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SYNTHESIS, CHARACTERIZATION, AND BIOLOGICAL ACTIVITY OF ORGANOTIN(IV) COMPLEXES DERIVED FROM N-PHTHALOYLAMINE LIGANDS

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ABSTRACT

N-Phthaloylamine derivatives were synthesized through the condensation of an equimolar amount of phthalic anhydride with 2,4-dinitrophenylhydrazine, aniline, and hydrazine (1:1 molar ratio) in glacial acetic acid using excess tetrahydrofuran (THF) as solvent. The reactions afforded the corresponding ligands in good yields (60–68.6%) after purification by column chromatography and recrystallization. The synthesized ligands were subsequently complexed with organotin(IV) compound, including dibutyltin(IV) dichloride, to afford stable organotin(IV) complexes. The compounds were characterized by melting point determination, thin-layer chromatography (TLC), column chromatography, and Fourier Transform Infrared (FT-IR) spectroscopy. The IR spectra of the free ligands exhibited characteristic imide carbonyl bands in the 1715–1730 cm^{-1} region and N–H stretching bands at 3200–3300 cm^{-1}. Upon complexation, the carbonyl stretching frequencies shifted to lower wavenumbers (1680–1700 cm^{-1}), indicating coordination through the carbonyl oxygen atom. The appearance of new bands in the 450–550 cm^{-1} region, assignable to Sn–O and Sn–N vibrations, supports ligand coordination. Changes in the N–H region suggest possible involvement of the amide nitrogen in bonding, suggesting coordination through oxygen donor atoms. Antimicrobial screening against *Klebsiella pneumoniae*, *Escherichia coli*, and *Staphylococcus aureus* revealed enhanced activity of the complexes compared to the free ligands. The ligands exhibited inhibition zones of 12–18 mm, whereas the organotin(IV) complexes showed larger zones of 16–22 mm, with maximum activity observed against *Klebsiella pneumoniae*. These findings demonstrate that complexation with organotin(IV) significantly enhances the antimicrobial efficacy of the ligands.

Keywords: Ligand, N-phthaloylamine, Dibutyltin(IV) dichloride, Infrared spectroscopy, Antimicrobial activity

INTRODUCTION

Organotin(IV) compounds continue to attract significant attention because of their diverse structural chemistry and broad spectrum of biological and industrial applications. Their ability to adopt various coordination numbers (four to seven) and geometries enables the formation of stable complexes with oxygen-, nitrogen-, and sulfur-donor ligands. Biologically, organotin(IV) derivatives have demonstrated antiviral, antibacterial, antifungal, antiparasitic, and antitumor activities (Awang & Baba, 2012; Yang et al., 2009; Iornumbe et al., 2021; Javed et al., 2023). Gwaram et al. (2021) successfully synthesized a Schiff base ligand and its corresponding Tin (IV) complex using an eco-friendly "green" grinding method, which yielded high-purity products with significant percentage yields. Analytical characterization and antimicrobial testing confirmed that the complex coordinated through the azomethine nitrogen and phenolic oxygen, exhibiting superior antibacterial and antifungal activity against various pathogens compared to the free ligand.

In particular, di- and triorganotin compounds have shown promising cytotoxic effects against several cancer cell lines, often attributed to their capacity to interact with DNA, proteins, and mitochondrial pathways. Mechanistic studies suggest that the biological activity of organotin compounds is strongly influenced by their lipophilicity, which enhances membrane penetration and facilitates intracellular accumulation. However, concerns regarding organotin toxicity—especially their persistence and environmental impact following applications such as antifouling agents—have prompted efforts to design complexes with improved selectivity and controlled biological profiles.

The biological properties of organotin(IV) carboxylates are closely related to their coordination environment and ligand framework. Carboxylate ligands can coordinate in monodentate, bidentate, or bridging modes, which significantly affects their stability and bioavailability. Several studies have shown that chelation enhances biological activity by increasing complex stability and facilitating transport across biological membranes. For example, organotin complexes containing heterocyclic and Schiff base ligands, including thiosemicarbazones (Salam et al., 2012), have demonstrated enhanced antimicrobial and anticancer activities compared to their parent ligands. These findings highlight the importance of rational ligand design in modulating organotin bioactivity.

Experimental

All reagents and solvents were of analytical grade and used without further purification. Phthalic anhydride, 2,4-dinitrophenylhydrazine, aniline, hydrazine hydrate, and dibutyltin(IV) dichloride (Bu_2SnCl_2) were commercially obtained. Tetrahydrofuran (THF), methanol, ethanol, chloroform, ethyl acetate, diethyl ether, acetone, and n-hexane were used as supplied. Melting points were determined using a Stuart SMP3 melting point apparatus and are uncorrected. Infrared (FT-IR) spectra were recorded on a PerkinElmer Spectrum GX FT-IR spectrometer (4000–400 cm^{-1}) using KBr pellets. UV–Visible spectra were recorded in methanol (200–800 nm) to monitor ligand-to-metal charge transfer transitions. Elemental analyses (C, H, N) were performed using a CHN analyzer to confirm purity and stoichiometry. Thin-layer chromatography (TLC) was carried out on silica gel 60 F254 plates using ethyl acetate:n-hexane

(3:2) as eluent unless otherwise stated. Rf values were recorded consistently for all compounds.

Organotin(IV) compounds are toxic and potentially bioaccumulative. All syntheses were carried out in a fume hood using gloves and protective eyewear. Waste containing organotin residues was collected separately and disposed of according to hazardous chemical regulations (Priyanka et al., 2020).

Synthesis of the Ligands

Synthesis of N-2,4-dinitrophenylphthalamic acid ($C_{14}H_{10}N_4O_6$)

The ligands and their complexes were synthesized based on previous methods (Graisa et al., 2009; Dolan et al., 2014; Javed et al., 2023). Phthalic anhydride (5 mmol, 0.741 g) was dissolved in 15 mL of glacial acetic acid. 2,4-Dinitrophenylhydrazine (5 mmol, 0.991 g) was dissolved separately in 15 mL of glacial acetic acid. The two solutions were combined in a 250 mL round-bottom flask and stirred magnetically for 24 h at room temperature. Excess tetrahydrofuran (THF) was added as co-solvent. The reaction mixture was concentrated using a rotary evaporator and allowed to crystallize. The product was filtered, washed with n-hexane, and dried.

Yield: 67.5%; **Melting point:** 188–190 °C; **Colour:** Golden yellow; **Rf:** 0.54; **FT-IR (KBr, cm^{-1}):**
ν(O–H) 3649, ν(N–H) 3265, ν(C=O)acid 1737, ν(C=O)amide 1606, ν(NO_2) 1508

Synthesis of N-phenylphthalamic Acid ($C_{14}H_{11}NO_3$)

Phthalic anhydride (14 mmol, 2.07 g) and aniline (14 mmol, 1.30 g) were dissolved separately in 15 mL glacial acetic acid and combined. The mixture was stirred for 24 h, concentrated, and crystallized. The synthesized ligand was found to be soluble in ethyl acetate, diethyl ether, tetrahyrofuran and chloroform.

Yield: 68.6%; **Melting point:** 209–211 °C; **Rf:** 0.51; **FT-IR (KBr, cm^{-1}):** ν(N–H) 3468, ν(C=O)acid 1703, ν(C=O)amide 1699

Synthesis of N-aminophthalamic Acid ($C_8H_8N_2O_3$)

Phthalic anhydride (14 mmol, 2.07 g) and 14 mmol (0.7 mL) of hydrazine were dissolved in 15 mL glacial acetic acid. The mixture was poured into a 250 mL round-bottom flask with addition of excess tetrahydrofuran as solvent. The procedure used in 2.1.1 above was also followed.

The synthesized ligand was found to be soluble in Tetrahydrofuran, ethyl acetate, diethyl ether, ethanol, methanol, and acetone.

Yield 60.1%; Melting point 206 – 208 ^oC; Colour: light yellow; Appearance: Crystalline. FT-IR (KBr, cm^{-1})ν_{max}: ν(N-H) 3186, ν(N–N) 1006, ν(C-C) 1489, ν(C=O) acid 1701, ν(C=O) amide 1654.

Synthesis of organotin(IV) Complexes

Synthesis of $C_{14}H_9N_4O_6SnBu_2Cl_2$

The same procedure was used in the synthesis of the complexes, as reported in the literature (Affan et al., 2010; Shahzadi et al., 2010). 5 mmol (0.346 g) of the ligand (N-phthaloylamine) and 0.303 g of the (complex) dibutyltindichloride were measured and each dissolved in 15 mL of Tetrahydrofuran. The resulting solutions were poured into a 250 mL round bottom flask and was refluxed at a temperature of 70 ^0C for 3 hours in an oil bath. The colour change from light yellow to deep brown was observed. The solution was removed from the stirrer, filtered under gravity and left for a few days to crystallize. The dried product was recrystallized in ethanol, purified by thin-layer chromatography and column chromatography.

The synthesized complex was found to be soluble in methanol, ethanol, chloroform, ethyl acetate, and tetrahydrofuran. Yield: 67.5%, Colour: Golden Yellow, Appearance: Sticky solid crystal, R_f: 0.66. FT-IR (KBr, cm^{-1})ν_{max}: ν(C=O)amide 1645, ν(C=C) 1585, ν(C–C) 1498, ν(Sn–O) 465, ν(Sn–c) 557.

Synthesis of Organotin (IV) Complex of N-phenyl phthalamic Acid

Each of 1 mmole (0.241 g) of the ligand (N-phenylphthalamic acid) and 1mmole (0.304 g) of dibutyltin (IV) dichloride was dissolved in 10 mL THF respectively. The complex was then prepared using the same procedure for 2.1.1 above. The synthesized complex was found to be soluble in ethyl acetate, tetrahydrofuran, and acetone.

Yield was 58.9%, Melting point 209-210 ^oC, colour: white, Appearance: Tiny crystals.

FT-IR (KBr, cm^{-1})ν_{max}: ν(N-H) 3367, ν(C=C) 1595, ν(C-C) 1496, ν(C=O) acid, ν(C=O) 1735, ν(C-N) 1219, ν(C-O) 1116, ν(Sn-O) 457, ν(Sn-C) 530.

Synthesis of Organotin(IV) Complex of N-amino phthalamic Acid

An equimolar amount of the 1 mmol (0.18 g) of the ligand and 1 mmol (0.304 g) of dibutyltin (IV) dichloride as dissolved in 10 mL tetrahydrofuran. The same procedure for 2.1.1 above was followed in the synthesis. The synthesized complex was found to be soluble in methanol, ethanol, acetone and tetrahydrofuran.

Yield: 77.5%; Melting point: 198–200 ^oC; Colour: brownish yellow; Appearance: crystalline. FT-IR (KBr, cm^{-1})ν_{max}: ν(N-H) 3167, ν(C-H) 2960, ν(C=O) acid 1716, ν(C-C) 1489, , ν(C-N) 1263, ν(C-O) 1082, ν(Sn-O) 493, ν(Sn-C) 563.

RESULTS AND DISCUSSION

Infrared Spectra Data

The FT-IR spectra of the free ligands and their corresponding organotin(IV) complexes were recorded in the range 4000–400 cm^{-1}. Significant changes in characteristic absorption bands were observed upon complex formation, indicating coordination of the ligands to the tin center. The broad ν(O–H) stretching vibration of the free ligand appeared around 3095 cm^{-1}. In the complexes, this band either shifted or decreased in intensity, suggesting involvement of hydrogen bonding and/or deprotonation prior to coordination. The aliphatic ν(C–H) stretching vibrations were observed near 2958 cm^{-1}.

The carbonyl stretching vibration of the carboxylic group, ν(C=O), appeared at 1732 cm^{-1} in the free ligand and shifted slightly to 1735 cm^{-1} in the complex. The amide carbonyl stretching vibration was observed at 1712 cm^{-1} in the free ligand and shifted to lower frequencies (1661–1647 cm^{-1}) in the complexes, indicating coordination through the carbonyl oxygen. The ν(C–N) stretching vibration appeared at 1284–1286 cm^{-1} and exhibited slight shifts upon complexation, suggesting possible involvement of the amidic nitrogen in bonding. The aromatic ν(C=C) stretching vibration was observed at 1595 cm^{-1}, while ν(C–C) vibrations appeared near 1454 cm^{-1}. These values are comparable with literature reports (1555–1484 cm^{-1} range).

The ν(C–O) stretching vibration of the carboxylic group shifted from 1172 cm^{-1} in the ligand to 1184 cm^{-1} in the complex, consistent with deprotonation and coordination through oxygen. Similarly, Carboxylate ν(C–O) shifts suggest possible coordination through oxygen donor atoms.. New

bands appeared in the far-IR region at 451 cm^{-1} assigned to ν(Sn–O), and 567 cm^{-1} assigned to ν(Sn–C). These bands were absent in the free ligands and confirm the formation of organotin(IV) complexes. Notably, the ν(Sn–Cl) band expected around 350–330 cm^{-1} was absent in the spectra of the final products, supporting ligand substitution and successful complex formation.

The result of Biological Activity

The antibacterial activities of the synthesized ligand and its organotin(IV) complex were evaluated using the agar well diffusion method as previously described (Shah et al., 2010; Salam et al., 2012; Jimaa, 2021). Test organisms included are *Escherichia coli, Staphylococcus aureus, Klebsiella pneumoniae, Bacillus subtilis, and Pseudomonas aeruginosa.* Seven grams of nutrient agar were dissolved in 250 mL of distilled water and heated until completely homogenized. The medium was sterilized by autoclaving at **121 °C for 15 minutes.** The sterilized agar was poured into sterile Petri dishes and allowed to solidify under aseptic conditions. Sterile cotton swabs were dipped into standardized broth cultures (adjusted to 0.5 McFarland standard where applicable) and evenly spread across the agar surface. Wells were created using a sterile cork borer. The ligand and complex were prepared at concentrations of 50 ppm and 100 ppm. Each solution was introduced into separate wells using a micropipette. The plates were incubated at **37 °C for 24 hours.** Zones of inhibition were measured in millimeters (mm). The organotin(IV) complex exhibited higher antibacterial activity than the free ligand at both tested concentrations (50 and 100 ppm), as evidenced by larger zones of inhibition. The increase in activity was concentration-dependent, with 100 ppm generally producing greater inhibition. The observed enhancement in antibacterial activity upon complexation is consistent with previous reports on organotin(IV) complexes of carboxylate and heterocyclic ligands (Shah et al., 2010; Salam et al., 2012). Similar structure–activity relationships have been attributed to increased lipophilicity and metal-mediated biological interactions.

The inhibition growth in percentage was determined based on the average diameter of the bacterial colony on the growth medium compared to the respective control.

% inhibition = $\frac{(A - B) \times 100}{}$

A where A = Average diameter of growth organism in the control plate

B = Average diameter of organism in the test plate

Table 1: Antibacterial Activity Data for the Ligands and the Complexes

Ligand/Complex	Escherichia Coli		Staphylococcus aureus		Klebsiella pneumoniae		Bacillus subtilis		Psudonomas aureginosa	
	Zone of inhibition (mm)									
	50 Ppm	100 Ppm	50 Ppm	100 Ppm	50 Ppm	100 Ppm	50 Ppm	100 Ppm	50 Ppm	100 ppm
Acetone	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
$C_{14}H_{10}N_4O_6$	14	16	12	18	8	10	0	0	0	0
$C_{14}H_9N_4O_6SnBu_2Cl_2$	16	20	18	22	8	12	0	0	0	0
$C_{14}H_{11}NO_3$	10	15	16	19	18	22	19	22	-	-
$C_{14}H_{10}NO_3SnBu_2Cl_2$	13	17	23	26	25	27	20	24	-	-
$C_8H_8N_2O_3$	12	18	18	24	17	19	13	15	-	-
$C_8H_7N_2O_3SnBu_2Cl_2$	14	19	22	26	19	22	16	18	-	-

NB: NA means no activity and - means no clinical isolates for the bacteria specie.

Discussion

The observed spectral shifts confirm coordination of the ligand to the organotin(IV) center. The downward shift of the amide ν(C=O) band (1712 → 1661–1647 cm^{-1}) is characteristic of coordination through carbonyl oxygen due to reduction in bond order upon donation of electron density to tin. Similar trends have been reported for organotin(IV) carboxylate and amide complexes.

The shift in ν(C–O) and disappearance or weakening of the ν(O–H) band support deprotonation of the carboxylic/phenolic group prior to coordination, indicating formation of Sn–O bonds. The appearance of new bands at 451 cm^{-1} and 567 cm^{-1} confirms the formation of ν(Sn–O) and ν(Sn–C) bonds, respectively. These bands fall within the characteristic ranges reported for six-coordinate diorganotin(IV) complexes. The absence of ν(Sn–Cl) vibrations in the final complexes indicates successful substitution of chloride ligands by donor atoms of the ligand. Largely, the spectral evidence supports bidentate coordination via carbonyl oxygen and phenolic/carboxylate oxygen, possible secondary interaction through amidic nitrogen, and formation of a six-coordinate geometry around the Sn(IV) center.

CONCLUSION

The present study successfully reports the synthesis of novel organotin(IV) complexes derived from N-phthaloylamine ligands and their subsequent characterization using physicochemical and spectroscopic techniques. Infrared spectral analyses confirm that coordination occurs primarily through the carbonyl oxygen of the amide/carboxylate group, accompanied by deprotonation and formation of Sn–O bonds. The observed downward shift in ν(C=O) bands and the appearance of new ν(Sn–O) and ν(Sn–C) vibrations in the far-IR region are consistent with bidentate coordination behaviour and the formation of six-coordinate diorganotin(IV) species with a six-coordinate geometry around the Sn(IV) center. These findings agree with previously reported structural trends for organotin(IV) carboxylate and amide complexes (Shahzadi et al., 2010; Dolan et al., 2014; Hadi et al., 2019).

Biological evaluation revealed that the organotin(IV) complexes exhibit enhanced antibacterial activity compared to the free ligands against both Gram-positive and Gram-negative bacterial strains. The improved activity upon complexation can be attributed to chelation theory, whereby coordination reduces the polarity of the metal ion through partial sharing of its positive charge with donor atoms and increases lipophilicity, thereby enhancing membrane permeability and facilitating interaction with intracellular targets (Salam et al., 2012; Priyanka et al., 2020). Similar structure–activity relationships have been widely reported for diorganotin(IV) derivatives, where biological efficacy is strongly influenced by ligand environment and organotin

substituents (Javed et al., 2023; Adeyemi & Onwudiwe, 2020).

The enhanced antimicrobial performance of the synthesized organotin(IV) complexes suggests their potential as lead compounds for the development of new antibacterial agents, particularly in combating resistant bacterial strains. Beyond pharmaceutical prospects, organotin(IV) carboxylates are known to possess applications in materials science, including as PVC stabilizers, catalysts in esterification and transesterification reactions, and antimicrobial additives in coatings (Hadi et al., 2019; Priyanka et al., 2020). The incorporation of biologically active ligands such as N-phthaloylamines may therefore broaden the functional versatility of organotin systems, enabling dual roles in biomedical and industrial domains. In conclusion, the synthesized organotin(IV) complexes demonstrate promising structural integrity and enhanced antibacterial activity relative to their parent ligands. With further structural refinement and comprehensive biological evaluation, these compounds may serve as valuable scaffolds for future organometallic drug design and multifunctional industrial applications.

RECOMMENDATIONS

While the present work establishes structural and preliminary biological profiles, further investigations are required to fully elucidate the therapeutic potential and safety of these complexes. Future studies should include:

i. Detailed structural confirmation using multinuclear NMR spectroscopy (^{1}H, ^{13}C, ^{119}Sn) and single-crystal X-ray diffraction to unequivocally determine coordination geometry.
ii. Determination of minimum inhibitory concentration (MIC) and minimum bactericidal concentration (MBC) values for quantitative antimicrobial assessment.
iii. Cytotoxicity evaluation against mammalian cell lines to assess selectivity and therapeutic index.
iv. Mechanistic studies on DNA/protein binding interactions and membrane disruption pathways.
v. Investigation of structure–activity relationships (SAR) by varying organotin substituents (e.g., methyl, phenyl, butyl groups) to optimize biological performance while minimizing toxicity.

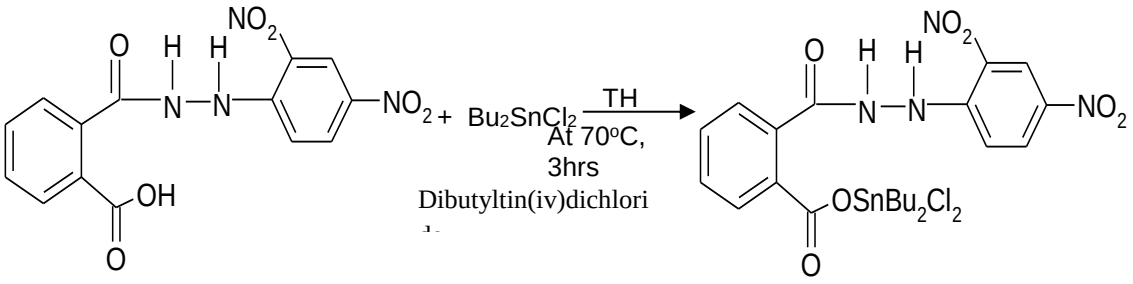
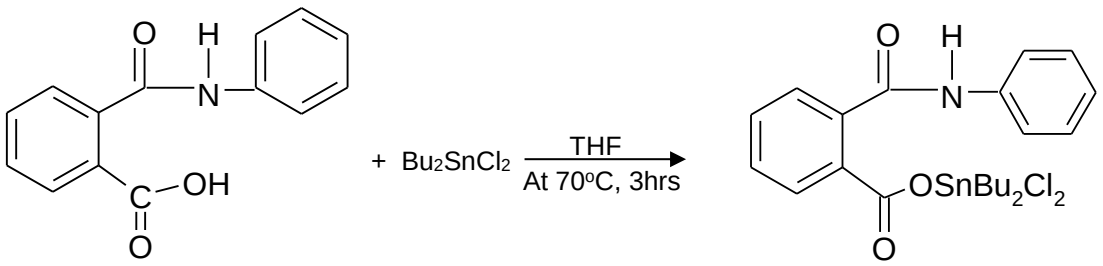


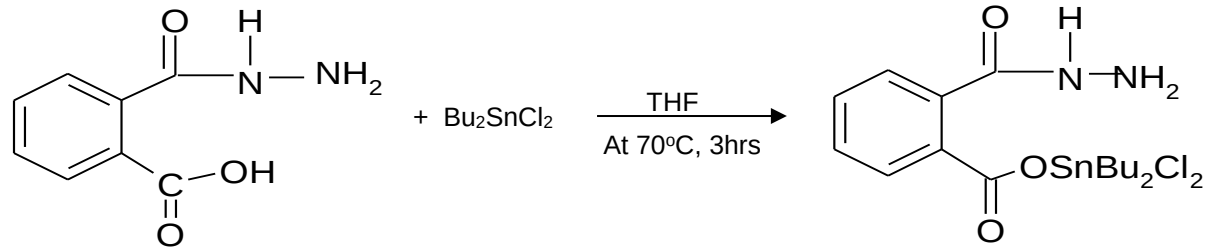
Figure 1: Synthesis Pathway of Organotin(IV) Complexes of N-2, 4-dinitrophenylanilidephthalamic Acid



N – Phenylphthalamic acid Dibutyltin(iv)dichloride

F

Figure 2: Synthesis Pathway of Organotin(IV) Complexes of N-phenylphthalamic Acid



N-Amino pthhalamic Acid Dibutyltin(iv)dichloride

Figure 3: Synthesis Pathway of Organotin(IV) Complexes of N-aminophthalamic Acid

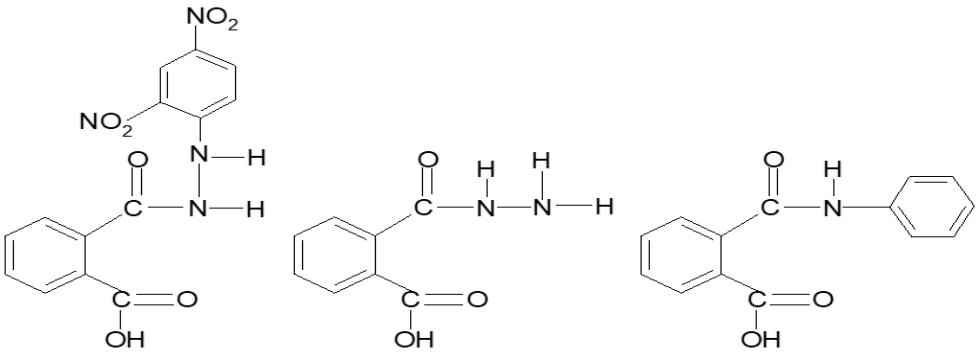
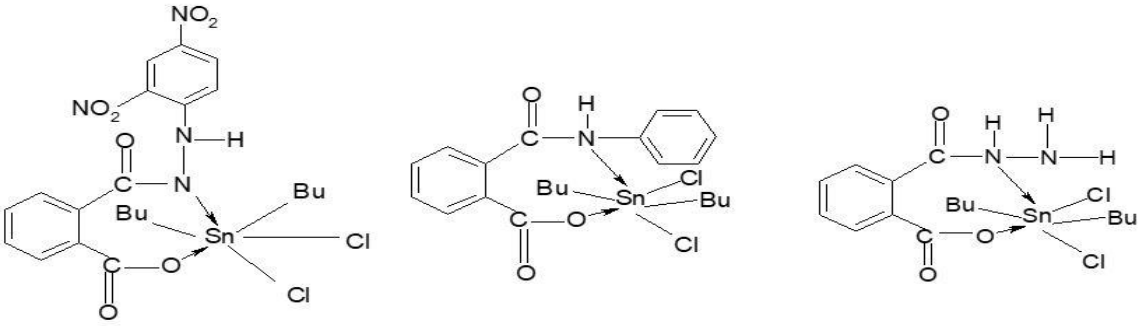


Figure 4: Proposed Structures for the Ligands



Bu = C_4H_9

Figure 5: Proposed Structures for the Organotin(IV) Complexes

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