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Synthesis and characterization of Fe(III)-azomethine complex

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ABSTRACT

The present study is being down on synthesis of chelating agent, which might have selectively for single metal ion. In the view of these consideration the chelating nature of azomethine derived from the para-aminobenzoic acid and substituted benzaldehyde towards the metal ions mainly Fe(III) is undertaken. Many fold aspects of complexasation of these azomethine is still lacking. Thus it was decided to undertake such work, the synthesis, characterization of some azomethine and study their magnetic susceptibility after complexarion. The synthesis of azomethine and Fe(III) complexes still not mentioned in any record because of the oxidizing nature of Fe(II) ion in solution. Therefore, the main aim of azomethine complexes and to study their magnetic susceptibility.

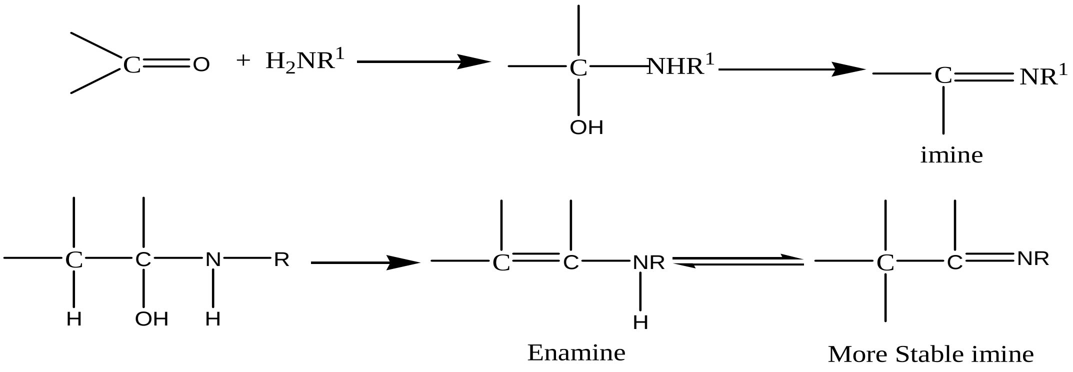
Keywords: Fe (III)-azomethine complex, synthesis, characterization, magnetic susceptibility.

INTRODUCTION

Chemistry of complex compounds which contain transition metal ion as nucleus have been intensively studied and this is an active and challenging field for theoretical and experimental research. Coordination chemistry is not important only for inorganic chemist; but it is also broad constituency of researchers ranging from biochemistry to material scientist. Thus coordination chemistry is not disciplinary but it is interdisciplinary in nature.

Azomethine ligands are able to coordinate metal through imine nitrogen and another group, usually linked to carbonyl group. In fact, azomethine ligands are able to coordinate many different metal and to stabilized them in various oxidation states. They have been widely studied because of their application in many field of fundamental and applied research. A number of azomethine used for the qualitative analysis[1,2,3] quantitative analysis[4,5,6]can also be applied for their agriculture uses[7],and in organic synthesis[8].

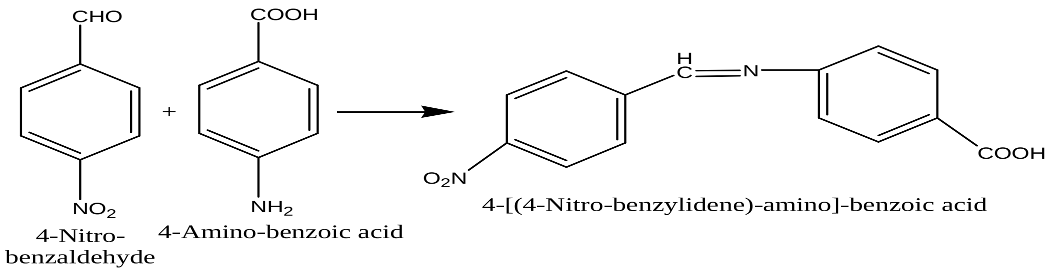
The interest in the field of coordination chemistry of azomethine arose out of the exploration of several chelating agents, which lead to the synthesis of several novel group of coordination complexes. Amine reacts with carbonyl compounds by nucleophilic addition. If amine is primary, the initial addition product undergoes dehydration to form a compound containing a carbon-nitrogen double bond, an imine. Elimination occurs with this orientation ever if the carbonyl compound containing as α-hydrogen, that is the preffered product is the imines rather than the enamine. If some enamine should be formed initially, it rapidly toutomerizes into more stable iminoform[9].



MATERIALS AND METHODS

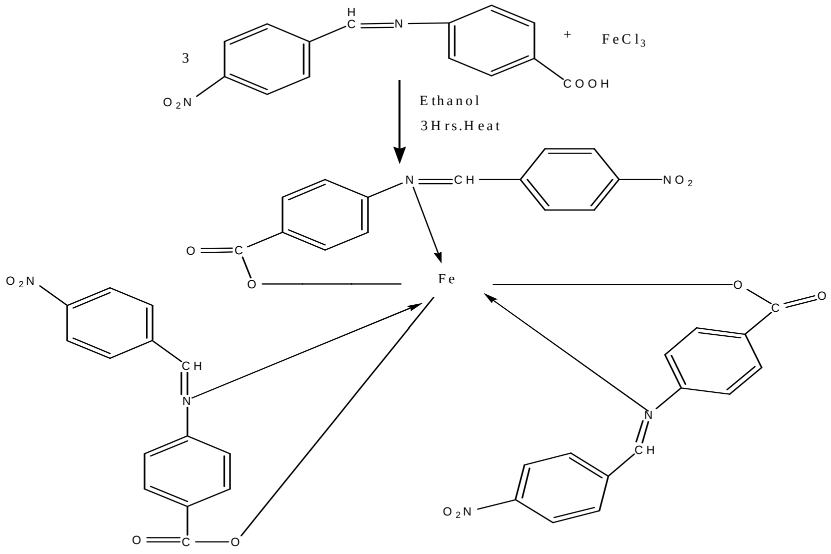
Synthesis of Azomethine

A mixture of 4-Nitrobenzaldehyde (0.5Mole) and 4-aminobenzoic acid (0.5Mole) and (0.5 mL) of Lemon Juice was grinding at room temperature for 20 minutes. After completion of the reaction as indicated by TLC, the reaction mixture was poured on to crushed ice and stirred for 5-10 minutes. The solid separated was filtered under suction, washed with ice cold water and then recrystallized from ethanol[10].



Synthesis of Fe(III) complex of Azomethine

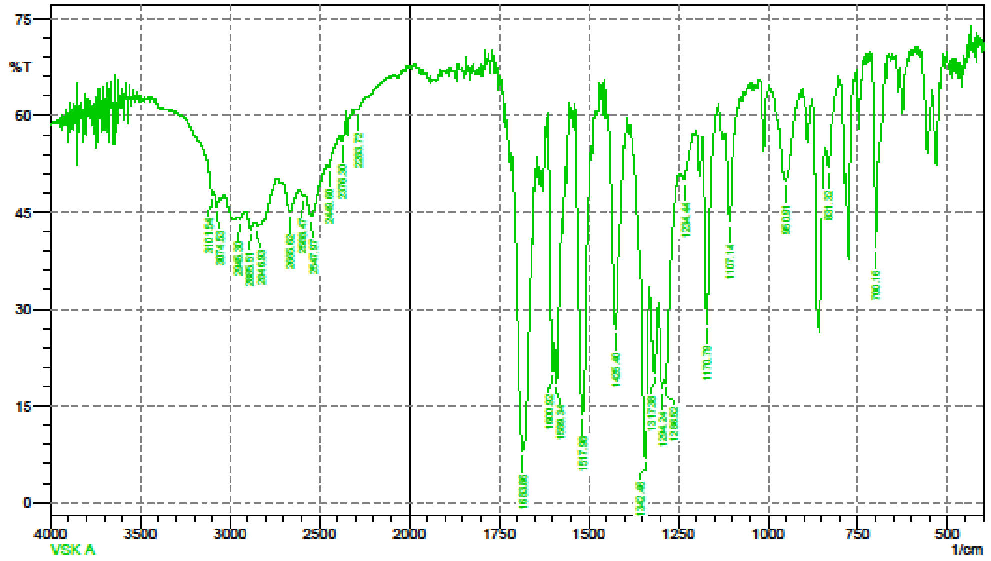
The complex was prepared by carrying out the insitu reaction of 4-[(4-Nitro-benzyledene)-amino]-benzoic acid and metal salt ferric chloride. 4-Nitrobenzaldehyde(1mole) and 4-aminobenzoic acid (1mole) were taken together in ethanol (30ml) and refluxed for an hour after which ferric chloride (1mole) in ethanol was added and refluxed or another 3 hours. On cooling microcrystalline iron complexes precipitated.filtered, washed thoroughly with ethanol and dried. The yield was quantitative.



CHARACTERIZATION

A) IR spectra of Azomethine

SHIMADZU



	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	700.16	39.546	29.634	713.66	673.16	10.161	3.581
2	831.32	51.932	6.039	840.96	804.32	8.555	0.578
3	950.91	49.876	14.077	995.27	914.26	19.696	3.993
4	1107.14	43.552	15.113	1118.71	1078.21	10.772	1.763
5	1170.79	27.87	30.169	1182.36	1139.93	13.842	4.196
6	1234.44	50.02	1.975	1240.23	1209.37	8.469	0.223
7	1286.52	18.551	2.18	1288.45	1249.87	17.265	0.246
8	1294.24	17.598	4.515	1305.81	1290.38	10.424	0.768
9	1317.38	20.172	11.035	1328.95	1307.74	12.984	2.246
10	1342.46	6.876	31.801	1382.96	1330.88	27.88	9.065
11	1425.4	26.899	32.537	1444.68	1392.61	18.961	7.212
12	1517.98	13.786	45.056	1537.27	1494.83	21.817	11.878
13	1589.34	18.648	8.879	1593.2	1575.84	9.216	0.878
14	1600.92	20.405	15.289	1612.49	1595.13	9.356	1.675
15	1683.86	8.012	38.235	1712.79	1658.78	36.914	18.706
16	2283.72	60.901	0.302	2287.58	2249	8.074	0.034
17	2376.3	56.049	1.979	2387.87	2351.23	8.832	0.38
18	2449.6	52.155	0.708	2459.24	2389.8	18.366	0.093
19	2547.97	44.404	4.534	2582.68	2461.17	39.04	2.364
20	2588.47	47.523	0.213	2613.55	2584.61	9.24	0.034
21	2665.62	45.145	4.056	2736.99	2615.47	39.043	1.744
22	2846.93	42.832	1.322	2860.43	2738.92	41.544	1.375
23	2885.51	42.434	1.782	2912.51	2862.36	18.147	0.386
24	2945.3	44.042	0.487	2958.8	2914.44	15.606	0.101
25	3074.53	45.774	1.956	3091.89	3055.24	12.104	0.311
26	3101.54	47.694	1.286	3174.83	3093.82	22.598	0.098

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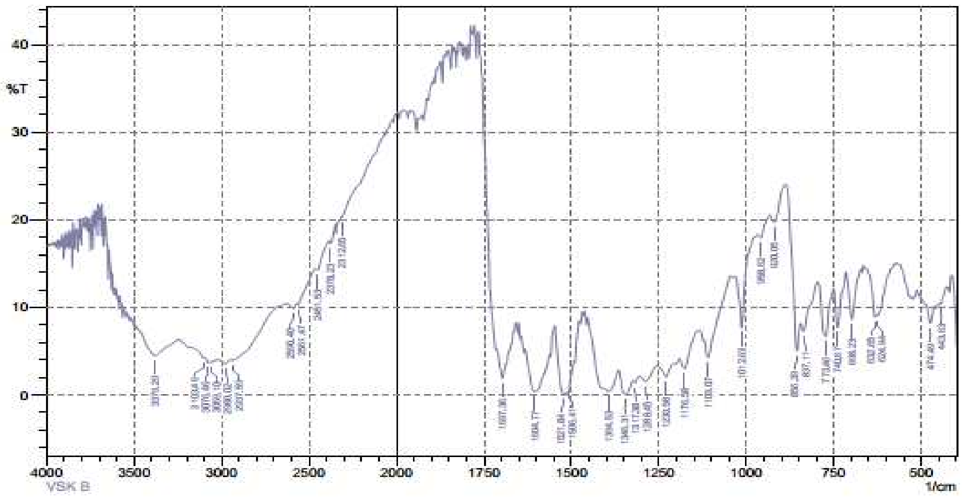
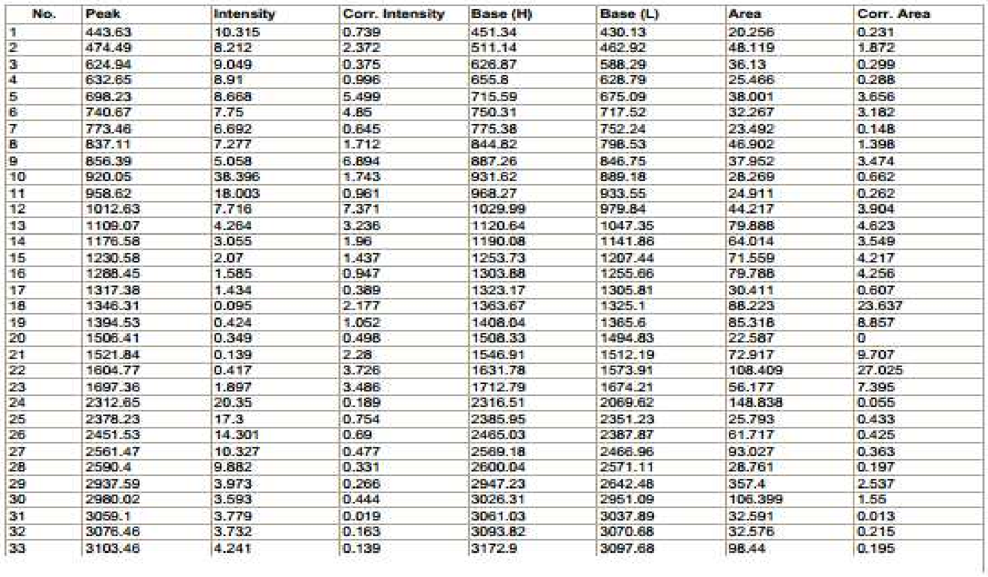
B)IR spectra of Fe(III) complex Azomethine





No.	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	443.63	10.315	0.739	451.34	430.13	20.256	0.231
2	474.49	8.212	2.372	511.14	462.92	48.119	1.872
3	624.94	9.049	0.375	626.87	588.29	36.13	0.299
4	632.65	8.91	0.996	655.8	628.79	25.466	0.288
5	698.23	8.668	5.499	715.59	675.09	38.001	3.656
6	740.67	7.75	4.85	750.31	717.52	32.267	3.182
7	773.46	6.692	0.645	775.38	752.24	23.492	0.148
8	837.11	7.277	1.712	844.82	798.53	46.902	1.398
9	856.39	5.058	6.894	887.26	846.75	37.952	3.474
10	920.05	38.396	1.743	931.62	889.18	28.269	0.662
11	958.62	18.003	0.961	968.27	933.55	24.911	0.262
12	1012.63	7.716	7.371	1029.99	979.84	44.217	3.904
13	1109.07	4.264	3.236	1120.64	1047.35	79.888	4.623
14	1176.58	3.055	1.96	1190.08	1141.86	64.014	3.549
15	1230.58	2.07	1.437	1253.73	1207.44	71.559	4.217
16	1288.45	1.585	0.947	1303.88	1255.66	79.788	4.256
17	1317.38	1.434	0.389	1323.17	1305.81	30.411	0.607
18	1346.31	0.095	2.177	1363.67	1325.1	88.223	23.637
19	1394.53	0.424	1.052	1408.04	1365.6	85.318	8.857
20	1506.41	0.349	0.498	1508.33	1494.83	22.587	0
21	1521.84	0.139	2.28	1546.91	1512.19	72.917	9.707
22	1604.77	0.417	3.726	1631.78	1573.91	108.409	27.025
23	1697.36	1.897	3.486	1712.79	1674.21	56.177	7.395
24	2312.65	20.35	0.189	2316.51	2069.62	148.838	0.055
25	2378.23	17.3	0.754	2385.95	2351.23	25.793	0.433
26	2451.53	14.301	0.69	2465.03	2387.87	61.717	0.425
27	2561.47	10.327	0.477	2569.18	2466.96	93.027	0.363
28	2590.4	9.882	0.331	2600.04	2571.11	28.761	0.197
29	2937.59	3.973	0.266	2947.23	2642.48	357.4	2.537
30	2980.02	3.593	0.444	3026.31	2951.09	106.399	1.55
31	3059.1	3.779	0.019	3061.03	3037.89	32.591	0.013
32	3076.46	3.732	0.163	3093.82	3070.68	32.576	0.215
33	3103.46	4.241	0.139	3172.9	3097.68	98.44	0.195

Observation And Calculation of Magnetic Susceptibility:

Calibration of Electromagnetic ($MnSO_4$) Known Solution:-

Sr. No	Current	MSR	VSR	Magnetic Field (H)Gauss
1	0 A	4.2	25	0
2	1.5 A	4.2	29	888.39 G
3	2 A	4.2	34	1329 G
4	2.5 A	4.2	43	1880 G
5	3 A	4.2	55	2427 G
6	3.5 A	4.2	63	2731
7	4 A	4.2	74	

Observation table for calculation of magnetic susceptibility of Fe(III)-Azomethine complex in Dimethyl Sulphoxide

Sr.No	Current	MSR cm	VSR	X_{mass}	X_{molar}	μ_0	No .of unpaired electron
1	0 A	4.2	42	48.92×10^{-6}	0.003371	1.09	1.17e⁻
2	1.5 A	4.2	32				
3	2 A	4.2	31				
4	2.5 A	4.2	28				
5	3 A	4.2	27				
6	3.5 A	4.2	25				
7	4 A	4.2	22				

Observation :

1. Weight of Density Bottle = 6.68
2. Weight of Density Bottle + Complex + Solvents = 19.49
3. Weight of Density Bottle + Water= 20.61

Calculation :

a) Calculation of Density of Solution by formula

$$\rho = \rho_{water} \frac{(c-a)}{(b-a)}$$

b) $X_{\text{molar}} = X_{\text{mass X Molecular weight}}$

c) The magnetic moment is calculated by using formula.

$\mu_{eff=2.84\sqrt{(xm)\times TB.M.}}$

$\mu_{eff=Us=\sqrt{n(n+2)B.M.}}$

$\mu eff= 2.84\ (T\ x\ X_{molar)}{}^{1/2}$

$\mu eff= [n(n+2)]^{1/2}$

The IR and magnetic susceptibility studies of the complex indicate that it is an octahedral complex of the type Fe-L3.

RESULTS AND DISCUSSION

From the two I.R. spectra of 4-[(4-Nitro-benzylidene)-amino]-benzoic acid i.e.Schiff Base and Fe(III)- azomethine complex shows the presence of C=N stretch (1683 cm^{-1}), C-N stretch (1294 cm^{-1}) and COOH (2945-2560 cm^{-1}). Where as there is difference in values between C=O group of both. Carbonyl group of azomethine absorb at (1710 cm^{-1}) which is carbonyl group carboxylic acid. Complex formation take place after deprotonation of carboxylic group therefor, it observed at (1735cm^{-1}), ester like frequency. Unless and until breaking of carboxylic O-H bond takes place, it is impossible to form carboxylic oxygen $-M^+$ metal covalent bond. Secondly as nitrogen from azomethine donating lone pair of electron to metal ion and from coordinate bond, its I.R. absorption value increases.

There fact clearly indicate the formulation of bidentate d^2sp^3, octahedral paramagnetic Fe(III) – azomethine complex.And also according to magnetic study Azomethine acts as strong field ligand and hence pairing of metal electron i.e. Fe(III) valence electron takes place. Magnetic study reveals that magnetic moment of Fe(III)-azomethine complex is nearly equal to one (1) and value of magnetic susceptibility is more than 0 and less than one (by Quinck's method).

Therefore, the present Fe(III)-azomethine complex is paramagnetic in nature. It has octahedral geometry having d^2sp^3 configuration i.e. inner-orbital complex or low spin complex.

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