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Synthesis, Spectral studies and Antimicrobial activity of Arsenic (III) Benzaldehyde Semicarbazone, Thiosemicarbazone and their Derivatives

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Abstract

The synthesis and spectral studies of the complexes of As(III) is reported in 1:2 with the general composition of $(L)_2$ AsCl (where L= Benzaldehydede semicarbazones and thiosemicarbazones) Complexes of Arsenic(III) with benzaldehyde semicarbazone, m-cyano benzaldehyde semicarbazone, o-nitro benzaldehyde semicarbazone, benzaldehyde thiosemicarbazone, m-cyano benzaldehyde thio semicarbazone and o-nitro benzaldehyde thiosemicarbazone have been synthesized and characterised by elemental analysis (C, H, N, Cl, S, O and As), molecular weight measurements and spectral studies(IR,^{1}HNMR,^{13}CNMR). These complexes were also screened for their antibacterial and antifungal activities. The result reveal that the metal chelates are more potent than parent ligand.

Keywords: Semicarbazone, thiosemicarbazone, benzaldehyde, antimicrobial activity, ligand.

Introduction

The semicarbazones and thiosemicarbazones complexes have been stimulated by their biological activity [1-3]. These compounds present a great variety of biological activity such as antibacterial, antifungal, antiviral and antiinflamatory[4-9]. The transition metal complexes of both ligands have appeared in the literature[10-14].But little is known about the complexing behaviour of Arsenic. Arsenic (III) tripyrazolinates and Arsenic (III) thiocarbamyls have been synthesized by the reaction of $AsCl_3$ with sodium salt of pyrazolinates and thiocarbamyls respectively and found greater activity of arsenic (III)tripyrazolenes and thiocarbamyls towards all tested bacteria and fungi than free ligand[15,16]. In Present communication we described the synthesis, spectral studies and antimicrobial activity of Arsenic (III) Benzaldehyde semicarbazone, thiosemicarbazone and their derivatives.

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Experimental

Chemicals and Solvent used were dried and purifies by standard methods before use[17]. Moisture was excluded from the glass apparatus using $CaCl_2$. All the chemical used were of analytical grade. Arsenic trichloride has been prepared by the reaction of arsenic trioxide with thionyl chloride. Ligands were prepared by the reported procedure[18].

Synthesis of Ligands

Synthesis of benzaldehyde semicarbazone:

0.1mol.(11.15g.) of semicarbazide hydrochloride and 0.1mol.(8.20g.) of crystaline sodium acetate were dissolve in required quantity of distilled water by warming and combine both solution in a beaker, then add 0.1mol. of corresponding benzaldehyde and shake

well on cooling yellow shiny solid separates out. Filtered and washed with cold water. It was recrystallised from ethanol.

Synthesis of benzaldehyde thiosemicarbazone:

To a hot solution of thiosemicarbazide (1.82gm., 20mmol) in 160 ml. methanol was added drop wise a solution of corresponding benzaldehyde (20mmol) in 70 ml. methanol during 30 minutes. The mixture was stirred and refluxed for 4 hrs. it was filtered and the filtrate was concentrated to half the volume under reduced pressure. After a slow evaporation of the concentrate at room temperature, crystals were collected by filtration, washed with cold ethanol and recrystallised from ethanol.

Synthesis of Complexes

Synthesis of $(R,ArC_2H_2N_3O)_2$ AsCl

Chloroarsenic (III) benzaldehyde semicarbazones have been synthesized by the reaction of arsenic trichloride with corresponding benzaldehyde semicarbazone in 1:2 molar ratios. The reaction mixture refluxed for 1-2 hours at 40°C, till the color of the reaction mixture underwent a change. Reaction mixture was filtered. The solvent was removed under reduced pressure from the filtrate. The brown colored solid thus obtained washed with alcohol and dried in vacuum to get the purified product. (Analytical data presented in Table1) compounds 1-3 prepared by the same route.

$$AsCl_3 + 2(R,ArC_2H_3N_3O) \xrightarrow{\text{Ethanol}} (R,ArC_2H_2N_3O)_2\,AsCl + 2HCl$$

Synthesis of $(R,ArC_2H_2N_3S)_2$ AsCl

Chloroarsenic (III) benzaldehyde thiosemicarbazones have been synthesized by the reaction of the solution of arsenic trichloride in ethanol was added drop wise to a stirred solution of the corresponding benzaldehyde thiosemicarbazones in methanol, in 1:2 molar ratios. Sodium acetate in 3ml. of water then added. The reaction mixture was refluxed for 2 hours and stirred of 24 hours at room temperature, till the colour of the reaction mixture underwent a change. Reaction mixture was filtered. The solvent was removed under reduced pressure from the filtrate. The light yellow coloured solid thus obtained washed with alcohol and dried in vacuum to get the purified product. (Analytical data presented in Table1) compounds 4-6 prepared by the same route.

$$AsCl_3 + 2(R,ArC_2H_3N_3S) \xrightarrow{\text{Ethanol}} (R,ArC_2H_2N_3S)_2\,AsCl + 2HCl$$

Physical Measurements

Chlorine was estimated by volhards method and arsenic was estimated iodometrically[19]. Infrared spectra were recorded on Perkin Elmer Model 557 FT-IR spectrophotometer using CsI cell in the range 4000-200 cm^{-1}. NMR spectra were recorded at room temperature on a Bruker DRX-300 spectrometer operated at 300 and 75.45 MHz for ^1H, ^{13}C, using TMS (tetramethylsilane) as internal standard. Molecular weights were determined on a knauer vapour pressure in $CHCl_3$at 45^0C[20]. The elemental analysis(C, H and N) was estimated by coleman analyser

Table1. Physical and Analytical data of $(R,ArC_2H_2N_3O)_2$AsCl and $(R,ArC_2H_2N_3S)_2$AsCl complexes

S. No	Product	Yield %	M.P. ^0C	M.W. (Calc) Found	Elemental Analysis % (Calculated) Found						
					C	H	N	O	S	Cl	As
1	$(R,ArC_2H_2N_3O)_2$AsCl	81	145	(434.7)	((44.21)	(3.71)	(19.33)	(7.36)	-	(8.16)	(17.23)
				433.8	44.36	3.55	19.51	7.24	-	8.61	16.73
2	$(R,ArC_2H_2N_3O)_2$AsCl	80	180	(524.7)	(36.62)	(2.69)	(21.36)	(18.30)	-	(6.76)	(14.28)
				523.9	37.12	2.59	21.45	18.40	-	6.66	13.84
3	$(R,ArC_2H_2N_3O)_2$AsCl	80	215	(484.7)	(44.60)	(2.91)	(23.12)	(6.60)	-	(7.31)	(15.46)
				484.2	44.16	3.1	22.67	7.1	-	7.21	15.76
4	$(R,ArC_2H_2N_3S)_2$AsCl	85	140	(466.8)	(41.16)	(3.45)	(18.00)	-	(13.74)	(7.59)	(16.05)
				466.7	41.07	3.85	18.19	-	13.64	6.69	16.55
5	$(R,ArC_2H_2N_3S)_2$AsCl	83	180	(556.8)	(34.51)	(2.53)	(20.12)	(11.49)	(11.53)	(6.37)	(13.45)
				557.2	35.12	2.37	20.75	11.37	10.62	6.82	12.95
6	$(R,ArC_2H_2N_3S)_2$AsCl	85	220	(516.9)	(41.83)	(2.73)	(21.68)	-	(12.41)	(6.86)	(14.50)
				515.9	41.24	3.13	21.87	-	12.31	6.46	15.00

Where R=H, Ar=C_6H_5 in1 and 4, $C_6H_4NO_2$ in 2 and5,C_6H_4CN in 3 and 6.

Antimicrobial studies

The antifungal and antibacterial activity of the parent ligand and their complexes was tested in vitro for the growth inhibiting potential against various fungal and bacterial strains using agar disk diffusion technique[21]. Inoculums of bacteria were prepared in nutrient broth and fungi in potato dextrose agar slant. The cultures were inoculated and incubated for 24 hrs. for bacteria and 72 hrs for fungi. For the preparation of seeded agar plate the molten medium was poured in sterile petridish to get a depth of 5mm. The medium was left to solidify and it was seeded with test organism. 5ml sterile water was added to agar slant culture of fungi to get suspension. A sterile cotton swab was dipped in suspension, lightly spread over the solidified medium. The petridish was left for a few minutes. 5mm. discs of whatmann filter paper no.1 were cut and sterilized. The filter paper discs were dipped in the solution of sample after that the disc was removed and left in a sterile petridish to permit the solvent to evaporate. After 10 minutes the discs were transferred to seeded agar plates. 2 discs were kept on the seeded agar plate. Finally dishes were incubated at 37^0C for 24hrs. (for bacteria) and at 30^0C for 72hrs. (for fungi), inhibition zones were detected around each disc.

Results and Discussion

General property

All the compounds of benzaldehyde semicarbazones with arsenic are light yellow colored and all the compounds of benzaldehyde thiosemicarbazone with arsenic are brown colored. All the compounds are amorphous and stable at room temperature. These are partial soluble in organic solvents (chloroform, acetone, benzene and ether) and soluble in coordinating solvents (Tetrahydro furan, Dimethyl Formamide and Dimethyl sulphoxide).

Table 2. IR spectral data (cm^{-1}) for $(R,ArC_2H_2N_3O)_2AsCl$ and $(R,ArC_2H_2N_3S)_2AsCl$ complexes

S.No.	Compounds	¬ (NH_2)	¬ (N-N)	¬ (C=N)	¬ (C-O)	¬ (C-S)	¬ (As-X)	¬ (As-N)
1.	$(R,ArC_2H_2N_3O)_2$ AsCl	Sy-3212 Asy-3424	1065	1560	1668	-	512	430
2.	$(R,ArC_2H_2N_3O)_2$ AsCl	Sy-3225 Asy-3420	1063	1566	1670	-	509	428
3.	$(R,ArC_2H_2N_3O)_2$ AsCl	Sy-3215 Asy-3422	1058	1558	1675	-	515	425
4.	$(R,ArC_2H_2N_3S)_2$ AsCl	Sy-3217 Asy-3423	1067	1565	-	805	355	426
5.	$(R,ArC_2H_2N_3S)_2$ AsCl	Sy-3216 Asy-3422	1062	1565	-	803	350	435
6.	$(R,ArC_2H_2N_3S)_2$ AsCl	Sy-3218 Asy-3425	1058	1555	-	810	357	426

Where R=H, Ar=C_6H_5 in1 and 4, $C_6H_4NO_2$ in 2 and 5, C_6H_4CN in 3 and 6, X=O in 1-3, S in 4-6.

Infrared Spectra

In IR spectra the bands of the –NH group observed at 3000-3195 cm^{-1} for the ligands, these bands are disappear in the spectra of complexes, which indicates the deprotonation of the –NH group[22,23]. Two sharp bands are observed at 3212-3225 and 3420-3425 cm^{-1} for semicarbazone and thiosemicarbazone ligand due to symmetrical and asymmetrical modes of $–NH_2$ group, remain unchanged indicates the non coordination of the $–NH_2$ group in coordination[24]. The strong band observed at 1600-1620 cm^{-1} range in the free ligands have been assigned to ν (C=N) stretching vibrations. On complexation, these bands were observed to lower frequency at 1555-1566 cm^{-1}, thus indicates the coordination of azomethine nitrogen to metal atom[25]. A sharp band observed at 1690-1720 cm^{-1} in semicarbazone mainly due to ν (C=O) stretching frequency. These bands are shifted to lower frequency side at 1668-1675 cm^{-1} in the spectra of the complexes due to coordination of the carbonyl oxygen to the metal atom[26]. All thiosemicarbazone ligands showed medium bands in the 790-900 cm^{-1} range ascribed to ν (C=S) vibrations, these absorption bands shift to lower frequency at 803-810 cm^{-1} on the coordination of the thiocarbonyl sulfur to metal ion[27] In the spectra of the complexes the presence of new bands in comparison of free ligands in the region of 509-515 cm^{-1} and 425-435 cm^{-1} and 350-357 cm^{-1} these bands show stretching vibrations of ν (As-O) and ν (As-N) and ν (As-S) respectively[15,22].

Table 3. ^{1}H & ^{13}C NMR spectral data for $(R,ArC_2H_2N_3O)_2AsCl$ and $(R,ArC_2H_2N_3S)_2AsCl$ complexes

S.No	Compound	^{1}H NMR Chemical Shift (¢ppm)	^{13}C NMR Chemical Shift (¢ ppm)
1.	$(R,ArC_2H_2N_3O)_2AsCl$	7.23-7.36 (10H, m, Ar-H) 8.25 (2H, s, H-C=N) 8.57(2H, s, NH_2)	126.7-135.4 (Ar-C) 152.0 (C=N) 155.2 (C-O)
2.	$(R,ArC_2H_2N_3O)_2AsCl$	7.55-8.24 (8H, m, Ar-H) 8.33 (2H, s, H-C=N) 8.51(2H, s, NH_2)	123.7-135.1 (Ar-C) 151.8 (C=N) 154.2 (C-O)
3.	$(R,ArC_2H_2N_3O)_2AsCl$	7.66-8.12 (8H, m, Ar-H) 8.31 (2H, s, H-C=N) 8.41(2H, s, NH_2)	129.8-134.8 (Ar-C) 150.3 (C=N) 156.5 (C-O)
4.	$(R,ArC_2H_2N_3S)_2AsCl$	7.39-7.44 (10H, m, Ar-H) 8.28 (2H, s, H-C=N) 8.56(2H, s, NH_2)	129.2-135.1 (Ar-C) 146.3 (C=N) 167.1 (C-S)
5.	$(R,ArC_2H_2N_3S)_2AsCl$	7.59-8.26 (8H, m, Ar-H) 8.29 (2H, s, H-C=N) 8.59(2H, s, NH_2)	124.2-134.5 (Ar-C) 147.3 (C=N) 167.5 (C-S)
6.	$(R,ArC_2H_2N_3S)_2AsCl$	7.60-8.10 (8H, m, Ar-H) 8.38 (2H, s, H-C=N) 8.51(2H, s, NH_2)	127.2-135.3 (Ar-C) 146.0 (C=N) 168.1 (C-S)

Where R=H, Ar=C_6H_5 in1 and 4, $C_6H_4NO_2$ in 2 and 5, C_6H_4CN in 3 and 6. s=singlet, t=triplet, m=multiplate.

Multinuclear NMR spectroscopy

The ^{1}H NMR spectra of the complexes exhibit aromatic proton signal at δ 7.23-8.24 ppm. The spectra of ligands exhibit the N-H proton signal in the range of δ 10.83- 10.90 ppm. The NH signal disappears in the spectra of the metal complexes indicating the chelation of the ligand moiety to arsenic through the nitrogen atom[11,28]. A sharp singlet is observed for the NH_2 proton at δ 8.25-8.59 ppm. in complexes. A comparison of spectra of the metal complex with those of the ligands provides very useful information about the mode of bonding. In semicarbazone complexes the signal for C=N carbon appears at δ 150.3-152.0 ppm and C-O carbon appears at δ 154.2-156.5 ppm. but these signals was appears at δ 153.40 ppm and δ 159.68 ppm in free ligands respectively this shows involvement N and O in chelation. In thiosemicarbazone the signal for C=N carbon appears at δ 146.0-147.3 ppm and C-S carbon appears at δ 167.1-168.1 ppm in complexes but these signals was appears at δ 152.40 ppm and δ 176.98 ppm. in free ligands respectively this shows involvement N and S in chelation. Where signal for aromatic carbon at δ 123.7-135.4 ppm[29,30].

Table 4. Antimicrobial activity of $(R,ArC_2H_2N_3O)_2AsCl$ and $(R,ArC_2H_2N_3S)_2AsCl$ complexes

S.No	Free ligands and their metal complexes	Zone of inhibitions in m.m.						
		Bacteria					Fungi	
		Echerichia coli	*Bacillus lichaniformis*	*Pseudomonas aeruginosa*	*Klebsiella pneumoniae*	*Staphylococcus aureus*	*Penicillium notatum*	*Aspergillus niger*
1.	$(R,ArC_2H_3N_3O)$	14	15	12	15	13	14	12
2.	$(R,ArC_2H_2N_3O)_2AsCl$	18	17	21	19	18	17	14
3.	$(R,ArC_2H_3N_3O)$	13	12	14	12	11	11	12
4.	$(R,ArC_2H_2N_3O)_2AsCl$	16	18	19	18	18	16	18
5.	$(R,ArC_2H_3N_3O)$	10	11	10	11	12	11	12
6.	$(R,ArC_2H_2N_3O)_2AsCl$	19	18	17	20	16	20	20
7.	$(R,ArC_2H_3N_3S)$	16	15	15	16	16	16	16
8.	$(R,ArC_2H_2N_3S)_2AsCl$	19	20	22	19	22	20	22
9.	$(R,ArC_2H_3N_3S)$	15	16	15	17	13	16	15
10.	$(R,ArC_2H_2N_3S)_2AsCl$	18	19	19	22	19	21	22
11.	$(R,ArC_2H_3N_3S)$	17	14	16	18	19	12	17
12.	$(R,ArC_2H_2N_3S)_2AsCl$	19	19	20	22	22	19	22
13.	R	23	24	24	24	24	24	25

Where R=H, Ar=C_6H_5 in1,2,7,8, $C_6H_4NO_2$ in 3,4,9,10, C_6H_4CN in 5,6,11,12, R=Chloramphenicol (antibacterial agent), Terbinafin (antifungal agent).

Microbial assay

The antimicrobial activity of free ligands and their metal complexes were tested against the bacterial species Staphylococcus aureus, Bacillus lichaniformis, Escherichia coli, Klebsiella pneumoniae, Pseudomonas aeruginosa and the antifungal activity were tested against Aspergillus niger and Penicillium notatum. The results have been compared with known drug chloramphenicol against bacteria and terbinafin against fungi. Semicarbazone and thiosemicarbazone are of considerable interest due to their biological activity. The detected antibacterial, antifungal activity of the organic molecule is usually increased on the complexation. The result reveals that the chelates are more potent than the parent ligands. The complexes of thiosemicarbazone were found to be more effective than semicarbazone complexes and free ligand. The results are shown in table4.

Structure

Present study describes the series of chloroarsenic (III) benzaldehyde semicarbazone, $(R,ArC_2H_2N_3O)_2$ AsCl, benzaldehyde thiosemicarbazone $(R,ArC_2H_2N_3S)_2$ AsCl and their derivatives. The bidentate behavior of these ligands has been confirmed by IR, ^{1}H NMR and ^{13}C NMR data. In these complexes the central arsenic (III) atom appears to acquire the coordination number five and most possible geometry around the arsenic atom is distorted octahedral fig1.

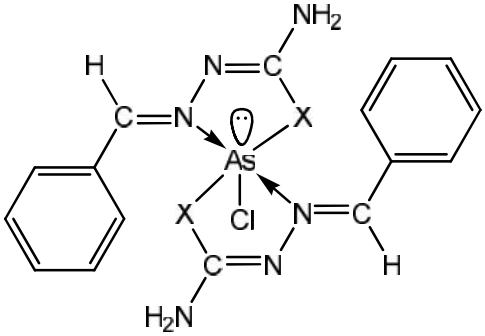

Figure 1: Structure of $(R,ArC_2H_2N_3X)_2$ AsCl where X=O for semicarbazone and S= thiosemicarbazone

Conclusion

It is very difficult to comment on the molecular structure of these compounds in solid state. However the bidentate behaviour of the ligands in $(R,ArC_2H_2N_3O)_2$AsCl and $(R,ArC_2H_2N_3S)_2$AsCl has been confirmed by IR, ^{1}H NMR and ^{13}C NMR data. In these complexes the central atom arsenic (III) appears to acquire the coordination five and most plausible geometry around the arsenic atom is distorted octahedral.

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