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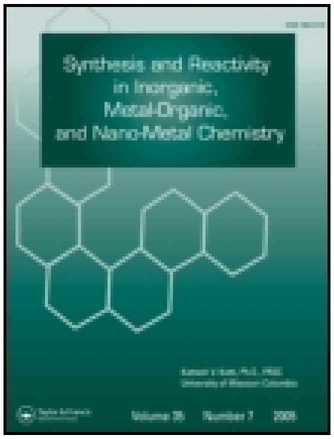

Synthesis and Reactivity in Inorganic and Metal-Organic Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/lsrt19

Synthesis and Characterization of Organotin(IV) 2,4-Dinitrophenoxides

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Published online: 15 Feb 2007.

To cite this article: N. Sharma, A. Kaistha, S. S. Bhatt & S. C. Chaudhry (2003) Synthesis and Characterization of Organotin(IV) 2,4-Dinitrophenoxides, Synthesis and Reactivity in Inorganic and Metal-Organic Chemistry, 33:3, 497-507, DOI: 10.1081/SIM-120020001

To link to this article: http://dx.doi.org/10.1081/SIM-120020001

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SYNTHESIS AND REACTIVITY IN INORGANIC AND METAL-ORGANIC CHEMISTRY
Vol. 33, No. 3, pp. 497–507, 2003

Synthesis and Characterization of Organotin(IV) 2,4-Dinitrophenoxides

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ABSTRACT

Complexes of the composition [$R_3Sn(DNP)$] and [n-$Bu_2SnCl_{2-x}(DNP)_x$] (R = Ph and Me; x = 1 and 2; DNP = anion of 2,4-dinitrophenol) have been synthesized by the silver metathesis method, involving the reaction of Ph_3SnCl, Me_3SnCl and n-Bu_2SnCl_2 with the silver salt of 2,4-dinitrophenol in tetrahydrofuran at room temperature and were characterized by elemental analyses, conductivity measurements, IR, and ^{1}H NMR studies. The IR spectral studies suggest a bidentate mode of coordination of phenol in these complexes. Double phenoxides of the type $M_2[R_3Sn(DNP)_3]$ (M = Na and K) have also been obtained by reacting the parent triorganotin(IV) phenoxides with alkali metal derivatives of the phenol in 1:2 molar ratio. Thermal investigations of these complexes depicted quite interesting behaviour, as the decomposition did not yield any residue, contrary to previous reports. The thermal behaviour of these complexes has shown their ability as high-energy materials.

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DOI: 10.1081/SIM-120020001
0094-5714 (Print); 1532-2440 (Online)

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INTRODUCTION

While nitrophenates of transition metals, because of their immense importance in explosive composition,[1−3] have been the subject of intensive research interest, the literature on analogous organotin(IV) complexes is rather scant.[4−7] Interestingly, the phenolic ligands containing a second donor atom incorporated at the *ortho* position are known to exhibit a unique bonding behaviour wherein an *ortho* donor atom may coordinate to a metal atom in addition to the phenolic oxygen[8,9] (Figure 1).

Hence, in the present investigation, 2,4-dinitrophenol has been chosen as an ancillary ligand towards the synthesis of new organotin(IV) complexes with the aim to study its coordination chemistry and to gain an insight into the structure and behaviour of these complexes. The reactivity of these complexes towards alkali metal derivatives has also been examined. A study of the thermal behaviour of these complexes has provided convincing evidence for their potential utility as high-energy materials.

EXPERIMENTAL

Materials and Methods

2,4-Dinitrophenol (85%, moist solid containing 15% water) (Merck) was recrystallized from THF and was then warmed to ~100 °C. The purity was checked by the melting point. The purity of Ph_3SnCl, Me_3SnCl and Bu_2SnCl_2 (Fluka and Merck) was checked both by chlorine analysis and melting point. The silver salt of 2,4-dinitrophenol was prepared by the reaction of an ethanolic solution of the phenol with an aqueous solution of silver nitrate in equimolor ratios by stirring at room temperature. Tin was determined gravimetrically as SnO_2. Chlorine was analyzed by Volhard's method. Microanalyses for carbon and hydrogen were performed on a Coleman CHN analyzer. The conductance measurements in nitrobenzene were made on an NDC-732 Elico digital conductivity bridge. Infrared spectra (KBr) were recorded from 4000-100 cm^{-1} with a Nicolet-Avtar 1000

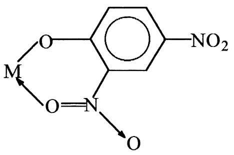

Figure 1. Bidentate mode of bonding of 2,4-dinitrophenol.

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FTIR spectrophotometer and ^{1}H NMR spectra on a Jeol JNM PMX 60 SI spectrometer using $CDCl_3$ as solvent. Thermograms were recorded on a simultaneous DT-40 DT-TG Shimadzu thermal analyzer in air; heating rate: 10 °C min^{-1}/ 20 °C min^{-1}; sample size: 1–4 mg; reference: Al_2O_3; thermocouple: Pt/ Pt-Rh 10%.

Preparation of Complexes [R_3Sn(DNP)] (R = Ph and Me)

To a solution of Ph_3SnCl (1.5 g, 3.8 mmol) and Me_3SnCl (1.5 g, 7.5 mmol) in THF (20 mL) was added an equimolar amount of AgDNP (1.106 g, 3.8 mmol and 2.18 g, 7.5 mmol), respectively, in THF (20 mL). Mixing of these solutions resulted in a colour change and an instantaneous separation of a white solid (AgCl) was observed. After stirring for 5–6 hours, it was filtered off and the filtrate was concentrated by evaporating the solvent under vacuum, when a yellow solid appeared, It was repeatedly washed with petroleum ether and dried under vacuum; the yields were 1.742 g for Ph_3Sn(DNP) and 2.134 g for Me_3Sn(DNP).

Preparation of Complexes [n-Bu_2SnCl_{2-x}(DNP)$_x$] (x = 1 or 2)

To a solution of *n*-Bu_2SnCl_2 (1.2 g, 3.8 mmol) in THF (25 mL) were added stoichiometric amounts of AgDNP (1.106 g, 3.8 mmol or 2.212 g, 7.6 mmol) dissolved in THF (25 mL) in separate experiments. An immediate formation of a white solid (AgCl) resulted. After stirring for 5–6 hours, AgCl was removed from the reaction mixture by filtration. The filtrate was reduced to half of its volume under vacuum and was then treated with petroleum ether (40–60 °C) to give pale yellow crystals which were subsequently dried under vacuum; the yields were 1.391 g for Bu_2SnCl(DNP) and 2.069 g for Bu_2Sn(DNP)$_2$.

Preparation of M_2[R_3Sn(DNP)$_3$] (M = Na, K; R = Ph and Me)

To a solution of Ph_3Sn(DNP) (2.06 g, 3.8 mmol) in THF (25 mL)was added a suspension of NaDNP (1.59 g, 7.7 mmol) in THF (20 mL) in the tin:alkali metal molar ratio 1:2. It was stirred for ~2 hours until the reactants completely went into the solution. The solvent was then removed under vacuum and the remaining concentrated solution was treated with petroleum ether when a bright yellow solid resulted. It was dried under vacuum. The yield of Na_2[Ph_3Sn(DNP)$_3$] was 3.19 g. The following complexes were prepared by similar method:

Na_2[Me_3(DNP)$_3$] from the reaction of Me_3Sn(DNP) (1.31 g, 3.7 mmol) with NaDNP (1.55 g, 7.4 mmol); yield 2.24 g. K_2[Ph_3Sn(DNP)$_3$] from Ph_3SnDNP (2.02 g, 3.7 mmol) with KDNP (1.68 g, 7.5 mmol); yield 3.0 g.

$K_2[Me_3Sn(DNP)_3]$ from Me_3SnDNP (1.32 g, 3.8 mmol) with KDNP (1.68 g, 7.6 mmol); yield, 2.67 g.

RESULTS AND DISCUSSION

Interaction between various organotin(IV) chlorides and the silver salt of 2,4-dinitrophenol in tetrahydrofuran in stoichiometric amounts at room temperature proceeds smoothly and quantitatively with instantaneous separation of silver chloride according to Eqs. 1 and 2:

$$R_3SnCl + AgDNP \xrightarrow[25\,^\circ C]{THF} [R_3Sn(DNP)] + AgCl\downarrow \quad (1)$$

(R = Ph and Me)

$$n\text{-}Bu_2SnCl_2 + xAgDNP \xrightarrow[25\,^\circ C]{THF} n\text{-}Bu_2SnCl_{2-x}(DNP)x + xAgCl\downarrow \quad (2)$$

(x = 1 and 2)

Silver chloride is easily removed by filtration, and the title complexes crystallized out in good yields from the filtrate. The elemental analyses support the stoichiometric formulations (Table 1). The compounds are light yellow to dark yellow, crystalline solids with sharp melting points and are readily soluble in common organic solvents such as CCl_4, C_6H_6, $C_2H_2Cl_2$. The molar conductances of the complexes in nitrobenzene (10^{-3} M solution) are very low, indicating the non-electrolytic nature of the complexes.

Infrared Spectra

Formation of the new complexes has been inferred from a comparison of IR spectra with those of free phenol, the silver salt and the previous data[7,8] reported for other tin(IV) and organotin(IV) complexes. The characteristic ligand bands for the complexes are observed in the fingerprint region. The most important and diagnostic band due to the phenolic ν(C–O) mode observed at 1255 cm^{-1} in the phenol under study has remained almost unchanged in the complexes, in contrast to a significant decrease in the frequency of this band reported to be observed in metal alkoxides and aryloxides.[10,11] The observed trend of ν(C–O) may be ascribed to the weaker coordination of the dinitrophenolic ligand perhaps due to ring conjugation. The occurrence of new vibrational modes in the region 520–

Table 1. Analytical data of the complexes.

Complex (empirical formula)	Formula weight	Color	Melting point (°C)	Yield (%)	% Found (calc.)				Λ_M in nitrobenzene (Ω^{-1} cm^2 mole^{-1})
					Sn	Cl	C	H	
$Ph_3Sn[OC_6H_3(NO_2)_2\text{-}2,4]$	533	Mustard yellow	80	86	21.9	-	53.8	3.1	1.1
($C_{24}H_{18}N_2O_5Sn$)					(22.4)		(54.0)	(3.3)	
$Me_3Sn[OC_6H_3(NO_2)_2\text{-}2,4]$	347	Lemon yellow	112	82	33.9	-	30.9	3.1	1.4
($C_9H_{12}N_2O_5Sn$)					(34.4)		(31.1)	(3.4)	
$Bu_2SnCl[OC_6H_3(NO_2)_2\text{-}2,4]$	451	Yellow	107	81	26.1	7.5	36.8	4.7	0.9
($C_{14}H_{21}N_2O_5ClSn$)					(26.3)	(7.7)	(37.2)	(4.6)	
$Bu_2Sn[OC_6H_3(NO_2)_2\text{-}2,4]_2$	599	Pale yellow	106	91	19.7	-	39.7	4.1	1.6
($C_{20}H_{24}N_4Sn$)					(19.8)		(40.0)	(4.0)	
$Na_2[Ph_3Sn\{OC_6H_3(NO_2)_2\text{-}2,4\}_3]$	945	Bright yellow-	-	87	12.1	-	45.6	2.6	5.2
($C_{36}H_{24}N_6Na_2O_{15}Sn$)					(12.5)		(45.7)	(2.7)	
$K_2[Me_3Sn\{OC_6H_3(NO_2)_2\text{-}2,4\}_3]$	789	Yellow	-	89	15.1	-	31.6	2.2	4.7
($C_{24}H_{18}K_2N_6O_{15}Sn$)					(15.0)		(31.9)	(2.2)	
$Na_2[Me_3Sn\{OC_6H_3(NO_2)_2\text{-}2,4\}_3]$	759	Bright yellow	-	78	15.0	-	33.1	2.1	4.3
($C_{21}H_{18}N_6Na_2O_{15}Sn$)					(15.6)		(33.2)	(2.3)	
$K_2[Ph_3Sn\{OC_6H_3(NO_2)_2\text{-}2,4\}_3]$	975	Yellow	-	81	12.0	-	43.9	2.1	3.8
($C_{36}H_{24}K_2N_6O_{15}Sn$)					(12.2)		(44.4)	(2.4)	

Λ_M = Molar conductance.

480 cm^{-1} (not present in the ligand) attributable to ν(Sn–O) stretching frequencies[12] has supported the linkage between oxygen and tin metal. The IR spectra of the complexes reveal that bands observed at ~1545 and 1342 cm^{-1} attributed to antisymmetric and symmetric stretching vibrations, respectively, of the NO_2 group in free phenol and in its silver salt have been found to shift to lower frequencies on complexation and have appeared in the regions 1542–1520 and at ~1332 cm^{-1}, suggesting coordination of the *ortho* NO_2 group through oxygen to the metal. These observations are thus indicative of a probable bidentate nature of the phenolic ligand. Nevertheless, a slight ambiguity in these assignments may be anticipated due to the overlapping of many other ligand vibrations in this region. The bands observed ~280 and 240 cm^{-1} in the far-IR spectra of these complexes have tentatively been assigned to ν_{asym} and ν_{sym} (Sn–C) modes, respectively.

^{1}H NMR Spectra

The ^{1}H NMR spectral data of free 2,4-dinitrophenol have provided a basis for interpreting the spectra of its organotin complexes. The spectra showed three distinct resonances due to aromatic protons which are attributed to three types of ring hydrogens, apart from the one due to hydroxyl (phenolic protons) at δ 4.65 ppm. The assignments for the three resonances observed at 9.1, 8.5 and 7.35 ppm were made by taking into account the electron withdrawing effect of the NO_2 substituents on the phenolic ring. These resonances have been attributed to two different kinds of *meta* protons, (*m*) having two neighboring NO_2 groups at 2 and 4-positions, *meta* (*m′*) protons with one adjacent NO_2–4 group and the third *ortho* to the phenolic group, respectively. In the ^{1}H NMR spectra of the complexes, the complete absence of a signal at δ 4.65 ppm due to the phenolic OH proton in free 2,4-dinitrophenol confirmed the deprotonation of this group.

The spectra of the triphenyltin(IV) complex indicated that (*m* and *m′*) protons are displaced slightly upfield compared to that of free phenol, which may be attributed to the shielding effect exerted on these protons by the aromatic protons of the phenyl ring attached to the tin metal. A complex pattern between 7.0 and 8.0 ppm as a result of superposition of phenyl protons attached to tin and phenolic *ortho* protons has been observed. In the ^{1}H NMR spectra of the Me_3Sn(IV) complex the signals due to aromatic protons have been observed to undergo a very small downfield shift amounting to 0.01 to 0.15 ppm. The signal due to the methyl protons is seen at 0.7 ppm.

In the spectra of the dibutyltin(IV) complexes, the signal assignments have been ascertained with respect to those of the starting materials, *n*-Bu_2SnCl_2 and phenol. In *n*-Bu_2SnCl_2, methyl and methylene resonances appear at ~0.9, 1.30 and 1.77 ppm. It is interesting to note that, compared to a moderate downfield shift observed for the *n*-Bu protons, the aromatic protons appear almost at the same position as in the free ligand. Nevertheless, integration supports the proposed formulations.

Based upon the available analytical and spectral data, a trigonal bipyramidal structure for triorganotin(IV) and chlorodibutyltin(IV) complexes and a distorted octahedral structure for the dibutylbis(2,4-dinitrophenoxo)tin(IV) complex may tentatively be suggested in accordance with earlier reports[13] (Figure 2).

Double Aryloxides

In the course of the present study, we have also investigated the reactivity of the title complexes towards alkali metal derivatives of 2,4-dinitrophenol with an objective of ascertaining their ability to form double aryloxides. Although our attempts to prepare complexes of the composition $M\{R_3Sn(DNP)_2\}$ were not successful, products of the composition $M_2\{R_3Sn(DNP)_3\}$ containing six-coordinating anions[14] were prepared by the following reaction:

$$R_3Sn(DNP) + MDNP \longrightarrow M_2[R_3Sn(DNP)_3] \qquad (3)$$

$$[R = Ph,\ Me;\ M = Na,\ K]$$

The complexes are bright yellow solids and are insoluble in common organic solvents. The molar conductance values of millimolar solutions of these complexes in nitrobenzene have indicated their appreciable electrolytic nature (Table 1). The IR spectra of the complexes have shown the characteristic stretching frequencies of phenolic bonding in the regions 1256–1108, 927–916 and 679–640 cm^{-1} supporting their formation.

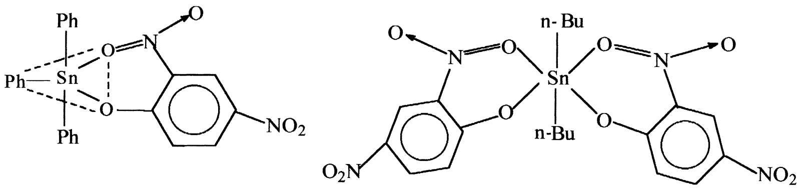

Figure 2. Proposed structures for the complexes.

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Thermal Studies

The decomposition behavior of the ligand 2,4-dinitrophenol (DNPH), the silver salt of 2,4-dinitrophenol, (AgDNP) and the organotin(IV) and bimetallic 2,4-dinitrophenoxides have also been investigated by thermogravimetric measurements and differential thermal analysis in view of the importance of substances containing NO_2 groups as flame sensitizers and high-energy materials or explosives. The results obtained from TG and DTA curves are summarized in Table 2. The TG/DT curves of 2,4-dinitrophenol indicate its melting temperature to be 111.1 °C, depicted as a sharp endotherm in the DT curve and its stability up to 121.8 °C, after which temperature it sublimes quantitatively. On the other hand, $AgOC_6H_3(NO_2)_2$-2,4 begins to decompose at $\sim$23.5 °C and undergoes explosive decomposition without leaving any residue. The possibility of the substance being blown out during the explosive decomposition can't be ruled out. The decomposition is accompanied by a sharp exothermic effect at 319.5 °C. No exotherm, however, was observed in the case of the free phenol. An induction period of $\sim$2 minutes for this exothermic change is suggestive of its being a high-energy material due to the dinitrophenolic ligand bonded to silver ion.

Thermograms of Ph_3SnDNP, Me_3SnDNP, *n*-$Bu_2SnClDNP$ and *n*-$Bu_2Sn(DNP)_2$ have shown these compounds to be stable up to 113.4, 113.2, 118.9 and 110.2 °C, respectively. The melting endotherms have been observed at 79.9, 112.0, 106.9 and 106.0 °C for the respective complexes. A striking feature of the decomposition of these complexes is that there is neither any explosion during decomposition nor any sharp exothermic peak in the DTA curves, as was observed in AgDNP. Hence, the 100% weight loss in organotin(IV) 2,4-dinitrophenoxides may be attributed to their volatile nature. These complexes, therefore, represent, to our knowledge, the first example of its kind, which have not yielded any residue from their thermal decomposition processes, contrary to previous reports[15,16] wherein SnO_2, SnO and Sn have resulted as end products.

A thorough scrutiny of the thermal decomposition behaviour of NaDNP, KDNP and the bimetallic phenoxides of the compositions $Na_2[Ph_3Sn(DNP)_3]$ and $K_2[Me_3Sn(DNP)_3]$ has offered some unique results: 1) an initial decomposition process is accompanied by a small weight loss and a weak endotherm; 2) the main decomposition process proceeds with a significant weight gain, subsequently followed by an abrupt weight loss over a very small range of temperature leaving no residue; 3) the major fast decomposition process in the DTA exhibits a very strong and sharp exothermic peak due to violent decomposition accompanied by a cracking

Table 2. Thermal decomposition data of the complexes.

		TG data		DTA	
Complex (sample weight)	IDT (°C) (initial decomp. temp.)	Decomp. range (°C)	% weight loss	Peak temp.,(°C)	Peak nature
2,4-DNP (4 mg)	121.8	121.8–220.4	99.75	111.1	Endo (Sharp)
				200.5	Exo
Ag-DNP (1 mg)	23.5	23.5–41.5	98.87	139.2	Endo (Weak)
				319.5	Exo (Sharp)
$Ph_3Sn(DNP)$ (4 mg)	113.4	113.4–291.3	99.87	79.9	Endo (Sharp)
				251.6	Exo (Sharp)
$Me_3Sn(DNP)$ (2 mg)	113.2	113.2–224.9	99.5	112.0	Endo (Sharp)
				202.8	Exo (Weak)
n-$Bu_2SnCl(DNP)$ (3 mg)	118.9	118.9–225.0	99.32	106.9	Endo (Sharp)
				200.9	Exo (Weak)
n-$Bu_2Sn(DNP)_2$ (2 mg)	110.2	110.2–220.4	100	106.0	Endo (Sharp)
				202.9	Exo (Weak)
Na(DNP) (3.53 mg)	111.9	111.9–136.9	6.37	125.2	Endo (Broad)
		136.9–290.9	–	–	–
		290.9–312.2	–	309.5	Exo (Sharp)
$Na_2[Ph_3Sn(DNP)_3]$ (1 mg)	119.8	119.8–129.1	3.73	124.1	Endo (Weak)
		129.1–298.1	–	–	–
		298.1–305.8	–	301.3	Exo (Sharp)
$K_2[Me_3Sn(DNP)_3]$ (1 mg)	113.4	113.4–130.6	3.75	–	–
		130.6–295.3	–	–	–
		295.3–311.4	–	310.1	Exo (Sharp)

DNP = anion of 2,4-dinitrophenol.

sound and 4) a weight gain in the TG and corresponding exothermic peak in DTA appear in close proximity to each other.

Of all the complexes, the violent decomposition was more pronounced with NaDNP and KDNP. Moreover, the present observations of the DTA indicate a close resemblance with the characteristic features of high-energy materials, such as TNT, HMX (octogen) and RDX (hexogen).

In conclusion, the present study has shown that novel tri and diorganotin(IV) complexes of 2,4-dinitrophenol can be synthesized by the reaction of the respective chlorides with the silver salt of the phenol by the silver metathesis method. Based on the thermal decomposition of these complexes involving 100 % weight loss, they are proposed to be volatile. Double phenoxides of these complexes, however, have been found to exhibit a significant explosive nature and are likely to be quite useful as high-energy materials in the future.

ACKNOWLEDGMENTS

The authors are grateful to Prof. M.S. Hundal, Department of Chemistry, Guru Nanak Dev University, Amritsar, for obtaining the FTIR spectra.

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Received: July 12, 2001 Referee II: J. M. Iriarte-Gross
Accepted: November 21, 2002 Referee III: F. P. Gabbai