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SYNTHESIS, CHARACTERIZATION, ANTIMICROBIAL AND PESTICIDAL ACTIVITY OF ORGANOARSENIC(III) AND ORGANOANTIMONY(III) COMPLEXES WITH N∩S DONOR LIGANDS

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Keywords:

Sb (III) and As (III) complexes, Spectroscopic techniques, Antimicrobial activity, Pesticidal activity

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ABSTRACT: Some new Sb(III) and As(III) complexes of N∩S donor ligands N'-[1-(2-oxo-2H-chrome-3yl-ethylidene] - hydrazinecarbodithionic acid methyl ester (L1H) and N' - [1-(2-oxo-2H-chrome-3yl - ethylidene] -hydrazinecarbodithionic acid benzyl ester (L2H) have been synthesized. All the synthesized compounds were characterized by elemental analyses, melting point determinations and a combination of electronic, IR, 1H NMR, and X-ray diffraction spectroscopic techniques. These studies showed that the ligands coordinated to organoantimony and organoarsenic in a monobasic bidentate manner through sulfur and nitrogen donor system. Thus, tetra and pentacoordinated environments around the antimony and arsenic atom have been proposed. In order to evaluate the effect of metal ions upon chelation, both the ligands and their complexes have been screened for their antimicrobial activity against the various pathogenic bacterial and fungal strains. The results indicate that the metal complexes are more potent antimicrobial agents than the parent ligands. Further, the ligands and their corresponding metal complexes have been tested for their pesticidal activity against the Corcyra cephalonica. The results of pesticidal screening are quite promising.

INTRODUCTION: Metal complexes of Schiff bases derived from S-alkyl esters of dithiocarbazic acid have been the subject of a large number of studies due probably to their interesting properties[1] and potentially beneficial biological activities[2-3]. Coumarin derivatives constitute an important class of heterocyclic compounds with anticoagulant[4], insecticide[5], antibacterial[6], and pharmacological properties. The number and diversity of nitrogen and sulfur chelating agents used to prepare new coordination and organometallic compounds have increased rapidly during the past few years[7-9]. Sulfur compounds and their metal complexes have antimicrobial activity and showed a high dependence on their substituents[10-11].

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Organic compounds containing $-C_6H_4S$ moiety are well known for their significant biological activities. The activity may be due to the presence of multi-coordination centers having the ability to form stable chelates with the essential metal ions which the organisms need in their metabolism. Interest in metal complexes of sulfur-nitrogen chelating agents, especially those formed from S-methyl/benzyl esters of dithiocarbazic acid has been stimulated by their interesting physicochemical properties and potentially useful pharmacological properties[12]. Organoarsenicals were the first compounds applied for the successful treatment of syphilis (they have since been replaced by penicillin) and have been used as feed additives in livestock to prevent bacterial and parasitic infections.

Organoarsenic(III) compounds are known to be biologically active. Antimicrobial activities of some arsenic(III) complexes with Schiff bases have been evaluated[13]. The toxicity as well as

therapeutic value of organometallics is well known. The introduction of metal ions within biological macromolecules such as proteins and nucleic acids is a continuing area of research. Arsenic is another metal known to the ancients with toxic as well as medicinal Properties. Arsenic was widely used as a pesticide in the form of calcium arsenate. Antimony had quite widespread use in pharmacology for the treatment of syphilis, fever, melancholy, pneumonia, epilepsy, and inflammatory conditions[14]. Organic antimony salts are used medically to treat some tropical diseases[15], especially in the treatment of all forms of leishmenasis[16].

Organoantimony compounds also exhibit significant antimicrobial[17] as well as antitumor activities[18], which is associated with cytostatic activity[19] similar to that for cisplatin. Antimony compounds have been used for nearly a century in the clinical treatment of the parasitic disease, leishmaniasis. About 70 years ago, trivalent antimonial drugs were substituted by less toxic pentavalent antimonials in the treatment of these diseases[20, 21]. In addition, organo-antimony derivatives were evaluated for their cytotoxic activity against tumor cell lines[22]. Silvestru *et al.* reported for the first time the antitumoral activity of organoantimony(III) derivatives[23,24]. The relatively high antitumoral activity of organo-antimony(V) derivatives has also been reported in literature[25-27]. The antifungal and antibacterial activities of some organoantimony(V) compounds have also been reported[28,29]. Despite these previous studies, organoantimony compounds still deserve further chemical and pharmacological investigations.

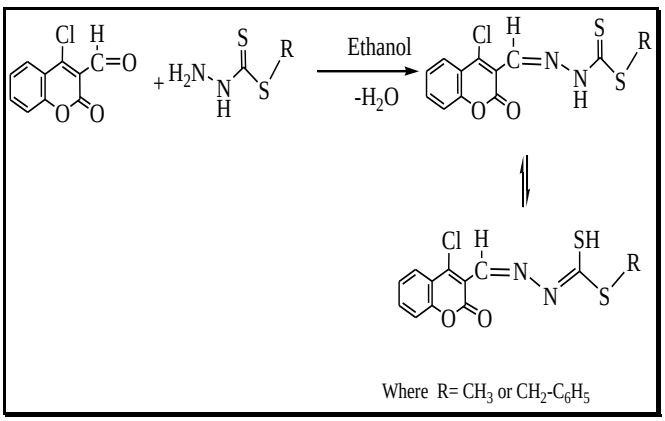
The focus of our present communication is on the exploration of the studies on the synthesis, structural, and biological aspects of Sb(III) and As(III) complexes of stereochemical as well as biological interest with monobasic bidentate hydrazinecarbodithioic acid ligands.

MATERIALS AND METHODS:

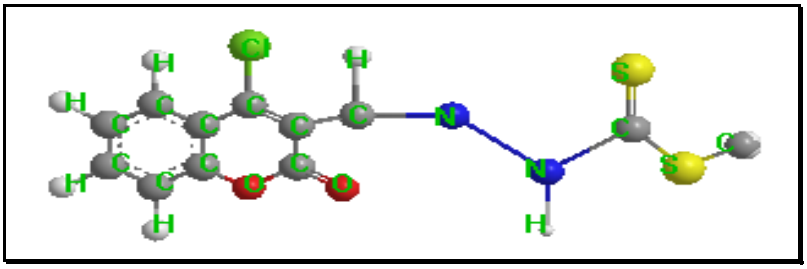
Triphenylantimony and triphenylarsene were obtained from Aldrich. 4-Hydroxycoumarin was obtained from Alfa Aesar. All chemicals used were of reagent grade. The reactions were carried out under anhydrous conditions. Molecular weights were determined by the Rast camphor method. Chlorine was estimated by Volhard's method. Arsenic and antimony was estimated iodimetrically. Nitrogen was estimated by the Kjeldahl method, and sulfur was estimated by the Messenger method. Carbon and hydrogen analyses were performed at the Saurashtra University, Gujarat, India. The infrared (IR) spectra have been recorded on a Nicolet Megna FTIR- 550 spectrophotometer using KBr pellets. Conductivity data were obtained with a Digimed DM31 apparatus equipped with a conductivity cell (C = 1.185 cm−1). Atomic absorption analyses of arsenic and antimony contents were carried out on a Bio-Age/752/28. ^{1}H- and ^{13}C-NMR were recorded at room temperature using a JEOL-AL-300 FT NMR spectrometer in DMSO-d_6, with TMS as internal standard. X-ray powder diffractogram of a representative compound was obtained on a Philip Model PW1840 automatic diffractogram using Cu(Kα) target with Mg filter. The wavelength used was 1.540598 A◦.

Preparation of the Ligands

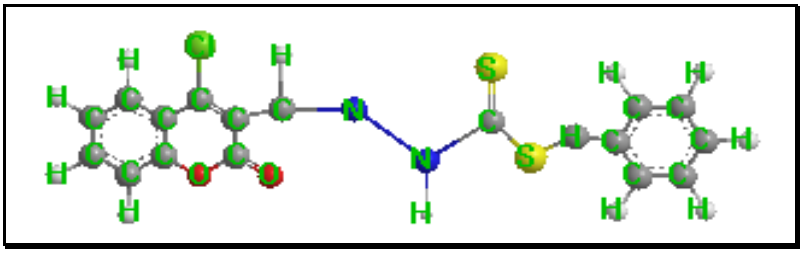
The ligands [1-(2-oxo-2H-chrome-3-yl-ethylidene] hydrazinecarbothionicacid benzyl ester (L^1H) and [1- (2 - oxo- 2H – chrome – 3 – yl - ethylidene] hydrazinecarbothionicacid methyl ester (L^2H) were prepared by the condensation of 4-chloro, 3-formylcoumarin (0.02mol) with, S-methyldithiocarbazate (0.02 mol) or S-benzyldithiocarbazate (0.02 mol) in 1:1 molar ratio. The resulting products were then recrystallized with alcohol and dried under vacuum. The synthetic route of ligands (L^1H and L^2H) is shown in **scheme 1**. Their physiochemical properties and analytical data are given in **Table 1**. The structures of ligands are shown in **Fig. 1.**



SCHEME 1. SYNTHETIC ROUTE OF LIGANDS



N'-[1-(2-oxo-2H-chrome-3yl-ethylidene]-hydrazinecarbodithionic acid methyl ester (L^1H)



N'-[1-(2-oxo-2H-chrome-3yl-ethylidene]-hydrazinecarbodithionic acid benzyl ester (L^2H)

FIG. 1 STRUCTURES OF LIGANDS L^1H AND L^2H

Preparation of the complexes

The complexes were synthesized by the reaction of the ligands with triphenylarsine and triphenylantimony in 1:1 and 1:2 molar ratios in dry benzene. The reaction mixture was heated under reflux for 10–15 h. The residue formed was separated out, filtered off, washed with n-hexane and the filtrate was dried under reduced pressure. The physiochemical properties and analytical data of these complexes are listed below in **Table-1**.

TABLE 1: ANALYTICAL DATA AND PHYSICAL PROPERTIES OF THE LIGANDS AND THEIR COMPLEXES

Compounds	Colour	Melting Point (^{0}C)	Found (Calculated.) (%)					Molar mass Found (Calc.)	Yield (%)
			C	H	N	S	M		
L^1H	Brown	140	69.07 (69.12)	4.08 (4.19)	23.46 (23.51)	20.43 (20.50)	-	312.32 (312.79)	86
L^2H	Sandy	139	55.16 (55.59)	3.29 (3.37)	7.08 (7.20)	16.28 (16.49)	-	388.56 (388.90)	84
$[Ph_2As(L^1)]$	Light yellow	200	53.10 (53.29)	3.16 (3.35)	4.80 (5.18)	11.79 (11.86)	13.56 (13.85)	540.50 (540.92)	70
$[PhAs(L^1)_2]$	Brown	210	46.32 (46.44)	2.64 (2.73)	7.18 (7.22)	16.34 (16.53)	9.60 (9.65)	775.81 (775.92)	75
$[Ph_2Sb(L^1)]$	Yellow	174	49.00 (49.04)	3.01 (3.09)	4.23 (4.77)	10.76 (10.91)	20.54 (20.71)	587.67 (587.75)	73
$[PhSb(L^1)_2]$	Light yellow	225	43.58 (43.81)	2.41 (2.57)	6.32 (6.81)	15.45 (15.59)	14.56 (14.80)	822.36 (822.43)	71
$[Ph_2As(L^2)]$	Orange	185	58.23 (58.40)	3.46 (3.59)	4.43 (4.54)	10.24 (10.39)	12.08 (12.14)	616.97 (617.02)	70
$[PhAs(L^2)_2]$	Orange	200	54.27 (54.37)	3.07 (3.15)	6.00 (6.04)	13.67 (13.82)	8.03 (8.07)	927.67 (927.81)	72
$[Ph_2Sb(L^2)]$	Light yellow	190	54.21 (54.28)	3.46 (3.58)	4.16 (4.22)	9.56 (9.66)	18.24 (18.34)	663.79 (663.85)	76
$[PhSb(L^2)_2]$	Light yellow	210	51.56 (51.76)	2.48 (2.79)	5.67 (5.75)	13.08 (13.16)	12.37 (12.49)	974.61 (974.63)	69

MICROBIOLOGICAL STUDIES

Antifungal activity

The antifungal activity of the standard fungicide (Fluconazole), ligands, and complexes was tested for their effect on the growth of microbial cultures and studied for their interaction with *Aspergillus niger* and *Fusarium oxysporum* using Czapek's agar medium having composition glucose 20 g, starch 20 g, agar–agar 20 g, and distilled water 1000 mL. To this medium was added requisite

amount of the compounds after being dissolved in methanol to get 100 and 200 ppm solution. The medium was then poured into Petri plates and spores of fungi were placed on the medium using inoculum's needle. These Petri plates were wrapped in polythene bags containing a few drops of alcohol and were placed in an incubator at 30±2º C. The controls were also run and three replicates were used in each case. The linear growth of the fungus was recorded by measuring the diameter of the fungal colony after 96 h and the percentage inhibition was calculated by the equation:

% Inhibition = (C-T) 100/ C

Where C and T are the diameters of the fungal colony in the control and test plates, respectively[30].

Antibacterial activity

The antibacterial activity was screened against Gram-positive (*P. aeruginosa*) and Gram-negative (*E. coli*) using the paper disc method[31]. Each compound was dissolved in methanol and the solutions of 500 and 1000 ppm were prepared separately. Paper discs of Whatman filter paper (No. 42) of 2 cm were cut and sterilized in an autoclave. Paper discs soaked in the desired concentration of the complex solutions were placed aseptically in Petri dishes containing nutrient agar media (agar 20 g+ beef extract 3 g + peptone 5 g) seeded with *E. coli* and *P. aeruginosa* bacteria, separately. The Petri dishes were incubated at 37ºC and the inhibition zones were recorded after 24 h of incubation. The antibacterial activity of Streptomycin was also recorded using the same procedure, concentrations, and solvent. The % activity index for the complex was calculated by the formula:

$$\% \text{ Activity Index} = \frac{\text{Zone of inhibition by test compound (diameter)} \times 100}{\text{Zone of inhibition by standard (diameter)}}$$

Insecticidal activity

A pest is an animal whose population buildup increases above a certain level of economic injury, and its existence conflicts with human welfare, convenience, and profit[32]. To control the insect pests, the man since long has been employing various strategies which include mechanical, physical, chemical, and biological methods.

By feeding method larvicidal efficacy of the synthesized chemicals was assessed. The last instar larvae (Corcyra cephalonica) were separated from subculture and kept in vials containing 5 g of topically treated wheat grains with 1 mL of chemicals. Until the pupal formation, larvae were allowed to continue their development on this diet, replicated thrice, each dose. The food was treated with solvent only in control. By Abott's formula, larval mortality and percentage of corrected mortality were calculated.

RESULTS AND DISCUSSION:

UV Spectra

Electronic spectra of the ligand in methanol display maxima at ~298 and ~320 nm, which are due to π– π* electronic transitions and remain almost unchanged in the spectra of the metal complexes. The electronic spectra of the ligands show a band due to the >C=N chromophore observed at 365nm and shows a bathochromic shift of 20–30nm after coordination of azomethine nitrogen to the metal, indicating delocalization of the electronic charge within the chelate ring and thereby stabilizing the resulting complexes.

IR Spectra

Absorption frequencies of ligands and their metal complexes along with their assignments are listed in table.2.The IR spectra of the free ligands L^1H and L^2H display absorption bands at 3150-3250, 1610-1630 and 1050-1080 cm−1 assigned to - (NH), (>C=N), and (>C= S), respectively. The broad band due to - (NH) vibrations, at 3298 cm^{-1} disappears in the spectra of the complexes, indicating the deprotonation of this group on coordination with the metal atom. The marked shift (10–20cm−1) of (>C=N) band observed in all the complexes indicates the involvement of azomethine nitrogen upon complexation. The bands at1735 cm-1 due to (>C=O) of lactone moiety of the ligands remain almost unchanged in the complexes indicating their non- involvement in complexation. The band due to (>C=S) is shifted towards lower frequencies in the complexes indicating coordination of sulfur to the central metal atom. So, the potential binding sites are sulfur and azomethine nitrogen atoms at the ligand molecules. Some new bands observed in the regions 430-438 cm^{-1} for ν (As←N) and 440-450 for ν (Sb←N)[33]. The As–Ph vibrations have been observed in the range 472–496 cm^{-1} [34].

^{1}H NMR Spectra

The ^{1}H NMR spectra of the ligands L^1H and L^2H show signals due to –NH protons at δ8.45 and 8.68 respectively. These disappear in the spectra of the complexes indicating deprotonation and simultaneous bond formation between thiolic sulfur and arsenic or antimony. The aromatic protons appear at 6.81-8.23. The ^{13}C NMR spectra of the ligands and their arsenic or antimony complexes were carried out in DMSO-d_6. The signal due to azomethine carbon in L^1H and L^2H appeared at 160.34 and 162.16 ppm, respectively and on complexation it shows peaks at δ163.0-168.54, ppm which indicates that nitrogen is involved in coordination. The signal due to lactone >C=O carbon atoms remains almost at the same position in the ^{13}C NMR spectra of the metal complexes while a mark shifts in the position of the carbon atoms attached to the sulfur atom shows that proton is lost via thioenolization (**Table .2**)

TABLE 2. IR (CM^{-1}) AND ^{1}H NMR (δ, PPM) SPECTRAL DATA OF THE LIGANDS AND THEIR COMPLEXES

	1H NMR spectral data					
Compounds	**(>C=N)**	***M←N***	***-NH***	***-CH$_3$***	***-NH$_2$***	**Aromatic protons (m)**
L^1H	1610	-	8.48	2.07	3.43	6.42–8.04
L^2H	1622	-	8.68	-	3.42	6.45–8.14
$[Ph_2As(L^1)]$	1590	320	-	2.11	3.46	6.46-8.12
$[PhAs(L^1)_2]$	1600	322	-	2.08	3.45	6.73-8.06
$[Ph_2Sb(L^1)]$	1595	325	-	2.04	3.38	6.74-8.08
$[PhSb(L^1)_2]$	1600	227	-	2.06	3.47	6.78-8.10
$[Ph_2As(L^2)]$	1595	408	-	-	3.46	6.79-8.13
$[PhAs(L^2)_2]$	1605	417	-	-	3.47	6.83-8.12
$[Ph_2Sb(L^2)]$	1605	440	-	-	3.39	6.82-8.14
$[PhSb(L^2)_2]$	1610	445	-	-	3.48	6.88-8.17

X-Ray structure determination

The possible lattice dynamics of the finely powdered product, [PhSb(L^1)2] has been deduced on the basis of X-ray powder diffraction studies. The observed interplanar spacing values (’d’ in Å) have been measured from the diffractogram of the compound and the Miller indices h, k and l have been assigned to each d value and 2-Theta angles are reported. The results show that the compound belongs to ’orthorhombic’ crystal system having unit cell parameters as a=12.3568, b=18.3144, c=8.659, maximum deviation of 2-Theta= 0.028 and Alpha= 90, Beta= 90, Gama=90 at the wavelength = 1.540598.

The synthesized complexes are soluble in methanol, DMF and DMSO. The molar conductance values of 10^{-3} M solutions of metal complexes lie in the range 10–15 ohm^{1}cm^2 mol^{-1}in dry DMF indicating their non electrolytic behavior.

Biological Results and Discussion:

The results of antimicrobial activity are shown in **Fig. 2** and **Fig. 3.** Both the ligands and their metal complexes were sensitive against all the fungal and bacterial strains. The antimicrobial screening data indicate that the metal complexes are more potent antimicrobial agents than the free ligands.

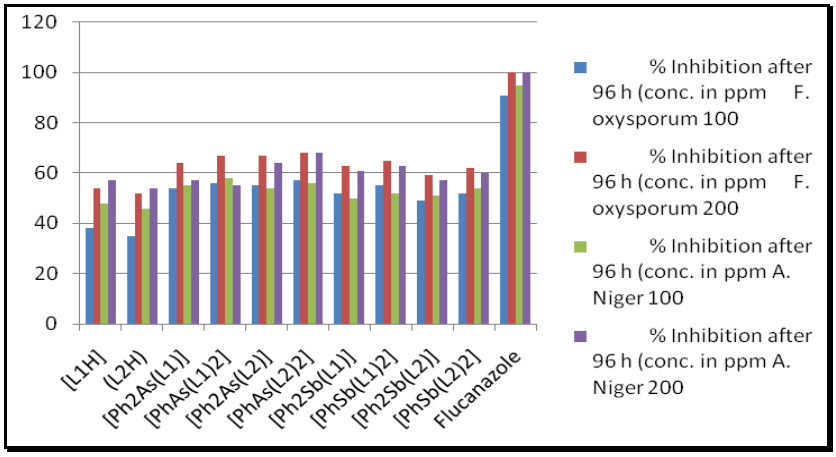


FIG. 2 ANTIFUNGAL ACTIVITY OF LIGANDS AND THEIR CORRESPONDING METAL COMPLEXES

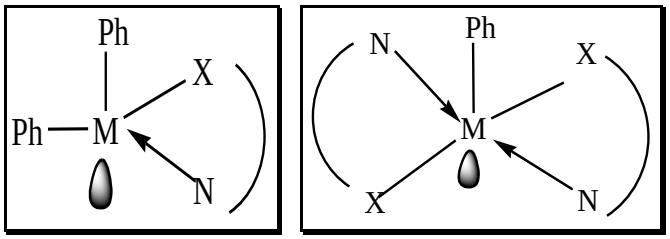
Pesticidal

Data reported in **Table 3** reveal that of the 10 compounds tested, $PhSb(L)_2$ was highly effective as insecticide with LC_{50} 100 mg L^{-1} against *C. cephalonica*. Other compounds showed good insecticidal activity. Broad conclusions may become possible only after a critical appraisal of a larger data set.

TABLE 3. PESTICIDAL DATA OF THE LIGANDS AND THEIR METAL COMPLEXES.

Compounds	Correct motility (%)	χ^2	LC_{50} (mgL^{-1})
L^1H	60.00	0.950	412
L^2H	59.99	0.275	632
$[Ph_2As(L^1)]$	67.77	0.304	306
$[PhAs(L^1)_2]$	72.22	0.737	210
$[Ph_2Sb(L^1)]$	77.77	0.196	351
$[PhSb(L^1)_2]$	83.33	0.154	240
$[Ph_2As(L^2)]$	61.11	0.242	165
$[PhAs(L^2)_2]$	77.77	0.572	135
$[Ph_2Sb(L^2)]$	88.88	0.117	200
$[PhSb(L^2)_2]$	89.00	0.162	100
Control	_	1.42	_

On the basis of spectral studies, tetra-coordinated and penta-coordinated structures may be proposed for the resulting complexes.



1:1 Complexes 1:2 Complex
(Where, X = N/SorO, M = Sb/As)

FIG.4: PROPOSED STRUCTURE OF METAL COMPLEXES

CONCLUSIONS: We describe the synthesis, characterization, and biological activity of As (III) and Sb(III) complexes. On the basis of analytical and spectral data a tetra- and penta-coordinated environment have been proposed around the metal ions for 1:1 and 1:2 complexes respectively. The results of antimicrobial activity indicated that the complexes showed promising antibacterial and antifungal activities.

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