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Research Article

SYNTHESIS, CHARACTERISATION AND ANTIMICROBIAL ACTIVITY OF GOLD (III) WITH 2-CHLOROQUINOLINE-3-CARBALDEHYDE THIOSEMICARBAZIDE {1-((2-CHLOROQUINOLINE-3-YL) METHYLENE THIOSEMICARBAZIDE (2-CHLORO-QAT)

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ABSTRACT
Synthesis of 2-Chloroquinoline-3-carbaldehyde Thiosemicarbazide (2-Chloro-QAT), Melting point Elemental analysis, XRD, Effect of diverse ion and Antimicrobial Activity are studied. Stability constant, Dissociation constant and Change in free energy are determined. Composition of metal and ligand has been determined by Job's variation and mole ratio method. The optimum conditions for complete colour development have been established by studying parameters like Effect of medium, Reagent concentration, Time period have been studied.

KEYWORDS -- Gold (III) , 2-Chloroquinoline-3-carbaldehyde Thiosemicarbazone , (2-Chloro-QAT), Spectrophotometry, Antimicrobial Activities.

INTRODUCTION

Gold is more noble. It occurs native and in sulphide and arsenide ore. It is used in dentistry and in corrosion resistant plating. The first and second ionization energies are 890 & 1970 KJ mole^{-1} [1]. Tertiary phosphine & Thiol gold complexes are used in the treatment of rheumatoid arthritis an inflammatory disease[2] that affects the tissue around joints. The inflammation arises by the action of hydrolytic enzymes in cell compartments known as lysosomes that are associated with the Golgi apparatus. Although the mechanism of action is not established. It is known that Au accumulates in the lysosomes & it is therefore possible that Au inhibits these hydrolytic enzymes. Another hypothesis is that Au (I) compounds deactivate singlet O_2 a harmful species that can be formed by oxidation of superoxides. The mechanisms for this reaction might involve promotion of intersystem crossing by the high spin-orbit coupling constant of the heavy elements[3,4].Commonly administered drugs include sodium aurothiomalate form linear linkage with gold. Many are water soluble polymers but they can not be administered orally due to acid hydrolysis in the stomach. By constrast the compound known as Auranofin can be given orally[5]. Goladis the best result obtained with quinol (1,4-dihydroxybenzene) as the reducing agent[6].

Thiosemicarbazones usually react as chelating ligands with tranisition metal ions by bonding through sulphur and nitrogen atoms[7]. Thiosemicarbazones have been frequently used for spectrophotometric determination of inorganic ions and their analytical potentialities have been reviewew[8,9]. Thiosemicarbazone are important organic analytical reagents for the determination of metal ions in microgram quantities. They form coloured complexex with many metal ions and act as good chealating agents. In addition to the analytical utility[10,16].Metal complexes formed with these reagents are of great medicinal value in the treatment of diseases like influenza[17] ,protozoa[18] ,smallpox[19],tumors[20] and pesticides[21] .A large number of thiosemicarbazides have been found to posses good antibacterial[22],antifungal[23,] herbicidal[24] and antiacetylchlinesterase[25,26] activities. The pharmacological importance of metal complex with heterocyclic thiosemicarbazone[27]. Synthesis and spectrophotometric determination of Cr(II) isonicotinohydrazide with glyoxal-bis-isonicotinyl hydrazone was studied[28].

MATERIALS AND METHODS

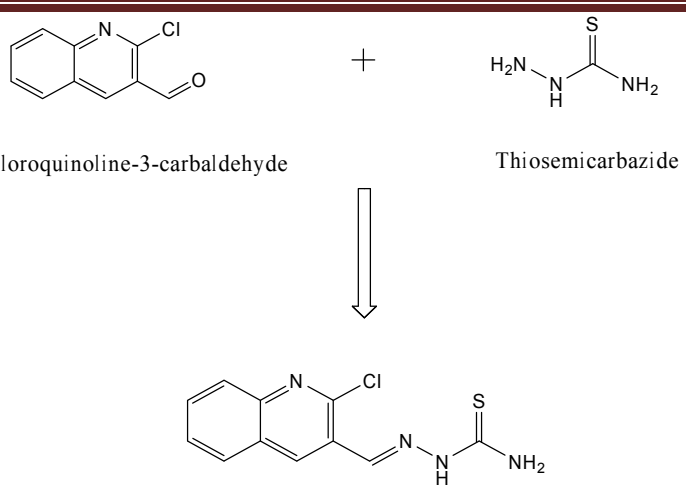
An Elico UV-visible spectrophotometer model UV-SL-164 equipped with 1 cm quartz cell used for spectrophotometeric measurements. An Elico pH meter LI-610 is used for the pH measurements. The chemicals used are of AR grade. X-RD was taken on PW 3710 diffractometer using CuK_2 radiation has been taken on the instrument BRUKER AC 300F.Elemental analysis and antimicrobial activity was done in Laboratory approved by Central Government for AGMARK. All chemical and solvents used were of analytical grade

EXPERIMENTAL WORK

SYNTHESIS AND CHARACTERISATION OF 2-CHLORO-QAT

Synthesis of 2-ChloroQAT

2-Chloroquinoline-3-CarbaldehydThiosemicarbazide{1-((2-Chloroquinolin-3-yl)methylene) thiosemicarbazide (2-Chloro-QAT) is prepared by taking equimolar quantity of 2-Chloroquinoline-3-Carbaldehyde & thiosemicarbazide in methanol and reflux 74 hours or more. It was allowed hydroxide to stand at room temp until the yellowish crystal were found.



2-Chloroquinoline-3-carbaldehyde

Thiosemicarbazide

(E)-1-((2-Chloroquinolin-3-yl)methylene)thiosemicarbazide

The crude product is crystallized in methanol. The recrystallized product has melting point is 227^0C and molecular weight by formula is 252.5.

Characterization of 2-Chloro-QAT

Absorption Spectra of 2-Chloro-QAT was recorded against a blank solution containing buffer (pH=2) and is shown in fig 1. Absorption spectra was recorded in the wave length range 250-570 nm. The complex shows an absorption maximum at 285 nm. At 285 nm wavelength the molar absorptivity of 2-Chloro-QAT is 0.9750 x 10^3 L.mol^{-1}.cm^{-1}.

Elemental Analysis of 2-Chloro-QAT

The elemental analysis of 2 -Chloro-QAT was done in Laboratory approved by Central Government for AGMARK. It shows the result of elemental analysis in Table 1.

X-RD of 2-Chloro-QAT

X-RD spectra of 2-Chloro-QAT was taken on PW 1710 diffractometer using CuK_2 radiation (wavelength 1.54060 to 1.54438 A^0). The X-RD diffraction of 2-Chloro-QAT was recorded at angle 2Ø from 20.000-80.000. The data of X-ray diffraction of 2-Chloro-QAT were presented in Table 2. And X-ray spectrum in fig.2. For the determination of structure Hesse-Lipson procedure is used[29].

Antimicrobial Activity of 2-Chloro-QAT

Antimicrobial Activity of 2-Chloro-QAT has been done in the Laboratory approved by Central Government through AGMARK. The result are noted in Table 3.

Effect of Reagent concentration

Effect of Reagent concentration was studied by taking varying amount of reagent and fixed amount of Au (III). Optimum pH of solution was maintained 2. It was noted that 0.125 ml reagent is sufficient for complete colour development. However by adding excess of reagent there is no substantial change in the absorbance value.Effect of reagent shown in fig. No.3.

Validity of Beer's Law

For the study of Beer's law the solutions were prepared which containing different amounts of Au (III) ,same amount of 2-chloro-QAT and 1 ml of pH 2. It indicate that It shows the result of elemental analysis in Table 1.

Composition of Complex

The composition of the Au (III)-2-chloro-QAT complex is found to be 1:1. It was determined by studying Job's method. The ratio of metal ion to ligand molecule in the coloured complex was found to be 1:1.Composition of complex shown in fig No.5.

Physico-chemical Characteristic of Au (III)-2-chloro-QAT

Physico-chemical and Analytical Characteristic of Au (III)-2-chloro-QAT was studied and given in Table 4.and Tolerance limit of diverse ions in the determination of 2-Chloro-QAT shown in Table No. 5.

RESULT AND DISCUSSION

2-Chloroquinoline-3-CarbaldehydThiosemicarbazide{1-((2-Chloroquinolin-3-yl)methylene) thiosemicarbazide (2-Chloro-QAT) is yellowish crystal . The recrystallized product has melting point is 227^0C and molecular weight by formula is 252.5. The complex shows an absorption maximum at 285 nm. At 285 nm wavelength the molar absorptivity of 2-Chloro-QAT is 0.9750 x 10^3 L.mol^{-1}.cm^{-1}.The elemental analysis of2-chloro-QAT shows that carbon 48.38 %,hydrogen 4.02 %,Sulphur 13.24 %,nitrogen 22.60 % & chlorine 14.33 %. XRD of 2-chloro-QAT shows the structure of the complex 1:1 as shown in fig 1. The ratio of metal ion to ligand molecule in the coloured complex was found to be 1:1. Antimicrobial activity of 2-chloro-QAT was studied for Klebsiella pneumoniae, Vibriae cholerease, Bacillus megaterium , Salmonalla typhi, Shigella flexneri. The Stability complex,Dissociation constant, Change in free energy & Sandell's sensitivity of 2-chloro-QAT is 2.85041 x 10^7, 3.50826 x 10^{-8}, -42.53 KJ/mole & 0.003321 µg /cm^2 respectively.

Table 1. Elemental Analysis of 2-Chloro-QAT

Sr.No.	Chemical Analysis	Percentage Found	Percentage Expected
1)	Carbon	48.38	47.52
2)	Hydrogen	04.02	03.56
3)	Sulphur	13.24	12.67
4)	Nitrogen	22.60	22.18
5)	Chlorine	14.33	14.06

Table 2. XRD for 2-Chloro-QAT (Powder method)

Sr.No.	2Ø	hkl	Sin^2 Ø Observed	Sin^2 Ø Calculated	d (A^0) Observed	d (A^0) Calculated
1)	25.695	211	0.04960	0.05162	3.4556	3.9556
2)	28.350	211	0.04653	0.05068	3.8159	4.0556
3)	30.950	220	0.07102	0.07998	2.8901	2.9102
4)	35.500	222	0.08065	0.07225	2.4279	2.3093
5)	44.730	210	0.03950	0.03950	4.6603	3.8753
6)	50.775	211	0.05980	0.04780	4.4828	4.5220
7)	53.330	110	0.06321	0.06821	3.8984	3.8779
8)	72.520	111	0.07881	0.07328	3.9806	3.9857
9)	74.720	111	0.08341	0.08333	4.0525	4.9765

a = 9.3682 b = 6.8990 c = 8.2506

Structure of Powder Crystal
Fig 1.

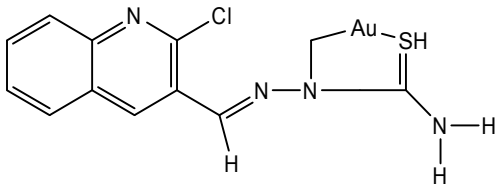


Table 3. Antimicrobial Activity of 2-Chloro-QAT

Sr.No.	Antimicrobial	Activity
1)	Klebsiella Pneumoniae	Nil
2)	Vibriae Cholerease	Nil
3)	Bacillus Megaterium	Nil
4)	Salmonalla typhi	Nil
5)	Shigella Flexneri	Nil

Table 4. Physico-Chemical and Analytical Characteristic of 2-Chloro-QAT

Sr.No.	Characteristics	Result
1)	Absorption Spectra	285 nm
2)	Molar absorptivity	0.9750×10^3 Lit. mol^{-1}. cm^{-1}
3)	pH range (optimum)	2.0
4)	Reagent required for maximum complexation	0.125 ml
5)	pKa	7.004×10^8
6)	Beer's law validity range (ppm)	11 ppm
7)	Composition of complex (M : L)	1:1
8)	Stability Constant	2.85041×10^7
9)	Dissociation Constant	3.50826×10^{-8}
10)	Change in free energy	-42.53 KJ/mole
11)	Sandell's Sensitivity (µg /cm^2)	0.003321 µg /cm^2

Table 5. Tolerance limit of diverse ions in the determination of 2-Chloro-QAT

Sr. No.	Metal ion	Salt	Interference
1)	Mg (II)	$MgSo_4$	240
2)	Cu (II)	$CuSo_4$	88
3)	Cd (II)	$CdCl_2$	40
4)	Mn (II)	$MnCl_2$	30
5)	Co (II)	$CoSo_4$	Interferes
6)	Ce (IV)	$Ce(So_4)_2$	14
7)	Ba (II)	$BaCl_2$	14
8)	Cr (III)	$K_2Cr_2O_7$	05
9)	Hg (II)	$HgCl_2$	02
10)	Ti (V)	K-titanyl oxalate	05
11)	Ni (II)	$NiCl_2$	04
12)	Sn (II)	$SnCl_2$	03
13)	Pb (II)	$PbSo_4$	75
14)	V (v)	V_2O_5	Interferes
15)	Zn (II)	$ZnSo_4$	43
16)	Al (III)	$AlCl_3$	Interferes
17)	Pd (II)	$PdCl_2$	Interferes
18)	Ni (II)	$NiCl_2$	04

Fig.1. Absorption Spectra of 2-Chloro-QAT

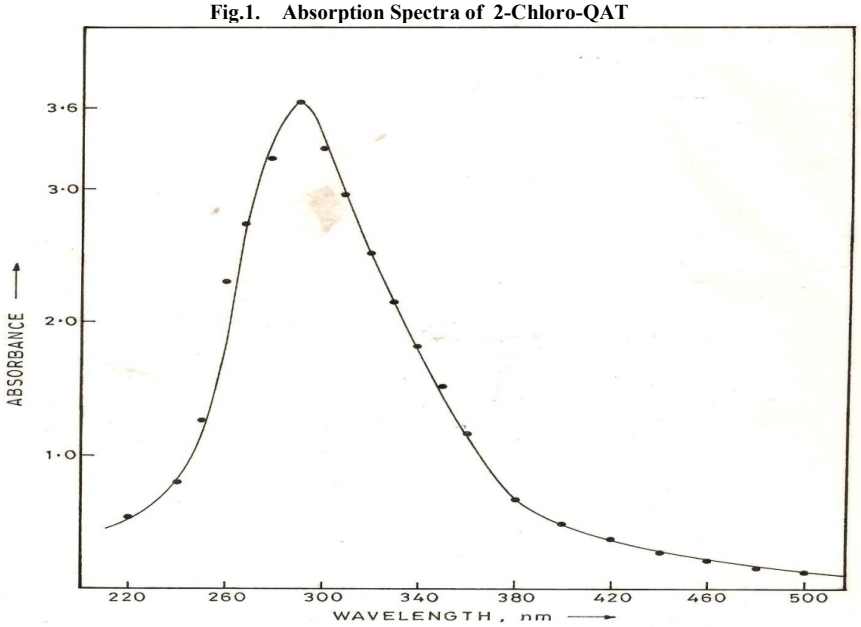


Fig.2. X-RD of 2-Chloro-QAT

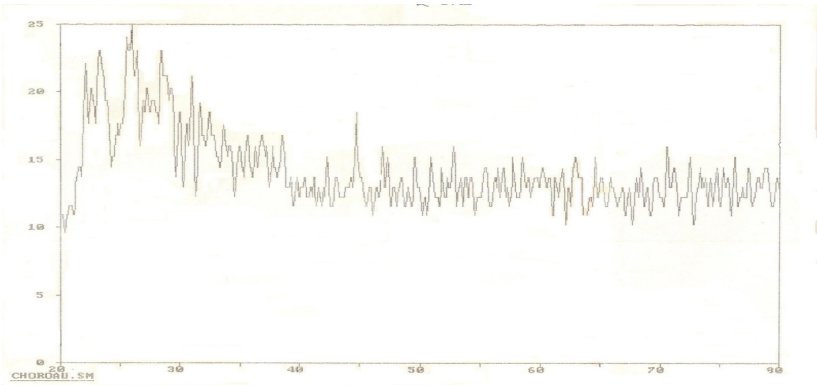


Fig.3 Effect of Reagent concentration

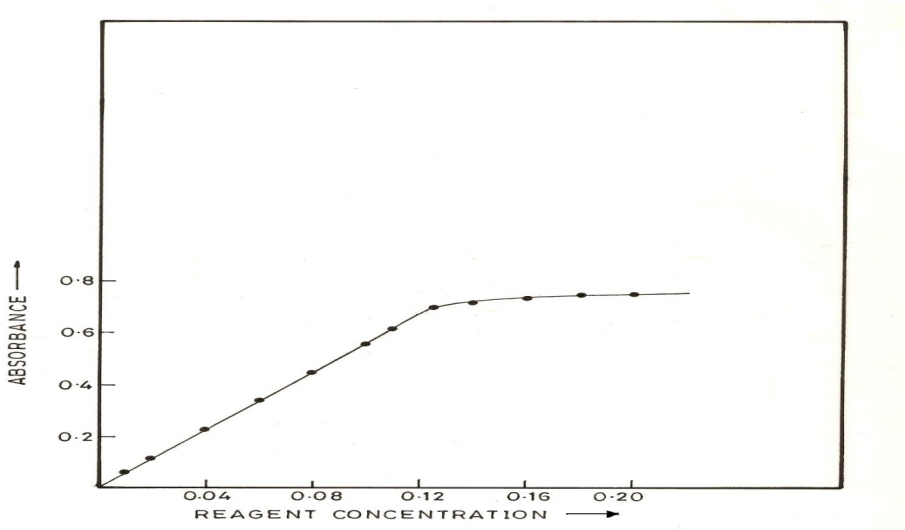


Fig.4. Validity of Beer's Law

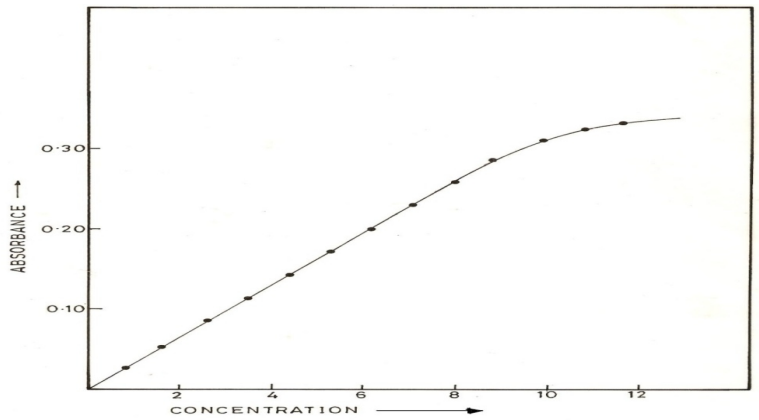
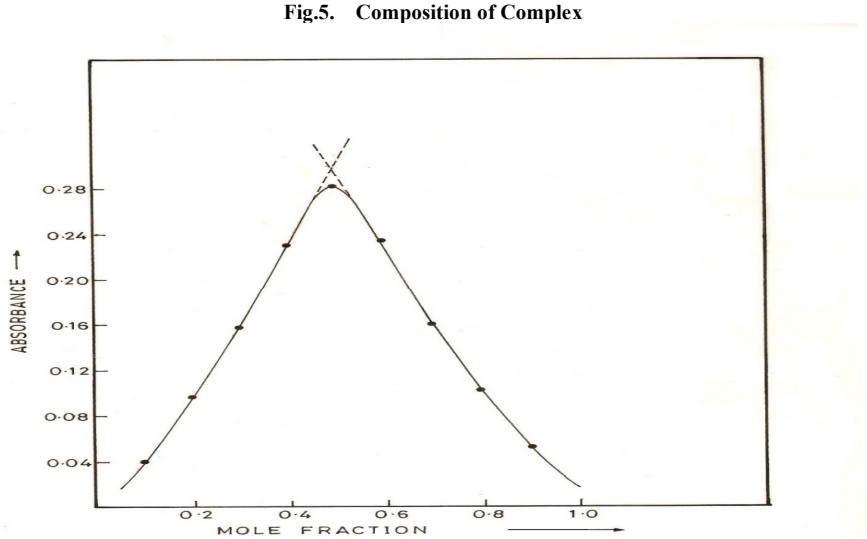


Fig.5. Composition of Complex



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