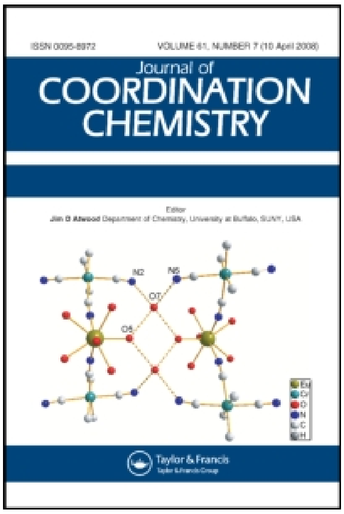

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Syntheses and structural characterization of New Tl+ and K+ complexes of 3,5-dinitrobenzoic acid (HDNB), [Tl(μ-DNB)]<i>_n and [K(μ-DNB)(μ-HDNB)]<i>_n

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Syntheses and structural characterization of New Tl^+ and K^+ complexes of 3,5-dinitrobenzoic acid (HDNB), $[Tl(\mu\text{-DNB})]_n$ and $[K(\mu\text{-DNB})(\mu\text{-HDNB})]_n$

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Two new Tl^+ and K^+ complexes of 3,5-dinitrobenzoic acid (HDNB), $[Tl(\mu\text{-DNB})]_n$ and $[K(\mu\text{-DNB})(\mu\text{-HDNB})]_n$ have been synthesized and characterized by elemental analysis, IR, ^1H NMR and ^{13}C NMR spectroscopy. Determination of the structure of the $[K(\mu\text{-DNB})(\mu\text{-HDNB})]_n$ and $[Tl(\mu\text{-DNB})]_n$ by X-ray crystallography shows that there are nine coordinate K atoms (KO_9) and eight coordinate Tl atoms (TlO_8). The $[Tl(\mu\text{-DNB})]_n$ and $[K(\mu\text{-DNB})(\mu\text{-HDNB})]_n$ complexes are 2D and 3D coordination polymers, respectively.

Keywords: Thallium; Potassium; 3,5-Dinitrobenzoic acid; Coordination polymer

1. Introduction

Tl chemistry parallels that of the alkali metals (and silver) in many ways. The solubility of the hydroxides, MOH, and its tendency to absorb CO_2 serve as examples. Many Tl and alkali-metal compounds crystallize isotypically, e.g., Tl_2SO_4 and K_2SO_4 [1]. Tl is able to replace K ions in biological systems [2], where it has an affinity of about 10 times that of potassium [3]. Therefore, it is of interest to study the interaction of Tl and K with H-acid ligands. Numerous compounds of potassium(I) [4–7] and thallium(I) [8–10] with H-acid ligands have been reported. The 3,5-dinitrobenzoic acid (HDNB) is an interesting ligand with one carboxylic and two nitro groups for coordination [11–13], a multifunction ligand that has a variety of binding geometries both as neutral (HDNB) and anionic (DNB^-) ligand. Its ability to form polynuclear complexes and spontaneous aggregation of several metal ions may also be of interest. The new Tl^+ and K^+ complexes of 3,5-dinitrobenzoic acid (HDNB), $[Tl(\mu\text{-DNB})]_n$ and $[K(\mu\text{-DNB})(\mu\text{-HDNB})]_n$ provide new 2D and 3D coordination polymers.

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2. Experimental

2.1. *Physical measurements*

IR spectra were recorded using Perkin-Elmer 597 and Nicolet 510P spectrophotometers. Microanalyses were carried out using a Heraeus C, H, N, O Rapid analyzer. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. ^{1}H and ^{13}C NMR spectra were measured with a BRUKER DRX-500 AVANCE spectrometer at 500 and 125 MHz, respectively.

2.2. *Preparation of [K(μ-DNB)(μ-HDNB)]$_n$*

The complex [K(μ-DNB)(μ-HDNB)]$_n$ was prepared by dissolving potassium hydroxide (0.056 g, 1 mmol) in ethanol (10 mL) and adding 3,5-dinitrobenzoic acid (0.106 g, 0.5 mmol). The resulting solution was stirred for 1 h at room temperature, and then allowed to stand for 10 days at room temperature. White crystals of the product precipitated, were filtered off, washed with acetone and ether, and dried in air, m.p. >350°C. Yield: 0.208 g, 45%.

Anal. Found: C, 36.70; H, 0.79; N, 12.60; Calcd for $C_{14}H_7KN_4O_{12}$: C, 36.33; H, 1.51; N, 12.11%. IR (KBr) selected bands: $\upsilon = 514$(w), 716(s), 1069(m), 1345(vs), 1375(s), 1440(m), 1510(vs), 1616(vs), 3065(m), 3420(b) cm^{-1}. ^{1}H NMR ([d$_6$]-DMSO): $\delta = 8.80$ (2H, t), 8.90 (1H, d) ppm. ^{13}C-{^{1}H} NMR ([d$_6$]-DMSO): $\delta = 118.75$, 128.79, 148.15, 164.29 and 178.36 ppm.

2.3. *Preparation of [Tl(μ-DNB)]$_n$*

The compound [Tl(μ-DNB)]$_n$ was prepared by dissolving 0.266 g (1 mmol) thallium(I) nitrate in distilled water and methanol and adding a mixture of 3,5-dinitrobenzoic acid (0.106 g, 0.5 mmol) and sodium hydroxide (0.04 g, 1 mmol) in methanol. The resulting mixture was stirred for 1 h. The solution was allowed to stand at room temperature for several days. After evaporation of the solvent, crystals were obtained (m.p. >300°C). Yield: 0.207 g, 50%.

Anal. Found: C, 20.10; H, 0.7; N, 6.73; Calcd for $C_7H_3N_20_6Tl$: found: C, 20.20; H, 0.90; N, 7.10. IR (cm^{-1}) selected bonds: $\upsilon = 660$(m), 750(s), 800(m), 1134(s), 1292(vs), 1318(vs), 1382(vs), 1473(vs), 1580(vs), 3040(w). $\delta = 8.85$ (2H, t), 8.95(1H, d) ppm. ^{13}C-{^{1}H} NMR ([d$_6$]-DMSO): $\delta = 118.70$, 128.72, 148.10, 164.28 and 178.30 ppm.

2.4. *X-ray crystallography*

X-ray measurements were made at 120(2) K using a Siemens R3m/V diffractometer. The intensity data were collected within the range $2.4 \leq \theta \leq 28°$ and $2.27 \leq \theta \leq 26.01°$ using graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) for [K(μ-DNB)(μ-HDNB)]$_n$ and [Tl(μ-DNB)]$_n$, respectively. Accurate unit cell parameters and an orientation matrix for data collection were obtained from least-squares refinement. The intensities of 5238 and 7121 unique reflections were collected, from which 3676 and 1612 with $I > 2\sigma(I)$ were used in the refinement for [K(μ-DNB)(μ-HDNB)]$_n$ and [Tl(μ-DNB)]$_n$, respectively. The structures were solved by direct methods and refined

Table 1. Crystal data and structure refinement for [K(μ-DNB)(μ-HDNB)]$_n$ and [Tl(μ-DNB)]$_n$.

Identification code	[K(μ-DNB)(μ-HDNB)]$_n$	[Tl(μ-DNB)]$_n$
Empirical formula	$C_{14}H_7KN_4O_{12}$	$C_{14}H_6N_4O_{12}Tl_2$
Formula weight	462.34	830.97
Temperature (K)	120(2)	120(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/n$
Unit cell dimensions (Å, °)		
a	14.7936(11)	4.8424(17)
b	5.8222(4)	10.413(4)
c	20.3933(15)	17.690(6)
β	94.8300(10)	94.056(7)
Volume (Å^3)	1750.3(2)	889.7(52)
Z	4	2
Density (calculated) (g cm^{-3})	1.755	3.102
Absorption coefficient (mm^{-1})	0.385	18.170
$F(000)$	936	752
Crystal size (mm^3)	0.30 × 0.30 × 0.24	0.20 × 0.15 × 0.1
θ range for data collection (°)	2.4–28.0	2.27–26.01
Index ranges	$-19 \geq h \geq 19$, $-7 \geq k \geq 7$, $-26 \geq l \geq 26$	$-5 \leq h \leq 5$, $-12 \leq k \leq 12$, $-21 \leq l \leq 21$
Reflections collected	5238	7121
Independent reflections	4208 [R(int) = 0.0216]	1612 [R(int) = 0.1444]
Absorption correction	Multi-scan	Semi-empirical from equivalents
Max. and min. transmission	0.913 and 0.893	0.913 and 0.893
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	4208/0/280	1612/6/145
Goodness-of-fit on F^2	1.036	1.050
Final R indices for ref1. [$I > 2\sigma(I)$]	$R_1 = 0.0373$, $wR_2 = 0.0874$	$R_1 = 0.0630$, $wR_2 = 0.1385$
R indices (all data)	$R_1 = 0.0418$, $wR_2 = 0.0897$	$R_1 = 0.0705$, $wR_2 = 0.1420$
Largest diff. peak, hole (e Å^{-3})	0.368, −0.262	3.461, −2.901

by full-matrix least-squares on F^2. The positions of hydrogen atoms were calculated at idealized geometrical positions and included in the structure factor calculation as fixed-atom contributions. All calculations were carried out with a PDP-11/23+ computer using the SDP-PLUS program package [14, 15].

3. Results and discussion

Upon reaction between DNBH and potassium hydroxide as well as a mixture of sodium hydroxide and thallium nitrate provided crystalline material analyzing as [K(μ-DNB)(μ-HDNB)]$_n$ and [Tl(μ-DNB)]$_n$, respectively. Crystal data and refinement parameters are given in table 1. Selected bond lengths and angles are given in tables 2 and 3. ORTEP diagrams and a perspective view of the packing in the unit cells are shown in figures 1–4. Determination of the structure of [K(μ-DNB)(μ-HDNB)]$_n$ by X-ray crystallography showed the complex in the solid state (figure 2) to be a 3D coordination polymer with the basic repeating monomeric [K(μ-DNB)(μ-HDNB)] moiety. On the other hand, the structure may be considered a coordination polymer of potassium(I) consisting of chains formed by bridging DNB$^-$ ligands. These chains are further bridged by neutral DNBH ligands resulting in a three-dimensional polymeric array.

Table 2. Selected bond lengths (Å) and angles (°) for [K(μ-DNB)(μ-HDNB)]$_n$.

K1–O2	2.5601(11)		
K1–O8	2.6150(12)		
K1–O3$^{\mathrm{ii}}$	2.9285(12)		
K1–O12$^{\mathrm{i}}$	2.9388(13)		
K1–O1	2.9430(12)		
K1–O9$^{\mathrm{ii}}$	2.9475(13)		
K1–O7	2.9604(13)		
K1–O11$^{\mathrm{ii}}$	3.0535(13)		
K1–O4$^{\mathrm{ii}}$	3.0588(13)		
O2–K1–O8	159.72(4)	O8–K1–O7	99.51(4)
O2–K1–O(3)$^{\mathrm{ii}}$	97.41(4)	O3$^{\mathrm{ii}}$–K1–O7	163.85(3)
O8–K1–O3$^{\mathrm{ii}}$	96.08(4)	O12$^{\mathrm{i}}$–K1–O7	115.91(3)
O2–K1–O12$^{\mathrm{i}}$	129.59(4)	O1–K1–O7	123.92(3)
O8–K1–O12$^{\mathrm{i}}$	70.07(4)	O9$^{\mathrm{ii}}$–K1–O7	61.26(3)
O3$^{\mathrm{ii}}$–K1–O12$^{\mathrm{i}}$	65.83(3)	O2–K1–O11$^{\mathrm{i}}$	123.13(4)
O2–K1–O1	95.69(4)	O8–K1–O11$^{\mathrm{i}}$	66.00(4)
O8–K1–O1	76.80(3)	O3$^{\mathrm{ii}}$–K1–O11$^{\mathrm{i}}$	108.07(3)
O3$^{\mathrm{ii}}$–K1–O1	63.97(3)	O12$^{\mathrm{i}}$–K1–O11$^{\mathrm{i}}$	42.28(3)
O12$^{\mathrm{i}}$–K1–O1	114.84(3)	O1–K1–O11$^{\mathrm{i}}$	141.15(3)
O2–K1–O9$^{\mathrm{ii}}$	82.77(4)	O9$^{\mathrm{ii}}$–K1–O11$^{\mathrm{i}}$	115.63(3)
O8–K1–O9$^{\mathrm{ii}}$	77.02(4)	O7–K1–O11$^{\mathrm{i}}$	74.89(3)
O3$^{\mathrm{ii}}$–K1–O9$^{\mathrm{ii}}$	127.22(3)	O2–K1–O4$^{\mathrm{ii}}$	66.74(4)
O12$^{\mathrm{i}}$–K1–O9$^{\mathrm{ii}}$	146.07(4)	O8–K1–O4$^{\mathrm{ii}}$	126.04(4)
O1–K1–O9$^{\mathrm{ii}}$	63.52(3)	O3$^{\mathrm{ii}}$–K1–O4$^{\mathrm{ii}}$	97.97(3)
O2–K1–O7	68.79(3)	O12$^{\mathrm{i}}$–K1–O4$^{\mathrm{ii}}$	69.15(4)

Symmetry codes, i: $-x+2$, $y+1/2$, $-z+3/2$; ii: $-x+1$, $-y+1$, $-z+1$.

Table 3. Intermolecular H-bonds in structure of the [K(μ-DNB)(μ-HDNB)]$_n$ complex.

D–H···A	D···A (Å)	D–H (Å)	H···A (Å)	DHA (°)
O7–H7O···O1(x, $y-1$, z)	2.482(2)	0.90	1.60	167

The potassium atoms are linked by oxygen atoms and can be considered to be nine-coordinate with K–O distances of K1–O2 = 2.5601(11), K1–O8 = 2.6150(12), K1–O3$^{\mathrm{ii}}$ = 2.9285(12), K1–O12$^{\mathrm{i}}$ = 2.9388(13), K1–O1 = 2.9430(12), K1O$^{\mathrm{ii}}$ = 2.9475(13), K1–O7 = 2.9604(13), K1–O1$^{\mathrm{ii}}$ = 3.0535(13), K1–O4$^{\mathrm{ii}}$ = 3.0588(13) Å. The intra-chain K···K distances by DNB$^-$ and "HDNB" ligands in the three-dimensional coordination polymer are 5.852, 8.397 and 9.980 Å.

The coordination modes of DNB$^-$ and HDNB ligands are different. The DNB$^-$ anion acts as a tetradentate ligand, connecting four K$^+$ ions. The carboxylate group of the DNB$^-$ ligand is bidentate, bridging two K$^+$ ions and one of the nitro groups is also bidentate bridging two other K$^+$ ions. The other nitro group of this ligand is not coordinated to K$^+$ (scheme 1a). The HDNB$^-$ ligand is also tetradentate, connecting four K$^+$ ions. The carboxylate group of the HDNB ligand is bidentate bridging two K$^+$ ions, and one nitro group is bidentate chelating to one K$^+$, whereas the other nitro group of this ligand is monodentate coordinated to K$^+$ (scheme 1b).

The complex is linked by intermolecular hydrogen bonding (table 3). The –O–H group is involved in hydrogen bonding acting as hydrogen-bond acceptors with O1

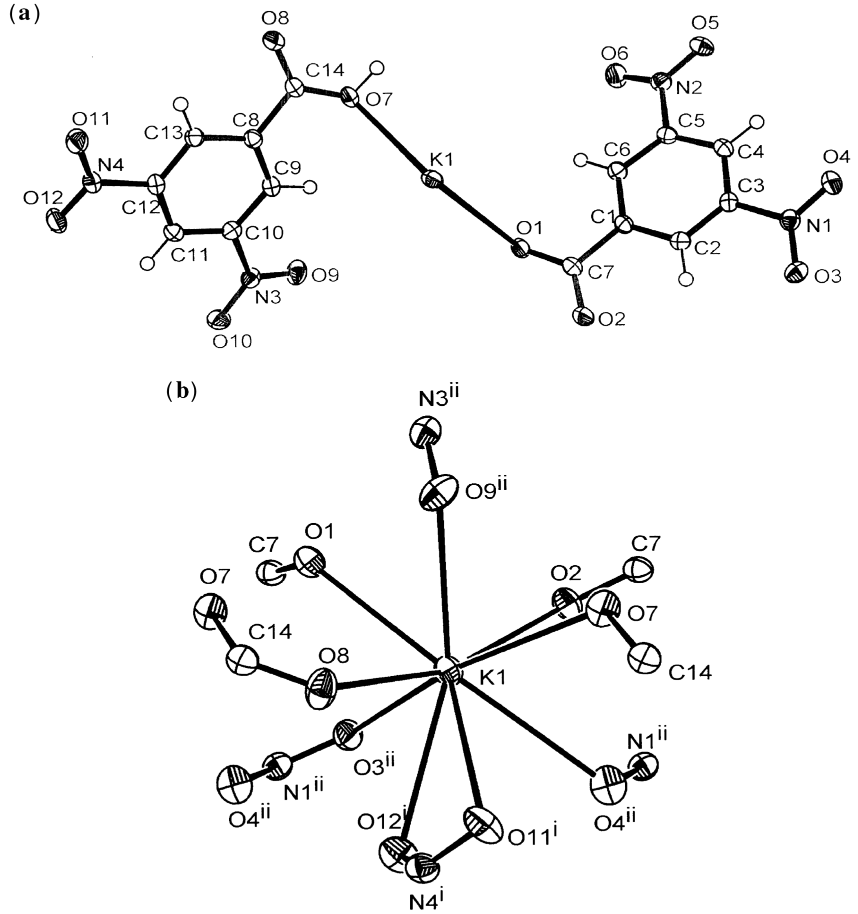


Figure 1. (a) X-ray crystal structure of $[K(\mu\text{-DNB})(\mu\text{-HDNB})]_n$ with ellipsoids of 30% probability; (b) Coordination environment around the K(I) atom.

from DNB^- as potential hydrogen-bond donors. There are also edge-to-face π–π stacking [8–9] interactions between phenyl groups of the DNB^- ligands belonging to adjacent chains in this network with the distance 3.312 Å, appreciably shorter than the normal π π stacking [16, 17].

Determination of the structure of the $[Tl(\mu\text{-DNB})]_n$ (table 1) showed the compound to be a novel two-dimensional polymer (figures 3 and 4) with six-coordinate Tl atoms (figure 3 and table 4). Each DNB^- anion is octadentate connecting eight Tl^I ions. The carboxylate groups of the DNB^- ligand are both bidentate chelating, and bridging where two oxygen atoms of the carboxylate group coordinate to a thallium(I) ion, also two of these oxygen atoms bridge to two other thallium atoms (scheme 2 and figure 4). One of the nitro groups of this ligand is only chelated to Tl^+ (bidentate); the other nitro the group of this ligand is only bridged to Tl^+ (scheme 2).

The arrangement of the oxygen atoms of the DNB^- ligands in the thallium complex suggest a vacant position in the coordination geometry around the metal ions, occupied possibly by a stereochemically-active lone pair of electrons on thallium. The observed

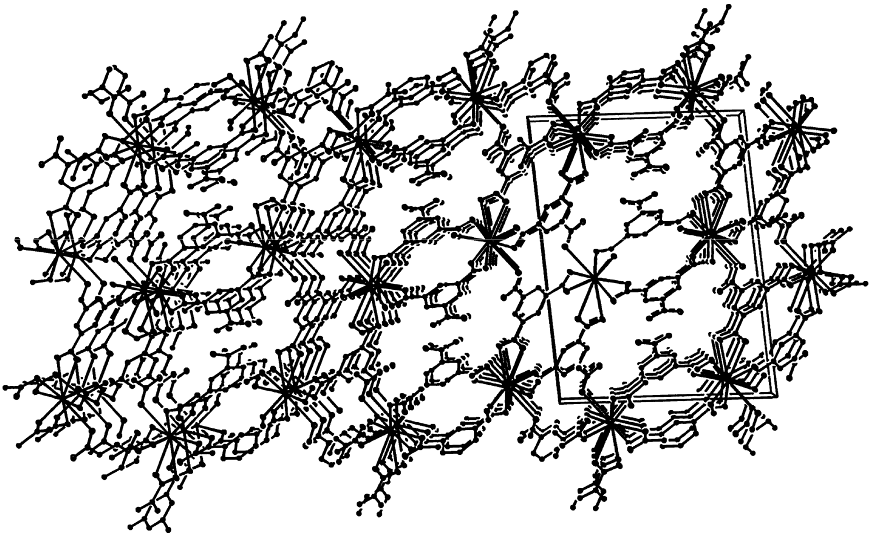


Figure 2. Packing diagram for the complex $[K(\mu_4\text{-DNB})(\mu_4\text{-HDNB})]_n$.

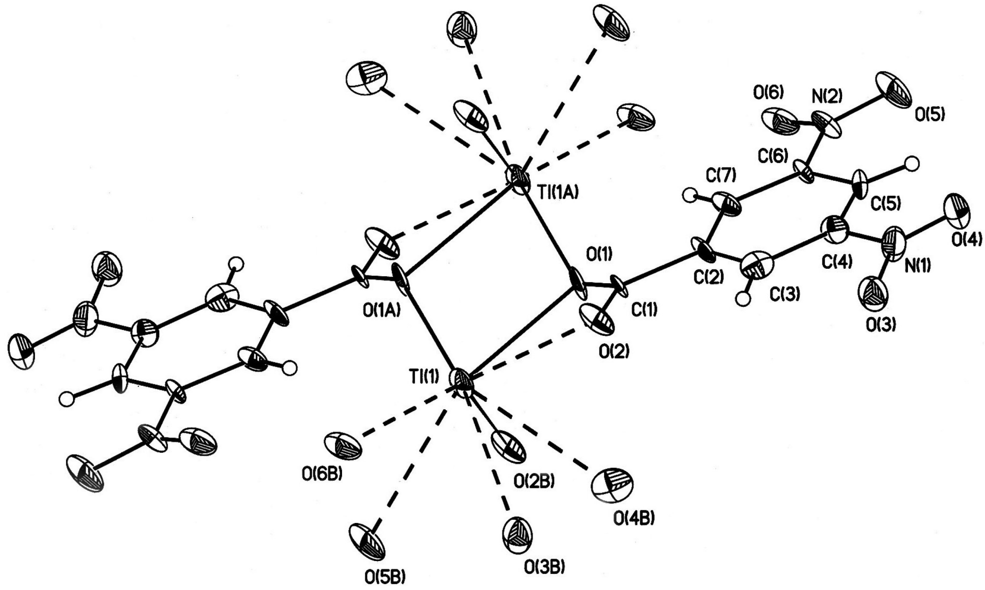


Figure 3. ORTEP diagram of $[Tl(\mu\text{-DNB})]_n$.

shortening of the Tl–O bond on the side of Tl opposite to the putative lone pair (Tl1–O = 2.562(9)–2.780(10) compared with Tl1–O = 3.038(11)–3.462(11) Å adjacent to the lone pair) supports the presence of this feature [18]. The presence of a lone pair on the thallium, is apparently the reason that the bridging interactions are so long. If the stereochemically active lone pair was not present, there might be more interactions between bridging atoms of thallium(I) with the neighboring oxygen atoms

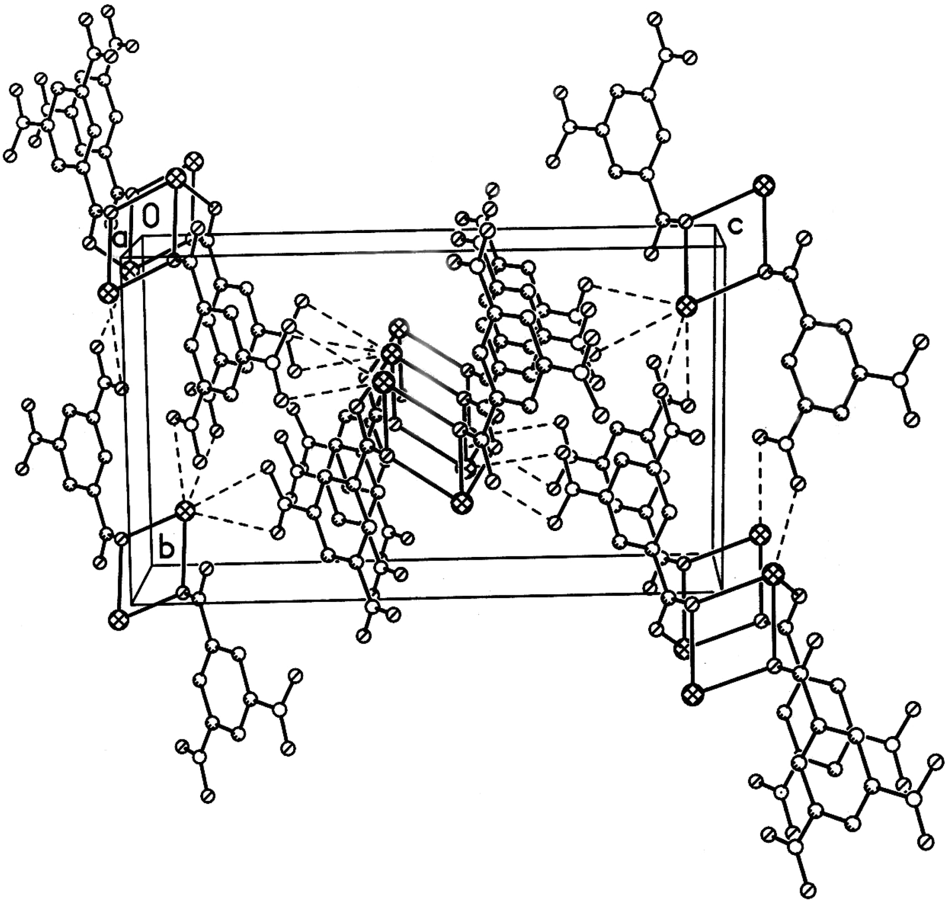


Figure 4. Unit cell of the [Tl(μ-DNB)]$_n$.

of the DNB^{-} ligands. However, the lone pair (6s^{2}) in this complex is active [19]. Hence, the geometry of the nearest coordination environment of every thallium atom is likely caused by the geometrical constraints of coordinated DNB^{-} ligands and by the influence of a stereo-chemically active lone pair of electrons. Such an environment leaves space for bonding of oxygen atoms of nitrite of adjacent molecules (figure 3).

In summary, the structures of [K(μ-DNB)(μ-HDNB)]$_n$ and [Tl(μ-DNB)]$_n$ represent new three-dimensional and two-dimensional polymers with unusual bridging of carboxylate and nitro groups with nine- and eight-coordinated K^{I} and Tl^{I}, respectively.

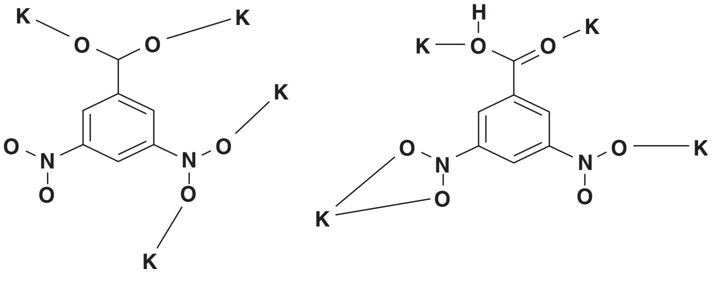
Supplementary material

Complete bond lengths and angles, co-ordinates and displacement parameters have been deposited at Cambridge Crystallography Data Centre. Supplementary data are available from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK on request,

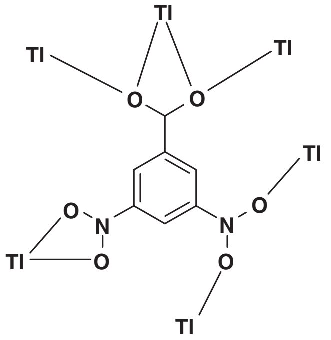
Table 4. Selected bond lengths (Å) and angles (°) for $[Tl(\mu\text{-DNB})]_n$.

Tl(1)–O(1)	2.562(9)
Tl(1)–O(2)#1	2.687(10)
Tl(1)–O(1)	2.780(10)
Tl(1)–O(2)	3.038(11)
Tl(1)–O(5)#1	3.060(12)
Tl(1)–O(4)#4	3.063(13)
Tl(1)–O(3)#4	3.210(12)
Tl(1)–O(6)#5	3.462(11)
O(1)–Tl(1)–O(2)#1	84.9(3)
O(1)–Tl(1)–O(1)#2	77.2(3)
O(2)#1–Tl(1)–O(1)#2	74.5(3)
O(1)–Tl(1)–O(2)	45.7(3)
O(2)#1–Tl(1)–O(2)	115.4(4)
O(1)#2–Tl(1)–O(2)	117.2(3)
O(1)–Tl(1)–O(5)#3	154.9(3)
O(2)#1–Tl(1)–O(5)#3	71.4(3)
O(1)#2–Tl(1)–O(5)#3	103.5(3)
O2)–Tl(1)–O(5)#3	139.2(3)
O(1)–Tl(1)–O(4)#4	76.3(3).
O(2)#1–Tl(1)–O(4)#4	73.3(3)
O(1)#2–Tl(1)–O(4)#4	139.6(3)
O(2)–Tl(1)–O(4)#4	58.8(3)
O(5)#3–Tl(1)–O(4)#4	88.7(4)
O(1)–Tl(1)–O(3)#4	111.0(3)
O(2)#1–Tl(1)–O(3)#4	96.1(3)
O(1)#2–Tl(1)–O(3)#4	167.3(3)
O(2)–Tl(1)–O(3)#4	74.3(3)
O(5)#3–Tl(1)–O(3)#4	64.9(3)
O(4)#4–Tl(1)–O(3)#4	40.1(3)
O(1)–Tl(1)–O(6)#5	136.1(3)
O(2)#1–Tl(1)–O(6)#5	136.4(3)
O(1)#2–Tl(1)–O(6)#5	121.1(3)
O(2)–Tl(1)–O(6)#5	94.0(3)
O(5)#3–Tl(1)–O(6)#5	65.5(3)
O(4)#4–Tl(1)–O(6)#5	99.1(3)
O(3)#4–Tl(1)–O(6)#5	60.2(3)

Symmetry codes, 1: $x+1$, y, z; #2: $-x+2$, $-y$, $-z+2$; #3: $x+1$, $y+1$, z; #4: $-x+1/2$, $y+1/2$, $-z+3/2$; #5: x, $y+1$, z.



Scheme 1. Potassium coordination to DNB (a) and HDNB (b).



Scheme 2. Thallium coordination to DNB$^-$.

quoting the deposition number 270823 for $[K(\mu\text{-DNB})(\mu\text{-HDNB})]_n$ and 288930 for $[Tl(\mu\text{-DNB})]_n$.

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