexes were recorded on Perkin Elmer UV-Vis spectrophotometer. WIN-EPR (BRUKER) spectrophotometer operating in the frequency range 8.8-9.6 GHz was employed in recording the ESR spectrum of Cu(II) complex in DMF at LNT.

III. Results And Discussion:

All the complexes are coloured, stable at room temperature and are non-hygroscopic. The ligand and its metal complexes are mostly insoluble in water, very slightly soluble in hot methanol and fairly soluble in dimethylformamide. The analytical and physical data of the metal complexes are given in Table-1.

Table-1: Analytical and physical data of metal complexes

Metal complex	Colour	Percent			Molar Conductance $\Omega^{-1}cm^2mol^{-1}$	μ_{eff} B.M.
		C	H	N		
Fe $(C_{11}H_{10}N_2O_3S)_2Cl_3$	Reddish brown	39.18 (39.87)	2.98 (3.04)	8.04 (8.45)	66	5.80
Ru $(C_{11}H_{10}N_2O_3S)_2Cl_3$	Black	37.02 (37.32)	2.72 (2.85)	7.42 (7.91)	54	1.82
Co $(C_{11}H_{10}N_2O_3S)Cl_2$	Dark brown	41.21 (41.92)	2.98 (3.20)	9.12 (9.35)	12	4.52
Ni $(C_{11}H_{10}N_2O_3S)(OAc)_2$	Dark brown	45.50 (46.10)	3.54 (3.87)	7.90 (8.27)	14	--
Cu $(C_{11}H_{10}N_2O_3S)_2Cl_2$	Gray	40.88 (41.61)	2.94 (3.17)	8.06 (8.82)	16	1.82
Pd $(C_{11}H_{10}N_2O_3S)Cl_2$	Black	38.16 (38.98)	2.84 (2.97)	7.89 (8.27)	13	--
Zn $(C_{11}H_{10}N_2O_3S)(OAc)_2$	Light black	44.96 (45.65)	3.14 (3.83)	7.86 (8.19)	16	--
Cd $(C_{11}H_{10}N_2O_3S)(OAc)_2$	Brown	42.04 (42.72)	3.06 (3.58)	7.02 (7.66)	17	--
Hg $(C_{11}H_{10}N_2O_3S)Cl_2$	Gray	33.76 (34.23)	2.22 (2.61)	6.92 (7.26)	18	--

Values in parentheses are the calculated ones.

The per cent values of the elements : carbon, hydrogen and nitrogen in the complexes have been calculated as per the composition given. It may be seen from the table that there is a fair agreement between the experimental and calculated values suggesting the composition as given. The molar conductance values indicate that the Fe(III), and Ru(III) complexes are 1:1 electrolytes while all others are non-electrolytes [6,7]. The magnetic studies reveal that the Fe(III), Ru(III), Co(II) and Cu(II) complexes are paramagnetic to the extent of respectively five, one, four and one unpaired electrons while others are diamagnetic[8].

The IR spectral data of FMABS and its complexes are presented in Table 2. The band that shows up around 1627 cm^{-1} in the ligand is due to azomethine group and it has been lower shifted by 15-20 cm^{-1} in the complexes indicating that the nitrogen of this group is coordinated to the metals.[9] The presence of SO_2 group in the ligand is indicated by two bands at 1160 and 1330 cm^{-1} due to its symmetric and asymmetric stretching vibrations respectively and they remain unshifted or higher shifted in the complexes indicating that the SO_2 group is not involved in coordination.[10] Further, the ligand shows a band at 883 cm^{-1} due to νC-O furan cyclic which has been lower shifted in its complexes indicating that furan oxygen is involved in coordination.[11] These observations suggest that the ligand acts as a neutral bidentate one towards the metal ions coordinating through azomethine nitrogen and furan oxygen[12-13].

Table-2 IR Spectral data of FMABS and its complexes :

S.No.	Compound	νC=N	ν $SO_{2\ Sy}$	ν $SO_{2\ Asy}$	νC-O
1	FMABS	1627	1160	1330	883
2	Fe-FMABS	1597	1203	1397	860
3	Ru-FMABS	1587	1205	1398	865
4	Co-FMABS	1594	1203	1398	858
5	Ni-FMABS	1605	1203	1398	864
6	Cu-FMABS	1594	1205	1400	862
7	Pd-FMABS	1602	1203	1397	868
8	Zn-FMABS	1600	1203	1398	865
9	Cd-FMABS	1602	1205	1398	860
10	Hg-FMABS	1583	1203	1400	858

The electronic spectral data of the Ru(III), Co(II), Ni(II), Cu(II) and Pd(II) complexes along with the assignment are given here under.

Complex	Frequency (cm^{-1})	Assignment
Ru-FMABS	10610	$^2T_{2g} \rightarrow {}^4T_{1g}$
	15110	$^2T_{2g} \rightarrow {}^4T_{2g}$
	22230	$^2T_{2g} \rightarrow {}^2A_{2g}$
Co- FMABS	10080	$^4A_2(F) \rightarrow {}^4T_2(F)$
	18250	$^4A_2(F) \rightarrow {}^4T_1(F)$
	26310	$^4A_2(F) \rightarrow {}^4T_1(P)$
Ni- FMABS	15230	$^1A_{1g}(D) \rightarrow {}^1A_{2g}(G)$
	21690	$^1A_{1g}(D) \rightarrow {}^1Eg(G)$
Cu- FMABS	15110	$^2B_{1g} \rightarrow {}^2E_g$
	21600	$^2B_{1g} \rightarrow {}^2E_g$
Pd- FMABS	15800	$^1A_{1g} \rightarrow {}^1A_{2g}$
	18650	$^1A_{1g} \rightarrow {}^1B_{1g}$
	26310	$^1A_{1g} \rightarrow {}^1E_g$

The transitions assigned are characteristic of octahedral geometry for Ru (III) (low spin) and Cu(II) complexes, tetrahedral for Co(II) complex and square planar for Ni(II) and pd(II) complexes[14-18].

The Fe(III), Zn(II), Cd(II) and Hg(II) complexes reveal no d-d bands owing to spin forbidden nature in the former and to the filled d configuration in the latter three. On the basis of other data obtained for them, the Fe(III) complex has been assigned high spin octahedral geometry and the Zn(II), Cd(II), and Hg(II) complexes, tetrahedral geometry.

The ESR spectrum of Cu(II) complex is anisotropic in nature with $g_{\|} > g_{\perp}$ indicating that the unpaired electron is present in the $d_{x^2-y^2}$ orbital giving $^2B_{1g}$ as the ground state[19-20].

Antimicrobial activity:

The compounds were assayed for their antibacterial and antifungal activities by seeded plate technique.[21] From the stock solutions of the test compounds in acetone, solutions, each of concentration 0.1mg/ml, were prepared by dilution with acetone.

The antimicrobial activity of FMABS and its Cu, Co, Pd and Hg complexes has been studied against bacteria: Bacillus subtilis, Staphylococcus aureus (Gram +ve), and Escherichia coli, Salmonella typhi (Gram –ve) and fungi: Penicilliumrubrum and Aspergillusniger wherein the zone of inhibition measured is recored in Table 3.

Table-3: Antimicrobial activity of FMABS and its complexes:

S.No.	Compound	Zone of inhibition (mm)					
		Gram **positive** Bacteria		Gram **negative** Bacteria		**Fungi**	
		B.subtilis	**S.aureus**	**E.coli**	**S.typhi**	**P.rubrum**	**A.niger**
1	FMABS	14	18	13	20	24	30
2	Cu- FMABS	12	17	10	16	12	13
3	Co- FMABS	30	28	15	21	--	42
4	Pd- FMABS	12	10	13	--	35	13
5	Hg- FMABS	28	30	22	30	30	22
6	Standard - Streptomycinsulphate	29	30	34	24	15	10

The results indicate that the complexes are in general more active than the free ligand. Further, Hg complex, of all the compounds, exerts highest activity on the bacteria as well as fungi studied.

IV. Conclusions

Based on the foregoing data, it may be concluded that the ligand acts as a neutral, bidentate one coordinating to the metals through nitrogen of azomethine group and furan oxygen. Fe(III), Ru(III) and Cu(II) complexes are octahedral, Co(II), Zn(II), Cd(II) and Hg(II) complexes are tetrahedral and Ni(II) and Pd(II) complexes are square planar in geometry. The metal complexes are more active in inhibiting the microbial growth than the ligand, the Hg complex exerting the highest activity.

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