

Synthesis and Structure of a Polymeric Homobimetallic Mercury Compound Constructed from (*E*)-Acetyloxy{4-[(pyridin-2-ylmethylene)amino]phenyl}mercury(II)

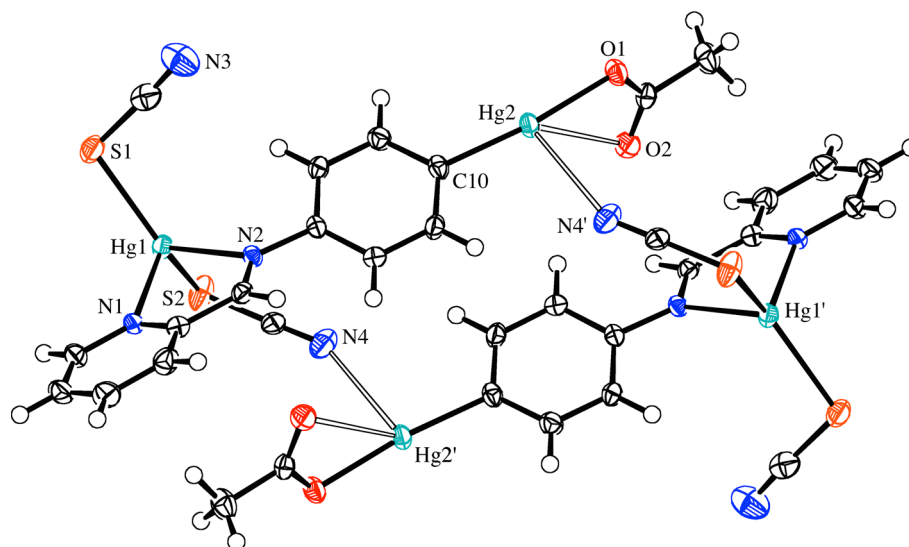
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Abstract A two-dimensional homobimetallic mercury coordination polymer (2-[[4-(acetyloxymethyl)phenyl]imino]methyl}pyridine- κ^2 N,N')dithiocyanatomercury(II) (**1**) was synthesized and characterized by FT-IR, ¹H-NMR spectroscopic and single crystal X-ray diffraction techniques. Crystallographic results reveal that the primary structural building unit in the structure is a centrosymmetric tetranuclear entity,

where long Hg–N bonds link a thiocyanate ligand of the dinuclear asymmetric unit to the acetate coordinated Hg-atom of its neighbor. These tetranuclear units are then linked into extended chains and the chains are cross-linked by weaker Hg...O interactions to give a two-dimensional polymeric structure.

Graphical Abstract Part of the structure of the 2-D



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bimetallic mercury coordination polymer constructed from the reaction of (*E*)-acetyloxy{4-[(pyridin-2-ylmethylene)amino]phenyl}mercury(II) with Hg(SCN)₂ is reported.

Keywords Single-crystal X-ray diffraction · Structural building unit · Organomercury · Homobimetallic · N, N'-donor · Organomercurio-ligand

Introduction

The coordination chemistry of mercury has received considerable attention, in part due to concerns regarding the impact on the environment and on health [1, 2]. Complexes of mercury(II) have been used for the detection of heavy metals and also for their removal. On the other hand, the coordination and structural chemistry of mercury is also well investigated: mercury(II) with a large ionic radius as a consequence of the filled 4*f* and 3*d* electron shells, displays highly varied coordination numbers and flexible and often distorted coordination geometries, although tetrahedral geometry appears frequently. Consequently, mercury can readily accommodate all kinds of architectures in coordination polymers, including 1D, 2D and 3D topologies [3, 4], with 1D coordination polymers occurring more frequently than 3D polymers [4]. Moreover, the effects related to the geometry, size, number of donor atoms and rigidity of ligands, as well as the number and size of anions, are important contributors to the greater number of one-dimensional coordination polymers [4].

In this perspective, mercury complexes of Schiff-base ligands, such as (*E*)-*N*-(pyridin-2-ylmethylene)arylamine, have received attention owing to their distinctive coordination and structural properties [5–7]. Recent X-ray crystallographic studies performed on a variety of such mercury(II) compounds, viz. $[\text{HgL}(\text{X})_2]$, $[\text{HgL}(\text{X})_2(\text{S})]$, $[\text{HgL}(\text{X})_2]_2$, $[\text{HgL}(\text{X})_2]_n$, $\{[\text{HgL}(\text{X})_2]_2\}_n$ and $\{[\text{HgL}(\text{X})_2]_2 \cdot \text{Hg}(\text{X})_2\}_n$ (*X* = halides, azides or nitrates and *S* = coordinating solvent; Scheme 1), revealed that these complexes are promising for the generation of interesting and diverse supramolecular assemblies. These assemblies are produced through the formation of coordination bonds, hydrogen bonds and other intermolecular interactions, thereby creating a large variety of supramolecular architectures [5–7]. Although the global topology (monomer, dimer, polymer, etc.) of the resulting compound is determined by the interplay between Hg^{2+} , X^- , and *L*, in the solid-state, the crystal structure is frequently controlled by weaker intermolecular interactions such as π – π and CH – π contacts [5–7]. We have recently described the isolation and characterization of an organomercurio-ligand: 2-[[4-(chloromercuryl)phenyl]iminomethyl]pyridine (*L*) which was used for the construction of bimetallic mercuric chloride compounds, (2-[[4-(chloromercuryl)phenyl]iminomethyl]pyridine- $\kappa^2\text{N},\text{N}'$)dichloromercury(II) (dimethylsulfoxide- κO) ($\text{LHgCl}_2 \cdot \text{DMSO}$) and (2-[[4-(chloromercuryl)phenyl]iminomethyl]pyridine- $\kappa^2\text{N},\text{N}'$)dichloromercury(II) (dimethylformamide- κO) ($\text{LHgCl}_2 \cdot \text{DMF}$), and cadmium chloride compound, (2-[[4-(chloromercuryl)phenyl]iminomethyl]pyridine- $\kappa^2\text{N},\text{N}'$)dichlorocadmium(II) (dimethylformamide- κO) ($\text{LCdCl}_2 \cdot \text{DMF}$) [8]. As a part of an on-going

investigation into the synthesis and structures of bimetallic mercury compounds, we now report the reaction of (*E*)-acetyloxy{4-[(pyridin-2-ylmethylene)amino]phenyl}mercury(II) [8] with $\text{Hg}(\text{SCN})_2$, which provided a polymeric homobimetallic compound (2-[[4-(acetyloxymercuryl)phenyl]iminomethyl]pyridine- $\kappa^2\text{N},\text{N}'$)dithiocyanatomercury(II) (**1**) (Scheme 2). The structure of **1** has been elucidated from spectroscopic and X-ray diffraction results.

Experimental

Materials

Caution! Compounds of mercury are highly toxic [9]. Care must be taken when handling samples, and appropriate disposal procedures are necessary. All chemicals were used as purchased without purification: pyridine-2-carbaldehyde (Merck), mercuric acetate (Loba chemicals), mercuric thiocyanate (Loba Chemie), except for aniline (Sd Fine), which was freshly distilled prior to use. Solvents were purified by standard procedures and were freshly distilled prior to use.

Physical Measurements

Melting points were recorded in capillary tubes on a Scanca apparatus and are uncorrected. Elemental analyses were performed using a Perkin Elmer 2400 series II instrument. IR spectra in the range 4000–400 cm^{-1} were obtained on a Perkin Elmer Spectrum BX series FT-IR spectrophotometer with samples investigated as KBr discs. The ^1H NMR spectra were recorded on a Bruker Avance II spectrometer and measured at 400.13 MHz. The ^1H chemical shifts were referenced to Me_4Si set at 0.00 ppm.

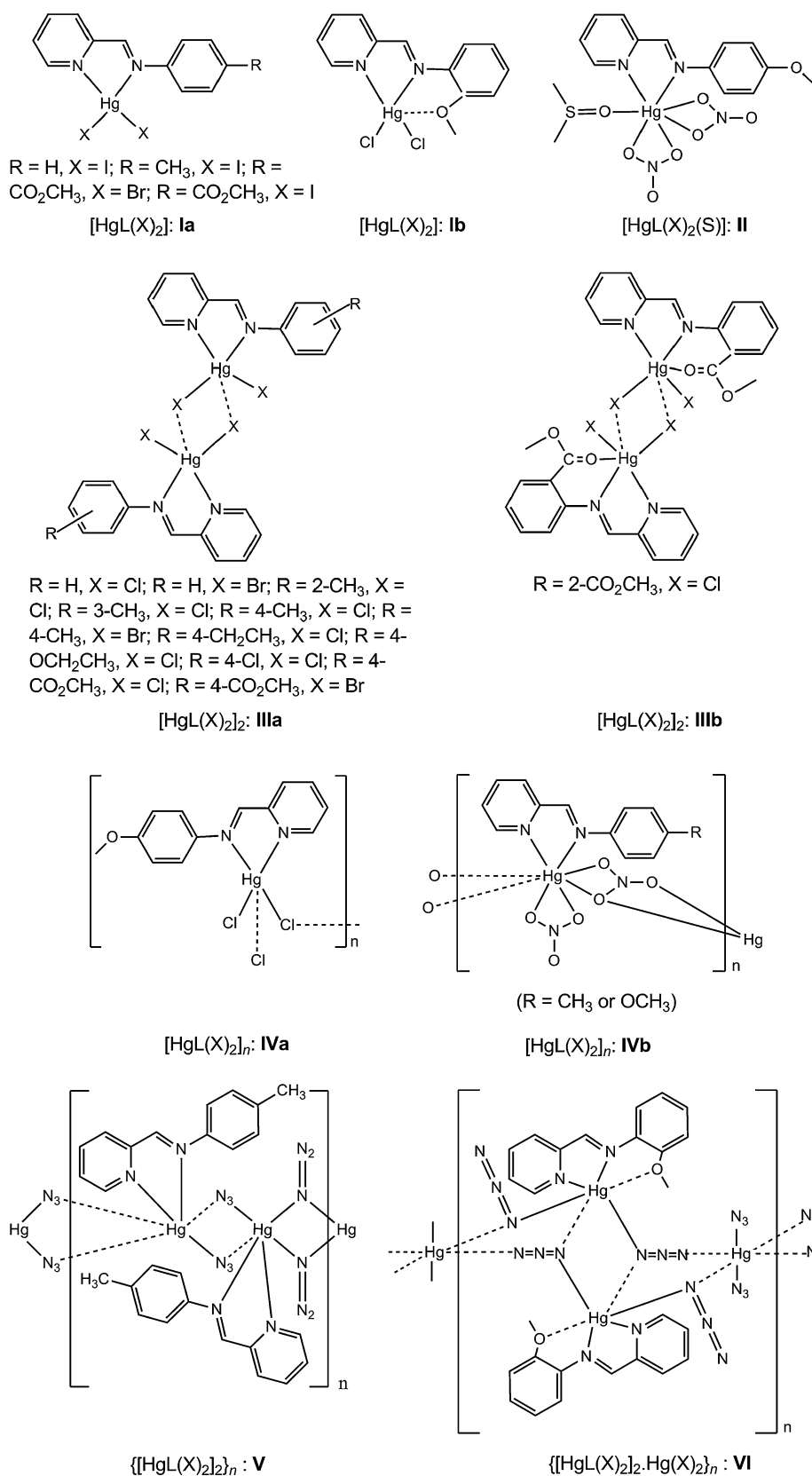
Synthesis of (*E*)-Acetyloxy{4-[(pyridin-2-ylmethylene)amino]phenyl}mercury(II)

The compound was prepared by reacting pyridine-2-carbaldehyde and acetyloxy(4-aminophenyl)mercury(II) in ethanol under reflux conditions [8]. The spectroscopic data of the compound reported in Ref. [8] correspond well within the experimental errors with those of this freshly prepared sample.

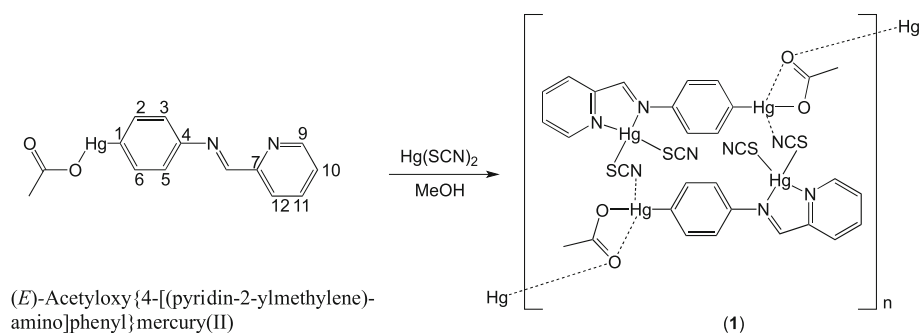
Synthesis of (2-[[4-(Acetyloxymercuryl)phenyl]iminomethyl]pyridine- $\kappa^2\text{N},\text{N}'$)dithiocyanatomercury(II) (**1**)

To a hot Solution of (*E*)-acetyloxy{4-[(pyridin-2-ylmethylene)amino]phenyl}mercury(II) (0.50 g, 1.13 mmol) in methanol (15 ml) was added a solution of $\text{Hg}(\text{SCN})_2$

Scheme 1 Schematic representation of various structural motifs (**I–VI**) observed in mercury(II) compounds with (*E*)-*N*-(pyridin-2-ylmethylidene)arylamine



Scheme 2 Reaction showing the synthesis of polymeric homobimetallic mercury thiocyanato compound **1**



(0.36 g, 1.13 mmol) in methanol (20 ml) under stirring conditions which resulted in the immediate formation of a pale yellow precipitate. The stirring was continued for 4 h. The precipitate was filtered and washed with methanol (3 × 5 ml) and dried in vacuo. The product was insoluble in common organic solvents. Yield: 78 % (0.67 g). M.p.: 431–433 K (dec.). Anal. Found: C, 25.50; H, 1.75; N, 7.60 %. Calc. for $C_{16}H_{12}Hg_2N_4O_2S_2$: C, 25.37; H, 1.60; N, 7.40 %. IR (KBr cm^{-1}): 2139, 2108 $\nu_{as}(CN(Hg-SCN))$, 1627 $\nu_{as}(OCO)$, 1586 $\nu_{as}(C(H)=N)$, 1483, 1440 $\nu(C=N)py$. 1H NMR (DMSO- d_6): 9.05 [s, 1H, CH=N], 8.89 [d, $J = 5.0$ Hz, 1H, H-9], 8.13 [t, 1H, H-11], 8.04 [d, $J = 7.0$ Hz, 1H, H-12], 7.76 [t, 1H, H-10], 7.51 [d, 4H, H-2,6,3,5], 1.98 [s, 3H, CH_3CO_2] ppm.

Crystal Structure Determination

Crystals of compound **1** suitable for an X-ray crystal-structure determination were obtained from the slow evaporation of an acetonitrile/DMF solution (1:1) of the compound at room temperature. The measurement was made at low temperature on an Agilent Technologies Super Nova area-detector diffractometer [10] using Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å) from a micro-focus X-ray source and an Oxford Instruments Cryojet XL cooler. Data reduction was performed with CrysAlisPro [10]. The intensities were corrected for Lorentz and polarization effects, and a numerical absorption correction [11] was applied. Equivalent reflections were merged. The data collection and refinement parameters are given in Table 1. The structure was solved by direct methods using SHELXS97 [12], which revealed the positions of all non-hydrogen atoms. The non-hydrogen atoms were refined anisotropically. All H-atoms were placed in geometrically calculated positions and refined by using a riding model where each H-atom was assigned a fixed isotropic displacement parameter with a value equal to $1.2U_{eq}$ of its parent C-atom ($1.5U_{eq}$ for the methyl group). The refinement of the structure was carried out on F^2 by using full-matrix least-squares procedures, which minimized the function $\sum w(F_o^2 - F_c^2)^2$. A correction for secondary

Table 1 Crystallographic data and structure refinement parameter for **1**

Empirical formula	$C_{32}H_{24}Hg_4N_8O_4S_4$
Formula weight	1515.19
Crystal size (mm)	$0.05 \times 0.14 \times 0.20$
Crystal color, habit	Red, tablet
Temperature (K)	160(1)
Crystal system	Monoclinic
Space group	$P2_1/c$
a (Å)	14.8405(2)
b (Å)	8.20466(10)
c (Å)	15.8137(2)
β (°)	105.4188(15)
V (Å ³)	1856.19(4)
Z	2
D_x (g cm ⁻³)	2.711
μ (mm ⁻¹)	16.765
Transmission factors (min, max)	0.081; 0.428
θ range for data collection (°)	2.6–29.0
Reflections measured	19,943
Independent reflections (R_{int})	4416 (0.028)
Reflections with $I > 2\sigma(I)$	4049
Number of parameters	236
$R(F)$ ($I > 2\sigma(I)$ reflns)	0.0213
$wR(F^2)$ (all data)	0.0492
$GOF(F^2)$	1.066
Max, min $\Delta\rho$ (e/Å ³)	3.07; -1.10

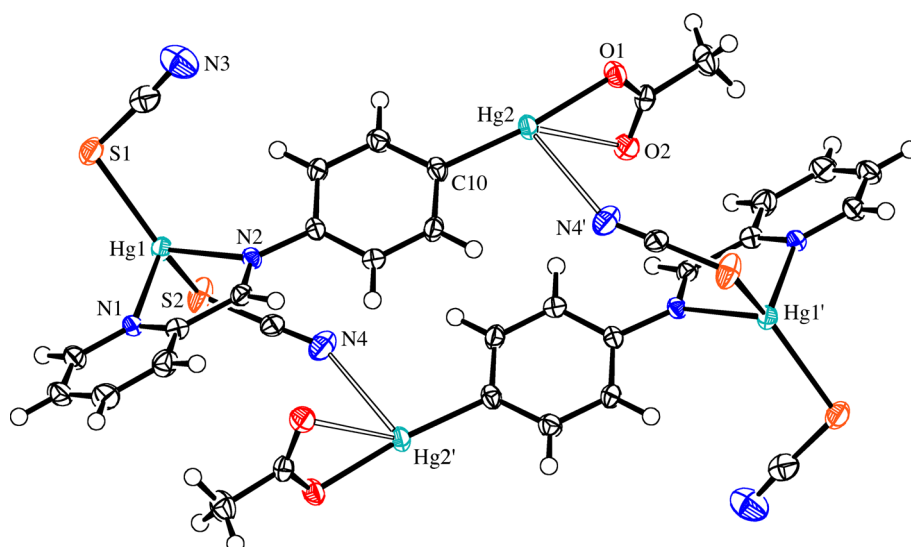
extinction was not applied. The SHELXL2013 program [13] was used for all calculations.

Results and Discussion

Synthetic and Spectroscopic Aspects

Recently, we have reported the synthesis and structures of the bimetallic compounds $LHgCl_2 \cdot DMSO$, $LHgCl_2 \cdot DMF$

Fig. 1 The structure of the tetranuclear SBU of **1** (50 % probability ellipsoids for the non-H atoms). The asymmetric unit is half of this. The symmetry operators for the primed atoms are as defined in Table 2



and $\text{LCdCl}_2\cdot\text{DMF}$, which were synthesized from the metallo-ligand precursor 2-[[4-(4-chloromercuryl)phenyl]iminomethyl]pyridine (L) [8]. In another endeavor, a metallo-ligand (*E*)-acetyloxy{4-[(pyridin-2-ylmethylene)amino]phenyl}mercury(II) was prepared by reacting acetyloxy(4-aminophenyl)mercury with pyridine-2-carbaldehyde in absolute ethanol according to the usual condensation reaction [8]. The metallo-ligand of the present investigation also contains imino- and pyridyl nitrogen atoms, which are capable of coordinating to additional metal atom. In view of this, reaction of the metallo-ligand with $\text{Hg}(\text{SCN})_2$ was attempted in order to obtain a bimetallic derivative. The compound (2-[[4-(acetyloxymercuryl)phenyl]iminomethyl]pyridine- $\kappa^2\text{N},\text{N}'$)dithiocyanatomercury(II) (**1**) (Scheme 2) has been isolated and characterized using IR and ^1H NMR spectroscopy. The ^1H NMR spectrum of **1** revealed the presence of the metallo-ligand skeleton. In the IR spectrum of **1**, the $\nu_{\text{as}}(\text{CN})$ stretch of the thiocyanate ligand appears at 2139 and 2108 cm^{-1} . This indicates S-coordination of the bound thiocyanate to the mercury atom [14, 15]. This hypothesis regarding the coordination behavior of the ambidentate pseudohalide is corroborated further by the single crystal X-ray crystallography.

Crystal Structure

Complex **1** is a two-dimensional coordination polymer. The asymmetric unit contains two chemically and structurally distinct Hg-atoms, two thiocyanate ligands, one acetate ligand and one imine ligand, L (Fig. 1). Within the asymmetric unit, atom Hg(1) is coordinated in a distorted tetrahedral fashion by the S-atoms of two thiocyanate ligands, and the two N-atoms of L acting in a bidentate chelating mode (Table 2). The small bite angle of

$71.89(11)^\circ$ of the chelating N-atoms contrasts with the large angle of $129.56(4)^\circ$ subtended by the two thiocyanate S-atoms at Hg(1). The primary coordination sphere of atom Hg(2) contains the carboxy O-atom, O(1), of the acetate ligand and the *para*-positioned C-atom of the phenyl ring of L disposed in an almost linear arrangement with the angle at Hg(2) being $173.82(14)^\circ$. However, atom Hg(2) is involved in three additional longer, but still significant interactions (Table 2), given that the sums of the van der Waals radii of Hg and N, and Hg and O are approximately 3.3 \AA [8]. The carbonyl O-atom, O(2), of the acetate ligand is $2.848(3)\text{ \AA}$ from Hg(2), thereby completing an asymmetric bidentate chelation of this ligand to Hg(2), while O(2) from another asymmetric unit, O(2''), is involved in a long $\text{Hg}\cdots\text{O}$ interaction of $2.888(3)\text{ \AA}$ to atom Hg(2); atom O(2) thus weakly bridges two Hg(2) atoms. The fifth interaction with Hg(2) is a long $\text{Hg}(2)\text{--N}(4')$ bond of $2.726(4)\text{ \AA}$ involving a thiocyanate N-atom from a third asymmetric unit. The overall coordination geometry at Hg(2) cannot easily be described by any classical shape, but is perhaps most closely associated with a distorted square pyramid where atom N(4') is at the apex. A similar asymmetric geometry for a bidentate acetate ligand and long linking Hg–N distance were observed in the polymeric chain structure of acetyloxy(4-aminophenyl)mercury(II) [8].

The primary structural building unit (SBU) in the structure can be considered to be a centrosymmetric tetranuclear entity, where the $\text{Hg}(2)\text{--N}(4')$ bond links a thiocyanate ligand of the dinuclear asymmetric unit to the acetate-coordinated Hg-atom, Hg(2), of its centrosymmetrically-related neighbor and vice versa (Fig. 1). These SBUs are then linked into extended chains running parallel to the [010] direction by the weak $\text{Hg}(2)\cdots\text{O}(2'')$ type

Table 2 Selected bond lengths (Å) and angles (°) in the structures of compound **1** and the related bimetallic compounds LHgCl₂·DMSO, LHgCl₂·DMF and LCdCl₂·DMF

	1	LMgCl ₂ ·DMSO	LMgCl ₂ ·DMF	LCdCl ₂ ·DMF
M(1)–N(1)	2.363(3)	2.310(4)	2.296(3)	2.319(2)
M(1)–N(2)	2.353(3)	2.464(4)	2.505(3)	2.417(2)
M(1)–X(1)	2.4624(12)	2.4679(14)	2.4283(8)	2.4426(6)
M(1)–X(2)	2.4433(11)	2.4172(14)	2.4629(8)	2.4544(7)
Hg(2)–C(10)	2.046(4)	2.057(5)	2.062(3)	2.057(3)
Hg(2)–O(1)	2.085(3)	–	–	–
Hg(2)···O(2)	2.848(3)	–	–	–
Hg(2)–N(4')	2.762(4)	–	–	–
Hg(2)···O(2'')	2.888(3)	–	–	–
Hg(1)···N(3*)	3.082(5)	–	–	–
N(1)–M(1)–X(1)	105.12(9)	112.42(12)	134.11(7)	132.32(6)
N(1)–M(1)–X(2)	116.76(8)	132.92(12)	116.44(7)	117.22(6)
N(2)–M(1)–X(1)	113.63(8)	97.77(11)	105.46(6)	104.08(5)
N(2)–M(1)–X(2)	105.45(8)	107.24(11)	105.90(6)	101.10(5)
N(1)–M(1)–N(2)	71.89(11)	70.82(15)	70.87(9)	71.64(8)
X(1)–M(1)–X(2)	129.56(4)	114.37(5)	108.60(3)	110.25(2)
N(2)–Hg(1)···N(3*)	155.18(12)	–	–	–
Hg(1)···N(3*)–C(15*)	93.3(3)	–	–	–
C(10)–Hg(2)–O(1)	173.82(14)	–	–	–
C(10)–Hg(2)···O(2)	133.01(12)	–	–	–
C(10)–Hg(2)···O(2'')	99.74(12)	–	–	–
C(10)–Hg(2)–N(4')	98.64(15)	–	–	–
O(1)–Hg(2)···O(2)	50.77(10)	–	–	–
O(1)–Hg(2)···O(2'')	79.03(10)	–	–	–
O(1)–Hg(2)–N(4')	86.70(12)	–	–	–
O(2)–Hg(2)···O(2'')	123.04(7)	–	–	–
O(2)–Hg(2)–N(4')	80.24(11)	–	–	–
N(4')–Hg(2)···O(2'')	70.70(10)	–	–	–
Hg(2)–O(2)···O(2'')	109.63(10)	–	–	–

M = Hg for LHgCl₂·DMF, LCdCl₂·DMF and **1** and Cd for LCdCl₂·DMF; X = Cl for LHgCl₂·DMSO, LHgCl₂·DMF and LCdCl₂·DMF and X = S for **1**

Primed atoms refer to the molecule in the following symmetry related positions for **1**: ' 1 – x, –y, 1 – z; '' 1 – x, – $\frac{1}{2}$ + y, $\frac{3}{2}$ – z; * –x, $\frac{1}{2}$ + y, – $\frac{1}{2}$ – z

Refer to Ref. [8] for details of compounds LHgCl₂·DMSO, LHgCl₂·DMF and LCdCl₂·DMF

interactions. Each SBU is involved at each end in two such chain-forming interactions, with the net result that a two-dimensional coordination polymer is formed with the sheets lying parallel to (100) (Fig. 2). The sheets are linked into the overall three-dimensional supramolecular framework by weak C–H···N interactions involving atoms C(5) and N(3*) of adjacent sheets with a H···N distance of 2.69 Å and a C–H···N angle of 137° (Fig. 3; see Table 2 for the symmetry operator for N(3*)). Across the same gap between the sheets, atom N(3*) is only 3.082(5) Å from the opposing atom Hg(1) and the thiocyanate ligand containing atom N(3*) lies partially within the opening at Hg(1) afforded by the wide S(2)–Hg(1)–S(2) angle. Potentially, this contact could also be contributing to the three-

dimensional coherence of the structure, although the Hg(1)···N(3*)–C(15*) angle is rather acute at 93.3(3)°. If this contact is considered in the outer coordination sphere of Hg(1), the coordination geometry could be described as highly distorted trigonal bipyramid.

Compound **1** is a further example of the series of similar bimetallic complexes whose crystal structures were reported earlier (compounds LHgCl₂·DMSO, LHgCl₂·DMF and LCdCl₂·DMF) [8]. In these complexes, chloride ligands were present instead of thiocyanate and either dimethylformamide (DMF) or dimethylsulfoxide, the solvents used for crystallization, had become coordinated to atom Hg(1) (atom Cd(1) in LCdCl₂·DMF), which was thus five-coordinate. In **1**, despite being crystallized from DMF/

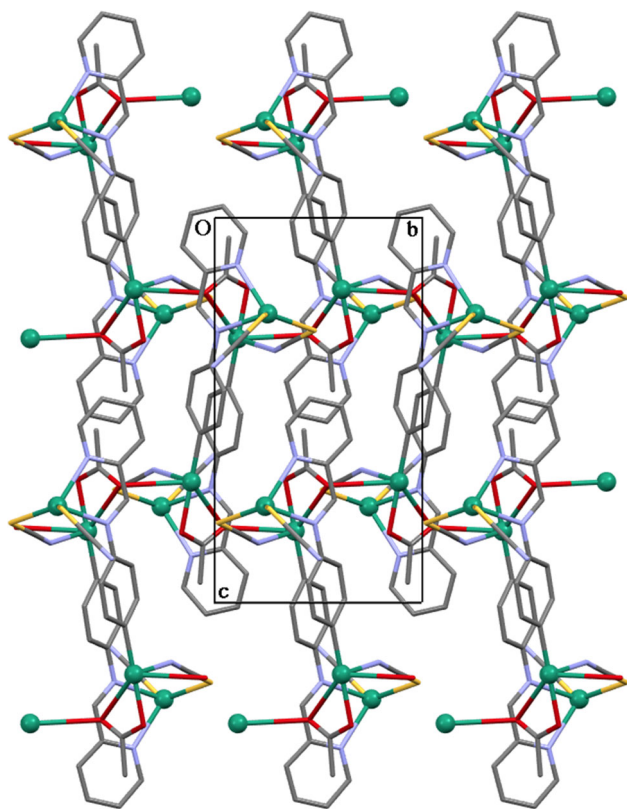


Fig. 2 One layer of the two-dimensional coordination polymer in the structure of **1**, viewed down the *a*-axis. The Hg atoms are the *cyan*-colored spheres and the Hg...O linkages between SBUs are clearly visible as the near-horizontal *cyan-red* bonds, some of which are terminating in this view (Color figure online)

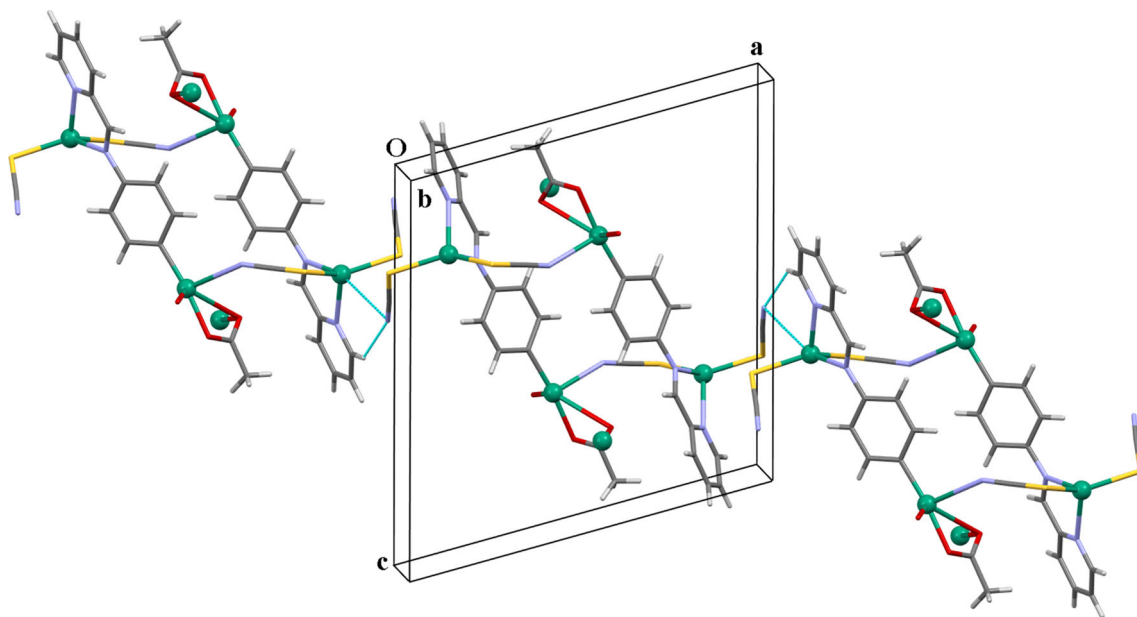


Fig. 3 The weak Hg...N and C-H...N interactions (*blue dashed lines*) linking the tetranuclear SBUs of **1** to complete the three-dimensional supramolecular framework. The two-dimensional sheets resulting from

acetonitrile, DMF is not incorporated into the complex and Hg(1) is four-coordinate with a wide S–Hg–S angle accommodating the weak inter-sheet interactions described above. Atom Hg(2) in **1** is five-coordinate, including three weaker long interactions, whereas in LHgCl₂·DMSO, LHgCl₂·DMF and LCdCl₂·DMF, this atom is four-coordinate with two weak interactions to neighboring asymmetric units. Whereas **1** is a two-dimensional coordination polymer in which all linkages within the polymer involve only atom Hg(2), LHgCl₂·DMSO, LHgCl₂·DMF and LCdCl₂·DMF are one-dimensional polymers in which the connections involve a route through both Hg-atoms (the Hg and Cd-atoms in LCdCl₂·DMF) [8]. One can speculate upon the cause of the different polymeric structure found for **1** and surmise that the steric bulk of the thiocyanate ligands, compared with the chloride ligands present in the other structures, might be a contributing factor.

There are two further notable differences between the structure of **1** and those of LHgCl₂·DMSO, LHgCl₂·DMF and LCdCl₂·DMF. In the L ligand of LHgCl₂·DMSO, LHgCl₂·DMF and LCdCl₂·DMF, the imine Hg–N bonds were found to be consistently longer than the pyridine Hg–N bonds (Table 2), whereas the Hg–N distances in **1** are equivalent. The imine ligand in **1** is twisted significantly from a planar conformation, with the dihedral angle between the planes of the phenyl and pyridyl rings being 37.6(2)°, compared with a more planar arrangement in the other structures, for example the corresponding dihedral angle in LHgCl₂·DMF is 11.5(3)°. The planes of the

the Hg(2)···O(2'') interaction lie parallel to the *bc*-plane and the linkages to the Hg(2) atom of the adjacent SBUs in the sheet are included. The Hg atoms are the *cyan*-colored spheres (Color figure online)

chelating five-membered ring and the pyridine ring in **1** have a dihedral angle of $6.8(3)^\circ$, similar to the values found in the other structures.

Supplementary Materials

CCDC-1035223 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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