Studies on Organ Antimony Compounds

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ABSTRACT:

Organometallic compounds can be defined as any member of a class of substance containing at least one metal to carbon bond in which carbon is a part of organic group. In these compounds metal act as main group compound or transitional metal compound. The main group metals of organometallic compounds are typically considered to be those of S block (gp1 and gp2) and heavier elements of p block (gp 13, 14, 15) respectively of the periodic table.

The properties of organometallic compounds depend upon the type of Carbon-metal bonds involved. Some are ordinary covalent bond (in which pairs of electron is shared between atoms). Some are ionic bond (in which pair of bonded electron is donated by only one atom) making the bond polarized where one bond is more negative that the other. Thus in organometallic compounds the metal atom is less electro negative than the carbon atom [1]. The polarity of M-C bond makes the organometallic compound a good substrate for studying electrophillic substitution at the metal atom. Due to this polarity, Organometallic compounds has a nature to get attached by wide variety of reagents, which opens a vast area for the synthesis of various type of new organometallic compounds.

INTRODUCTION:

Generally Organometallic like compounds exist in solid state, especially the compounds in which the hydro-carbon groups are aromatic or have a ring structure. From past studies it has been observed that organometallic chemstry plays important role in other branch of chemistry such as biological, analytical and medicinal chemistry, as organometallic compounds are found to be toxic to human body.

The biological aspects of organometallic compounds especially arsenic [2] and antimony [3] compounds were used extensively in medicines till today. A comprehensive structure-activity relationship on the reported potential bioacitve organotin compounds reveal that the activity of an organometallic compounds is highly affected by; the nature of ligands, presence of fluoro substituents for hydrophilic and lyphophilic character and the hydroytic stability of metal-carbon bond [4]

Morever, recent studies have shown that Antimony compounds especially containing thiolates and Nitrogen containing derivatives are better antifungal and antitumor agents respectively [5,6,7]. Looking deeper we recently found that fluoro based organoantimony compounds are biological very active [8]

The present investigation deals with synthesis of several organoantimony (III) compounds of R_2SbX (R=aryl group) and (X= succinimide and pthalimide) respectively. These compounds were found to be potentially active against cell lines and human pathogenic fungal and bacterial strains.

EXPERIMENTS:

The synthesis of organoantimony (III) amides were carried out by the reported method [9] The diarylorgano antimony (III) chloride R_2SbCl was prepared by redistribution reactions of R_3Sb and $SbCl_3$ in heat and crystallized in dichloromethane solution in inert atmosphere.

$$2R_3Sb + SbCl_3 \xrightarrow{\text{Heat}} 3R_2SbCl$$

R= (C_6H_5, C_6F_5, C_6H_4F)

The molecular weights were determined cryoscopically in benzene solution. The IR spectra of new organoantimony compounds were recorded in perkin – Elmer spectophotometer in 4000 - 4200 cm^{-1} range. The NMR spectral and structural determination was done by normal computation programming.

Preperation of organoantimony (III) Amides.

1. **Reaction of (C_6F_5) SbCl with succinimide**
   In an inert atmosphere, a solution of bis (pentaflurophenyl) antimony (III) chloride (1mmol) in benzene solution and succinimide (0.099g; 1mmol) were stirred together in presence of triethyl amine at room temperature for 6 hr. $Et_3N.HCl$ was formed and filtered off. The filterate on evaporation in vacuum gave white colour crystalline solid. Which was resrystallized from pet ehter to give bis (penta flurophenyl)
   antimony III succinimide (40-60).

2. **Reaction of $(C_6F_5)_2$ Sbcl with pthalimide**
   In an inert atmosphere, a solution of bis (pentaflurophenyl) antimony (III) chloride (1mmol) in benzene solution and succinimide (0.099g; 1mmol) were stirred together in presence of triethyl amine at room temperature for 6 hr. $Et_3N.HCl$ was formed and filtered off. The filterate on evaporation in vacuum gave white colour crystalline solid. Which was resrystallized from pet ehter to give bis (penta flurophenyl) antimony (III) succinimide (40-60).

3. **Reaction of $(C_6H_5)_2$ SbCl with succinimide**
   In an inert atmosphere, a solution of bis (pentaflurophenyl) antimony (III) chloride (1mmol) in benzene solution and succinimide (0.099g; 1mmol) were stirred together in presence of triethyl amine at room temperature for 6 hr. $Et_3N.HCl$ was formed and filtered off. The filtrate on evaporation in vacuum gave white colour crystalline solid. Which was recrystallized from pet ether to give bis (penta flurophenyl)
   Antimony (III) succinimide (50 - 80).

4. **Reaction of $(C_6H_5)_2$ SbCl with pthalimide.**
   In an inert atmosphere, a solution of bis (pentaflurophenyl) antimony (III) chloride (1mmol) in benzene solution and pthalimide (0.099g; 1mmol) were stirred together in presence of triethyl

amine at room temperature for 6 hr. $Et_3N.HCl$ was formed and filtered off. The filtrate on evaporation in vacuum gave white colour crystalline solid. Which was recrystallized from pet ether to give bis (penta flurophenyl)
antimony (III) pthalimide (60-80).

5. **Reaction of $(C_6H_4F)_2SbCl$ with succinimide.**

In an inert atmosphere, a solution of bis (pentaflurophenyl) antimony (III) chloride (1mmol) in toulene solution and succinimide (0.099g; 1mmol) were stirred together in presence of triethyl amine at room temperature for 6 hr. $Et_3N.HCl$ was formed and filtered off. The filtrate on evaporation in vacuum gave white colour crystalline solid. Which was recrystallized from pet ether to give bis(penta flurophenyl)
Antimony (III) succinimide (40-60).

Results and discussion:

Physicochemical properties of organoantimony compounds.

S.No.	Molecular Formula	Elemental Analysis			Infra Real cm^{-1}		
		C%	H%	N%	Vasy(CO)	Vsym[CO]	Colour
1	$C_{16}H_{14}NO_2Sb$	50.27	3.29	3.29	1700	1300	White
2	$C_{20}H_{14}NO_2Sb$	55.00	3.12	3.12	1741	1300	White
3	$C_{16}F_2H_{12}NO_2Sb$	46.44	2.83	3.22	1725	1329	White
4	$C_{20}F_2H_{12}NO_2Sb$	51.82	2.50	3.00	1720	1320	White
5	$C_{16}F_{16}H_4NO_2Sb$	34.21	0.71	2.45	1730	1330	White
6	$C_{20}F_{10}H_4NO_2Sb$	40.00	0.59	2.15	1729	1330	Red

Table 1

Analytical Data of organoantimony compounds.

S.No.	Formula	Melting Point		Solvent of Crystallization
		C%	Yield%	Pet ether
1	$C_{16}H_{14}NO_2Sb$	150	79	40-60
2	$C_{20}H_{14}NO_2Sb$	115	84	40-50
3	$C_{16}F_2H1_2NO_2Sb$	120	62	50-80
4	$C_{20}F_2H_{12}NO_2Sb$	110	68	60-80
5	$C_{16}F_{10}H_4NO_2Sb$	60	70	40-60

Table 2

The reaction is given below.

$$R_2SbCl + HL \xrightarrow{Et_3N} R_2SbL + Et_3N.Hcl$$

R= (C_6H_5), (C_6F_5), (C_6H_4F)

HL= succinimide, pthalimide

IR SPECTRA:

The IR spectra of all the synthesized compounds show absorption bands due to the presence of pentaflurophenyl group. The absorption frequencies are fully assigned. Sb-C vibration in case of pentaflurophenyl derivative appear in the range of 440 – 468 cm^{-1} (Wadell 1982) V(Sb-N) band cannot be assigned with certainty due to the complex nature of the spectra. The absorption frequencies in amido derivatives, bearing carboxyl group have been assigned as Vasy(CO) and Vsym(CO). The appearance of a strong band ranging between 1700-1730 cm^{-1} which clearly suggests the presence of ester type CO group.

Biological importance of organoatimony compounds.

The discovery of a synthetic arsenical, "salvarsan" in 1910 found as an effective medicines against syphilis, led to an extensive investigation on the synthesis and biological studies of organoarsenic compounds [11]. Later on in some reviews and books it was published that organometallic compounds of group 15 elements show higher activity against bacterial, fungal and viral strains of micro organism [12].

The organoantimony compounds also played important role against microorganism. They also proved effective against infections caused by Trypasnosomas and leishmania organism. It was found that the organoantimony (III) derivatives show important anti bacterial and antifungal activities which have been studied by some workers.

ORGANOATIMONY (III) COMPOUNDS AS ANTI-TUMOUR

It was found that the cyclophosphamide is an alkylating agent, which possess significant antitumour activity in selected malignant neoplastic cells (13). However, its 1:1 adduct formed with SbCl3 showed no activity against L-1210 leukemia and Ehrlich ascites tumour (14). The other metal coordination compounds containing cyclophosphamide were also found incactive. A series of organoantimony (III) compounds with polydentate carboxylate has also been investigated for their antitumour potentiality.

Although, the full details are yet to be published, preliminary, results have indicated that coordination of antimony (III) by these ligands results in compounds of greater potency than that displayed by the uncoordinated ligands, indicating the importance of the presence of antimony (III) for the activity. The metals ions investigated with ligands of such types, displayed better biological activity. By the use of colorimetric methods, the cytotoxicity of organoantimony (III) compounds were examined in human promylecytic leukemia HL-60 cell line. After 24 hours exposure at conc of 100, 10, and 1 mg/ml, inhibition of cell growth was 100, 70 and 18% respectively. The antitumour activity against mice inoculated with S-180 solid tumours showed a reduction in tumour weight to 74% of the control values after 9 days lapsed. It was noticed that the mentioned antimony (III) compounds have Sb-O and/or Sb-N bonds, but the next class of compound described have Sb-S bounds.

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