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Research Paper

Synthesis of Novel p-fluorophenyl derivatives of Group-15 Elements (As, Sb, Bi) and their efficacy against Antimicrobial Resistance

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Abstract: The present invention deals the synthesis of a novel p-fluorophenyl derivative of group-15 elements (As, Sb, Bi) through modified method followed by their characterization for their antimicrobial activity against pathogenic strains of bacteria and fungi at different concentrations to find out their efficacy against Antimicrobial Resistance (AMR). It was found that these compounds show remarkable antimicrobial activity and shows effective against Antimicrobial Resistance with different Structure-Activity Relationship.

Keywords: p-fluorophenyl, pathogenic, antimicrobial resistance, antibacterial, antifungal.

Introduction:

The importance of metal based drugs lies in the fact that they are essential components for various physico-chemical processes occurring in living system (Rambert, 1991). The spectrum of the metal based drugs has been expanded as metals can do better due to generation of reactive oxygen species, which help in the treatment of cancer and other acute diseases (Ravi Kant *et. al*., 2008). The organometallic complexes of group-15 elements (As, Sb, Bi) are used for the treatment of various acute and pathogenic diseases in case of human beings (Dominico *et. al*., 1997; Veldhuyzen and Sherman, 1994: Lambert and Midolo, 1997). Due to higher interest in medicinal application of metal based drugs, the present investigation was undertaken to explore the biomedicinal studies of some new organometallic complexes of group-15 elements (As, Sb, Bi) which show water and lipid solubility (Dominico *et. al*., 1997; Stratton *et. al*., 1999;, Ravi Kant *et. al*., 2008; 2009). The interest in organometallic chemistry of fluorine containing compounds gained momentum in recent past due to the unusual character of fluorine and the intrinsic properties shown by fluorocarbon based organometallics (Tripathi et. al., 2012; Ravi Kant et al., 2019; Tripathi et. al., 2020). Besides this perfluoroalkyl and perfluoroaryl derivatives of metals and non-

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metals provide much instructive comparison with compounds based on hydrocarbon residue (Tripathi et. al., 2020). The presence of fluorine atom either in organic group bound to metal or fluorine substituted ligand facilitate the solubility in lipid as well as in water and thus enhancing their bioavailability. In past two decades pentafluorophenyl derivatives of group 15 elements have been explored for their biological potential (Singhal et. al., 2003) and in fact have shown promising trends related to antimicrobial and antitumor activity (Singhal et. al., 2003).

Experimental:

The synthesis of organoarsenic was performed by following methods (Nath et. al., 2004) under oxygen free nitrogen atmosphere.

Reaction of *p*-fluorophenylarsenic(III)dichloride with glycine

In the stirring solution of *p*-fluorophenylarsenic(III)dichloride (1mmol), glycine (1mmol) was added in the presence of trimethylamine (1ml) in toluene and stirred under anhydrous oxygen free nitrogen atmosphere for 6 hr followed by refluxing for 2 more hr to ensure the completion of the reaction. The flocculent white precipitate of $Et_3N.HCl$ (M.P. 240˚C) was formed and filtered off. This filtrate on concentration under vacuum condition gives a light off white solid which was recrystallized by petroleum ether (40-60˚C).

Reaction of *p*-fluorophenylantimony(III)dichloride with glycine

In the stirring solution of *p*-fluorophenylantimony(III)dichloride (1mmol), glycine (1mmol) was added in the presence of trimethylamine (1ml) in toluene and stirred under anhydrous oxygen free nitrogen atmosphere for 6 hr followed by refluxing for 2 more hr to ensure the completion of the reaction. The flocculent white precipitate of $Et_3N.HCl$ (M.P. 240˚C) was formed and filtered off. This filtrate on concentration under vacuum condition gives a light off white solid which was recrystallized by petroleum ether (40-60˚C).

Reaction of *p*-fluorophenylbismuth(III)dichloride with glycine

In the stirring solution of *p*-fluorophenylbismuth(III)dichloride (1mmol), glycine (1mmol) was added in the presence of trimethylamine (1ml) in toluene and stirred under anhydrous oxygen free nitrogen atmosphere for 6 hr followed by refluxing for 2 more hr to ensure the completion of the reaction. The flocculent white precipitate of $Et_3N.HCl$ (M.P. 240˚C) was formed and filtered off. This filtrate on concentration under vacuum condition gives a off white solid which was recrystallized by petroleum ether (40-60˚C).

Antibacterial Activity

Antibacterial activity of the synthesized compound was carried out by disc diffusion method (Singhal et. al., 2004) using ampicilin as standard. The filter paper (Whatman No.1) sterile disc of 5 mm diameter, impregnated with the test compounds (10 □g/ml of ethanol) along with standard were placed on the nutrient agar plate at 37□C for 24 hrs in BOD incubator. The inhibition zone around the dried impregnated disc was measured after 24 hrs.

Antifungal Activity

The antifungal activity of the compound was tested by agar plate diffusion method (Verma and Imam, 1973), using ampicilin as standard wherein concentrations of the test compounds viz., 50 and 100 □g/ml were prepared and tested against two pathogenic fungal strains, *Aspergillus flavus* and *Aspergillus niger*. The 1 ml of each compound was poured into a petri dish containing 20-25 ml of molten potato dextrose-agar medium. As the medium

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solidify, petri dishes were incubated at 37□C for 96 hrs in BOD incubator. After 96 hrs the colony diameter was measured and % inhibition was calculated using standard method.

Results and Discussions:

All the reactions were conducted at room temperature and the final products were recrystallized in petroleum ether (40-60°C) or in benzene. The complexes were light brown, light off-white and off-white color, obtained as a sticky mass which on treatment with dry benzene gets solidified and subsequently crystallized with benzene/pet-ether. The complex has sharp melting point and soluble in chloroform and acetonitrile.

IR Spectra

As expected infrared absorptions inherent to p-fluorophenyl group bound to metal have no difference appreciably. The Infrared absorptions having diagnostic value related to the ligand, has been identified which on preliminary stage indicates the mode of bonding with ligand. The characteristic ν**(OH)** absorption band of ligand which appeared around **3400cm^{-1}** in the free ligand, was found missing in the newly synthesized complexes.

^{1}HNMR Spectra

^{1}H NMR spectra of the compound was recorded in $CDCl_3$ using TMS as an internal reference at 25°C. The disappearance of OH proton signals (δ9.1 ppm) present in the ligand clearly indicates the formation of glycine derivative. The appearance of singlet for -CH_3 protons at δ4.85 ppm showed the ligand is in one plane. The phenyl protons for the derivatives appear as multiplets in the range δ7.80-7.20 ppm.

UV Spectra

The electronic spectra obtained for representative compound was recorded in chloroform in the range 200-400nm. The UV absorption due to COO group appears at 274±6 and 294±2. On the basis of IR, NMR and UV spectral analysis data, it may be concluded that the present study behaves as a monodentate ligand.

Antibacterial activity

The compounds show higher to moderate activity against the bacterial strains. It was found that compound with water and lipid solubility is more effective. It generally form complexes with metaloenzymes, particularly those which responsible in basic physiology such as *cytochrome oxidase*. The compound may react with peptidoglycan layer of bacterial cell wall and damage it by penetrating in such a manner that the phenyl ring gets entered inside the cell by puncturing it followed by death of bacterial cell. Sometimes these compounds in low concentration may cause bacteriostatic condition by slow down the growth of bacteria.

Antifungal Activity:

The activity of compound was found variable at 50□g/ml concentration but at higher concentration compound show moderate to high activity against fungal strains. Presence of nitrogen, phenyl ring along with metal in +3 oxidation state are considered for fungal activity. The role of ligand was also commendable. These compounds generally damage the fungal strains by puncturing the cell wall similarly as in the case of bacteria.

Table: 1. Physico Chemical Properties of *p*-fluorophenyl derivatives of Group-15 Elements (As, Sb, Bi)

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Table: 2. Antibacterial Activity of compounds

S.N.	Compounds	Control Formula Weight	*Pseudomonas aeruginosa* Molecular Weight	M.P. (^{0}C)	*Staphylococcus aureus* Elemental Analysis C%		*Klebsiela pneumoniae*	
1	$C_8H_8ClNO_2FAs$	–	+++	+++	+++	H%	+++	N%
2	$C_8H_8ClNO_2FSb$	263.5	264.0	+++	124	36.43	3.03	+++ 5.31
3	$C_8H_8ClNO_2FBi$	310.5	310.0	+++	118	30.91	2.57	+++ 4.50
3	$C_8H_8ClNO_2FBi$	397.5	398.0		112	24.15	2.01	3.52

Table: 3. Antifungal Activity of compounds at 50µg/ml conc

S.N.	Compounds	*Aspergillus flavus* Col. Dia. (mm)	% Inhibition	*Aspergillus niger* Col. Dia. (mm)	% Inhibition
1	$C_8H_8ClNO_2FAs$	0.7	76.6	0.4	80.0
2	$C_8H_8ClNO_2FSb$	0.8	73.3	0.8	60.0
3	$C_8H_8ClNO_2FBi$	0.8	73.3	0.8	60.0
4	Control	3.0	-	2.0	-

Table: 4. Antifungal Activity of compounds at 100µg/ml conc

S.N.	Compounds	*Aspergillus flavus* Col. Dia. (mm)	% Inhibition	*Aspergillus niger* Col. Dia.(mm)	% Inhibition
1	$C_8H_8ClNO_2FAs$	0.2	93.3	0.4	80.0
2	$C_8H_8ClNO_2FSb$	0.1	96.7	0.2	90.0
3	$C_8H_8ClNO_2FBi$	0.1	96.7	0.1	95.0
4	Control	3.0	--	2.0	--

Conclusion:

The newly synthesized p-fluorophenyl derivatives of group-15 elements (As, Sb, Bi) are novel having pyramidal geometry. They show prominent antimicrobial activity against pathogenic bacterial and fungal strains and shows prominent efficacy against antimicrobial resistance.

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