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Syntheses and characterization of thallium(I) complexes with 3-nitrophenoxide [Tl(3-np)], 4-nitrobenzoate [Tl(4-nb)] and 2,4-dinitrophenoxide [Tl(2,4-dnp)]: X-ray crystal structures of $[Tl(3-np)]_n$ and Tl(2,4-dnp) (two new polymeric compounds)

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Abstract

Complexes thallium(I)3-nitrophenoxide [Tl(3-np)], thallium(I)2,4-dinitrophenoxide [Tl(2,4-dnp)] and thallium(I)4-nitrobenzoate [Tl(4-nb)] have been synthesized using a direct reaction between TlNO₃ and the appropriate ligand. The complexes have been isolated and characterized by IR spectra and CHN elemental analyses. The structures of [Tl(3-np)]_n and [Tl(2,4-dnp)] have been confirmed by X-ray crystallography. The single crystal X-ray crystallography of [Tl(3-np)]_n shows the complex to be a one-dimensional polymer as a result of bridging 3-nitrophenoxide ligands. The Tl atoms have an unsymmetrical three-coordinate, O₃ geometry (three oxygen atoms of the 3-nitrophenoxide ligand). The crystal structure of [Tl(2,4-dnp)] shows the complex to be a three-dimensional polymer as a result of bridging 2,4-dinitrophenoxide ligands. The Tl atoms have an unsymmetrical two-coordinate, O₂ geometry (two oxygen atoms of the 2,4-dinitrophenoxide ligand). The arrangement of the 3-nitrophenoxide and 2,4-dinitrophenoxide ligands suggests a gap in coordination geometry around the Tl(I) ions, occupied possibly by a stereoactive lone pair of electrons on Tl(I). There is a π - π stacking interaction between the parallel aromatic rings belonging to adjacent chains in the compounds that may help to increase the 'gap' in coordination geometry around the Tl(I) ions.

Keywords: Thallium(I) complexes; Crystal structure; Nitrophenoxide complexes

1. Introduction

Thallium(I) complexes are interesting because of their unique characteristics such as the potential ability to form metal-metal bonds and the presence of a lone pair in the valence shell [1,2]. An issue frequently discussed in considering the coordination and stereoactivity of heavy metals is that of the "stereochemical activity" of valence shell lone electron pairs. It appears that in complexes of Pb(II) [3–7], Tl(I) [8–10] and Bi(III) [11–13], the nature and form of the coordination sphere is generally determined by a number of factors, possibly lone pair-bond pair repulsions of comparable influence, so

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that seemingly minor differences in ligands or in the crystal array can have quite marked effects upon the coordination stereochemistry. Since the presence of a lone pair is not directly detected but is inferred on the basis of the spatial distribution of atoms assumed to be donors to the central metal ion, the identification of these donor atoms is fundamental to the analysis of any particular system. Interestingly, this alone is not a straightforward process. The lone pair activity can be dependent on the following factors: (1) low and high coordination number, (2) hard or soft ligands, (3) attractive or repulsive interactions among ligands, (4) lone pair has p or no p character, (5) fewer or more electrons transferred from ligands to metal [14].

The salts of 2-,3-,4-nitrophenol, 2-npH, 3-npH and 4npH, have more recently been recognized as members of a broad family of weak acid derivatives [15,16]. [Tl(2np)] and [Tl(4-np)] have been previously described [8]. In

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the present paper, we want to show the nature of adducts formed between thallium(I) nitrate and 3-nitrophenoxide, 2,4-dinitrophenoxide and 4-nitrobenzo-ate ligands.

2. Experimental

2.1. Physical measurements

IR spectra were recorded as Nujol mulls using Perkin–Elmer 597 and Nicolet 510P spectrophotometers. Microanalyses were carried out using a Heraeus CHN-O-Rapid analyzer.

2.2. Preparation of Tl(3-np)

The complex Tl(3-np) was prepared by dissolving 0.266 g (1 mmol) thallium(I) nitrate in distilled water and adding a mixture of 3-nitrophenol (0.139 g, 1 mmol) and potassium hydroxide (0.057 g, 1 mmol) in ethanol (10 ml). The resulting solution was stirred for 1 h at room temperature, then it was allowed to stand for 2-3 days in a refrigerator. Yellow crystals of the desired product precipitated, were filtered off, washed with acetone and ether and air dried (0.239 g, yield 70%), m.p. 190 °C.

Anal. Found: C, 19.65; H, 1.29; N, 4.31. Calc. for C₁₂H₈N₂O₆Tl₂: C, 21.05; H, 1.17; N, 4.05%.

IR (cm^{-1}) selected bands: 720(s), 850(s), 1380(vs), 1590(s), 1612(s), 2980(w), 3140(w).

2.3. Preparation of Tl(2,4-dnp)

An ethanolic solution of 2,4-dinitrophenol (0.368 g, 2 mmol) and potassium hydroxide (0.114 g, 2 mmol) was added to a solution of thallium(I) nitrate (0.532 g, 2 mmol) in water. The resulting solution was concentrated on a water bath at 80 °C and cooled to room temperature (~ 25 °C), when a red microcrystalline compound was separated. The compound was filtered off, washed with cold ethanol and dried over silica gel (0.387 g, yield 50%). The red single crystals were isolated by the following branch tube method. The complex (0.3 g)was placed in one arm of the branched tube. Methanol was carefully added to fill both arms, then the tube was sealed and the complex-containing arm immersed in a bath at 60 °C while the other was at ambient temperature. After 10 days, red crystals (m.p. 240 °C) had deposited in the cooler arm. Anal. Found: C, 18.3; H, 0.9; N, 7.3. Calc. for C₆H₃N₂O₅Tl: C, 18.59; H, 0.77; N, 7.23%.

 $IR(cm^{-1})$ selected bands: 720(w), 850(w), 1180(s), 1350(vs), 1385(vs), 1560(s), 1590(s), 1640(vs), 3030(w).

2.4. Preparation of Tl(4-nb)

The complex Tl(4-nb) was prepared by dissolving 0.266 g (1 mmol) thallium(I) nitrate in distilled water and adding an ethanolic solution of 3-nitrobenzoic acid (0.167 g, 1 mmol) and potassium hydroxide (0.057 g, 1 mmol). The resulting solution was stirred for 2 h at room temperature, and then it was allowed to stand for 3-4 days in a refrigerator. A white powder of the desired product precipitated, which was filtered off, washed with acetone and ether and air dried (0.230 g, yield 55%), m.p. 208 °C.

Anal. Found: C, 22.15; H, 1.13; N, 3.82. Calc. for C₇H₄NO₄Tl: C, 22.67; H, 1.08; N, 3.78%.

IR(cm⁻¹) selected bands: 717(vs), 817(s), 1331(vs), 1375(vs), 1507(vs), 1544(s), 2980(w), 3045(w).

2.5. Crystallography

2.5.1. Crystal data and refinement details

[Tl(3-np)]. C₁₂H₈N₂O₆Tl₂, *M* 684.94, triclinic, space group *P*I, *a* = 3.7649(15), *b* = 9.400(4), *c* = 10.068(4) Å, $\alpha = 85.159(8)^{\circ}$, $\beta = 86.133(8)^{\circ}$, $\gamma = 78.749(7)^{\circ}$, *V* = 347.8(2) Å³, *D*_{calc} (*Z* = 1 f.u.) 3.271 Mg m⁻³, *F*(000) 304. Specimen: 0.10 × 0.1 × 0.20 mm; *T*_{max,min} 0.761, 0.265, *N* 2891, *N*₀ 1359, *R* 0.0468, *R*_w 0.0835.

Tl(2,4-dnp)]. C₆H₃N₂O₅Tl, *M* 387.47, monoclinic, space group *P*2(1)/*c*, *a* = 5.7649(15), *b* = 19.261(11), *c* = 7.636(4) Å, $\alpha = 90^{\circ}$, $\beta = 91.857(11)^{\circ}$, $\gamma = 90^{\circ}$, *V* = 802.4(8) Å³, *D*_{calc} (*Z* = 4 f.u.) 3.208 Mg m⁻³, *F*(000) 696. Specimen: 0.05 × 0.2 × 0.30 mm; *T*_{max,min} 0.1553, 0.0294, *N* 9196, *N*₀ 2339, *R* 0.0500, *R*_w 0.1046.

2.5.2. Determination of the structures

Crystallographic measurements were made at 140(2) K for [Tl(3-np)] and 120(2) K for [Tl(2,4-dnp)] using a Siemens R3m/V diffractometer. The intensity data were collected within the range $2.03^{\circ} \le \theta \le 27.03^{\circ}$ for [Tl(3-np)] and $3.41^{\circ} \le \theta \le 30.06^{\circ}$ for [Tl(2,4-dnp)] using graphite monochromated Mo K α radiation ($\lambda = 0.71073$ Å). Accurate unit cell parameters and an orientation matrix for data collection were obtained from least-squares refinement. Intensities of 2891 and 9196 unique reflections were measured, from which 1359 and 2339 with $I > 2\sigma(I)$ were used in the refinement for [Tl(3-np)] and [Tl(2,4-dnp)], respectively. The structures have been solved by direct methods and refined by full-matrix least-squares techniques on F^2 .

The positions of hydrogen atoms were idealized and included in the calculations of the structure factors as fixed contributions. Each hydrogen atom was assigned an isotropic thermal parameter. R, R_w , with goodness-of-fit on F^2 0.897 and 0.754 are 0.0468, 0.0835 and 00.0385, 0.0962 for [Tl(3-np)] and [Tl(2,4-dnp)], respectively. The final difference density map showed a maximum peak and hole of 1.992, -1.495 e Å⁻³ for

[Tl(3-np)] and 2.507, -2.103 e Å⁻³ for [Tl(2,4-dnp)]. Corrections for Lorentz and polarization effects as well as the empirical correction for absorption using the SADABS programs were applied. All structural calculations were carried out with a PDP-11/23 + computer using the SDP-PLUS program package [17,18].

Crystal data and structure refinement are given in Tables 1 and 2. Selected bond lengths and angles are given in Tables 3 and 4. Anisotropic thermal parameters, observed and calculated structure factors, full lists of bond distances, bond angles and torsion angles are given in the supplementary material. ORTEP diagrams and a perspective view of the packing in the unit cells are shown in Figs. 1-4.

3. Discussion

3.1. Synthesis

Reaction between 3-nitrophenoxide, 4-nitrobenzoate and 2,4-dinitrophenoxide ligands provided powdered

Table 1 Crystal data and structure refinement for $Tl(3-NO_2C_6H_4O)$

Identification code	$Tl(3-NO_2C_6H_4O)$
Empirical formula	$C_{12}H_8N_2O_6T_{12}$
Formula weight	684.94
Temperature (K)	140(2)
Wavelength (Å)	0.71073
Crystal system	triclinic
Space group	$P\bar{1}$
Unit cell dimensions	
a (Å)	3.7649(15)
b (Å)	9.400(4)
c (Å)	10.068(4)
α (°)	85.159(8)
β (°)	86.133(8)
γ (°)	78.749(7)
V (Å ³)	347.8(2)
Ζ	1
D_{calc} (Mg m ⁻³)	3.271
Absorption coefficient (mm^{-1})	23.171
F(000)	304
Crystal size (mm)	0.1 imes 0.1 imes 0.2
Theta range for data collection (°)	2.03-27.03
Index ranges	$-4 \le h \le 4, -12 \le k \le 12,$ $-12 \le l \le 12$
Reflections collected	2891
Independent reflections	1359 $[R_{int} = 0.0708]$
Completeness to theta = 27.03°	89.6%
Absorption correction	SADABS program
T_{\min}/T_{\max}	0.265/0.761
Refinement method	full-matrix least-squares on F^2
Data/restraints/parameters	1359/0/100
Goodness-of-fit on F^2	0.897
Final <i>R</i> indices [for 1008 reflections	$R_1 = 0.0468, wR_2 = 0.0835$
with $I > 2\sigma(I)$]	
R indices (all data)	$R_1 = 0.0661, wR_2 = 0.0877$
Largest difference peak and hole (e ${\rm \AA}^{-3})$	1.992 and -1.495

Crystal data and structure refinement for Tl(2,4-dnp)

Empirical formula	C ₆ H ₃ N ₂ O ₅ Tl
Formula weight	387.47
Temperature (K)	120(2)
Wavelength (Å)	0.71073
Crystal system	monoclinic
Space group	P2(1)/c
Unit cell dimensions	
a (Å)	5.458(3)
b (Å)	19.261(11)
c (Å)	7.636(4)
α (°)	90
β (°)	91.857(11)
γ (°)	90
$V(Å^3)$	802.4(8)
Ζ	4
D_{calc} (Mg m ⁻³)	3.208
Absorption coefficient (mm^{-1})	20.126
F(000)	696
Crystal size (mm)	$0.05 \times 0.2 \times 0.3$
Theta range for data collection (°)	3.41-30.06
Limiting indices	$-7 \le h \le 7, \ -27 \le k \le 27,$
	$-10 \le l \le 10$
Reflections collected/unique	9196/2339 [$R_{\rm int} = 0.0637$]
Completeness to $\theta = 30.06^{\circ}$	99.3%
Absorption correction	Semi-empirical from equivalents
Max/min transmission	0.1553, 0.0294
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2339/0/127
Goodness-of-fit on F^2	0.754
Final R indices [for 1862 reflections	$R_1 = 0.0385, wR_2 = 0.0962$
with $1.2\sigma(I)$]	
R indices (all data)	$R_1 = 0.0500, wR_2 = 0.1046$
Largest difference peak and hole	2.507 and -2.103
$(e Å^{-3})$	

Table 3 Selected bond lengths (Å) and angles (°) for Tl(3-np)

Bond lengths		Bond angles	
Tl(1)-O(1)	2.499(8)	O(1)-Tl(1)-O(1)#1	73.0(3)
Tl(1)-O(1)#1	2.562(7)	O(1)-Tl(1)-O(1)#2	82.3(3)
Tl(1)-O(1)#2	2.717(10)	O(1)#1-Tl(1)-O(1)#2	90.9(3)
Tl(1)-O(2)#3	3.005(8)	O(1)-Tl(1)-Tl(1)#5	95.88(19)
Tl(1)-O(1)#4	3.396(11)	O(1)#1-Tl(1)-Tl(1)#5	133.8(2)
Tl(1)-O(3)#3	3.515(10)	O(1)#2-Tl(1)-Tl(1)#5	42.88(16)
Tl(1)-O(2)#4	3.554(9)	O(1)-Tl(1)-Tl(1)#6	84.12(19)
Tl(1)-Tl(1)#2	3.9308(14)	O(1)#1-Tl(1)-Tl(1)#6	46.2(2)
Tl(1)-Tl(1)#1	4.0696(14)	O(1)#2-Tl(1)-Tl(1)#6	137.12(16)
Tl(1) - Tl(1)#5	3.7649(15)	C(1) - O(1) - Tl(1)	126.0(7)
Tl(1)-Tl(1)#6	3.7649(15)	C(1)-O(1)-Tl(1)#1	123.7(7)
O(1)-Tl(1)#1	2.562(7)	C(1)-O(1)-Tl(1)#2	98.7(7)
O(1)-Tl(1)#2	2.717(10)	Tl(1)-O(1)-Tl(1)#1	107.0(3)
		Tl(1)-O(1)-Tl(1)#2	97.7(3)
		Tl(1)#1-O(1)-Tl(1)#2	90.9(3)

Symmetry transformations used to generate equivalent atoms: #1 -x+1, y+2, -z, #2 - x, -y+2, -z, #3 - x+1, -y+1, -z, #4-x, -y+1, -z, #5 x+1, y, z; #6 x-1, y, z.

Table 4 Selected bond lengths (Å) and angles (°) for Tl (2,4-dnp)

Bond lengths		Bond angles	
Tl(1)-O(1) Tl(1)-O(1)#1 Tl(1)-Tl(1)#2	2.601(5) 2.626(5)	$\begin{array}{c} O(1)-Tl(1)-O(1)\#1\\ O(1)-Tl(1)-Tl(1)\#2\\ O(1)\#-Tl(1)-Tl(1)\#2\\ C(1)-O(1)-Tl(1)\\ C(1)\#-O(1)-Tl(1)\#1\\ Tl(1)-O(1)-Tl(1)\#1\\ \end{array}$	75.41(18) 127.69(11) 141.24(11) 124.6(4) 130.6(4) 104.59(18)

Symmetry transformations used to generate equivalent atoms: #1 -x+1, -y+1, -z+2, #2 -x+1, -y+1, -z+1.

materials analyzing as [Tl(3-np)], [Tl(4-nb)] and [Tl(2,4dnp)], respectively. Attempts to isolate suitable single crystals for X-ray crystallography for [Tl(4-nb)] were not successful and each time the powdered compound was isolated.

3.2. Crystal structure of [Tl(3-np)]

Determination of the structure of [Tl(3-np] by X-ray crystallography (Table 1) showed the complex in the solid state (Fig. 2) to be a polymeric species with various similarities to the polymeric structures of [Tl(2-np)] and [Tl(4-np)], and it is really a novel one-dimensional polymer. The thallium atoms are linked within a classical 'stair-polymer' array by phenoxide oxygen atoms, each $Tl(\mu-O)_2Tl$ unit having an inversion point at its center, and a pair of them making up the unit b component of the polymer.

Thus, each thallium atom can be considered to be three-coordinate with an O_3 donor atom array (three



Fig. 2. A view perpendicular to a polymeric strand of the solid showing the chelating and bridging functions of the 3-nitrophenoxide ligand and the full donor atom environment of the Tl atoms in Tl(3-np).

oxygen atoms of the 3-nitrophenoxide ligand with Tl-O distances of T11···O1 = 2.499, T11···O1 (-x+1, -y)+2, -z = 2.562 and Tl1...O (-x, -y+2, -z) =2.717 Å), and the weak interaction of thallium(I) with oxygen atoms of an adjacent molecule produces polymer units in the solid state. In fact each Tl atom in this structure alone with three normal bonds forms four "weak" Tl...O bonds and the exact distances are Tl1···O2 (-x+1, -y+1, -z) = 3.005, Tl1···O3 (-x, -y+1, -z) = 3.396 Å, Tl1···O3 (-x+1, -y+1)1, -z) = 3.515 and Tl1···O2 (-x, -y+1, -z) = 3.554 Å (Table 3). The presence of a lone pair of electron on the thallium atom is apparently the reason that the bridging interactions cannot come closer together. If the stereochemically active lone pair was not present, it might create more interaction between the bridging atoms of thallium(I) with the neighboring oxygen atoms



Fig. 1. The ORTEP diagram of the Tl₂O₂ rhomb forming the basic link of the polymeric structure of [Tl(3-np)]₂.



Fig. 3. The ORTEP diagram of Tl(2,4-dnp).



Fig. 4. A view perpendicular to a polymeric strand of the solid showing the chelating and bridging functions of the 2,4-dinitrophenoxide ligand and the full donor atom environment of the Tl atoms in Tl(2,4-dnp).

of nitrophenoxide. Consequently, this phenomenon could lead to more symmetry.

The arrangement of the oxygen atoms of nitrophenoxide suggest a gap or hole in the coordination geometry around the metal ion, occupied possibly by a stereoactive lone pair of electrons on thallium(I). The observed shortening of the Tl-O bond on the side of the Tl(I) ion opposite to the putative lone pair (2.499 compared with 2.717 Å adjacent to the lone pair) supports the presence of this feature. There are some "weak" Tl...Tl bonds in the polymeric state and the exact distances are Tl1···Tl1 (-x, -y+2, -z) =3.9308 Å, T11...T11 (-x+1, -y+2, -z) = 4.0696 Å, Tl1···Tl1 (x+1, y, z) = 3.7649 Å and Tl1···Tl1 (x-1, z) = 3.7649 Å (v, z) = 3.7649 Å (Table 3). The Tl···Tl across the rhomb (3.7649 \AA) is appreciably shorter than the normal of approximately 4.0696 Å proposed as typical of TI...TI in $Tl(\mu-O_2)Tl$ rhomb. The nakedness of the resulting thallium environment possibly suggests a stereochemically active lone pair.

3.3. Crystal structure of [Tl(2,4-dnp)]

Determination of the structure of [Tl(2,4-dnp] by Xray crystallography (Table 2) showed the complex in the solid state (Fig. 4) to be a polymeric species with various similarities to the polymeric structures of [Tl(2-np)], [Tl(4-dnp)] and [Tl(3-dnp)], and it is really a novel threedimensional polymer. The thallium atoms are linked within a classical 'stair-polymer' array by phenoxide oxygen atoms. Each thallium atom can be considered to be two-coordinate with an O_2 donor atom array (two oxygen atoms of the 2,4-nitrophenoxide ligand) with TI-O distances of TI1 \cdots O1 = 2.601, and TI1 \cdots O1 (-x+1, -y+2, -z+2) = 2.626, and the weak interaction of thallium(I) with oxygen atoms of the nitro group of an adjacent molecule produces polymer units in the solid state (Table 4). There are "weak" $TI \cdots TI$ bonds in the polymeric state and the exact distances are Tl1...Tl1 (-x+1, -y+1, -z+1) = 3.740 Å (Table 4). The presence of a lone pair of electrons on the thallium atom is apparently the reason that the bridging interactions cannot come closer together. The arrangement of oxygen atoms of nitrophenoxide suggest a gap or hole in the coordination geometry around the metal ion, occupied possibly by a stereoactive lone pair of electrons on thallium(I). If the stereochemically active lone pair was not present, it might create more interaction between the bridging atoms of thallium(I) with the neighboring oxygen atoms of nitrophenoxide. Consequently, this phenomenon could lead to more symmetry.

The strikingly similar feature of the $[Tl(3-np)]_n$ complex with the $[Tl(2,4-dnp)]_n$ complex is that there is a $\pi-\pi$ stacking [19,20] interaction between the parallel aromatic rings belonging to adjacent chains in the two compounds. The question is, does the $\pi-\pi$ stacking between the parallel aromatic rings help to increase the "gap" in coordination geometry around the metal ion or does the 'gap' arise from the "stereochemical activity" of valence shell lone electron pairs that then help to form $\pi-\pi$ stacking.

The strikingly different feature of the $[Tl(3-np)]_n$ complex with the $[Tl(2,4-dnp)]_n$ complex is that the $[Tl(3-np)]_n$ complex is a one-dimensional polymer whereas the $[Tl(2,4-dnp)]_n$ complex is a three-dimensional polymer. This point must relate to the additional nitro group in the nitrophenoxide ligand. The $[Tl(2-np)]_n$ and $[Tl(4-np)]_n$ complexes [8] are one-dimensional polymers and support the presence of this feature.

Crystallographic data, selected bond lengths and angles are given in Tables 1–4. Atomic coordinates, anisotropic thermal parameters, observed and calculated structure factors, full lists of bond distances, bond angles and torsion angles are given in the supplementary material. A perspective view of the ORTEP diagram and the packing in the unit cell are shown in Figs. 1–4.

4. Supplementary material

Complete bond lengths and angles, co-ordinates and displacement parameters have been deposited with the Cambridge Crystallography Data Centre, CCDC Nos. 174597 for [Tl(3-np)] and 183434 for [Tl(2,4-dnp)]. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@cdc.cam.ac.uk or www: http:// www.ccdc.cam.ac.uk).

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