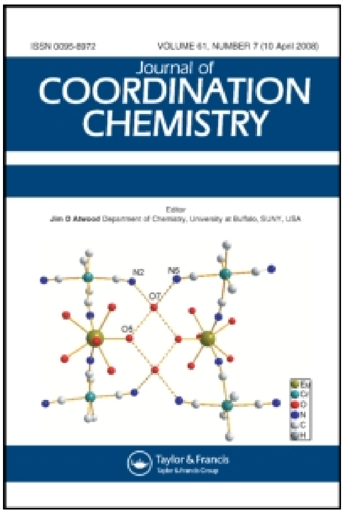

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Thermal and structural studies of two new Tl^I three-dimensional coordination polymers, $[Tl_2(\mu\text{-CSB})]_n$ and $[Tl_2(\mu\text{-ADC})]_n$, CSB^{2-} = 4-[(4-carboxyphenyl) sulfonyl]-1-benzenecarboxylate and ADC^{2-} = acetylendicarboxylate

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Two new 3D coordination polymers, $[Tl_2(\mu_{10}\text{-CSB})]_n$ (**1**) [CSB = 4-[(4-carboxyphenyl)sulfonyl]-1-benzenecarboxylate] and $[Tl_2(\mu_9\text{-ADC})]_n$ (**2**) [ADC = acetylendicarboxylate], have been synthesized and characterized by elemental analysis, and IR spectroscopy. Thermal behavior and structures of **1** and **2** have been studied. Single-crystal X-ray data of **1** show that the thallium atoms have an irregular coordination sphere containing a stereochemically active lone pair with TlO_5 environment. The single-crystal X-ray data of **2** showed that there are two different Tl environments. One Tl is six-coordinate with a weak $Tl \cdots Tl$ interaction with a distance of 3.904 Å. The other type of Tl in the TlO_5C_2Tl units is eight-coordinate with one weak $Tl \cdots Tl$ and two short $Tl \cdots C$ interactions.

Keywords: Thallium; 4-[(4-Carboxyphenyl)sulfonyl]-1-benzenecarboxylate; Acetylendicarboxylate

1. Introduction

Thallium(I) is a low-valence p-block metal with a closed sub-shell (s^2). Yet its structural chemistry features unusual patterns of aggregation in the solid state because of unique characteristics such as the ability to form metal–metal bonds, metal–carbon bonds, high coordination number because of the large size of thallium(I), and the presence of a lone pair of electron in the valence shell of the Tl^I cation [1–7]. Thallium(I) coordination may be built on several different types of metal–ligand and metal–metal interactions [9–38]. From structural studies of some Tl^I complexes [1–6], it has been argued that "stereo-chemical activity" and polyhapto-aromatic interactions play important roles in determining the solid-state geometry of these compounds.

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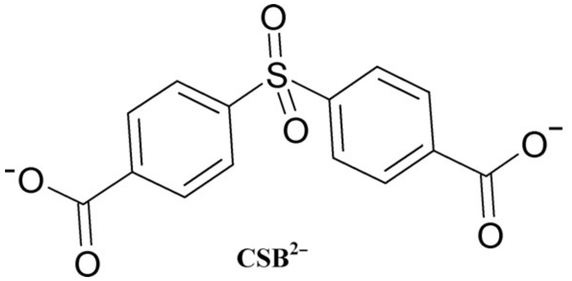
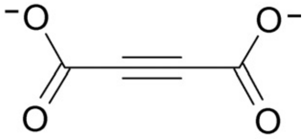


CSB^{2-}



ADC^{2-}

For further study, we used 4-[(4-carboxyphenyl)sulfonyl]-1-benzenecarboxylate (CSB) and acetylendicarboxylate (ADC) anions which contain both aromatic rings and linear triple bonds. Because these ligands can form polymeric compounds, investigation of the metal–metal and metal–carbon interactions may be of interest in polymeric and supramolecular compounds.

2. Experimental part

2.1. *Physical measurements*

All reagents and solvents for the synthesis and analysis were commercially available and used as received. IR spectra were recorded using Perkin-Elmer 597 and Nicolet 510P spectrophotometers. Microanalyses were carried out using a Heraeus CHN-O-Rapid analyzer. Thallium content was determined using ICP method. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. The thermal behavior was measured with a PL-STA 1500 apparatus. The reason for the relatively high values of absorption is the poor quality of the crystal and there is a high positive residual density of 4.79 e Å at 0.81 Å near Tl1 due to considerable absorption effects in **2**, which could not be completely corrected.

2.2. *Preparation of [Tl_2(μ-CSB)]$_n$ (1)*

Complex **1** was prepared by dissolving 0.532 g (2 mmol) of $TlNO_3$ in 20 mL MeOH and 5 mL water. In another beaker 0.306 g (1 mmol) of potassium-4-[(4-carboxyphenyl) sulfonyl]-1-benzenecarboxylic acid was dissolved in 25 mL MeOH. Both solutions were mixed slowly. After a week colorless crystals appeared which were collected and air-dried (0.320 g yield 45%). The melting point of this compound is over 300°C. IR (selected bands; in cm^{-1}): 621 m, 738 s, 1361 s, 1154 s, 1292 s, 1369 vs, 1506 vs, 1582 m, 3055 w. Anal. Calcd for $C_{14}H_8O_6STl_2$: C 23.56, H 1.12, Tl 57.22; found: C 23.80, H 1.26, Tl 57.80.

2.3. *Preparation of [Tl_2(μ-ADC)]$_n$ (2)*

Complex **2** was prepared by dissolving 0.266 g (1 mmol) of $TlNO_3$ in 20 mL MeOH and a little water. In another beaker 0.114 g (2 mmol) of KOH was dissolved in 10 mL

MeOH and added to 0.114 g (1 mmol) of acetylendicarboxylic acid that was dissolved in MeOH. Then the beakers were added to each other and mixed and heated for an hour. After several days colorless crystals were collected (0.286 g, yield 55%), m.p: 145°C. IR (selected bands; in cm^{-1}): 666 m, 770 m, 818 m, 938 m, 1380 vs, 1607 vs, 1758 w. Anal. Calcd for $C_4O_4Tl_2$: C 9.21, Tl 78.34; found: C 9.50, Tl 78.60.

2.4. *X-ray crystallography*

X-ray measurements were made at 100(2) K for **1** and 213(2) K for **2** using a Siemens R3m/V diffractometer. The intensity data were collected within the range $2.86 \leq \theta \leq 29.06°$ for **1** and $3.14 \leq \theta \leq 27.94°$ for **2** using graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). Accurate unit cell parameters and an orientation matrix for data collection were obtained from least-squares refinement. The intensities of 1952 and 781 unique reflections were collected, from which 1749 and 645 with $I > 2\sigma(I)$ were used in the refinement for **1** and **2**, respectively. The structures were solved by direct methods and refined by full-matrix least-squares on $|F|^2$. The positions of hydrogen atoms were calculated at idealized geometrical position and included in the structure-factor calculation as riding on the parent carbon atoms. Corrections for Lorentz and polarization effects as well as a semi-empirical absorption correction were applied. There is a high positive residual density near the Tl1 centers in the compounds **1** and **2** that due to considerable absorption effects which, could not be completely corrected. All calculations were carried out with a PDP-11/23+ computer using the SDP – PLUS program package [39, 40]. The ORTEP diagrams of compounds are shown in figures 1 and 2.

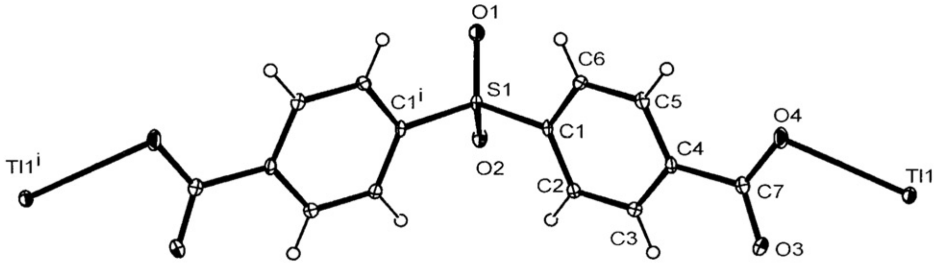


Figure 1. ORTEP representation of $[Tl_2(\mu\text{-CSB})]_n$ (**1**) i: $x, -y+1/2, z$.

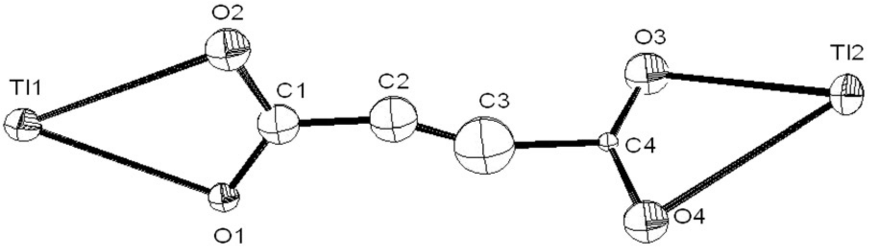


Figure 2. ORTEP representation of $[T1_2(\mu\text{-ADC})]_n$ (**2**).

3. Results and discussion

The reaction between the 4-[(4-carboxyphenyl)sulfonyl]-1-benzenecarboxylate (CSB^{2-}) and acetylendicarboxylate (ADC^{2-}) anions with $Tl^{I}(NO_3)$ provided crystalline materials of the general formula $[Tl_2(\mu\text{-CSB})]_n$ (**1**) and $[Tl_2(\mu\text{-ADC})]_n$ (**2**) (figures 1 and 2), respectively.

IR spectra display characteristic absorption bands for CSB^{2-} and ADC^{2-} ligands. Relatively weak absorption bands around $3055\,cm^{-1}$ are due to the C–H modes of the aromatic ring hydrogen atoms in **1**. Absorption bands with variable intensity in the frequency range $1407–1582\,cm^{-1}$ correspond to ring vibrations of the pH of the CSB^{2-} ligand. The weak absorption band at $1758\,cm^{-1}$ is due to the –C≡C– bond in **1**. The characteristic bands of the carboxylate groups appear at 1506 $\upsilon_{as(C–O)}$ and 1369 $\upsilon_{sym(C–O)}\,cm^{-1}$ in **1** and 1607 $\upsilon_{as(C–O)}$ and 1380 $\upsilon_{sym(C–O)}\,cm^{-1}$ in **2**. The $\Delta\upsilon$ value ($\upsilon_{as} - \upsilon_{sym}$) of 125 and $227\,cm^{-1}$, indicates that the carboxylate groups coordinate to the metal ions in both chelating and bridging mode [41–45], which is confirmed by the crystal structure. Results of TGA show one main weight loss, from 410 to 450°C and 430 to 690°C for **1** and **2**, respectively, during which the compounds decompose to Tl_2O. The total weight losses of 41.06 and 18.43% are close to the calculated value of 40.44 and 18.60% for **1** and **2**, respectively.

Determination of the structure of **1** and **2** by X-ray crystallography showed the compounds are three-dimensional polymer (figures 3 and 4). In **1** the thallium atoms are five-coordinate (figure 5) and in **2** there are two types of Tl^{+}-ions with coordination numbers of six $Tl1O_6$ and five $Tl2O_5$ (figure 6a and b). The Tl1 and Tl2 atoms in **2** interact with one neighboring thallium atom. The separation $Tl1 \cdots Tl2^{ii}$ and $Tl2 \cdots Tl1^{ii}$ is 3.904 Å, which is less than twice the van der Waals radius of thallium ca 3.92 Å [5] and is proposed to represent a weak attractive interaction [6]. Hence one $Tl \cdots Tl$ interaction for Tl1 and Tl2 atoms in **2** may be considered, giving environments of Tl1 and Tl2 in **2** as $Tl \cdots Tl1O_6$ and $Tl \cdots Tl2O_5$ (figure 6a and b). Each carboxylate group of "CSB^{2-}" anion in **1** acts as a pentadentate ligand, connecting four Tl^{I} ions (scheme 1a). In **2**, one carboxylate group of "ADC^{2-}" is pentadentate, connecting four Tl^{I} ions, and other carboxylate group of "ADC^{-2}" is tetradentate, connecting three Tl^{I} ions (scheme 1b). The carboxylate groups of "CSB^{2-}" and "ADC^{2-}" act as both bidentate chelating, and bridging where two oxygen atoms of the carboxylate group coordinate to a thallium(I) ion, and also bridge to other thallium atoms, interesting behavior of carboxylate (scheme 1a and b).

The arrangement of O-atoms in complexes **1** and **2** suggest a gap or hole in coordination geometry around the Tl^{I} coordination sphere, (figures 5–6), a gap possibly occupied by a 'stereo-active' electron lone pair. The geometry of the nearest coordination environment of every Tl^{I} is likely caused by geometrical constraints of coordinated O-atoms, $Tl \cdots Tl$ interactions, and by the influence of a stereo-chemically 'active' electron lone pair in a hybrid orbital on the metal. The observed shortening of the Tl–O bond on the side of Tl^{I} opposite to the putative lone pair (Tl1–O4 = 2.633(3) compared with Tl1–O3 = 2.972(3) Å adjacent to the lone pair in **1** and Tl1–O1 = 2.69(3) compared with Tl1–O2 = 2.91(4) Å adjacent to the lone pair of Tl1 in **2** as well as Tl2–O3 = 2.67(4) compared with Tl2–O4 = 3.00(3) Å adjacent to the lone

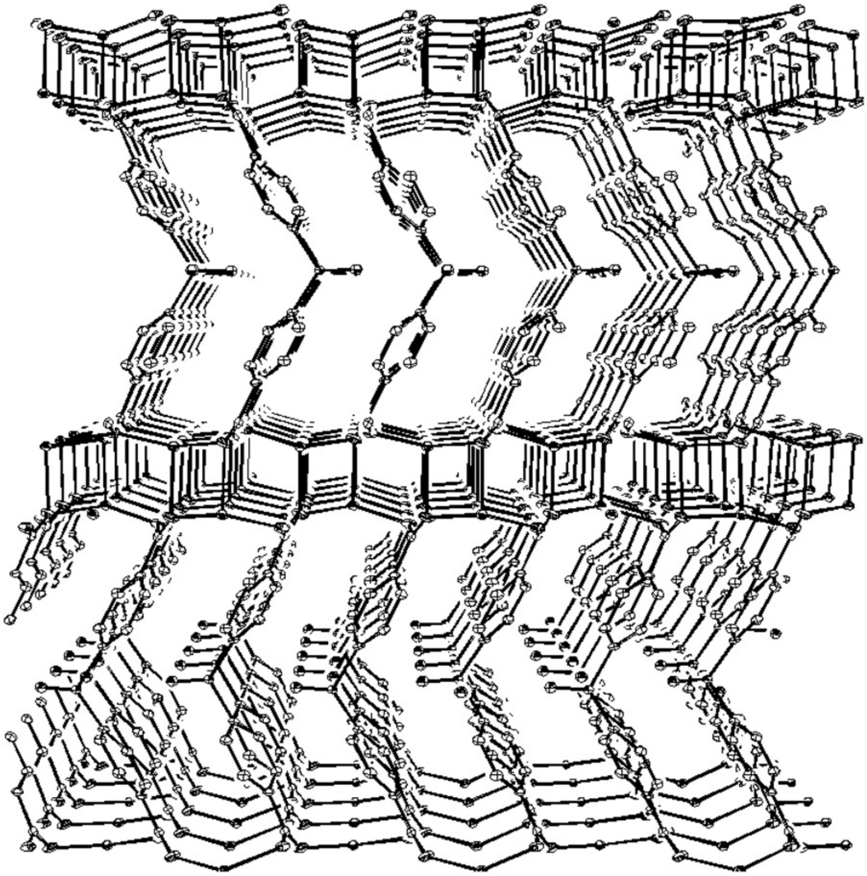


Figure 3. The packing of $[Tl_2(\mu\text{-CSB})]_n$ (**1**).

pair of Tl2 in **2** supports the presence of this feature [46]. In complexes **1** and **2**, the lone pair of Tl(I) is stereochemically 'active' in the solid state.

To find any other potential donor center, it is necessary to extend the bonding limit. A search was made generally for Tl···C approaches and it appears that Tl2 in **2** may also be involved in an η^2 interaction with acetylene of neighboring molecules. Thus, the Tl2 atoms in **2** are linked to two carbon atoms of neighboring acetylene groups, with distances $Tl2\cdots C2^{iii}$ and $Tl2\cdots C3^{iii}$ of 3.398(2), and 3.628(5) Å, respectively (figure 6b). Hence, the Tl^I coordination sphere is completed and rather than a $Tl\cdots Tl2O_5$ interaction the complex can be considered to contain a bihapto ($Tl\cdots Tl2O_5C_2$) with an irregular eight coordination. Reported Tl···C separations range from 3.20–4.00 Å in recent reported species [5, 25] and the sum of the van der Waals radii of carbon and Tl atoms is 3.66 Å [47]. Thus, bihapto coordination of Tl^I in **2** appears to be yet another factor which can make varying contributions to the stability of complexes of this metal ion. The ADC^{2-} ion should be linear, but in the compound **2** the conformation is non-linear and the dihedral angle of C1–C2–C3–C4 is 130.90°. The presence of strong Tl-acetylene interaction,

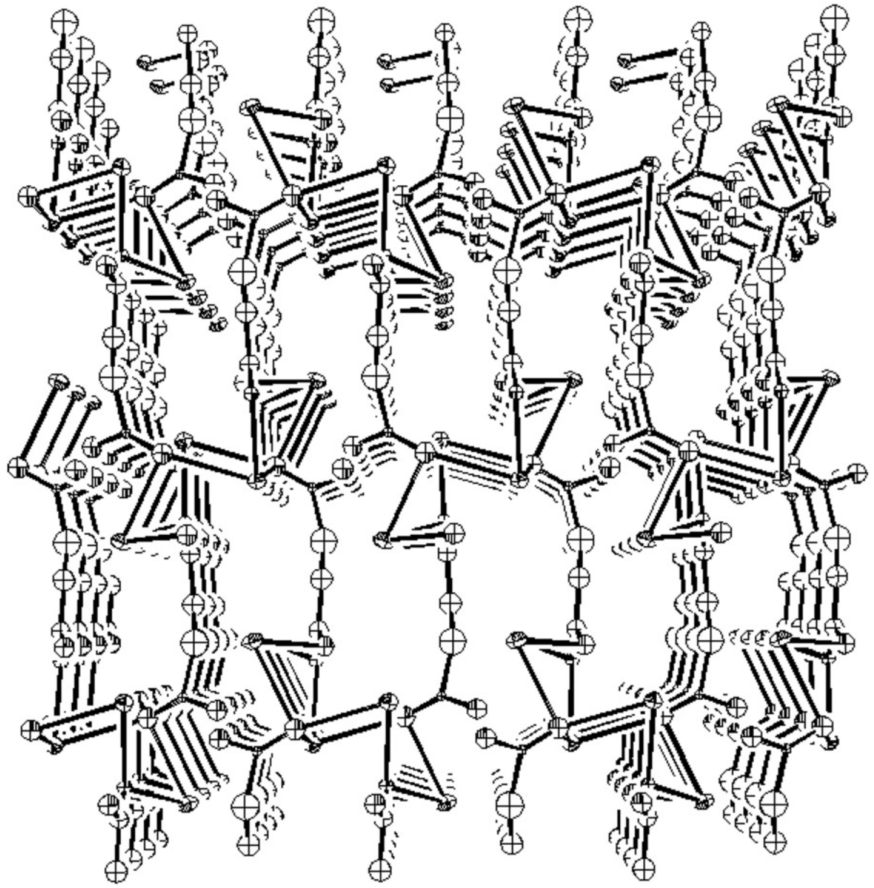


Figure 4. The packing of $[\mathrm{Tl}_2(\mu\text{-ADC})]_n$ (**2**).

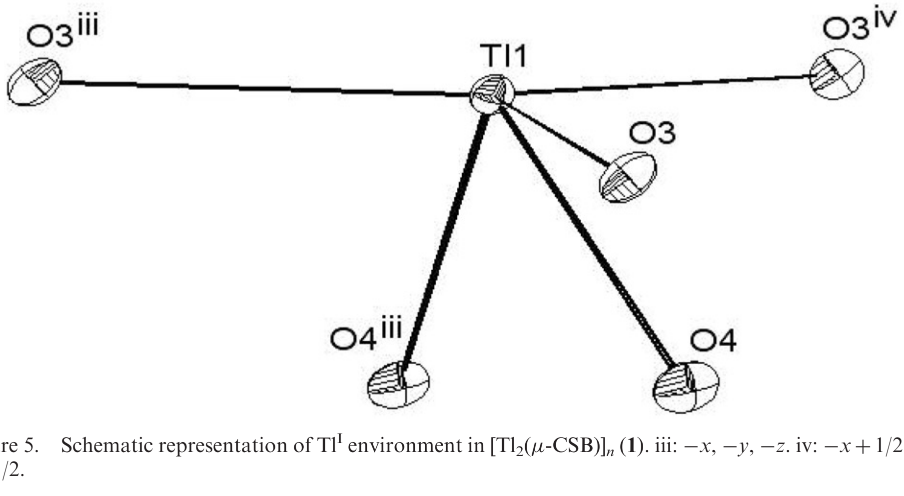


Figure 5. Schematic representation of Tl^{I} environment in $[\mathrm{Tl}_2(\mu\text{-CSB})]_n$ (**1**). iii: $-x$, $-y$, $-z$. iv: $-x+1/2$, $-y$, $z+1/2$.

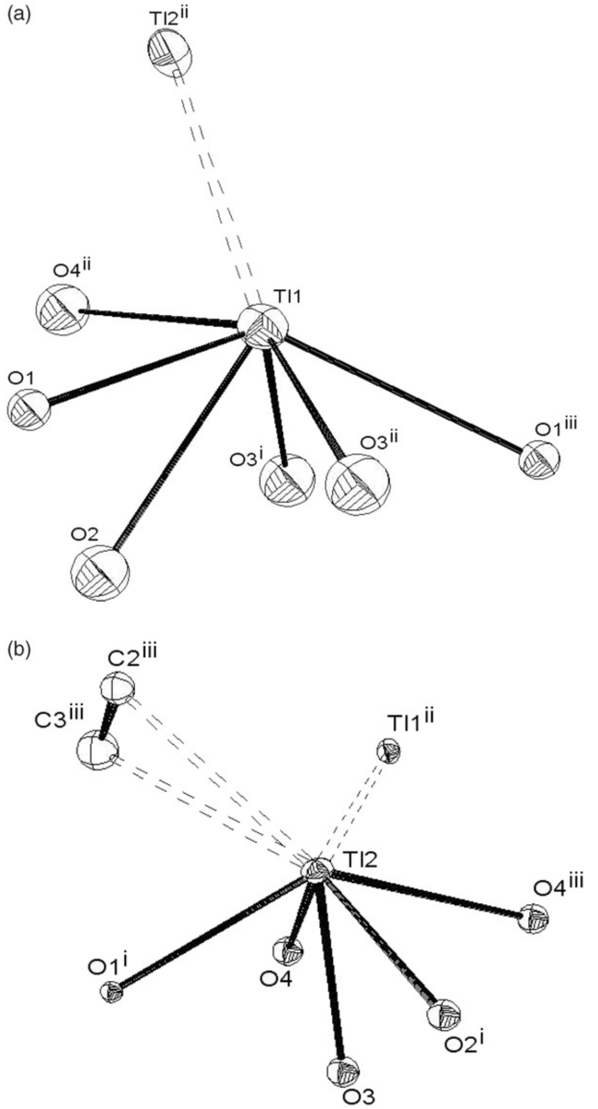
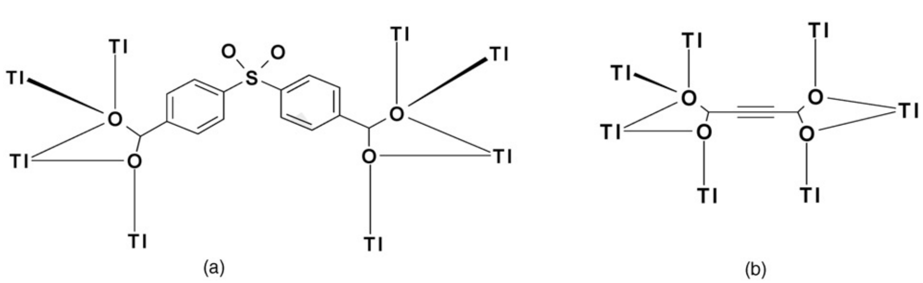


Figure 6. Schematic representation of (a) $Tl1^I$ and (b) $Tl2^I$ environment in $[Tl_2(\mu\text{-}ADC)]_n$ (**2**) i: $-x+1/2$, $-y$, $z+1/2$; ii: $-x$, $y+1/2$, $-z+1/2$; iii: $x+1/2$, $-y+1/2$, $-z$.



Scheme 1. Coordination mode of ligands (a) CSB^{2-} and (b) ADC^{2-}.

Tl2 $\cdots$ C2^{iii} and Tl2 $\cdots$ C3^{iii}, is apparently the reason for the large deviation of linearity and a zig-zag conformation of the ADC^{2-} in compound **2**.

Supplementary data

The crystallographic data (excluding structure factors) for **1** and **2** have been deposited with the Cambridge Crystallographic Data Centre (CCDC) as supplementary publication number CCDC-278655 for **1** and 278656 for **2**. Copies of the data can be obtained, free of charge, by application to CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336033; Email: data_request@ccdc.cam.ac.uk), or via the internet (http://www.ccdc.cam.ac.uk/products/csd/request).

Table 1. Crystal data and structure refinement for $[Tl_2(\mu\text{-CSB})]_n$ and $[Tl_2(\mu\text{-ADC})]_n$.

Identification code	$[Tl_2(\mu\text{-CSB})]_n$	$[Tl_2(\mu\text{-ADC})]_n$
Empirical formula	$C_{14}H_8O_6STl_2$	$C_4O_4Tl_2$
Formula weight	713.00	520.78
Temperature (K)	100(2)	213(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Orthorhombic	Orthorhombic
Space group	*Pnma*	$P2_12_12_1$
Unit cell dimensions (Å)		
a	8.7301(5)	6.2146(12)
b	28.4851	7.2413(14)
c	5.8705(4)	14.670(3)
Volume (Å^3)	1459.86(16)	660.2(2)
Z	4	4
Density (calculated) (mg m^{-3})	3.244	5.239
Absorption coefficient (mm^{-1})	22.219	48.698
F(000)	1272	872
Crsytal size (mm^3)	0.24 × 0.21 × 0.18	0.18 × 0.12 × 0.11
θ range for data collection (°)	2.86–29.05	3.14–27.94
Index ranges	$-11 \leq h \leq 11$, $-38 \leq k \leq 38$, $-8 \leq l \leq 8$	$0 \leq h \leq 8$, $0 \leq k \leq 9$, $0 \leq l \leq 19$
Reflections collected	14062	781
Independent reflections	1952 [*R*(int) = 0.0443)	81 [*R*(int) = 0.0000]
Completeness to θ	98.7%	82.1%
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	1952/0/109	781/5/51
Goodness-of-fit on F^2	1.026	1.000
Final *R* $[I > 2\sigma(I)]$	Indices for 1749 refl. $R_1 = 0.0255$, $wR_2 = 0.0565$	Indices for 645 refl. $R_1 = 0.0860$, $wR_2 = 0.1830$
R indices (all data)	$R_1 = 0.0296$, $wR_2 = 0.0581$	$R_1 = 0.1010$, $wR_2 = 0.1859$
Largest diff. peak, hole (e Å^{-3})	2.185, −1.050	4.794, −3.607
Flack's parameter	–	0.19(17)

Table 2. Selected bond lengths (Å) and angles (°) for [$Tl_2(\mu$-CSB)]$_n$ (**1**).

Tl1–O3	2.972(3)	O4–Tl1–O4$^{\mathrm{iii}}$	76.27(11)
Tl1–O3$^{\mathrm{iv}}$	2.797(3)	O3–Tl1–O4$^{\mathrm{iii}}$	117.05(11)
Tl1–O4	2.633(3)	O4$^{\mathrm{iii}}$–Tl1–O4$^{\mathrm{iv}}$	165.87(11)
Tl1–O3$^{\mathrm{iii}}$	3.024(3)	O4$^{\mathrm{iii}}$–Tl1–O3$^{\mathrm{iii}}$	82.77(11)
Tl1$\cdots$O4$^{\mathrm{iii}}$	2.718(4)	O4$^{\mathrm{iii}}$–Tl1–O3$^{\mathrm{iv}}$	87.23(11)
S1–O1	1.432(5)		
S1–O2	1.436(5)		
S1–C1	1.769(4)		

Symmetry operations: iii: $-x, -y, -z$; iv: $-x+1/2, -y, z+1/2$.

Table 3. Selected bond lengths (Å) and angles (°) for [$Tl_2(\mu$-ADC)]$_n$.

Tl1–O1	2.69(3)	O1–T1–O3$^{\mathrm{i}}$	108.8(9)
Tl1–O3$^{\mathrm{i}}$	2.71(4)	O1–Tl1–O3$^{\mathrm{ii}}$	69.5(10)
Tl1–O3$^{\mathrm{ii}}$	2.73(3)	O3$^{\mathrm{i}}$Tl1–O3$^{\mathrm{ii}}$	105.1(11)
Tl1–O2	2.91(4)	O1–Tl1–O2	47.1(8)
Tl1–O1$^{\mathrm{iii}}$	2.97(4)	O1–Tl1–O4$^{\mathrm{ii}}$	82.6(9)
Tl1–O4$^{\mathrm{ii}}$	2.92(4)	O3$^{\mathrm{i}}$–Tl1–O4$^{\mathrm{ii}}$	62.7(10)
Tl2–O3	2.67(4)	O3$^{\mathrm{ii}}$–T11–O4$^{\mathrm{iii}}$	144.3(11)
Tl2–O2$^{\mathrm{i}}$	2.71(4)	O2–Tl1–O4$^{\mathrm{iii}}$	73.5(10)
Tl2–O1$^{\mathrm{i}}$	2.73(3)	O1–Tl1–O1$^{\mathrm{iii}}$	126.8(5)
Tl2–O4$^{\mathrm{iii}}$	2.98(3)	O3$^{\mathrm{i}}$–Tl1–O1$^{\mathrm{iii}}$	65.6(9)
Tl2–O4	3.00(3)	O3$^{\mathrm{ii}}$–T11–O1$^{\mathrm{iii}}$	62.5(10)
		O2–Tl1–O1$^{\mathrm{iii}}$	94.9(9)
		O4$^{\mathrm{ii}}$–Tl1–O1$^{\mathrm{iii}}$	126.7(9)
		O3–Tl2–O2$^{\mathrm{i}}$	67.0(10)
		O2$^{\mathrm{i}}$–Tl2–O1$^{\mathrm{i}}$	96.4(9)
		O3–Tl2–O4$^{\mathrm{iii}}$	62.3(10)
		O2$^{\mathrm{i}}$–Tl2–O4$^{\mathrm{iii}}$	75.5(11)
		O1$^{\mathrm{i}}$–Tl2–O4$^{\mathrm{iii}}$	127.3(8)
		O3–Tl2–O4	49.3(7)
		O2$^{\mathrm{i}}$–Tl2–O4	114.6(9)
		O1$^{\mathrm{i}}$–Tl2–O4	73.0(10)
		O4–Tl2–O4$^{\mathrm{iii}}$	64.6(3)

Symmetry operations: i: $-x+1/2, -y, z+1/2$; ii: $-x, y+1/2, -z+1/2$; iii: $x+1/2, -y+1/2, -z$.

Acknowledgements

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