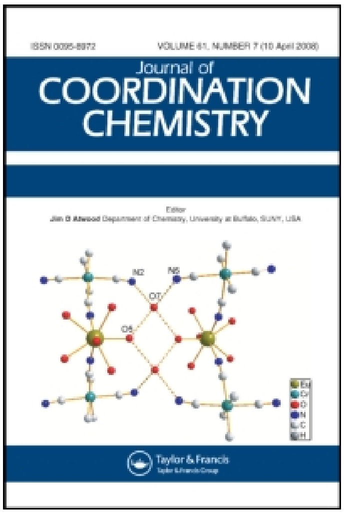

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Mercury(II) and Lead(II) complexes with 2,2′-Bis (4,5-dimethylimidazole) – syntheses, characterization and crystal structures of [M(DmImH)(SCN)$_2$], (M = Hg^{2+} and Pb^{2+})

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Mercury(II) and lead(II) complexes with 2,2′-bis(4,5-dimethylimidazole) (DmImH), [Hg(DmImH)(SCN)$_2$] and [Pb(DmImH)(NCS)$_2$], have been synthesized and characterized by IR specta and elemental analyses. The molecular structure of [Pb(DmImH)(NCS)$_2$]$_n$ is polymeric with four-coordinate lead atoms. The [Hg(DmImH)(SCN)$_2$] complex is built up of monomeric Hg(SCN)$_2$ units with one "DmImH" ligand coordinated to the Hg atom *via* the two N atoms in a distorted tetrahedral environment. The thiocyanate ligands are coordinated to lead *via* nitrogen, but to mercury *via* the sulfur. There are π–π stacking interactions between the parallel aromatic rings in the mercury(II) complex.

Keywords: Lead(II); Mercury(II); Crystal structure; 2,2′-Bis(4,5-dimethylimidazole) ligand

1. Introduction

The ability of mercury(II) and lead(II) to form a wide variety of 1 : 1 and 1 : 2 complexes with neutral ligands is well known [1–5]. Most of these complexes contain halide or thiocyanate [6–8]. The latter is an ambidentate ligand and can coordinate *via* both the N atom and the S atom. The coordination mode depends on the nature of the metal center, hence N-donor atoms are found in hard acid complexes such as Zn^{2+}, while in soft acid complexes such as Hg^{2+}, the S atom is the ligating site [9]. In complexes of the lead(II) and mercury(II), the electronic configuration difference is in the 6s^2 orbital. This lone pair exhibits inert pair properties and may act as holodirected or hemidirected in lead(II) complexes [10]. In the present work, comparison of the structures of Hg(II) and Pb(II) complexes of the 2,2′-bis(4,5-dimethylimidazole) (DmImH) ligand provides Hg(II) as a soft acid and Pb(II) as a hard acid exhibiting lone pair activity.

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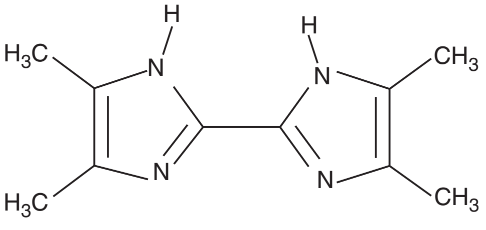

DmImH

2. Experimental

2.1. *Physical property 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. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected.

2.2. *Preparation of [Hg(DmIMH)(SCN)$_2$]*

The complex was prepared by dissolving mercury(II) thiocyanate (0.316 g, 1 mmol) in distilled water and adding an ethanolic solution of 2,2′-bis(4,5-dimethylimidazole) (0.190 g, 2 mmol). The resulting solution was stirred for 5 h at room temperature, and then allowed to stand for 2–3 days in a refrigerator (ca. 6°C). Black crystals of the product precipitated were filtered off, washed with acetone and ether and air dried (0.354 g yield 70%, m.p. 250°C). (Found C, 28.61; H, 2.68; N, 16.43: calculated for $C_{12}H_{14}HgN_6S_2$; C, 28.40; H, 2.76; N, 16.56%). -IR (KBr) selected bonds: $\nu = 612$(s), 1015(w), 1470(s), 1510(w), 1515(s), 1590(s), 1648(s), 2090(vs), 2980(w), 3042(w), 3248(s) cm^{-1}.

2.3. *Preparation of [Pb(DmImH)(NCS)$_2$]*

(a) The complex was prepared by dissolving lead(II) nitrate (0.33 g, 1 mmol) and potassium thiocyanate (0.194 g, 2 mmol) in distilled water and adding an alcoholic solution of 2,2′-bis(4,5-dimethylimidazole), (0.380 g, 2 mmol). The resulting solution was stirred for 2 h at room temperature, and then allowed to stand for 2–3 days in a refrigerator (ca. 6°C). Yellow crystals of the product precipitated, were filtered off, washed with acetone and ether and air dried (0.379 g yield 80%, m.p. 230°C). (Found C, 30.11; H, 2.75; N, 17.63: calculated for $C_{12}H_{14}N_6PbS_2$: C: 30.38; H: 2.95; N: 17.72%). -IR (KBr) selected bonds: $\nu = 611$(s), 1010(w), 1480(s), 1503(w), 1518(s), 1680(s), 2020(vs), 2980(w), 3040(w), 3250(s) cm^{-1}. ^{207}Pb NMR (DMSO, δ): 337.066 ppm.

(b) Branched tube method 2,2′-Bis(4,5-dimethylimidazole) (0.190 g, 1 mmol) was placed in one arm of the branched tube and a mixture of lead(II) nitrate (0.165 g, 0.5 mmol) and potassium thiocyanate (0.097 g, 1 mmol) in the other. Methanol was carefully added to fill both arms. The tube was sealed and the ligand-containing

Table 1. Crystal data and structure refinement for [Hg(DmImH)(SCN)$_2$] and [Pb(DmImH)(NCS)$_2$].

Empirical formula	$C_{12}H_{14}HgN_6S_2$	$C_{12}H_{14}N_6PbS_2$
Formula weight	507.00	513.60
Temperature (K)	293(2)	293(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P2_1/n$
Unit cell dimensions (Å, °)	$a = 7.7321(15)$	$a = 11.268(2)$
	$b = 10.252(2)$	$b = 7.3792(15)$
	$c = 11.802(2)$	$c = 19.400(4)$
	$\alpha = 65.57(3)$	$\alpha = 90$
	$\beta = 72.19(3)$	$\beta = 95.87(3)$
	$\gamma = 88.13(3)$	$\gamma = 90$
Volume (Å^3)	806.3(3)	1604.6(6)
Z	2	4
Density (calculated) (g cm^{-3})	2.09	2.126
Absorption coefficient (mm^{-1})	9.8	10.8
$F(000)$	480	968
Crystal size (mm^3)	0.4 × 0.3 × 0.2	0.35 × 0.20 × 0.10
θ range for data collection (°)	2.26 to 28.06	3.31 to 27.06
Index ranges	$0 \leq h \leq 10$, $-13 \leq k \leq 13$, $-14 \leq l \leq 15$	$-1 \leq h \leq 14$, $-1 \leq k \leq 9$, $-24 \leq l \leq 24$
Reflections collected	4204	3782
Independent reflections	3922 [R(int) = 0.0466]	3520 [R(int) = 0.0162]
Completeness to theta (%)	99.9	99.6
Absorption correction	Psi-scan	Psi-scan
Max. and min. transmission	0.675 and 0.260	0.42 and 0.69
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/parameters	3922/192	3520/190
Goodness-of-fit on F^2	1.039	1.070
Final R [$I > 2\sigma(I)$]	Indices for 3367 refl $R_1 = 0.0476$, $wR_2 = 0.1175$	Indices for 2585 refl $R_1 = 0.0576$, $wR_2 = 0.1060$
R indices (all data)	$R_1 = 0.0623$, $wR_2 = 0.1233$	$R_1 = 0.0912$, $wR_2 = 0.1189$
Largest diff. Peak, hole (e Å^{-3})	1.701, −2.940	1.288, −0.923

arm immersed in a bath at 60°C while the other arm was at ambient temperature. After 15 d, yellow crystals, (m.p. 230°C) deposited in the cooler arm.

2.4. *Crystallography*

The intensity data were collected at 293(2) K using a Siemens $R3m/V$ diffractometer with graphite monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). Accurate unit cell parameters and an orientation matrix for the data collection were obtained from least-squares refinements. 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 displacement parameter. Corrections for Lorentz and polarization effects as well as an empirical correction for absorption using the Psi-scan programs were applied. All structural calculations were carried out with a PDP–11/23+ computer using the SDP–PLUS program package [11, 12].

Crystal data and structure refinement details are given in table 1. Selected bond lengths and angles are given in tables 2 and 3. Anisotropic thermal parameters,

Table 2. Selected bond lengths (Å) and bond angles (°) for $[Pb(DmImH)(NCS)_2]$.

Pb(1)–N(3′)	2.492(8)	N(3′)–Pb(1)–N(3)	68.5(2)
Pb(1)–N(3)	2.506(7)	N(3′)–Pb(1)–N(5)	86.5(3)
Pb(1)–N(5)	2.544(10)	N(3)–Pb(1)–N(5)	78.2(3)
Pb(1)–N(4)#1	2.777(10)	N(3′)–Pb(1)–N(4)#1	95.9(3)
Pb(1)–S(1)	3.230(3)	N(3)–Pb(1)–N(4)#1	73.3(3)
Pb(1)–S(2)#1	3.279(3)	N(5)–Pb(1)–N(4)#1	148.2(3)
S(2)–Pb(1)#2	3.279(3)	N(3′)–Pb(1)–S(1)	91.0(2)
		N(3)–Pb(1)–S(1)	148.7(2)
		N(5)–Pb(1)–S(1)	77.0(2)
		N(4)#1–Pb(1)–S(1)	134.5(2)
		N(3′)–Pb(1)–S(2)#1	164.2(2)
		N(3)–Pb(1)–S(2)#1	96.8(2)
		N(5)–Pb(1)–S(2)#1	84.8(2)
		N(4)#1–Pb(1)–S(2)#1	84.6(2)
		S(1)–Pb(1)–S(2)#1	99.9(1)

#1: $-x+3/2$, $-y+1/2$, $-z+1/2$; #2: $-x+3/2$, $y-1/2$, $-z+1/2$.

Table 3. Selected bond lengths (Å) and bond angles (°) for $[Hg(DmImH)(SCN)_2]$.

Hg(1)–N(3)	2.250(5)	N(3)–Hg(1)–N(1)	74.24(2)
Hg (1)–N(1)	2.403(6)	N(3)–Hg(1)–S(2)	140.54(2)
Hg (1)–S(2)	2.408(10)	N(1)–Hg(1)–S(2)	110.88(1)
Hg (1)–S(1)	2.539(10)	N(3)–Hg(1)–S(1)	102.98(2)
		N(1)–Hg(1)–S(1)	106.05(2)
		S(2)–Hg(1)–S(1)	112.19(7)

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 figures 1–5.

3. Discussion

3.1. *Syntheses*

Reaction between 2,2′-bis(4,5-dimethylimidazole) and lead(II) thiocyanate and mercury(II) thiocyanate provided crystalline material analyzing as $[Pb(DmImH)(NCS)_2]$ and $[Hg(DmImH)(SCN)_2]$, respectively. The IR spectrum of $[Hg(DmImH)(SCN)_2]$ hows ν(SCN) at ca. 2090 cm^{-1} significantly different than $[Pb(DmImH)(NCS)_2]$ (2020 cm^{-1}), suggesting that the thiocyanate anion may be coordinated through sulfur in the mercury compound and through nitrogen in the lead compound. The IR spectra of both complexes show absorption bands resulting from the skeletal vibrations of the aromatic rings in the 1470–1690 cm^{-1} range. The relatively weak bands around 2980 and 3040 cm^{-1} are assigned to the $\nu(CH_3)$ and ν(CH) modes of the "DmImH" aromatic rings, respectively. The absorption bands of the NH groups in the two complexes are observed as a strong band centered around 3248 cm^{-1}, significantly shifted to lower frequency, when compared to the free ligand (3275 cm^{-1}). The relatively low frequency of this band is indicative of hydrogen bonding, which is confirmed by the crystal structure of these complexes.

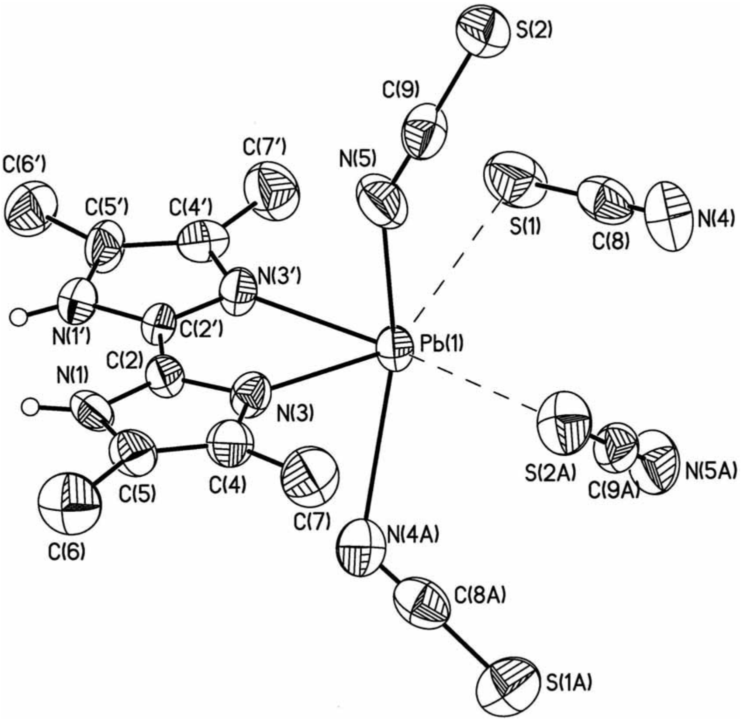

Figure 1. ORTEP diagram with ellipsoids 50% probability of [Pb(DmImH)$(NCS)_2$].

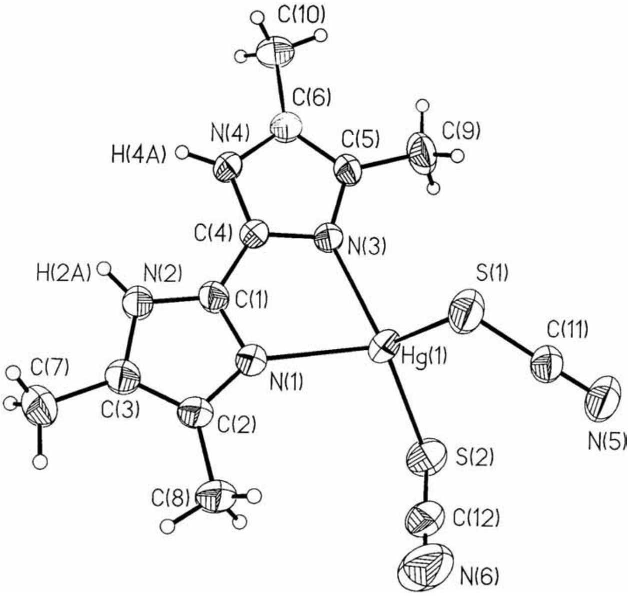

Figure 2. ORTEP diagram with ellipsoids 50% probability of [Hg(DmImH)$(SCN)_2$].

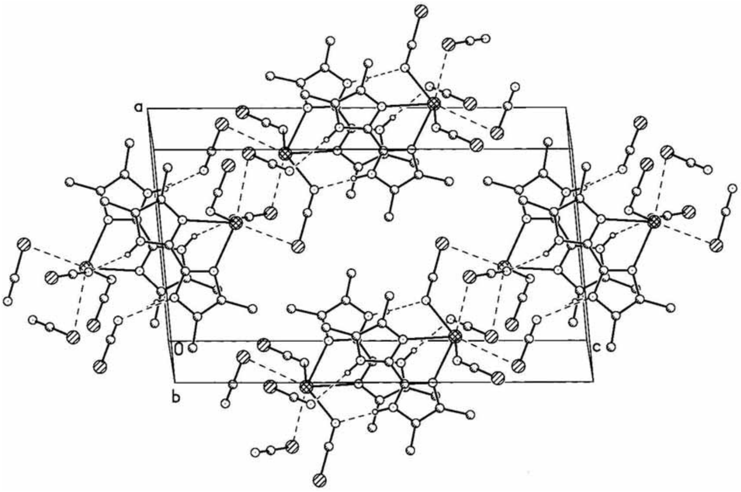

Figure 3. The unit cell of [Pb(DmImH)(NCS)$_2$].

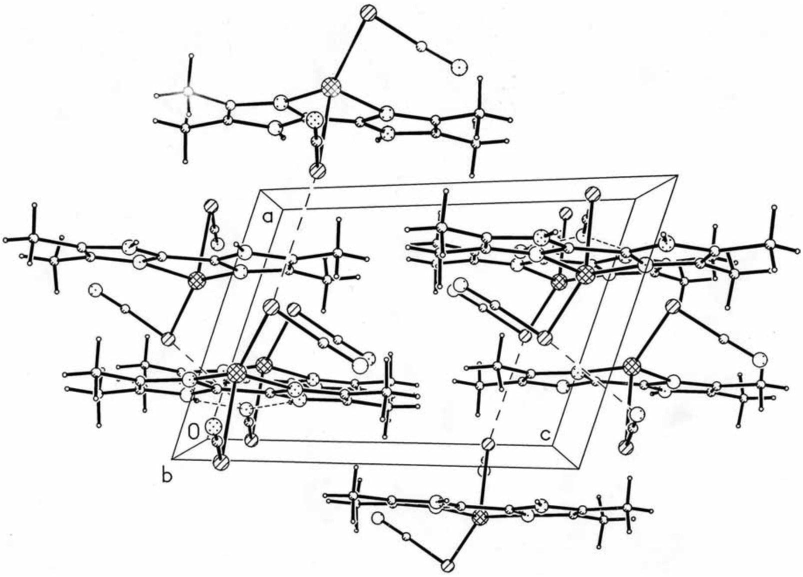

Figure 4. The unit cell of [Hg(DmImH)(SCN)$_2$].

3.2. *Crystal structure of [Pb(DmImH)(NCS)$_2$]*

The molecular structure (figure 1) is polymeric, with similarity to the polymeric structure of (L)Pb(SCN)$_2$, (L = 2,2′-bipyridyl [13], 1,10-phenanthroline and 4,4′-bithiazole [14]) in which the lead atoms are six-coordinate, PbN$_4$S$_2$. Two coordination sites are occupied by nitrogen atoms of the bidentate 2,2′-bis(4,5-dimethylimidazole) on one pole of the symmetry axis, with the two sulfur atoms on the opposite side with rather long Pb–S distances (3.230–3.279 Å). The arrangement of the two SCN$^-$ anions suggests a gap in coordination geometry around the metal ion

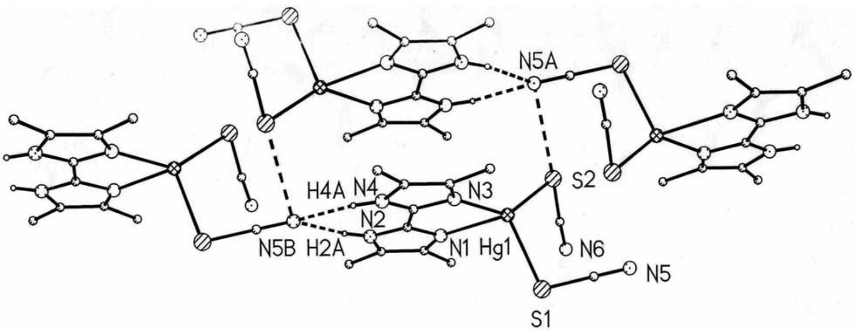

Figure 5. Hydrogen bonding, π–π stacking interaction in [Hg(DmImH)(SCN)$_2$].

Table 4. Hydrogen bond parameters (distances [Å] and angles [°]) for [Pb(DmImH)(NCS)$_2$].

D–H	d(D–H)	d(H···A)	d(D···A)	∠(DHA)	A
N(1)–H(1A)	0.88	2.43	3.141(11)	140(2)	N(5) [$-x+1$, $-y+1$, $-z$]
N(1′)–H(1′A)	0.88	2.04	2.866(11)	161(2)	N(4) [$x+1/2$, $-y+3/2$, $z+1/2$]

(angle N(5)–Pb(1)–N(4A) is 148.2°), possibly occupied by a stereo-active lone pair. The observed shortening of the Pb–N bond on the side of the Pb(II) opposite to the lone pair [2.492(8) compared with 2.777(10) Å adjacent to the lone pair] supports the presence of a stereo-active lone pair [15]. Hence, the coordination environment of every lead atom is caused by geometrical constraints of the coordinated 2,2′-bis(4,5-dimethylimidazole) ligand, two SCN$^-$ anions and also by the influence of the lone pair. Such an environment leaves space for bonding of sulfur atoms. The presence of the lone pair is apparently the reason that the bridging atoms cannot come closer together. If the stereo-chemically active lone pair were not present, more bridging interactions could occur. However, the coordination around the lead atoms is hemidirected with a significant gap trans to the chelating ligand [10] (figure 3).

The complexes are linked by hydrogen bonding. The coordinated 2,2′-bis(4,5-dimethylimidazole) is involved in hydrogen bonding, acting as hydrogen-bond donors with coordinated N atoms as potential hydrogen-bond acceptors. The hydrogen bond parameters {distances [Å], d(D–H), d(H···A), d(D···A), and angles [°], ∠(DHA)} are given in the table 4. As shown in figure 3, the hydrogen bonding yields infinite chains parallel to the *a* direction. Each molecule is bonded to one neighbor. Both amine H atoms are hydrogen bonded to N atoms.

3.3. *Crystal structure of [Hg(DmImH)(SCN)$_2$]*

The complex is built up of monomeric Hg(SCN)$_2$ units [Hg(1)–S(1) 2.539(2) and Hg(1)–S(2) 2.408(2) Å], with one 2,2′-bis(4,5-dimetylimidazole) ligand coordinated to the Hg atom *via* the two N atoms giving a five-membered chelate ring [Hg(1)–N(1) 2.250(5) Å and Hg(1)–N(3) 2.403(6) Å], in a distorted tetrahedral environment. The smallest and largest bond angles around the Hg atoms are N(3)–Hg(1)–S(2) 140.54(15)° and N(1)–Hg(1)–N(3) 74.24(19)°, respectively.

Table 5. Hydrogen bond parameters (distances [Å] and angles [°]) for [Hg(DmImH)(SCN)$_2$].

D–H	d(D–H)	d(H···A)	d(D···A)	∠(DHA)	A
N(2)–H(2A)	0.866	2.094	2.914(5)	157.72	N(5) [x, $y-1$, z]
N(4)–H(4A)	0.867	2.157	2.971(5)	156.18	N(5) [x, $y-1$, z]

The complexes are linked by hydrogen bonding. The coordinating 2,2′-bis(4,5-dimethylimidazole) molecule is a hydrogen-bond donor with coordinated N atoms of the thiocyanate ions. As shown in figures 4 and 5, the hydrogen bonding yields infinite chains parallel to the *b* direction. Each complex is bonded to two neighbors. Both amine H atoms are hydrogen bonded to N atoms.

Recently, we reported some analogous complexes such as [Hg(DPBTZ)(SCN)$_2$] (DPBTZ = 2,2′-diphenyl-4,4′-bithiazole) [16], [Hg(DABTZ)(SCN)$_2$] (DABTZ = 2,2′-diamino-4,4′-bithiazole) [17] or [Hg(L)(SCN)$_2$] (L = N-(2-pyridyl)carbonylaniline) [18]. The thiocyanate anions in all complexes are coordinated to mercury(II) *via* the sulfur atom. The S–Hg–S bond angle between the two sulfur atoms of two coordinated thiocyanate ligands in [Hg(DPBTZ)(SCN)$_2$], [Hg(DABTZ)(SCN)$_2$] and [Hg(L)(SCN)$_2$] are 147.46, 128.4 and 142.9°, respectively. Also the N–Hg–N bite angle in [Hg(DPBTZ)(SCN)$_2$], [Hg(DABTZ)(SCN)$_2$] and [Hg(L)(SCN)$_2$] are 70.10, 72.5, and 67.9°, respectively. These bond angles (S-Hg-S and N-Hg-N) in the [Hg(DmImH)(SCN)$_2$] complex are 112.19 and 74.24°. Comparison of the S–Hg–S angle of [Hg(DPBTZ)(SCN)$_2$], [Hg(DABTZ)(SCN)$_2$] and [Hg(L)(SCN)$_2$] with that in the reported complex in this article reveals the effect of the size of bite angle (N–Hg–N angle) on the size of the S–Hg–S angle. It seems that with decreasing S–Hg–S angle, the bite angle slightly increases. The environment in the [Hg(DPBTZ)(SCN)$_2$] [16] and [Hg(L)(SCN)$_2$] [18] complexes leaves space between two sulfur atoms for bonding of sulfur {in [Hg(DPBTZ)(SCN)$_2$]} or nitrogen {in [Hg(L)(SCN)$_2$]} atoms, thus increasing the Hg environment from a distorted tetrahedron to a distorted pseudo-octahedral geometry. The obvious question then is whether coordinate bonds have been stretched resulting in increased S–Hg–S angle or whether the increase of the S–Hg–S angle has imposed a positioning of the donor atoms such that weak interactions are possible.

The common feature of the two reported complexes is that hydrogen bonds (tables 4 and 5) exist between the N atoms of the thiocyanate ions and the H atoms of the "DmImH" ligand, as shown in figures 2 and 5. Hydrogen bonding clearly plays a role in geometry of two complexes and, the solid state structure of these complexes can be considered as a coordination polymer.

In the lead(II) complex and others [19–21], the thiocyanate ions are coordinated *via* the N atom. Indeed, lead as a borderline metal ion has a high affinity for nitrogen donors and appears to be ideal to bind to harder Pearson bases and the mercury ion as a soft acid appears to be ideal to bind to softer Pearson bases. This shows that the Hg^{2+} is softer than Pb^{2+}.

Comparing [Hg(DmImH)(SCN)$_2$] and [Pb(DmImH)(NCS)$_2$] show π–π stacking [22, 23] interactions between parallel aromatic rings that belong to adjacent chains in the mercury(II) complex, as shown in figures 4 and 5. The mean molecular planes are close to parallel and separated by a distance of ~3.5 Å, similar to graphite. Parallel arrays of planes of aromatic moieties indicate that these interactions are "π-stacking",

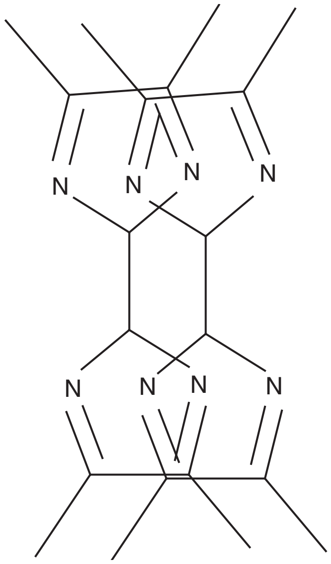

Figure 6. Projection of nearest neighbor pairs in the π–π stacks of heteroaromatic bases in [Hg(DmImH)(SCN)$_2$].

rather than "edge-to-face" or "vertex-to-face" (figure 6) [24–27]. In the mercury(II) structure reported here, the interplanar distance is 3.45 Å, appreciably shorter than the normal π–π stacking [28–29].

Thus two factors, hydrogen bonding and π–π stacking, control the coordination sphere of the mercury(II) complex. Whether hydrogen bonding stretches coordinate bonds resulting in ligand stacking or whether the stacking interaction, imposes a positioning of the donor atoms for hydrogen bonding is not clear. Since the packing molecules include equal or almost equal electron-deficient, or electron-rich rings, it can be expected that face-to-face π-stacking interactions must be disfavored due to dominance of π–π repulsion. Consequently, hydrogen bonding may be the most important factor.

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, quoting the deposition number 195763 for [Hg(DmImH)(SCN)$_2$] and 191963 for [Pb(DmImH)(NCS)$_2$].

Acknowledgements

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