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Synthesis, Characteristic Spectral Studies and in Vitro Antibacterial Activity of a Novel Stannane

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Abstract—*Stannanes are chemical compounds having tin bonded to some hydrocarbon substituents. These complexes fall under the category of organometallics. Stannanes can have various biological applications when the ligand used itself has antiseptic and disinfectant characteristics. In the present work, 1,3-dihydroxybenzene abbreviated as RCL by the common name Resorcinol would undergo complex formation with Dibutyltin oxide by the azeotropic removal of water molecule using benzene-ethanol medium in completely dried reaction system. The complex thus synthesized was characterized by various spectroscopic techniques. The Ultraviolet-visible spectra depicted a bathochromic shift from the electronic transitions in ligand due to complex formation. The FT-IR spectra have shown the expected functional groups peaks for Sn-O bond formation as well as other relevant peaks. H^1NMR and C^{13}NMR were recorded which duly confirms the successful synthesis of complex by the above quoted method. Powder XRD is one of the most important characterization tool and thus XRD pattern was also recorded for the solid orange colored complex. Since each crystalline solid has its unique XRD characteristic for its identification and thus we can determine the size and shape of unit cell for any compound or complex. The light orange colored complex thus obtained was screened for in vitro antibacterial activity against both gram-positive & gram-negative bacteria and found to be more biologically active than ligand. Stannanes inhibited the growth of microorganisms, it has been assumed that the production of an enzyme is being affected hence the organisms were less able to metabolize nutrients and consequently growth is ceased. The Complex exhibited a good antibacterial and antifungal potency higher than that of the corresponding ligand but lower than that of the parent stannane, hence can be potential anti-biofouling agents, bactericides or fungicides.*

1. INTRODUCTION

Stannanes are chemical compounds containing tin bonded to the hydrocarbons. These complexes fall under the category of organometallics. Stannanes are generally classified according to their oxidation states as they exists in various stable oxidation states.Tin(IV) complexes are much more useful and effective as stabilizers ,industrial biocides, wood preservative, antifouling agents etc. The entire series $R_{4-n}SnCl_n$ are known for many R groups and values of *n* up to 4. The tetragonal derivatives are invariably tetrahedral. The parent member of the series, stannane (SnH_4), is an unstable colorless gas. The main class of stannanesare diorganotindithiolates with the formula $R_2Sn(SR')_2$. The Sn-S bond is the reactivee component. Diorganotin carboxylates, e.g., dibutyltindilaurate, are used as catalysts for the formation of polyurethanes, for vulcanization of silicones, and transesterification. "Tributyltins" are used as industrial biocides, e.g. as antifungal agents in textiles and paper, wood pulp and paper mill systems, breweries, and industrial cooling systems.

2. EXPERIMENTAL WORK

2.1 Materials and methods

All the chemicals were of Analytical Grade and obtained from commercial sources(Merck specialitics,Spectrochemand Fischer scientific). Solvents used for the synthesis were dried and purified by standard procedures.The UV visible spectra studies were measured usingUV instrument **SHIMADZU UV 1800** ,200 -600 in ethanol. The ^{1}H NMR spectra (in DMSO – d6 solution) were recorded on a **JEOL ECX-400P NMR** at 400 MHz and 100 MHz, respectively at USIC, TMS was used as internal standard. The FT-IR studies were done by using **PERKIN ELMER SPECTRUM VERSION 10.03.06.**

Synthesis of Dibutyltin(IV) Complex–3,3' ((dibutylstannanediyl) bis (oxy)bis(1-hydroxybenzene)) ($C_{20}H_{28}O_4$): A stoichiometric amount of ligand (0.22g, 2mmol) was dissolved in a mixture of dry benzene (60 ml) and absolute ethanol (20 ml) and dibutyltin(IV) oxide (0.249 g,1mmol) as required for 1:2 (metal:ligand) molar ratio was added. The reaction mixture was then refluxed azeotropically with a Dean Stark separator over the heating mantle. Dibutyltin (IV) oxide goes in to solution within 20-25 minutes to give a clear solution. Refluxing was further continued for 5-6 hours and the contents were then cooled. Excess of solvent was removed under reduced pressure by a rotator evaporator to leave behind a solid complex. The solid was filtered and washed with chloroform and dried in vacuum. Recrystallisation was done from ethanol. The general reaction sequence is given as follows

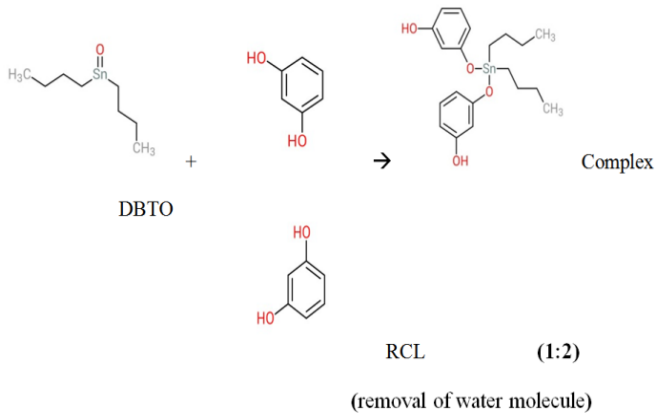


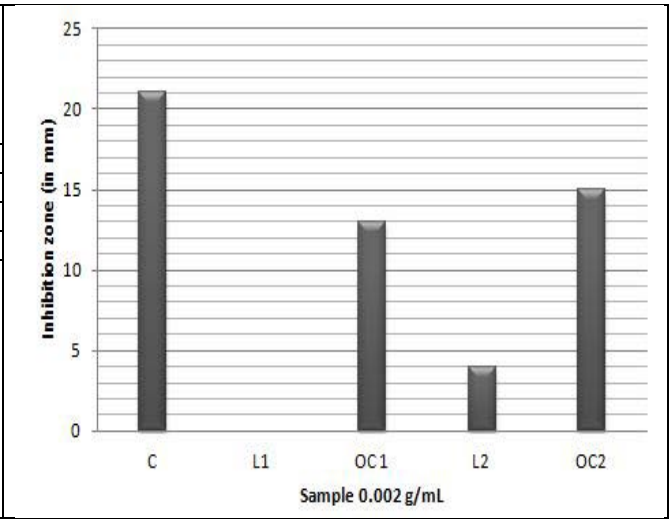
Fig. 1: Reaction pathway for the formation of Organotin(IV) complex

2.2 BIOLOGICAL ACTIVITYAntibacterial Bioassay (*in vitro*)

The synthesized complex was screened in vitro for its antibacterial activity against Gram-negative (*E coli*) and Gram-positive (*S aureus*) bacterial strains using **Disc Diffusion Method**.

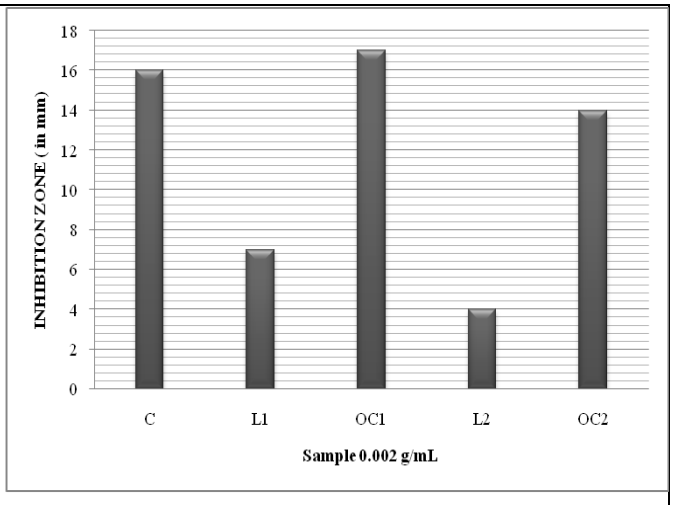
BACTERIA - Escherichia coli

SAMPLE 0.002 g/100 mL	DIAMETER OF INHIBITION ZONE (in mm)	INTERPRETATION
C	21	Active
L1	-	Nil
OC 1	13	Moderate
L2	4	Weak
OC2	15	Moderate



BACTERIA-Staphylococcus aureus

SAMPLE 0.002 g/100 mL	DIAMETER OF INHIBITION ZONE (in mm)	INTERPRETATION
C	16	Moderate
L1	7	Weak
OC1	17	Active
L2	4	Weak
OC2	14	Moderate



3. RESULTS AND DISCUSSION

3.1 ELECTRONIC SPECTRA

The electronic spectra analyses of ligands and its organotin(IV) complexes were carried out in ethanol at room temperature .The Ligand showed band at λ_{max}276.00 nm,205.00 nm; Absorbance0.256119, 2.089767 which is assigned to π-π* transition. After complexation, the λ_{max} value of the organotin complex was shifted to λ_{max}282nm ,276.00 nm ;Absorbance 0.005875 .This is due to the coordination of ligand to the DBTO. New peaks in the range λ_{max} 282.00 nm, 276.00 nm (**Redshift)** in the Organotin complex is attributed to the n-π* transition of band which is referred to the ligand metal charge transfer(LCMT).

3.2 ^{1}HNMR SPECTRA

The ^{1}H NMR spectra (in DMSO –d^{6}solution) of Organotin(IV)complexes were recorded.

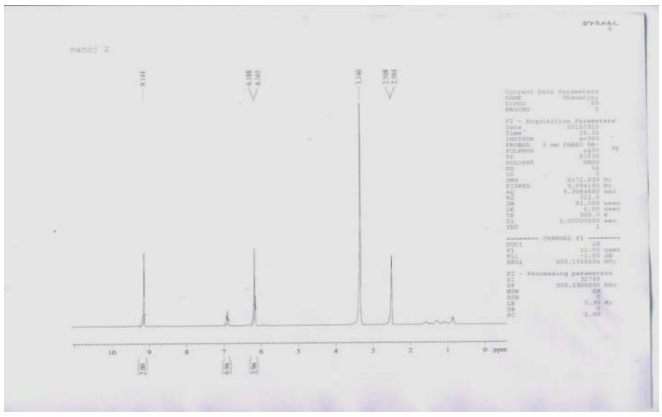


Fig. 2: ^{1}HNMR Spectra of TC1

3.3 ^{13}CNMR SPECTRA

The ^{13}CNMR spectra (in DMSO-d^{6} solution) of Organotn(IV) complexes were recorded.

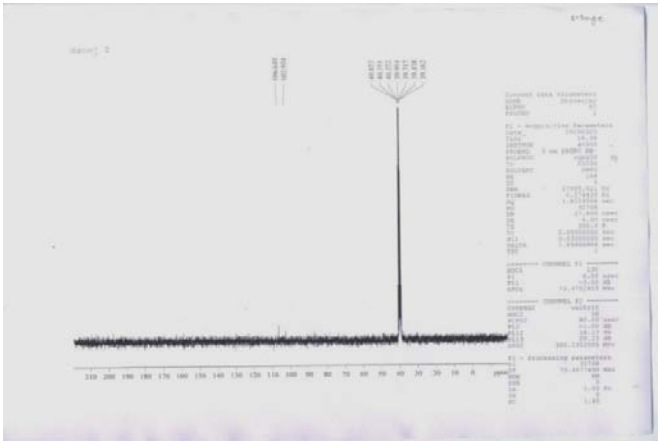


Fig. 3: ^{13}CNMR Spectra of TC1

3.4 FT-IR STUDIES

The FT-IR spectra have shown the expected functional groups peaks for new Sn-O bond formation as well as other relevant peaks: Alkyl C-H stretch : 2957-2857cm^{-1}, Aromatic C=C bond : ~1586 cm^{-1},C-O stretch : ~1150 cm^{-1}, Sn-O bond : ~460 cm^{-1}, Aromatic C-H stretch : ~680-773 cm^{-1}

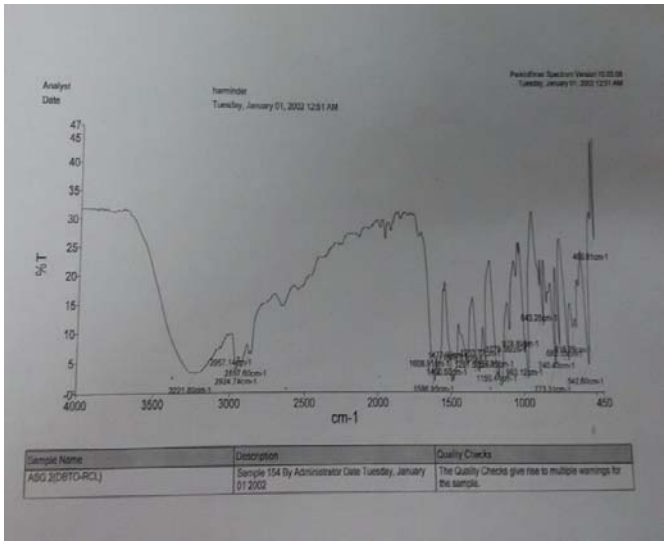


Fig. 4: FT-IR peaks for TC1

3.5 X-RAY DIFFRACTION

X-ray diffraction has been in use in two main areas, for the fingerprint characterization of crystalline materials and the determination of their structure. Each crystalline solid has its unique characteristic X-ray powder patternwhich may be used as a "fingerprint" for its identification. X-ray diffraction provides most definitive structural information & Interatomic distances and bond angles.**Theta value** is found to be 18 **(**For TC1**)**

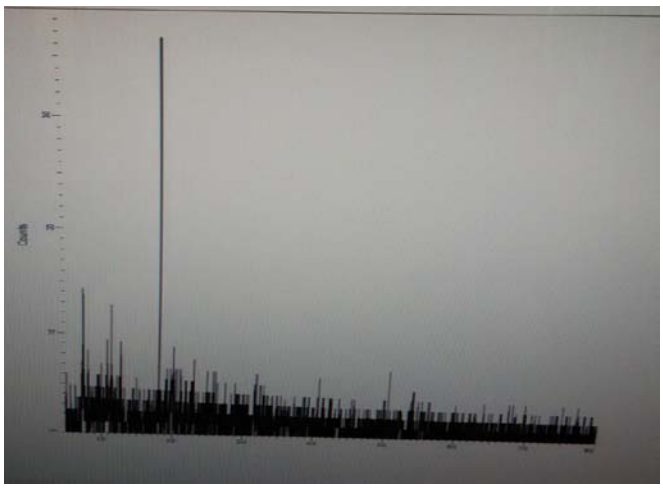


Fig. 5: XRD peak of TC1

4. CONCLUSION

Stannanes inhibited the growth of microorganisms, it has been assumed that the production of an enzyme is being affected hence the organisms were less able to metabolize nutrients and consequently growth is ceased. The Complex exhibited a good antibacterial and antifungal potency higher than that of the corresponding ligand but lower than that of the parent stannane, hence can be potential anti-biofouling agents, bactericides or fungicides.

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