International Journal of Science and Research (IJSR)
ISSN (Online): 2319-7064
Index Copernicus Value (2013): 6.14 | Impact Factor (2015): 6.391

Synthesis, Characterization and Microbial Studies of Platinum Complex of Thiazolidinone of Ethylene-Bis-Anil

Vibha

Department of Chemistry, H.S. Degree College, NaglaRund, Hathras

Abstract: ***The solution of K_4PtCl_6, thiazolidinone in acetone/ methanol were mixed together in 5:2 molar ratio and refluxed. From the concentrated reaction mixture, solid was isolated on crystallization. Thiazolidinone has been synthesized from 1,2 - ethyl- dianil (which is the condensation product of 1,2 diamino ethane and o-hydroxy phenyl glyoxal) in dry methanol and thioglycolic acid. The synthesized complex has been characterized by some physico-chemical studies namely, I.R., magnetic conductometric and elemental analysis.***

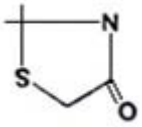
Keywords: Platinum complex, Thiazolidinone of ethylene-bis-anil, glyoxal and physico- chemical studies

1. Introduction

Schiff's bases product of primary amine and carbonyl compounds also called as anils characterized by the presence of azomethine (>C=N) auxochromic group are coloured organics owing to distinguished physico- chemical and biological features of Scheff's bases[1][4] Viz. high electron donar ability at their nitrogen, bathochromic property, resonance property in conjugation with other unsaturated group's and biological properties, their diversified applications are recorded in the field of industry[5], agriculture and medical sciences.

Ketoazomethines are Ketoanils product of glyoxals and primary amines, Charaterized by presence of ketonic group adjacent to azomethine group are special class of schiff's bases of biological and distinguished physico-chemical properties leading to their use in medical sciences, in apparel industry as dyes[6], in co-ordination chemistry as noval ligand in forming complex of unusual co-ordination number and isomeric structures, in synthetic organic chemistry as intermediates and in analytical chemistry as gravimetric and chromatographic reagents.

Thiazolidinone characterized by presence ofmoiety, well known for their multifarious roles in diverse field of development viz. Medicalsciences (anticancer[12] [14], anticonvulsant [15][16],anti-inflammatory[17] properties), agriculture (bactericiidal, fungicidal)[18][20] analytical and co-ordination chemistry.



2. Materials and Methods

Materials:- Selenium dioxide CDH (India), O-hydroxyacetophenone (Qualigens), Methanol (CDH), Sodium bicarbonate (CDH), thioglycolic acid, K_4PtCl_6 and Acetone.

2.1 Preparation of Glyoxal

O-Hydroxyphenylglyoxal was prepared by reported method, a solution of O-Hydroxyacetophenone (1 Mol in 50 ml. alcohol mixed with selenium dioxide solution (111 gm. in 500 ml. alcohol) was refluxed for 5hr. and mustard yellow reaction mixture was decanted and solvent was driven off on water bath. Dark viscous residue was distilled under reduced pressure to make it free from selenium metal.

2.2 Synthesis of Ketoanil

Solution of O-hydroxy phenyl glyoxal (0.30 mol) and 1.3 diamino propane (0.15 Mol) in ether were mixed together with vigorous stirring at room temperature, product Ketoanil precipitated after some time (~1 hr.) were washed with ether repeatedly.

2.3 Synthesis of thiazolidone

To the solution of Ketoanil in dry methanol (0.03 mol) thioglycolic acid (0.06 mol, CDH, 98%) was mixed and reaction mixture was refluxed for 6-8 h, concentrated and neutralized with aqueous sodium bicarbonate solution and precipitated solid was filtered, washed with water repeatedly and dried in hot air at 80^0C.

2.4 Preparation of platinum complex of thiazolidinone of Ethylene-Bis-Anil

For the preparation of complex solution of K_4PtCl_6 and thiazolidinone in acetone/methanol were mixed together in 5:2 molar ratio and refluxed for 4-5 h. From the concentrated reaction mixtures solid was isolated on crystallization. Product washed with water repeatedly to remove unreacted metal salt if any and finally with methanol were dried in air.

3. Result and Discussion

3.1Physico Chemical Studies

Table 1.1 comprising analytical and physical data clearly reveals consistence of theoretically proposed molecular

formulae with experimental data of Ketoanil, its thiazolidinone and Pt complex of this thiazolidinone.

3.2 I.R. Studies

Perusal of I.R. spectra of Ketoanil reveals a well defined peak at 1591cm^{-1} characteristic to CH=N gp. and another peak in 1616 cm^{-1}-1676cm^{-1} range assigned to Ketonic group adjacent o azomethine group, Benzene ring stretching vibration of C-H and C=C are displayed in the region 3098 cm^{-1} - 3226 cm^{-1} and 1447 cm^{-1} - 1450cm^{-1} respectively.

In cyclo condensation of Ketoanil with thioglycolic acid cyclization occurs at azomethine group and a water molecule, comprising one hydrogen atom of -SH and -OH of carboxylic group of thioglycolic acid eliminates. Thus in product thiazolidinone identification of C-S-C, C=O and C-N characteristic group of heterocyclic ring would indicate success of the cyclocondensation reaction I.R. spectra of thiazolidinone are in conformity of cyclization of Ketoanil at their azomethine group. General structure proposed for thiazolidinone exhibit tautomerism and structures of different tautomeric forms could be shown on page number-3

Although each of the tautomeric structure of thiazolidinone show ten coordinating site, two nitrogen two sulphur and two oxygen atoms of heterocyclic rings and two oxygen of chain carbonyl and two of phenolic groups but perusal of i.r. spectra of thiazolidinone and its complex reveals co-ordination structure (iii) through its deprotonated chain enolic groups and heterocyclic ring nitrogen.

I.R. band at 541 cm^{-1} and 420 cm^{-1}altributed to υM-O and υM-N respectively. Disappearance of Phenolic group peaks corresponding to υOH and δOH of ligand in its Pt complex also support this inference, regarding presence of quinonoid structure of ligand in the coordination zone of metal.

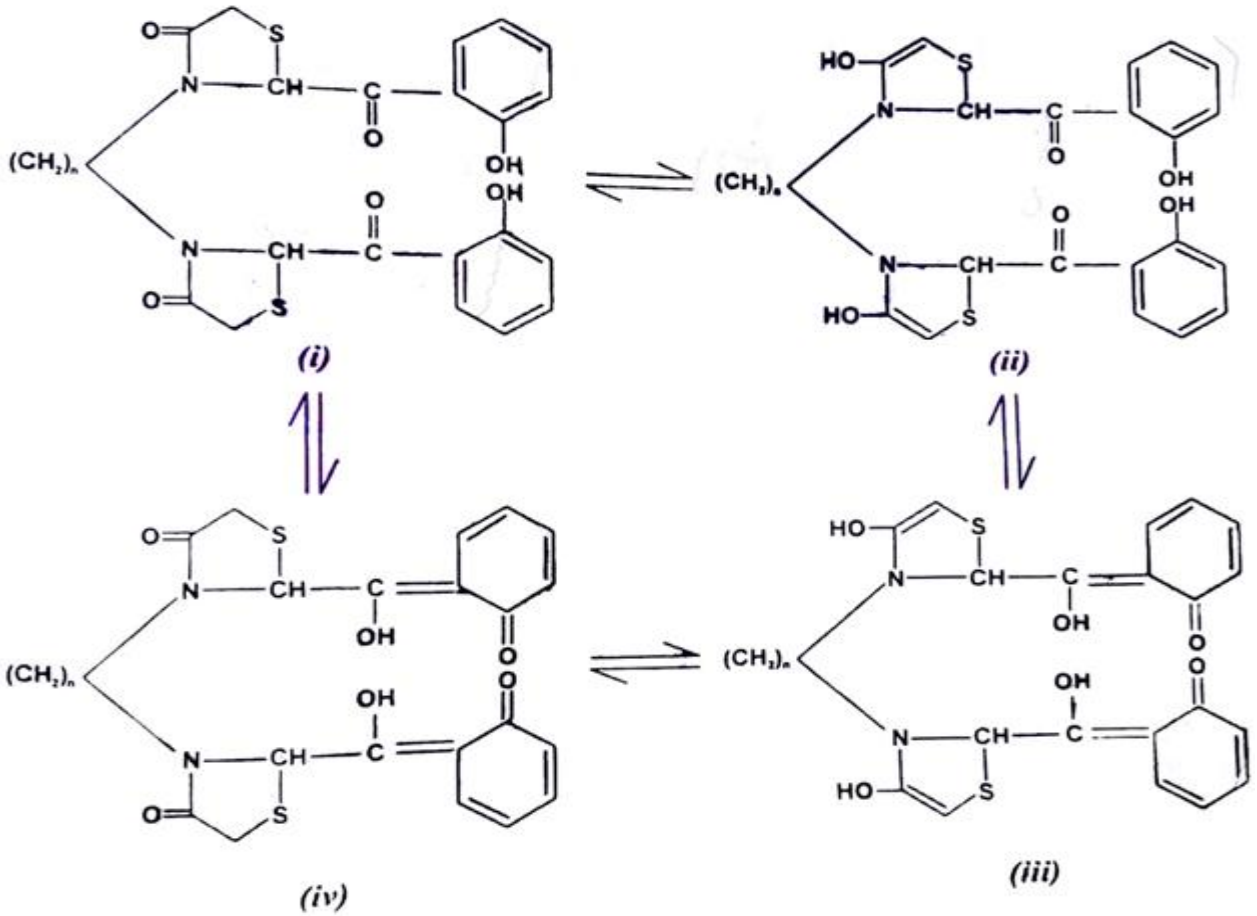
Pt (II) complex show strong band at 3437 cm^{-1} and 1616 cm^{-1} assigned υsymmetric & asymmetric and δsymmetric & asymmetric H_2O vibrations respectively suggest water of cryctallization in the complex.

Molar conductance reveals non-electrolytic nature of Pt(II) complex.

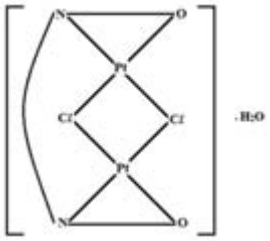
3.3 Electronic Spectral Studies

Pt(II) complex is diamagnetic and has a d^8 configuration implying a square planar geometry. Further electronic spectra of this complex positively affirm the presence of square planar geometry. Three d-d- spin allowed transition bands are expected corresponding to the transitions from the three lower lying d-levels to the empty dx^2-y^2orbital's. The electronic spectral bands at 26881 and 27777 cm^{-1} may be assigned to $^1A_{1g} \rightarrow {}^1B_g$ transition and $^1A_{1g} \rightarrow {}^1E_g$ transition respectively.

From above discussion general structure proposed for thiazolidinone exhibit tautomerism could be shown on page no. - 3.



Only one tautomeric structure of ligand may be proposed $[Pt_2(C_{22}H_{18}O_6N_2S_2)Cl_2]$.



3.4Anti-Bacterial Studies

From the antibacterial activity data platinum compound shown afficacy against staph Awrens, Ecoli ESS 2231, Protect Vulgaris and AspergillusFumigatus.

3.5Anti-Fungi Studies

From the anifungal data platinum compound show highest fungicidal property against candida Albicans, Candida Krusei and Candida Glabrate.

Conclusion

The synthesized complex has been characterized by some physico-chemical studies match from the hypothetical platinum complex. It has high antifungal and antibacterial properties.

Table 1.1: Physico - Chemical Data of Ketoanil and its Thiazoltdinone

Compound	Colour	M.P.	Molecular Wt.		Yield	Elemental Analysis							
						C%		H%		N%		S%	
			Calcd.	Found		Calcd.	Found	Calcd.	Found	Calcd.	Found	Calcd.	Found
$C_{18}H_{16}O_4N_2$ Ketoanil	Yellow Brown	190^0C	324	-	61.70	66.60	66.89	4.93	4.84	8.64	9.02	-	-
$C_{22}H_{20}O_6N_2S_2$ Thiazolidinone	Brown	275^0C	472	-	30.50	55.93	48.11	4.23	3.72	5.93	7.50	13.5	6.45
$[Pt_2(C_{22}H_{18}O_6N_2S_2)$ **Cl_2**$].H_2O$ Pt(II) Complex	Brown	260^0C	913	910	21.20	27.82	28.35	2.11	1.93	2.95	3.0	6.74	6.87

Table 1.2: I.R. Frequencies $(CM)^{-1}$

Compound	υC=O chain	υC=O ring	υC-N & υC-0 ring	υCH=N	C-S-C	δC-H O-Position	υ(OH) Intra Molecular bonding	υ(OH) Phenolic +υC-O of Phenolic	Benezene rng		δC-H (CH_2) Alkane	υC-H (CH_2) Alkane	υM-O	υM-N	Symm + AsyυH_2O	Symm + AsyδH_2O	υM-cl-M
									υC=C	υC-H							
$C_{18}H_{16}O_4N_2$	1676	-	-	1591	-	763s,d	3206s	1345s	1486s	3098s	1450s	2874s 2934s	-	-	-	-	-
$C_{22}H_{20}O_6N_2S_2$	1654s, br, Sh	1654s	1318m 1237m	-	644w	754m	3296s	1286m 1374s	1447d, s	3141s	1447s	2935m	-	-	-	-	-
$[Pt_2(C_{22}H_{18}O_6N_2S_2)Cl_2]H_2O$	1616vs	1616vs	1292s	-	677w, Sh	759m, Sh	-	-	1468m, Sh 1449s	3226s	1449s	2926s	541s	420m	3437s, br	1616v, Sh	246s 224s

Table 1.3: Magnetic Susceptibility and Magnetic Moment Data Of Complex

Complex	Wt. of Complex (gm.)	Length of Complex (Cm.)	Susceptib-ility of empty tube (R)	Susceptibility of tube + Complex (R_1)	Gram Susceptibility (χ x 10^6) (C.G.S.)	Molecular weight of Complex (gm)	Molecular Susceptibility (χm x 10^6) C.G.S.	Calculated diamagnetic Correction x 10^6	Effective Molecular susceptibility (χ^1m x10^6) C.G.S.	Effective magnetic moment μeff. (B.M.)	Hybridization
$[Pt_2(C_{22}H_{18}O_6N_2S_2)Cl_2]$ H_2O	0.13	1.2	-32	-123	-1.023	949	-970.9	242.14	-1213.04	Diamagnetic	dsp^2

Table 1.4: Conductometric Data and Electrolytic Nature of Complex

Compound	Volume containing 1 gmmol of complex (ml)	Conductance due to Complex x 10^6 (Mhos)	Specific Conductance (Conductance due to Complex x cell const.) (mhos)	Molar Conductance (Λ_m) (Specific Conductance x V) -Ω^2Cm^{-2}	Electrolytic Nature
$[Pt_2(C_{22}H_{18}O_6N_2S_2)Cl_2]$ H_2O	949000	13.8	0.01318 x 10^{-3}	1309	Non-Electrolyte

Table 1.5: Electronic Spectral Band Frequencies and their Assignments

Complex	Spectral Bands (cm^{-1})	Assignment
$[Pt_2(C_{22}H_{18}O_6N_2S_2)Cl_2]$ H_2O	26881(1674) 27777 (2182)	$^1A_{1g} \rightarrow {}^1B_{1g}$ $\rightarrow {}^1E_g$

Table 1.6: Antibacterial Activity Data

Complex	Antibacterial Activity									
	Conc. (μg/ml)	Staph Awrens	Conc. (μg/ml)	E Coli. Ess 2231	Conc. (μg/ml)	Protect Vulgaris	Conc. (μg/ml)	Klesiella Pneumoniae	Conc. (μg/ml)	Aspergillus Fumigatus
$[Pt_2(C_{22}H_{18}O_6N_2S_2)Cl_2]$ H_2O	90	18	120	16	230	16	80	14	120	18

Table 1.7: Antifungal Activity Data

Complex	Antifungal Activity					
	Conc. (μg/ml)	Candida Albicans	Conc. (μg/ml)	Candida Krusei GO_3	Conc. (μg/ml)	Candida Glabrate HO_3
$[Pt_2(C_{22}H_{18}O_6N_2S_2)Cl_2]$ H_2O	100	16	120	18	80	16

References

[1] GadadaNaganagoulda, ReinoutMeijboom and AmornPetsom (2014) Advance in Chemistry Volume 2014, Article ID. 921857.
[2] International journal of Inorganic Chemistry (2012) Vol. 2012, Article ID 372141.
[3] S. Yadav and R.V. Siingh (2011) SpectrochimicaActa Part A, Vol. 78, P (298-306).
[4] A. Kulkarni, P.G. Avaji, G.B. Bagihalli, S.A. Patil and P.S. Badami (June 2009) Vol. 6 issue 2, P. (259-270).
[5] Applications of Metal Complex of Schiff's Bases) Shalin Kumar, DurgaNathDhar and P.N. Saxena (March 2009) Journal of Scientfic and Industrial Research Vol. 68 P. (181-187).
[6] KaremaMasoudeAbuamer, Abdessalam Ali Maihub, Mari Miloud El-Ajaily, Abdunnaser Mohamed Etorki, Martaja Mohamed Abou-Krisha, MajdaAlbashirAlmagani (2014) IInternational Journal of Organic Chemistry, P.(7-15)
[7] ML. Tong, XM Chen, BH Ye, LN Ji (1999) Chemie, International Edition.
[8] E. Nataliya, Borisova, P. Marina Rshetova and A Yuri, Usynuk (2007) Chem. Rev. 107, 46-79.
[9] R.K. Upadhyay, M. Verma, M.K. Sharma and Madhu (1997) J. Indian chem. Soc., 74, 139.
[10] I Musmuka, A. Caroli, A. Mai, N KaushikBasu, (2010) J. Chem. ACS Publication.
[11] Nabil Ramadan Bader (2010) Rasayan J. Chem. Vol. 3 No. 4
[12] DmytroHavrylyuk, LudmylaMosula, Roman Lesyk, (Nov. 2010) European Journal of Medicinal Chem., Vol. 45 (11).
[13] Synthesis and Anticancer activity of 4-thiazolidinones (Nov. 2010) European Journal of Medicinal Chemistry 45(11) : 5012-21.
[14] Abhishek Kumar Jain, AnkurVaidya, VeeraSamyRavichandran, Sushil Kumar Kashaw, Ram KishorAgrawal, (2012) Elsevier, Bioorg. Med. Chem.
[15] A Gursoy, N Terzioglu (2005), Turkish Journal of Chemistry.
[16] M.R. Shiradhar, M. Ghodake, KG Bothara, SV Bhandari (2007), ARKI VOC, Arkat-USA Org.
[17] Synthesis and Biological evaluation of Somethizol. (2008) Eur J. Med. Chem.

[18] Deepak K. Aneja, PoonamLohan, SanjivArora, Chetan Sharma, Kamal R. Aneja and Om Prakash (2011) Organic and Medicinal Chemistry Letters.
[19] P. Vicini, A Geroni Kaki, K Anastosia, M Incerti (2006) Bioorganic & Medicinal Chem. Elsevier.
[20] VR Avupatii, RP Yejella, AAkula, GS Guntu Ku (2012) Bioorganic & Medicinal Elsivier.

Author Profile

Vibha is serving in Chemistry Department H.S. Mahavidhyalaya, NaglaRund, Sasni, Hathras (UP). She is pursuing Ph.D. from C.C.S. University, Meerut Under Supervision : R.K. Upadhyay (D.SC.). She is Ex. Principal, N.R.E.C. College, Khurja (Bulandshahr) UP