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Complexes of silver picrate with Succinylsulfathiazole and Sulfisoxazole

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Abstract: Molecular complexes of silver picrate (AgP) with succinylsulfathiazole (SSTZ) and sulfisoxazole (SUL) were studied by elemental analyses, and infrared spectroscopy. Ag and NO₂ group of AgP acceptor are involved in complexation with both donors (SSTZ and SUL). Carboxylate, amide nitrogen, sulfonamide oxygen and nitrogen of SSTZ while sulfonamide oxygen, nitrogen of aniline as well as isoxazole ring of SUL participates in complexation with AgP.

Keywords : Succinylsulfathiazole, sulfisoxazole, silver picrate, complexes.

1. Introduction

The compounds of silver are used primarily for their activity as germicides and antiseptics. These include, for example, silver nitrate, silver acetate, silver citrate, silver picrate as well as silver sulphadiazine. Silver nitrate yield silver ions which precipitates proteins. Silver picrate (silver trinitrophenolate) combines the antiseptic activity of silver with that of picric acid. Silver picrate's bacteriostatic effect is a function of free silver ions released from the combination used as antiseptics. Succinylsulfathiazole $[C_{13}H_{13}N_3O_5S_2, 4-oxo-4-(\{4-[(1,3-thiazol-2-ylamino)sulfonyl]$

phenyl}amino) butanoic acid, SSTZ, Fig. 1] is an antibacterial drug, derived from sulfonamide and used in the prevention and treatment of gastrointestinal infections. Sulfisoxazole [4-amino-N-(3, 4- dimethyl-5-isoxazolyl) benzenesulfonamide] (SUL, Fig.2) is used to treat urinary tract infections.

Metal sulfonamides have received much attention due to the fact that sulfonamides were the first effective chemotherapeutic agents to be employed for the prevention and cure of bacterial infections in humans ¹. It has been reported that the biological activity of sulphur-containing ligands gets enhanced on undergoing complexation with metal ions ²⁻⁴.



Fig.1. Structure of succinylsulfathiazole (SSTZ)



Fig. 2. Structure of sulfisoxazole (SUL)

Silver sulfanilamide products were useful as antiseptics and bactericides ⁵. Few reports on solid metal complexes of SSTZ and SUL, as well as on complexation equilibria of metal-SSTZ and metal-SUL systems have been reported ⁶⁻¹⁰. *myo*-Inositol derived podands bind very well with silver picrate ¹¹. Molecular complexes of antibacterial and antiamoebic drugs with picric acid and silver picrate were reported earlier ¹². The present investigations have been carried out to synthesize and characterize the complexes of sulfa drugs, namely succinylsulfathiazole (SSTZ) and sulfisoxazole (SUL) with silver picrate (AgP).

2. Experimental

The ligands namely succinylsulfathiazole and sulfisoxazole, were of Sigma grade. All other chemicals used for the preparation of complexes were Analar grade quality and were used without further purification. Silver picrate was prepared by literarure method ¹³.

2.1. Preparation of sulfisoxazole-silver picrate complex

Silver picrate (0.2mM) was dissolved in7 ml ethanol. To this solution, 0.2mM SUL in ethanol was added slowly. The final pH of mixture solution was about 4. Light yellow precipitates formed were filtered and washed thrice with ethanol and dried.

2.2. Preparation of succinylsulfathiazole-silver picrate complex

Succinylsulfathiazole (0.2mM) solution in ethanol was added slowly to 0.2mM silver picrate ethanolic solution. The final pH of mixture solution was about 6. Light yellow precipitates formed were filtered and washed thrice with ethanol and dried.

Physico-chemical measurements:

IR spectra of ligands and their AgP complexes were recorded in KBr matrix on Perkin-Elmer Spectrophotometer and elemental analysis of C, H, and N were carried out at RSIC, Chandigarh.

3. Results and Discussion

The elemental analysis data of the SSTZ-AgP and SUL-AgP complexes are presented in Table 1. The stiochiometry of the complexes as deduced from elemental analysis has found to be 1:1 in each case.

When the IR spectra of charge transfer complexes are compared with those of isolated molecules which form the complexes, three type of changes may occur (i) the vibrational frequencies in donor or acceptor (or both) may be shifted (ii) the intensities of the bands may be changed considerably and (iii) new low frequency bands appear due to vibrations of one molecule in the complex against the other ^{14,15}. The band shift on complexation has been used for investigations as a basis for the classification of donor-acceptor complexes.

In the present study, IR spectra of complexes compounds of SUL and SSTZ as donor and AgP as an acceptor are analyzed in order to establish the mode of interaction and the nature of the complex formed 12 . The analysis of the different regions of the spectrum expected to show changes in the position and intensity of the bands on molecular complexation, has been carried out. The major bands considered in the case of acceptor AgP are v_{NO2} , $v_{C=C}$ and v_{C-O} . The important bands of donor molecules have also been assigned. In the case of SSTZ $\nu_{\text{N-H}}$ amide, $\nu_{\text{C=O}}$ amide, $\nu_{\text{SO2}},$ ν_{NH} sulfonamide, v $_{C=0}$ carboxylic have been especially considered. The ν_{NH2} (aniline group), v_{NH} (sulfonamide), v_{SO2} etc. bands of SUL have been considered in order to establish the nature of interactions in the molecular complexes. In general, the change in the position as well as in the intensity of the band can be fixed as a criterion to study the type of interaction between the donor and acceptor molecule forming the complex.

The NO₂ asymmetric and symmetric bands of AgP disappear in the SSTZ-AgP and SSTZ-AgP complexes. The peaks due to $v_{c=0}$ of carboxylic stretching vibration (1701.4 cm⁻¹) and v_{N-H} of sulfonamide at 3319.8 cm⁻¹ disappear while two new peaks at 1691.6 cm $^{-1}$ (v_{coo- asym}) and 1453.3 cm $^{-1}$ (v_{coo-} svm) were observed in IR spectra of SSTZ-AgP complex. The stretching vibration band at 3476.6 cm⁻¹ (v_{N-H}) shift to 3337.4 cm⁻¹ on complexation. $v_{C=N}$ of imidazole ring of SSTZ shows a negative shift of 13.4 cm $^{-1}$ (1540.7 cm $^{-1}$ to 1527.3 cm $^{-1}$). v $_{SO2\ asym}$ and v $_{SO2}$ sym shift to lower wave number. The disappearance of NO₂ asymmetric and symmetric bands of AgP observed at 1623.3 and 1327. 7 respectively in both systems suggest involvement of NO₂ group in complexation. The binding of S atom to Ag has been reported and four Ag⁺ cations linked two ligands (amphiphilic thiophosphoryl derivative of calix-4resorcinarene) by P=S...Ag...S=P bonds ¹⁶. Two different types of compounds were reported in case of sulfanilamide derivatives. In this direction. coordination of Ag(I) with the anion of a sulfanilamide (L) usually produced a non-conducting compound of the composition AgL. However, with sulfamethoxypyrazine and sulfacetamide an ionic compound [AgL₂] Ag was formed 17 .

Complex	Elemental Analysis (%)						
2:2	Calculated			Experimental			
	С	Н	Ν	С	Н	Ν	
SSTZ	43.99	3.74	11.88	43.93	3.68	11.82	
SSTZ-AgP	33.00	2.18	12.15	33.21	2.46	12.23	
SUL	49.48	4.91	15.64	49.42	4.90	15.72	
SUL-AgP	33.84	2.50	13.93	34.01	2.98	13.96	

Table 1: Elemental analysis data for SSTZ-AgP and SSTZ-AgP complexes.

Table 2. Assignment of some of the characteristic vibrational (cm⁻¹) bands of succinylsulfathiazole-silver picrate [SSTZ –AgP] system.

SSTZ (cm ⁻¹)	AgP	$SSTZ - AgP (cm^{-1})$	Assignment
3476.6m		3337.4	$v_{\text{N-H}}$ amide
1675.9		1691.6s	$v_{C=0}$ amide
1540.7s		1527.3s	In plane N-H bending
1251.6m		1275.3s	C-N-H vibrations (amide)
694.1m		-	Out plane N-H bending
646.3m		645.0m	O-C-N bending vibrations
3319.8s		3107.3w	v N-H sulfonamide
742.8w		705.8	v C-S thiazole
1495		1453	v C=N thiazole
1587.1s		1591.3s	C=C str. phenyl ring
1300.7s		1275.3s	v asym. SO_2
1145.3s		1137.8vs	v sym. SO ₂
1227m		1318.1s	v C-N sulfonamide
1701s		-	v C=O carboxylic
945.9s		941.4s	O-H out of plane bending vib.Carboxylic
3028w		3028vw	v O-H carboxylic
	1623.3m		v asym. NO ₂
	1556.7m		
	1327.7s		v asym. NO ₂
	1482.9vs		C=C str.

vs - very strong, s - strong, m - medium, w - weak, vw - very weak, sh - shoulder

Table 3. Assignment of some of the characteristic vibrational (cm ⁻¹) ba	ands of
sulfisoxazole-silver picrate [SUL-AgP] system.	

SUL (cm ⁻¹)	AgP	SUL-AgP (cm ⁻¹)	Assignment
3485.5m		-	vNH_2 aniline
3381.1s		3364.6w	
1647.5s		-	δNH_2 isoxazole ring
1630.9vs		1620s	v C=N isoxazole ring
	1623.3m	-	v asym. NO ₂
	1556.7m	-	
	1327.7s	-	v asym. NO ₂
	1482.9vs	1473.7	C=C str.
1596.0s		1598.8sh	C=C str. phenyl ring
1345.6s		1310.2m	v asym. SO_2
1163.9vs		1160.5sh	v sym. SO_2
1324.4sh		-	v C-N aniline
1300.2sh		1300sh	v C-N sulfonamide
841.5m		832m	v C-H disubstituted para benzene

vs - very strong, s - strong, m - medium, w - weak, vw - very weak, sh - shoulder

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