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Synthesis and Spectral Characterization of Complexes Tin(II) With 4-Nitrophenol

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Abstract

Tin(II)4-nitrophenoxide complexes with the formulas $SnCl(OC_6H_4NO_2\text{-}4)$ and $Sn(OC_6H_4NO_2\text{-}4)_2$ were prepared through the reaction of stannous chloride ($SnCl_2$) with 4-nitrophenol ($HOC_6H_4NO_2\text{-}4$) in 1:1 and 1:2 molar ratios, respectively. The reaction was performed in tetrahydrofuran (THF) with diethylamine serving as the base. The resulting compounds were characterized through elemental analysis, determination of molecular weight, molar conductance measurements, and spectroscopic methods including IR and ^1H NMR spectroscopy. Current investigations are directed toward assessing the stability of these complexes and examining their potential antimicrobial activity. This study enhances insight into the coordination characteristics and reactivity of tin(II) with organic ligands, thereby contributing to the progress of coordination chemistry and its practical applications. The synthesized complexes were obtained in good yield under controlled experimental conditions and appeared as stable colored solids with appreciable solubility in common organic solvents. Infrared spectral data confirmed the coordination of the phenolic oxygen atom to the tin center through noticeable shifts in the characteristic stretching frequencies of the nitrophenoxide ligand. Proton NMR analysis further supported the proposed structures by displaying expected aromatic proton resonances corresponding to the coordinated ligand environment. Molar conductance values indicated the non-electrolytic nature of the complexes, suggesting that the compounds exist as neutral species in solution. The observed analytical and spectral findings collectively support the formation of monomeric tin(II) complexes with probable distorted coordination geometries around the metal center. These findings provide a useful basis for further exploration of organotin derivatives in synthetic and biological applications.

Keywords: Tin(II) aryloxides, 4-nitrophenol, reactivity, NMR, IR spectra

INTRODUCTION

One of the most dynamic and widely explored areas in main-group chemistry is the chemistry of tin. Continuous academic and industrial interest in tin(II) compounds arises from their broad applications in catalysis, materials science, and biological systems [1–3]. The capacity of tin to exhibit variable coordination numbers and geometrical arrangements has enabled the synthesis of a wide variety of tin-based compounds [4]. In particular, tin–oxygen bonded compounds have attracted significant attention because of their structural diversity and notable reactivity. Among these, tin phenoxides form an important subclass, as the ligand effectively stabilizes the tin center in different coordination environments through strong Sn–O interactions. Earlier investigations on tin phenoxides have demonstrated the existence of both monomeric and polymeric architectures, largely influenced by the nature of the ligand, substituents, and reaction conditions [5–7]. Phenols as a ligand represent an especially attractive class of oxygen donors because of their strong σ donating ability and potential for π interactions with central metal ion. The presence of aromatic ring with some

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substituent allows electronic modulation through substituent effects [8–10]. Reports describing the synthesis and applications of tin complexes, comprehensive studies correlating their molecular structures with spectroscopic behavior and electronic effects remain limited in particular, the influence of substituent on the ring and nature of organic groups attached to tin on the coordination environment, aggregation state and Sn-O bonding parameters. In this regard, the present work describes the preparation and detailed characterization of tin phenoxide complexes.

EXPERIMENTAL

Materials

4-Nitrophenol was purified by recrystallization from diethyl ether (m.p. 114°C) prior to use. Anhydrous stannous chloride was prepared according to the procedure described in the literature [11] and latter purity of compounds were checked by elemental analysis and melting point determination. Solvents were purified through distillation and were kept over activated molecular sieves in properly stoppered bottles before use. The sodium salt of 4-nitrophenol ($NaOC_6H_4NO_2$-4) was synthesized as a solid by reacting equimolar quantities of sodium metal and 4-nitrophenol in an organic solvent, with continuous vigorous stirring for three hours.

Synthesis of $Sn(OC_6H_4NO_2\text{-}4)_2$ and $SnCl(OC_6H_4NO_2\text{-}4)$

Under dry, oxygen-free conditions, a solution of stannous chloride (0.5 mmol) was prepared in THF (20 mL). This solution was added dropwise to a magnetically stirred solution of 4-nitrophenol (1.0 mmol or 0.5 mmol) in THF (20 mL) containing diethylamine (1.0 mmol or 0.5 mmol), which served as the base. The reaction mixture was maintained at room temperature with continuous stirring for 6 hours. The resulting diethylamine hydrochloride precipitate was removed by filtration under anhydrous conditions.The filtrate was then concentrated by removing most of the solvent through distillation and left undisturbed overnight. Dark brown and brown solid products formed in the respective reaction vessels. These solids were collected by filtration, washed with THF, and dried under reduced pressure to yield the tin(II) complexes $Sn(OC_6H_4NO_2\text{-}4)_2$ and $SnCl(OC_6H_4NO_2\text{-}4)$.

CHARACTERIZATION

The tin content in the synthesized compounds was estimated gravimetrically as SnO_2. Molecular weights were determined by the cryoscopic method based on depression in freezing point. A Beckman thermometer was employed for molecular weight measurements in nitrobenzene as the solvent. Molar conductance measurements were carried out in the same solvent using 10^{-3} M solutions with an Elico conductivity bridge (CM Type 82-T). Infrared spectra were recorded on a Nicolet 5700 FT-IR spectrometer. The ^{1}H NMR spectra were obtained on a Bruker AC 400 MHz spectrometer using tetramethylsilane (TMS) as an internal standard.

RESULTS AND DISCUSSION

The straightforward reaction between anhydrous stannous chloride in tetrahydrofuran and 4-nitrophenol did not produce the anticipated release of hydrogen chloride gas, even when the mixture was heated under reflux. Consequently, a different method was employed: stannous chloride was reacted with the stoichiometric quantity of sodium 4-nitrophenolate in tetrahydrofuran and then refluxed. Under these conditions, the generation and precipitation of sodium chloride were clearly observed, as described below:

$$SnCl_2 + 2NaOC_6H_4NO_2 - 4 \xrightarrow[\text{Reflux}]{\text{THF}} Sn(OC_6H_4NO_2 - 4)_2 + 2NaCl$$

$$SnCl_2 + NaOC_6H_4NO_2 - 4 \xrightarrow[\text{Reflux}]{\text{THF}} SnCl_1OC_6H_4NO_2 - 4\Big) + NaCl$$

The reactants were taken in 1:1:1 and 1:2:2 molar ratios, respectively. Each component was dissolved separately in tetrahydrofuran and then combined under continuous stirring. During the course of the reaction, a quantitative amount of a white solid separated from the reaction mixture. This solid was later identified as diethylamine hydrochloride ($Et_2NH{\cdot}HCl$) based on its melting point. The formation of the desired compound can be represented by the following reaction:

$$SnCl_2 + HOC_6H_4NO_2\text{-}4 + Et_2NH \xrightarrow{THF} SnCl(OC_6H_4NO_2\text{-}4) + Et_2NH.HCl$$

$$SnCl_2 + 2\ HOC_6H_4NO_2\text{-}4 + 2Et_2NH \xrightarrow{THF} Sn(OC_6H_4NO_2\text{-}4)_2 + 2Et_2NH.HCl$$

Analytical data of Tin(II) Aryloxides

The stoichiometric composition of the isolated solid compounds was confirmed through elemental analysis. The compounds do not display a sharp melting point; rather, they undergo decomposition at temperatures exceeding 50°C (Table 1).

Table 1. Physical appearance and elemental analysis data of synthesized organotin compounds showing observed and calculated percentages of tin (Sn), chlorine (Cl), carbon (C), and hydrogen (H). Values in parentheses represent the calculated elemental composition.

Compound	Color	Elemental Analysis% Found (Calcd)			
		Sn	***Cl***	***C***	***H***
$SnCl(OC_6H_4NO_2\text{-}4)$	Brown	39.78 (40.70)	11.92 (11.98)	23.92 (24.7)	1.31 (1.38)
$Sn(OC_6H_4NO_2\text{-}4)_2$	Dark Brown	29.88 (30.1)	-	35.99 (36.5)	2.02 (2.04)

They are only slightly soluble in methanol and acetonitrile. Molar conductance measurements carried out in nitrobenzene showed very low values, suggesting that the compounds are non-electrolytes. Notably, the compounds do not readily redissolve in tetrahydrofuran after isolation, suggesting the possibility of polymerization in the solid state.

I.R.Spectr: Evidence for the formation of $SnCl(OC_6H_4NO_2\text{-}4)$ and $Sn(OC_6H_4NO_2\text{-}4)_2$ was obtained from their infrared spectra recorded in the 4000–250 cm^{-1} region. In the spectrum of free 4-nitrophenol, a broad absorption band observed in the 3600–3200 cm^{-1} region, assigned to the phenolic ν(OH) stretching vibration [12], disappears in the spectra of both complexes. This absence confirms deprotonation of the phenolic hydroxyl group during complex formation.

The bands in the 1610–1370 cm^{-1} region, attributed to aromatic ν(C=C) stretching vibrations, show a slight shift toward higher wave numbers upon complexation, possibly due to enhanced ring conjugation resulting from coordination.

A significant diagnostic feature is the ν(C–O) stretching vibration of the phenolic group, which appears at 1284 cm^{-1} in free 4-nitrophenol. In the complexes, this band shifts markedly to lower wave numbers, appearing at 1223 cm^{-1} for $Sn(OC_6H_4NO_2\text{-}4)_2$ and 1225 cm^{-1} for $SnCl(OC_6H_4NO_2\text{-}4)$. This downward shift indicates coordination of the phenolic oxygen to the tin center.

Further support for Sn–O bond formation is provided by the appearance of new bands in the 620–490 cm^{-1} region, which are assigned to ν(Sn–O) stretching modes [13, 14]. Additionally, a band observed in the 340–360 cm^{-1} region in $SnCl(OC_6H_4NO_2\text{-}4)$ is attributed to the ν(Sn–Cl) vibration. The absence of this band in $Sn(OC_6H_4NO_2\text{-}4)_2$ confirms the complete removal of Sn–Cl bonds in the latter complex.

IR SPECTRAL DATA (CM^{-1})

4-Nitrophenol ($HOC_6H_4NO_2\text{-}4$)

3420, 3390, 3200, 3070, 3020, 3000, 1610, 1585, 1580, 1515, 1428, 1370, 1340, 1325, 1284, 1260, 1225, 1210, 1180, 1120, 850, 825, 750, 720, 700, 657, 600.

$SnCl(OC_6H_4NO_2\text{-}4)$

3104, 2991, 2925, 2854, 2471, 2082, 1775, 1739, 1644, 1585, 1493, 1445, 1393, 1225, 1165, 1112, 1045, 1015, 972, 943, 865, 761, 695, 621, 550, 315.

$Sn(OC_6H_4NO_2\text{-}4)_2$

3101, 2993, 2927, 2854, 2473, 2079, 1777, 1738, 1644, 1588, 1495, 1443, 1392, 1223, 1166, 1110, 1044, 1015, 969, 940, 864, 759, 697, 661, 625, 555.

^{1}H NMR Spectra

Additional confirmation for the formation of tin(II) aryloxides was obtained from their room-temperature ^{1}H NMR spectra. Comparison of the proton resonance signals of the complexes with those of the corresponding free phenol revealed noticeable changes. These variations in chemical shifts and signal patterns provide clear evidence for complex formation and coordination of the phenolic ligand to the tin(II) center.

The ^{1}H NMR spectra of $SnCl(OC_6H_4NO_2\text{-}4)$ and $Sn(OC_6H_4NO_2\text{-}4)_2$ did not display any signal at δ11.0ppm due to phenolic OH group in pure 4-nitrophenol, suggesting its deprotonation during the complex formation. Further examination of the aromatic proton signals reveals that, in free 4-nitrophenol, two doublets of equal intensity appear at δ 7.06 and δ 8.15 ppm. In the complex $SnCl(OC_6H_4NO_2\text{-}4)$, these signals shift to δ 6.92 and δ 8.08 ppm, respectively, while in $Sn(OC_6H_4NO_2\text{-}4)_2$ they appear at δ 6.90 and δ 8.06 ppm. The observed upfield shifts may tentatively be attributed to changes in the overall π-bonding characteristics of the ligand upon coordination to the tin center, as previously reported in the literature [15]. These spectral changes further support the formation of the tin(II) aryloxide complexes.

CONCLUSION

In summary, new tin(II) complexes were successfully synthesized and comprehensively characterized using spectroscopic techniques such as ^{1}H NMR and IR spectroscopy, which provided valuable insights into their structural and electronic features. The successful formation of these complexes demonstrates the versatile coordination behavior of tin(II) and its ability to form stable aryloxide derivatives. These results enhance the understanding of tin-based coordination chemistry and suggest potential applications in catalysis, materials science, and related fields. Future studies will focus on evaluating the catalytic activity and stability of these complexes under various reaction conditions, as well as exploring their possible industrial applications.

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