Organotin(IV) Derivatives of $N$-Tolyl-$m$-methoxybenzohydroxamic Acid: 
Synthesis and Structural Elucidation

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Abstract
A series of organotin (IV) complexes of the type $R_2SnL_2$, where $R$ = phenyl, butyl and methyl and $HL = N$-Tolyl-$m$-methoxybenzohydroxamic acid were synthesized and characterized by physico-chemical (elemental analysis, and electrolytic conductance) and spectral Infrared and $^{1H}$, $^{13}C$ and $^{119}Sn$ NMR techniques. Monomer structures for the complexes, bidentate and octahedral geometry was proposed for the complexes prepared.

Keywords: $N$-Tolyl-$m$-methoxybenzohydroxamic acid, Diorganotin(IV) Complexes, Spectral studies

1. Introduction
In the past decades the chemistry of tin compounds has gained considerable importance, both in basic research and in industrial applications. There are many interesting aspects of inorganic and organic tin chemistry discussed in various reviews. Tin (IV) and organotin (IV) compounds, a deceptively simple area of inorganic and metal-organic chemistry, have been receiving more attention due to the important industrial (Tammy and Georges, 2005) and environmental applications. Nitrogen, oxygen, and sulfur donor ligands have been used to enhance the biological activity of organotin derivatives (Mohammad et al., 2004; Jason et al., 2000). Also organotin compounds with such ligands have widely been tested for their possible use in cancer chemotherapy (Shang et al. 2008; Zhou et al. 2005). The coordination chemistry of tin is extensive with various geometries and coordination numbers known for both inorganic and organometallic complexes (Katsoulakou et al. 2008; Baul et al. 2007; Farina et al. 2008). Hydroxamic acids constitute a very important class of chelating agents with versatile biological activity (Farkas et al. 2002; Wang et al. 2003).

In view of the diverse fields of applications of organotin complexes, we have synthesized new ligand $N$-methyl-$m$-nitrobenzohydroxamate(HL) and its organotin(IV) compounds Diphenyltin(IV) Bis($N$-Tolyl-$m$-methoxybenzohydroxamate) ($Ph_2SnL_2$), Dibutyltin (IV) Bis ($N$-Tolyl-$m$-methoxybenzohydroxamate)(Bu$_2SnL_2$) and Dimethyltin(IV) Bis ($N$-Tolyl-$m$-methoxybenzohydroxamate)(Me$_2SnL_2$)

2. Materials and Methods
2.1 Synthesis of $N$-Tolyl-$m$-methoxybenzohydroxamic acid
An ether solution of $m$-methoxybenzoyl chloride (0.01 mole) was added dropwise to a stirred cold ethereal solution of $N$-Tolylhydroxylamine (0.01 mole) containing sodium hydrogen carbonate (0.01 mole). The precipitate was filtered and washed with cold ethanol. Good quality crystals suitable for X-ray analysis were obtained by recrystallization from ethyl acetate.
2.2 Preparation of Complexes

Complexes were synthesized by dissolving the free ligand N-Tolyl-methoxybenzohydroxamic acid (5 mmol) in hot toluene and adding the organotin (2.5 mmol) to the solution. The solution was refluxed for 6 hours with magnetic stirrer and then cooled and filtered. The filtrate was reduced under vacuum to a small volume and solid was precipitated by the added of petroleum ether (60-80 °C).

3. Instrumentation

The percentage compositions of the elements (CHN) for the compounds were determined using an elemental analyzer CHNS Model Fison EA 1108. Molar conductance measurements were made in anhydrous DMF at 25 °C using Inolop-Cond Level 1 WTW. The infrared spectra were recorded as potassium bromide discs using a Perkin-Elmer spectrophotometer GX. The ¹H and ¹³C nuclear magnetic resonance spectra were recorded using the JEOL JNM-ECP 400 spectrometer. Electronic UV-Vis spectra were recorded with 1650 PC SHIMADZU Spectrophotometer in the range 200-400 nm. And for ultraviolet using Shimadsu-UV-Vis spectrophotometer UV -2450, DMSO used as solvent. Crystals structures determination were carried out on a Bruker Smart APEX CCD area detector diffractometer equipped with graphite monochromatised Mo-Kα (λ=0.71073Å) radiation in each case. All data collection was carried out at room temperature. The program SMART (Siemens 1996) was used for collecting frames of data, indexing reflections and determination of lattice parameters, SAINT (Siemens 1996) for absorption correction, and SHELXTL (Sheldrick 1997).

4. Results and Discussion

The ligand was prepared by the reaction of m-methoxybenzoyl chloride with one mole N-Tolyl -m-hydroxylamine in presence of sodium hydrogen carbonate as a catalyst. The purity of the ligand and its complexes were checked by TLC using silica gel-G as adsorbent. The conductance of these complexes has been recorded in DMF at room temperature in the range 10-19 ohm⁻¹ cm² mol⁻¹, suggesting their non-electrolytic nature. Their physical properties and analytical data are recorded in Table (1). The calculated values were in a good agreement with the experimental values.

4.1 Infra-Red Spectroscopy

Solid state infrared spectra of the N- Tolyl-methoxybenzohydroxamic acid are recorded in the range 4000-370 cm⁻¹ and the most important bands are presented in the below table studied here. In agreement for diagnostic purpose, the principal infrared absorption bands are those due to -OH, C=O, C-N and N-O stretching vibrations of the hydroxamate group free hydroxamic acids have been shown to exist principally in the keto form. In compound (C=O) group is positioned at 1617cm⁻¹ significantly, below the typical ketonic (C=O) of 1600 cm⁻¹. The (O-H) band is located at 3251 cm⁻¹ as broad band. The presence of the carbonyl group at lower frequency where together with the broad OH band. In general, the (C-N) and (N-O) bands occur as a sharp peak in the ranges 1429, 953 cm⁻¹ respectively (Shahid et al.2002).

On complexation, there are clear differences between the infrared spectra of the free ligand and the diorganotin(IV) complexes. In all cases, the most important features of the infrared spectra are the absence of the (OH) bands due to the complexation of the metal to the ligand through oxygen of the carbonyl group. This suggests the deprotonation of the hydroxamate group on complex formation, and (C=O) group is shifted to lower frequencies in the range 1624-1691 cm⁻¹ significantly, below the typical ketonic (C=O) of 1600 cm⁻¹. The (O-H) band is located at 3251 cm⁻¹ as broad band. The presence of the carbonyl band at lower frequency where together with the broad OH band. In general, the (C-N) and (N-O) bands occur as a sharp peak in the ranges 1429, 953 cm⁻¹ respectively (Shahid et al.2002).

4.2 Nuclear Magnetic Spectroscopy

The ¹H NMR spectra for all compounds were recorded in [²H₆] DMSO using tetramethylsilane as the internal standard. The data are compiled in Table (3). The conclusion drawn from ¹H NMR studies of a few compounds lend further support to suggested formation of N-Tolyl-methoxybenzohydroxamic acid. Ligand (HL) give a singlet –OH resonance near δ 10.64 ppm due to hydroxy group. The hydroxy resonances is absent in the spectra of the complexes. The hydroxy resonances is absent in the spectra of the complexes. The aromatic protons in Ph₂SnL₂, Bu₂SnL₂ and Me₂SnL₂ Show additional signals. The aromatic protons in Ph-Sn appears in the 7.16-8.18 ppm (Shahid et al.2002).

The ¹⁳C NMR spectra for all compounds were recorded in [²H₆] DMSO using tetramethylsilane as the internal standard. The data are compiled in Table (3). The conclusion drawn from ¹H NMR studies of a few compounds lend further support to suggested formation of N-Tolyl-methoxybenzohydroxamic acid. Ligand (HL) give a singlet –OH resonance near δ 10.64 ppm due to hydroxy group. The hydroxy resonances is absent in the spectra of the complexes. The hydroxy resonances is absent in the spectra of the complexes. The aromatic protons in Ph-Sn appears in the 7.16-8.18 ppm (Shahid et al.2002).

The ¹⁹F NMR spectra for all compounds were recorded in [²H₆] DMSO using tetramethylsilane as the internal standard. The data are compiled in Table (3). The conclusion drawn from ¹H NMR studies of a few compounds lend further support to suggested formation of N-Tolyl-methoxybenzohydroxamic acid. Ligand (HL) give a singlet –OH resonance near δ 10.64 ppm due to hydroxy group. The hydroxy resonances is absent in the spectra of the complexes. The hydroxy resonances is absent in the spectra of the complexes. The aromatic protons in Ph-Sn appears in the 7.16-8.18 ppm (Shahid et al.2002).

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ppm. It is most likely that shift is due to the decrease of electron density at carbon atoms when oxygen is bonded to metal ion (Saad et al. 2003), (Figure 9 to 12).

On the basis of the observed spectral evidence, the following structure suggested for the prepared complexes

<Scheme 2>

4.3 X-ray structural studies of N-tolyl m-methoxybenzohydroxamic acid

The structure contain an C\textsubscript{15} H\textsubscript{15} N O\textsubscript{3} ligand, which is a molecule of 3-methoxybenzoyl chloride bonded with N-Tolyl hydroxylamine. The bond angles which will connect Sn atom with the ligand are: C(7)-N(1)-O2 = 117.43(16) , O(1)-C(7)-N(1)=120.14(15), respectively, giving the conformation of an octahedral with the oxygen atoms later at the bottom and the tin atom at the top. This coordination sphere is completed by the both oxygen atoms of the ligands and will be cis positions covering the oxygen atoms. The methoxybenzoyl chloride and tolyl hydroxylamine are chelated by carboxyl groups as shown bellow. And other data in table below.

<Scheme 3>

5. Conclusion

The ligand N- Tolyl-m-methoxybenzohydroxamic acid was successfully synthesized. The ligand was treated to different diorganotin(IV) oxide metal to afford the corresponding complexes. It may conclude that the ligand coordinated through oxygen to the Tin atom leading to the formation of five member ring chelate. Octahedral geometry was proposed for the prepared complexes.

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![Scheme 1](image1.png)

Scheme 1.

![Scheme 2](image2.png)

Scheme 2.
Table 1. Physical data for preparation ligand and the complexes prepared.

| Compound   | Colour  | Yield % | M.P(ºC)  | Found        | (Calcd)(%)     |
|------------|---------|---------|----------|--------------|----------------|
| HL         | Yellow  | 85      | 137-138  | 69.46        | 5.13           |
|            |         |         |          |              | (70.02)        |
|            |         |         |          |              | (5.88)         |
|            |         |         |          |              | 4.90           |
|            |         |         |          |              | (5.44)         |
| Ph₂SnL₂    | Yellow  | 67      | 142-143  | 62.17        | 3.90           |
|            |         |         |          |              | (64.22)        |
|            |         |         |          |              | (4.88)         |
|            |         |         |          |              | 3.09           |
|            |         |         |          |              | (3.57)         |
|            |         |         |          |              | 14.01          |
|            |         |         |          |              | (15.11)        |
| Bu₂SnL₂    | Yellow pale | 71   | 189-190  | 60.89        | 5.78           |
|            |         |         |          |              | (61.22)        |
|            |         |         |          |              | (6.22)         |
|            |         |         |          |              | 3.14           |
|            |         |         |          |              | (3.76)         |
|            |         |         |          |              | 15.22          |
|            |         |         |          |              | (15.92)        |
| Me₂SnL₂    | Brown   | 69      | 122-123  | 57.49        | 4.98           |
|            |         |         |          |              | (58.12)        |
|            |         |         |          |              | (5.18)         |
|            |         |         |          |              | 3.85           |
|            |         |         |          |              | (4.24)         |
|            |         |         |          |              | 16.89          |
|            |         |         |          |              | (17.95)        |

Table 2. Infrared Spectral Data for the ligand and its complexes

| Compound   | v (O-H) cm⁻¹ | v (C=O) cm⁻¹ | v (C-N) cm⁻¹ | v (N-O) cm⁻¹ | v (Sn-C) cm⁻¹ | v (Sn-O) cm⁻¹ |
|------------|--------------|--------------|--------------|--------------|---------------|---------------|
| HL         | 3251         | 1617         | 1429         | 953          | -             | -             |
| Ph₂SnL₂    | -            | 1724         | 1442         | 922          | 569           | 443           |
| Bu₂SnL₂    | -            | 1691         | 1428         | 927          | 573           | 454           |
| Me₂SnL₂    | -            | 1693         | 1430         | 934          | 545           | 455           |
Table 3. $^1$H NMR spectral data (δ, ppm) of the ligand and complexes

| Compound   | -OH(s) | Aromatic     | -O-CH$_3$(s) | -CH$_3$(s) |
|------------|--------|--------------|--------------|------------|
| HL         | 10.64  | 8.48-7.27    | 3.52         | 3.74       |
| Ph$_2$SnL$_2$ | -     | 8.12-7.16    | 3.52         | 3.71       |
| Bu$_2$SnL$_2$ | -     | 8.44-7.21    | 3.79         | 3.81       |
| Me$_2$SnL$_2$ | -     | 8.14-7.39    | 3.79         | 3.79       |

Table 4. $^{13}$C NMR spectral data (δ, ppm) of the ligand and complexes

| Compound   | C-N     | Aromatic     | O-C, Ph-C   | 119Sn    |
|------------|---------|--------------|-------------|----------|
| HL         | 158.61  | 113.67-139.73| 55.24       | -_       |
| Ph$_2$SnL$_2$ | 159.25 | 113.80-132.19| 55.27       | -404.75  |
| Bu$_2$SnL$_2$ | 159.44 | 114.13-133.49| 55.26       | -382.85  |
| Me$_2$SnL$_2$ | 159.18 | 113.77-129.95| 55.23       | -443.56  |
Table 5. Crystal data and structure refinement $N$-tolyl $m$-methoxybenzohydroxamic acid

| Property                                      | Value                                      |
|-----------------------------------------------|--------------------------------------------|
| Empirical formula                             | $C_{15}H_{15}NO_3$                        |
| Formula weight                                | 257.28                                    |
| Temperature                                   | 298(2) K                                  |
| Wavelength                                    | 0.71073 Å                                 |
| Unit cell dimensions                          |                                           |
| $a$ = 11.326(2) Å                            | $\alpha$ = 90°                            |
| $b$ = 7.9503(17) Å                           | $\beta$ = 106.369(4)°                    |
| $c$ = 15.560(3) Å                            | $\gamma$ = 90°                            |
| Volume                                        | 1344.3(5) Å                               |
| Z, Calculated density                         | 4, 1.271 Mg/m³                            |
| Absorption coefficient                        | 0.089 mm⁻¹                                |
| $F$(000)                                      | 544                                       |
| Crystal size                                  | 0.47 x 0.36 x 0.27 mm                     |
| Theta range for data collection               | 1.87 to 26.00 °                           |
| Limiting indices                              | -7 ≤ h ≤ 13, -9 ≤ k ≤ 9, -19 ≤ l ≤ 15    |
| Reflections collected/unique                  | 7258/2636[R (int) = 0.0232]               |
| Completeness to theta                         | 26.00                                    |
| Refinement method                             | Full-matrix least-squares on $F^2$        |
| Data / restraints / parameters                 | 2636 / 0 / 172                            |
| Goodness-of-fit on $F^2$                      | 1.158                                     |
| Final R indices [I>2sigma (I)]                | R1 = 0.0588, wR2 = 0.1212                 |
| R indices (all data)                          | R1 = 0.0767, wR2 = 0.1286                 |
| Largest diff. peak and hole                   | 0.176 and -0.164 e.A⁻³                   |
Table 6. Bond lengths [Å] and angles [deg] for N-tolyl m-methoxybenzohydroxamic acid

| Bond | Length/deg |
|------|------------|
| O(1)-C(2) | 1.416(3) C(4)-C(3)-C(2) | 120.2 |
| O(1)-C(7) | 1.236(2) C(4)-C(3)-H(3B) | 120.2 |
| C(2)-C(8) | 1.4007(19) C(2)-C(3)-H(3B) | 121.4(2) |
| O(3)-N(1) | 0.8200 C(5)-C(4)-C(3) | 119.3 |
| O(3)-H(3A) | 1.331(2) C(5)-C(4)-H(4A) | 119.3 |
| N(1)-C(8) | 1.425(2) C(3)-C(4)-H(4A) | 119.31(19) |
| N(1)-C(9) | 1.379(3) C(4)-C(5)-C(6) | 120.3 |
| C(1)-C(6) | 1.383(3) C(4)-C(5)-H(5A) | 120.3 |
| C(1)-C(2) | 0.9300 C(6)-C(5)-H(5A) | 119.59(18) |
| C(1)-H(1A) | 1.385(3) C(1)-C(6)-C(5) | 117.03(17) |
| C(2)-C(3) | 1.378(3) C(1)-C(6)-C(8) | 123.03(17) |
| C(3)-C(4) | 0.9300 C(5)-C(6)-C(8) | 109.5 |
| C(3)-H(3B) | 1.373(3) O(1)-C(7)-H(7A) | 109.5 |
| C(4)-C(5) | 0.9300 O(1)-C(7)-H(7B) | 109.5 |
| C(4)-H(4A) | 1.386(3) H(7A)-C(7)-H(7B) | 109.5 |
| C(5)-C(6) | 0.9300 O(1)-C(7)-H(7C) | 109.5 |
| C(5)-H(5A) | 1.493(3) H(7A)-C(7)-H(7C) | 109.5 |
| C(6)-C(8) | 0.9600 H(7B)-C(7)-H(7C) | 120.34(17) |
| C(7)-H(7A) | 0.9600 O(2)-C(8)-N(1) | 120.66(17) |
| C(7)-H(7B) | 0.9600 O(2)-C(8)-C(6) | 118.92(17) |
| C(7)-H(7C) | 1.377(3) N(1)-C(8)-C(6) | 120.52(18) |
| C(9)-C(14) | 1.378(3) C(14)-C(9)-C(10) | 118.90(17) |
| C(9)-C(10) | 1.380(3) C(14)-C(9)-N(1) | 120.55(17) |
| C(10)-C(11) | 0.9300 C(10)-C(9)-N(1) | 119.1(2) |
| C(10)-H(10A) | 1.387(3) C(9)-C(10)-C(11) | 120.4 |
| C(11)-C(12) | 0.9300 C(9)-C(10)-H(10A) | 120.4 |
| C(11)-H(11A) | 1.381(3) C(11)-C(10)-H(10A) | 121.6(2) |
| C(12)-C(13) | 1.505(3) C(10)-C(11)-C(12) | 119.2 |
| C(12)-C(15) | 1.379(3) C(10)-C(11)-H(11A) | 119.2 |
| C(13)-C(14) | 0.9300 C(12)-C(11)-H(11A) | 117.72(19) |
| C(13)-H(13A) | 0.9300 C(13)-C(12)-C(11) | 121.5(2) |
| C(14)-H(14A) | 0.9600 C(13)-C(12)-C(15) | 120.8(2) |
| C(15)-H(15A) | 0.9600 C(11)-C(12)-C(15) | 121.6(2) |
| C(15)-H(15B) | 0.9600 C(14)-C(13)-C(12) | 119.2 |
| C(15)-H(15C) | 117.77(19) C(14)-C(13)-H(13A) | 119.2 |
| C(2)-O(1)-C(7) | 109.5 C(12)-C(13)-H(13A) | 119.37(19) |
| N(1)-O(3)-H(3A) | 117.34(15) C(9)-C(14)-C(13) | 120.3 |
| C(8)-N(1)-O(3) | 130.83(16) C(9)-C(14)-H(14A) | 120.3 |
| C(8)-N(1)-C(9) | 111.54(14) C(13)-C(14)-H(14A) | 109.5 |
| O(3)-N(1)-C(9) | 121.02(19) C(12)-C(15)-H(15A) | 109.5 |
| C(6)-C(1)-C(2) | 119.5 C(12)-C(15)-H(15B) | 109.5 |
| C(6)-C(1)-H(1A) | 119.5 H(15A)-C(15)-H(15B) | 109.5 |
| C(2)-C(1)-H(1A) | 115.84(18) C(12)-C(15)-H(15C) | 109.5 |
| O(1)-C(2)-C(1) | 125.03(19) H(15A)-C(15)-H(15C) | 109.5 |
| O(1)-C(2)-C(3) | 119.12(19) H(15B)-C(15)-H(15C) | 109.5 |
| C(1)-C(2)-C(3) | 119.6(2) |

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds geometry for N-tolyl m-methoxybenzohydroxamic acid [Å and deg.].

| No  | D—H···A D—H H···A D···A D—H···A | Symmetry code(i) |
|-----|-------------------|------------------|
| 1   | O1—H1···O2i 0.86 1.99 2.699 139 | -x+1,-y+1,z+1. |
Figure 1. Infrared Spectra of N-tolyl m-methoxybenzohydroxamic acid

Figure 2. Infrared Spectra of dibutyltin (IV) bis(N-tolyl m-methoxybenzohydroxamate)
Figure 3. Infrared Spectra of dimethyltin (IV) bis (N-tolyl-m- methoxybenzohydroxamate)

Figure 4. Infrared Spectra of diphenyltin (IV) bis (N- tolyl m- methoxybenzohydroxamate)
Figure 5. $^{13}$C NMR spectrum of N- tolyl m- methoxybenzohydroxamic acid

Figure 6. $^1$H NMR spectrum of N- tolyl m- methoxybenzohydroxamic acid
Figure 7. $^1$H NMR spectrum of diphenyltin (IV) bis(N- tolyl m- methoxybenzohydroxamate

Figure 8. $^{13}$C NMR spectrum of diphenyltin (IV) bis(N- tolyl m- methoxybenzohydroxamate
Figure 9. $^{13}$C NMR spectrum of dibutyltin (IV) bis(N-tolyl m-methoxybenzohydroxamate

Figure 10. $^1$H NMR spectrum of dibutyltin (IV) bis(N-tolyl m-methoxybenzohydroxamate
Figure 11. \(^1\)H NMR spectrum of dimethyltin (IV) bis(N-tolyl m-methoxybenzohydroxamate

Figure 12. \(^{13}\)C NMR spectrum of dimethyltin (IV) bis(N-tolyl m-methoxybenzohydroxamate