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Cationic and neutral phenylmercury(II) complexes with heterocyclic thione ligands. X-ray structures of $[HgPh(dmpymtH)][BF_4] \cdot H_2O$ and $[\{HgPh\}_2(\mu\text{-dtu})]$

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ABSTRACT

The neutral complex [HgPh(dmpymt)] **1** (dmpymtH = 4,6-dimethylpyrimidine-2(1H)-thione) reacts with HBF_4 to give the cationic complex $[HgPh(dmpymtH)][BF_4]$ **2.** The X-ray molecular structure of the later revealed a [2+1] coordination sphere about the mercury(II) atom (C–Hg–S and Hg···N). In the dinuclear complex $[(HgPh)_2(\mu\text{-dtu})]$ **3** [$dtuH_2$ = 2,4(1H,3H)-pyrimidinedithione or dithiouracil] the coordination spheres are also [2+1] although dissimilar regarding the Hg···N secondary bonds. NMR spectroscopy (^1H, ^{13}C and ^{199}Hg) studies were undertaken in solution and the results discussed in the light of the X-ray structures.

1. Introduction

The establishment of the coordination number of the Hg^{II} atom in its complexes is crucial to understanding their properties. It has been noticed, mainly by X-ray crystallography, that the mercury(II) atom is, in addition to two strong bonds, usually being engaged in intra- and/or intermolecular secondary bonds, giving rise to various supramolecular arrangements. Grdenić [1] has proposed two kinds of coordination numbers to better structurally describe such complexes: (i) the primary coordination number (mercury-donating atom bond distance appropriate for their covalent radii) and (ii) effective coordination number (mercury-donating atom interactions that are less than the sum of their van der Waals radii). It has been pointed out that secondary bonds might be implicated in the easy mobility of organylmercury(II) ion in living organisms [2].

The study of monoorganylmercury(II)-thione complexes has been devoted mainly to the methylmercury(II) cation, probably because of its relevance in natural systems due to biological methylation of the mercury atom [3]. Usually the organylmercury(II) moiety links primarily to sulfur atoms present in the cysteinyl groups of polypeptides and completes its coordination sphere by establishing secondary bonds with nearby donating atoms. Heterocyclic compounds like pyrimidinethione/thiol have been considered good candidates for modeling this scenario [4]. In their complexes, usually the mercury atom bounds to the sulfur atom, enabling the nitrogen atoms to establish secondary interactions. In order to illustrate the structural diversity of organylmercury(II) derivatives containing S, N or O donating atoms, originated mainly due to secondary bonds, we will present hereafter some typical cases. In addition to the primary coordination, C–Hg–S, the complex methyl(pyridine-2-thiolato)mercury(II) exhibits intra-Hg···N and intermolecular Hg···S interactions [5]; the methyl(2-mercapto-4-methylpyrimidinato)mercury(II) presents intermolecular Hg···S and Hg···Hg interactions [6]. Not surprisingly, the complex methyl(2-methylthio-4-pyrimidinonato)mercury(II) is C–Hg–N primarily coordinated with additional intra-Hg···O and intermolecular Hg···N secondary interactions [7]. The complex phenyl (diacetylmonoximemorpholine *N*-thiohydrazonate)mercury(II) is C–Hg–S primarily coordinated with additional intramolecular Hg···N interaction [8].

Here, we present the preparation and structural studies both in the solid state and in solution for some mono- and dinuclear complexes of the phenylmercury(II) cation with pyrimidinethione derivatives as ligands.

2. Experimental details

2.1. Materials

Phenylmercury(II) acetate and $dtuH_2$ was purchased from Aldrich and dmpymtH was prepared following a reported method [9].

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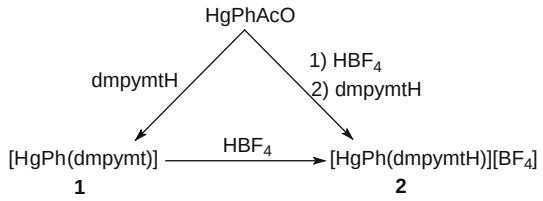
Table 1
Crystallographic data for [HgPh(dmpymtH)][BF_4] · H_2O **2** and [{HgPh}$_2$(μ-dtu)] **3**.

	2	**3**
Molecular formula	$C_{12}H_{15}BF_4HgN_2OS$	$C_{16}H_{12}Hg_2N_2S_2$
Formula weight	522.72	697.58
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/n$
Temperature (°C)	20(2)	20(2)
Unit cell dimensions		
a (pm)	741.00(2)	1621.93(7)
b (pm)	1263.80(6)	540.75(3)
c (pm)	1728.50(8)	2293.50(10)
α (°)	90	90
β (°)	95.166(3)	98.873(3)
γ (°)	90	90
Volume (nm^3)	1.61212(12)	1.98746(16)
Z	4	4
$D_{calc.}$ (g/cm^3)	2.154	2.331
Absorption coefficient (mm^{-1})	9.717	15.637
F(000)	984	1256
Crystal size (mm^3)	0.04 × 0.10 × 0.17	0.02 × 0.08 × 0.16
θ Range for data collection (°)	3.22–27.12	3.15–27.57
Limiting indices (*h*, *k*, *l*)	−7 → 8, −14 → 16, −19 → 22	−21 → 21, −6 → 7, −29 → 29
Reflections collected	6848	20036
Reflections unique/R_{int}	3365/0.0296	4536/0.0624
Completeness to θ (°)	27.12 (94.3%)	27.57 (99.0%)
Data/parameters	3365/200	4536/199
Absorption correction		
Maximum and minimum transmission	0.466 and 0.177	0.645 and 0.173
Goodness-of-fit on F^2	1.049	1.083
Final *R* indices [$I > 2\sigma(I)$]	R_1 = 0.0334, wR_2 = 0.0814	R_1 = 0.0533, wR_2 = 0.1389
R indices (all data)	R_1 = 0.0462, wR_2 = 0.0861	R_1 = 0.0941, wR_2 = 0.1624
Largest difference in peak and hole (e Å^{-3})	0.730 and −0.591	1.849 and −1.351



Scheme 1. Summary of the synthetic procedure for obtaining the phenylmercury(II) complexes.

2.2. Instrumentation

Elemental analyses (C, H, N, S) were performed on a Fisions MOD EA 1108 analyser. The infrared spectra (KBr pellets, 4000–400 cm^{-1}) were recorded on BOMEM BM 100 FT-IR spectrometer. NMR spectra were recorded on a Varian MERCURY plus spectrometer, 7.05 T, operating at 300.07 MHz for ^{1}H, 75.46 MHz for ^{13}C, and 53.74 MHz for ^{199}Hg. Chemical shifts (δ) are given in ppm relative to $SiMe_4$ (internal reference for ^{1}H and ^{13}C) and net $HgMe_2$ (external reference for ^{199}Hg), checked against 1.0 molar solution of $HgCl_2$ in DMSO (δ −1501.0) by using the substitution method [10].

Crystallographic data were collected at room temperature on a Enraf–Nonius Kappa-CCD diffractometer, equipped with Mo Kα radiation (0.71073 Å) and graphite monochromator. The cell refinements were performed using the software Collect [11] and Scalepack [12], and the final cell parameters were obtained on all reflections. Data reduction was carried out using the software Denzo-SMN and Scalepack [12]. Since the absorption coefficients are significant, absorption corrections were applied.

The structures were solved with SHELXS97 by direct methods [13]. All non-hydrogen atoms of the molecules were clearly solved and full-matrix least-squares refinement of these atoms with anisotropic thermal parameters was carried on [14]. The C–H hydrogen atoms were positioned stereochemically and were refined with fixed individual displacement parameters [$U_{iso}(H) = 1.2U_{eq}$ (C_{sp}^2 and N_{sp}^2) or $1.5U_{eq}$ (C_{sp}^3 and OH)] using a riding model [14]. Tables were generated by WINGX [15] and the structure representations by PLATON [16]. Additional crystal data and more information about the X-ray structural analyses are shown in Table 1.

2.3. Syntheses of the complexes

2.3.1. [HgPh(dmpymt)] **1**

2.3.1.1. Method A. A suspension containing Hg(C_6H_5)($OOCCH_3$) (HgPhAcO) (168.3 mg, 0.5 mmol) and $C_6H_8N_2S$ (dmpymtH) (70.1 mg, 0.5 mmol) in toluene (20 mL) was heated to reflux under continuous stirring when a clear solution has developed. The reflux was continued for 2 h. The slow evaporation of the solution into a beaker at room temperature produced colorless plates after six weeks. Yield 186 mg (90%). M.p. 128–130 °C. Anal. Found: C, 34.76; H, 2.64; N, 6.80; S, 7.62%. $C_{12}H_{12}HgN_2S$ Calc.: C, 34.57; H, 2.90; N, 6.72; S, 7.69. IR (KBr): 3040(w), 1577(s), 1537, 1527(m), 1475(w), 1429(m), 1386(w), 1366(w), 1339(m), 1253(vs), 1023(w), 997(w), 950(w), 875(w), 726(s), 693(m), 669(w), 549(w), 445(w) cm^{-1}.

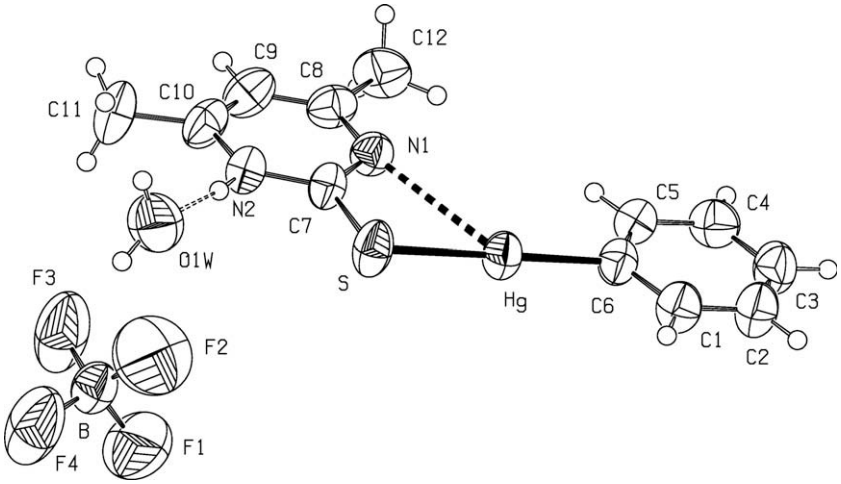


Fig. 1. ORTEP plot of [HgPh(dmpymtH)][BF_4] · H_2O **2** with the atom numbering. Thermal ellipsoids are scaled at 50%.

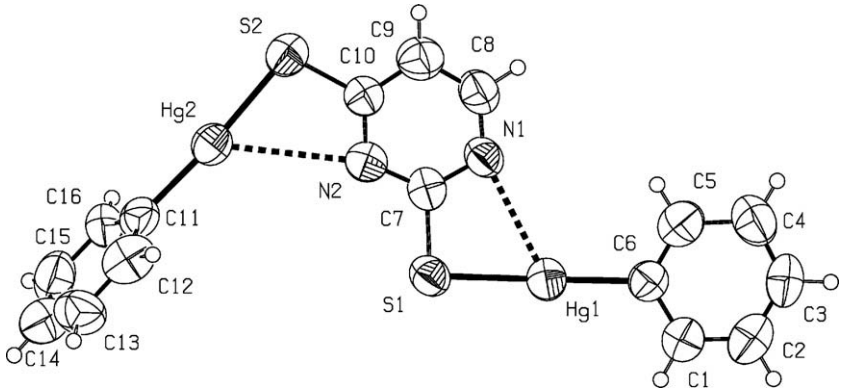


Fig. 2. ORTEP plot of [{HgPh}$_2$(μ-dtu)] **3** with the atom numbering. Thermal ellipsoids are scaled at 50%.

Table 2
Selected bond lengths (pm) and angles (°) for [HgPh(dmpymtH)][BF_4] · H_2O **2**.

Hg–S	236.7(2)	C(6)–Hg–S	177.3(2)
Hg–C(6)	207.1(6)	C(7)–S–Hg	95.5(2)
C(7)–S	171.5(6)	N(1)–C(7)–S	120.6(4)
C(7)–N(1)	133.9(7)	N(2)–C(7)–S	118.6(4)
C(7)–N(2)	135.6(7)		
Hg···N(1)	289.4(4)		

Table 3
Selected bond lengths (pm) and angles (°) for [{HgPh}$_2$(μ-dtu)] **3**.

Hg(1)–S(1)	236.6(4)	C(6)–Hg(1)–S(1)	178.3(4)
Hg(2)–S(2)	236.5(4)	C(11)–Hg(2)–S(2)	175.2(4)
Hg(1)–C(6)	205.8(14)	C(7)–S(1)–Hg(1)	93.6(5)
Hg(2)–C(11)	208.4(15)	C(10)–S(2)–Hg(2)	101.6(5)
C(7)–S(1)	175.5(13)	N(1)–C(7)–S(1)	117.2(10)
C(10)–S(2)	176.6(13)	N(2)–C(10)–S(2)	120.4(10)
C(7)–N(1)	131.9(16)	N(2)–C(7)–S(1)	115.8(10)
C(7)–N(2)	134.0(17)		
C(10)–N(2)	133.0(16)		
Hg(1)···N(1)	277.4(11)		
Hg(2)···N(2)	314.4(11)		

2.3.1.2. Method B. To a suspension of HgPhAcO (168.3 mg, 0.5 mmol) in acetonitrile (10 mL) was added a solution of dmpymtH (70.1 mg, 0.5 mmol) in acetonitrile (10 mL). A clear solution resulted in a few minutes. The solution was magnetically stirred for 2 h and filtered to remove any insoluble material. The solution was allowed to evaporate at room temperature to approximately 1 mL. Upon addition of hexane (3 mL) the product precipitated immediately. Yield 200 mg (95%).

2.3.2. [HgPh(dmpymtH)][BF_4] · H_2O ***2***

To a solution of **1** (208 mg, 0.5 mmol) in acetonitrile (20 mL) an aqueous solution of HBF_4 40% (0.08 mL, 0.5 mmol) was added under continuous magnetic stirring. The stirring was continued for 15 min and the clear solution was allowed to evaporate at room temperature resulting colorless needles after one week, one of which was selected for diffraction studies. Yield 225 mg (86%). M.p. 250–251 °C. Anal. Found: C, 27.81; H, 3.00; N, 5.96; S, 6.46%. $C_{12}H_{15}BF_4HgN_2OS$ Calc.: C, 27.57; H, 2.89; N, 5.36; S, 6.13%. IR (KBr): 3616–2577(w), 1633(s), 1575, 1480(w), 1434(m), 1396(w), 1370(w), 1333(w), 1256(s), 1167(w), 1088-1025(vs), 886(w), 741(s), 701(w), 544(w), 521(w), 451(m) cm^{-1}.

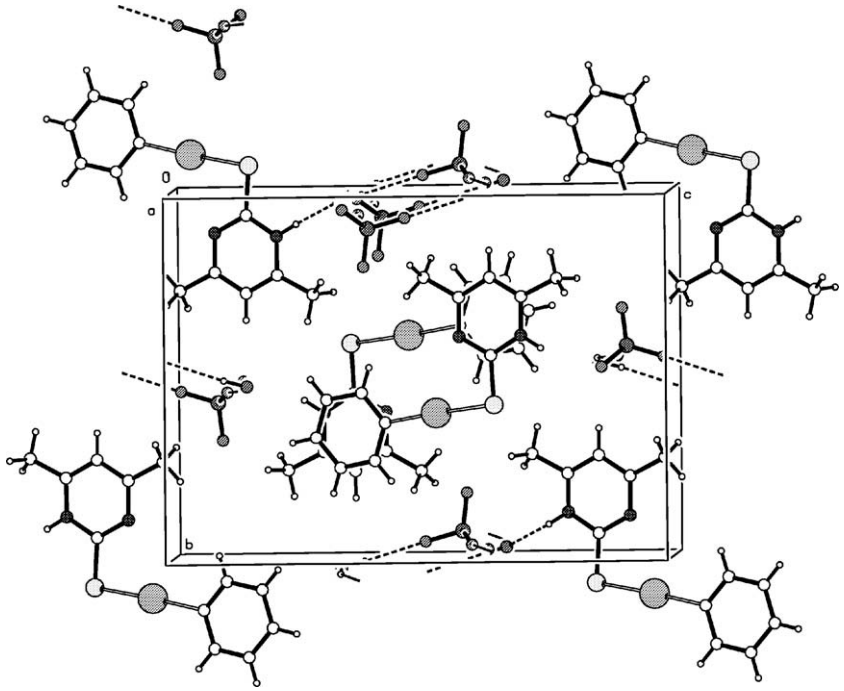


Fig. 3. Perspective view of the unit cell of [HgPh(dmpymtH)][BF_4] · H_2O **2**, showing the packing of the complex molecules parallel to the direction [100], in which the pyrimidine and phenyl rings from distinct molecules are superposed. The dashed lines indicate hydrogen bonds.

*2.3.3. [{HgPh}$_2$(μ-dtu)] **3***

Prepared according to method A in the proportion HgPhAcO (1 mmol) to dtuH$_2$ (0.5 mmol). Colorless plates were collected after two weeks, one of which was selected for diffraction studies. Yield 123.3 mg (35%). M.p. 181–183 °C. Anal. Found: C, 28.57; H, 2.14; N, 3.42; S, 8.59%. $C_{16}H_{12}Hg_2N_2S_2$ Calc.: C, 27.55; H, 1.73; N, 4.02; S, 9.19. IR (KBr): 3060(w), 3043(w), 1574(w), 1532(vs), 1507(s), 1476(w), 1429(m), 1384(s), 1308(s), 1188(s), 1147(m), 1091(w), 1021(w), 997(w), 807(s), 726(s), 693(s), 448(w) cm^{-1}.

3. Results and discussion

3.1. Syntheses

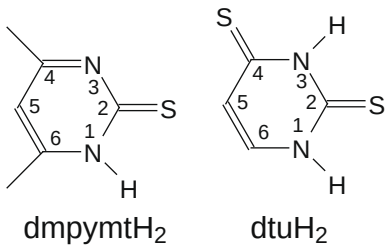
The neutral complexes have been synthesized in reasonable yields with displacement of acetic acid in mild conditions. We have proved the feasibility of the reversible protonation/deprotonation of the coordinated ligand in one typical case (Scheme 1).

3.2. X-ray structures

The atomic numbering used is shown in Figs. 1 and 2 for complexes **2** and **3**, respectively. Selected bond distances and bond angles are shown in Tables 2 and 3 for compounds **2** and **3**, respectively. The Hg–C bond distance of 207.1(6) pm observed in **2** is close to the average value 206 pm reported for Hg–C(Ph) compounds [17]. In the dinuclear complex **3** there is an asymmetry both in the Hg–C bond distances 205.8(14) and 208.4(15) pm and in the bond angles C–Hg–S being 178.3(4)° and 175.2(4)°. The Hg–S bond distances are similar in both compounds and close to 237.6(3), reported for [Hg(C$_6$H$_5$)(C$_5$H$_4$NS)] [18]. The C–S bond distances are those of the expected single bonds for the thiol form usually observed for such derivatives, particularly in the case of **2**, although in some cases the thione form can be stable enough to direct the metallation to the NH site as observed in [HgPh(DABRd)], where HDABRd is 5-(4-dimethylaminobenzylidene)rhodanine [19]. Assuming the van der Waals radius of mercury reported by Carty and Deacon [20] of 173 pm, the summation for Hg···S and Hg···N will be 353 and 328 pm, respectively. In complex **3**, the Hg(1)···N(1) and Hg(2)···N(2) distances are 274.4(11) and 314.4(11), respectively. In complex **2** this distance is 289.4(4) pm, supporting secondary bonds in both complexes. In addition, there is an array of hydrogen bonds, namely, N2–H2···O1W (DA 275.7(7), O1W–H11W···F1 (DA 277.2(8)) and O1W–H12W···F4 305.5(8) pm). The average deviation from ideal planarity considering all atoms in the cation species of complex **2** is 8.91 pm. The phenyl ring bound to Hg(2) in **3** forms an angle of 70.6(3)° with the plane formed by the remaining atoms of the complex molecule (see Fig. 2), which shows an average deviation of the fitted atoms of 4.55 pm from the planarity. The packing of the molecules in the crystal structure of **2** is shown in Fig. 3 (Supplementary material). The molecules are arranged in a head to tail form, so that the pyrimidine and phenyl rings from distinct molecules alternate in the staking parallel to the direction [10]. The distance between the molecules correspond to half of the cell constant *a*. The molecules of complex **3** are piled up parallel to the direction [10], as show in Fig. 4 (Supplementary material). The distance between the packed molecules in this case corresponds to the cell constant *b*.

3.3. NMR spectroscopy

Table 4 lists the NMR data of complexes **1–3**. One striking feature of ^{1}H NMR spectra of **1** and **2** is the absence of NH resonance



Scheme 2. Draw of the complexing agents (thione form) with numbering scheme for NMR purpose.

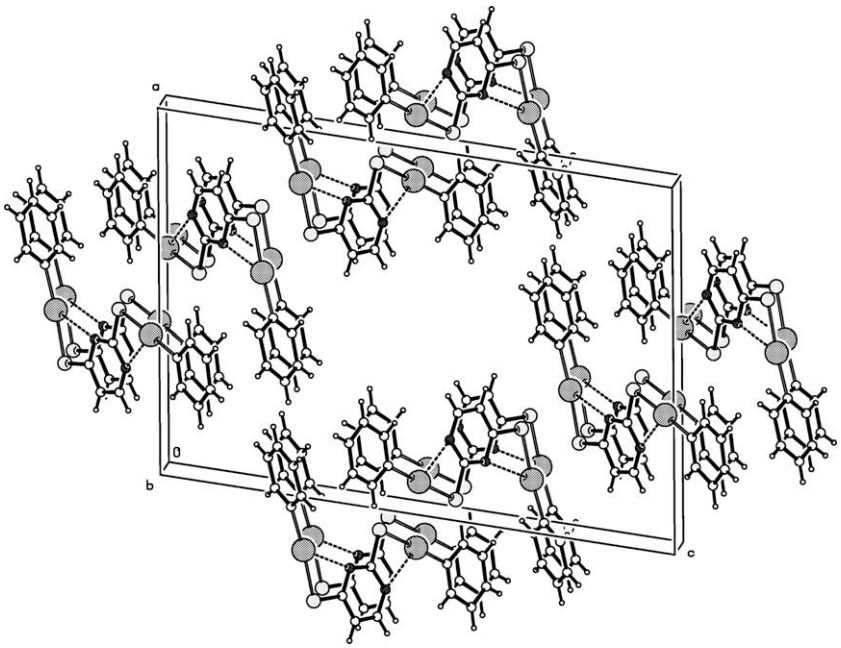


Fig. 4. Perspective view of the unit cell of [{HgPh}$_2$(μ-dtu)] **3**, showing the packing of the complex molecules parallel to the direction [0 1 0].

Table 4
^{1}H, $^{13}C\{^{1}H\}$, and $^{199}Hg\{^{1}H\}$ NMR data of complexes **1**– **3** in DMSO-d^6 solutions (δ in ppm; J in Hz).

Compound	δ ^{1}H	δ $^{13}C\{^{1}H\}$	δ $^{199}Hg\{^{1}H\}$[a]
1	2.276 (s, 6H, Me) 6.947 (s, 1H, C5H) 7.239 (tt, ^{3}J = 7.2, ^{4}J = 1.5, 1H, CH_p) 7.367 (t, ^{3}J = 7.5, 2H, CH_m) 7.481(dd, ^{3}J = 7.5, ^{4}J = 1.5, 2H, CH_o – flanked by $^{3}J(^{1}H–^{199}Hg)$ 168	23.47 (C4-Me/C6Me) 115.41 (C5H) 127.95 (CH_p) 128.53 (CH_m) 137.03 (CH_o) 166.20 (C4/C6) 175.96 (CS)	−1012.5 (82)
2	2.368 (s, 6H, Me) 7.178 (s, 1H, C5H) 7.250 (tt, ^{3}J = 7.2, ^{4}J = 1.5, 1H, CH_p) 7.479(dd, ^{3}J = 8.0, ^{4}J = 1.5, 2H, CH_o – flanked by $^{3}J(^{1}H–^{199}Hg)$ 180 9.02 (s, br, NH)	22.37 (C4-Me/C6Me) 116.22 (C5H) 127.97 (CH_p) 128.42 [CH_m, ^{3}J ($^{13}C–^{199}Hg$) = 192] 136.70 [CH_o, ^{2}J ($^{13}C–^{199}Hg$) = 112] 156.84 (CHi) 167.02 (C4/C6) 172.42 (CS)	−1080.5 (350)
3	7.069 (d, J = 5.5, 1H, C5H) 7.227 (tt, ^{3}J = 7.2, ^{4}J = 1.5, 2H, CH_p) 7.343 (t, ^{3}J = 7.5, 4H, CH_m) 7.485 (dd, ^{3}J = 6.6, ^{4}J not resolved, 4H, CH_o – flanked by $^{3}J(^{1}H–^{199}Hg)$ 168) 8.056 (d, J = 5.5, 1H, C6H)	116.78 125.20 127.76 128.09 128.25 128.78 136.89 (C5) 138.07 153.92 159.26 (C6) 174.39 (C2) 175.59 (C4)	−1027.5 (250)

[a] Values in parentheses are the line widths at half height (in Hz).

for the former while the later presents it as a broad signal at 9.02 ppm (see Scheme 2). Another characteristic is that in **2,** the methyl groups are magnetically equivalent both in the ^{1}H and ^{13}C NMR spectra, which is a consequence of chemical exchange of the NH group upon thione-coordination to the mercury(II) atom. The coupling constants $^{3}J(^{1}H–^{199}Hg)$ and $^{3}J(^{13}C–^{199}Hg)$ of the phenyl group attached to the Hg^{II} atom are in the range of reported values for other similar phenylmercury(II) complexes [21]. The ^{199}Hg NMR spectra of the analogous compounds [HgPh(HTu)] and [HgPh(Tu-SMe)] (H_2Tu = 2-thiouracil) in DMSO solutions showed resonances at 1072.7 and 1258.0 ppm, concluded to be S- and N-coordinated, respectively [7]. Usually one observe a low field shift of the ^{199}Hg resonance with increasing coordination number; the complex [$Hg(C_6H_4C_5H_4N$(Hstsc) showed a major peak at 868 ppm, which was attributed to the three-coordinated HgCNS isomer [22]. The ^{199}Hg NMR data of compounds **1–3** present only one reasonably sharp peak for each one. Based on the reported chemical shifts mentioned above it is concluded that, at room temperature DMSO solutions, the primary coordination number of the mercury atom is two (C−Hg−S) in all complexes, although coordinating solvents i.e. dimethylsulfoxide or acetonitrile might be interacting too. For instance, the ^{199}Hg chemical shift of **2** is −1105.5 ppm in acetonitrile and −1080.5 ppm in dimethylsulfoxide, at the same concentrations. Besides its resonance line width at half height in dimethylsulfoxide solution is about five times that observed in acetonitrile solution. It is worth mentioning that in **3** the ^{13}C NMR spectrum showed some nonequivalence of the phenyl groups, the peaks from (dtu) being identified by comparison with those of a dinuclear tin complex [23], whereas only one resonance was observed in the ^{199}Hg NMR spectrum. It can be explained by the almost equivalence of the mercury(II) sites [7].

3.4. Infrared spectroscopy

The comparison of the infrared spectra of **1** and **2** unequivocally shows that in the former the ligand is acting in its deprotonated form, i.e., dmpymt, as judged by the absence of bands in the 3190–2600 cm^{-1} region, assigned to the N–H and C–H vibrations of the free ligand. In complex **2** this region is rather complicated and extends from 3616 to 2577 cm^{-1}**.** The N–H stretching bands are also absent in complex **3**. All complexes exhibit the expected "NCS I, II and III bands" due to strong vibrational coupling effects [24]. Although complex **3** shows a strong absorption band at 807 cm^{-1} (absent in free $dtuH_2$), which could be related to C=S vibration [8,19], it most probably comes from nonequivalent phenyl groups. In the IR spectrum of complex **2,** the BF_4-absorption bands are seen in the region 1088–1025 cm^{-1} (νas) and 521 cm^{-1} (νs).

Acknowledgement

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Appendix A. Supplementary material

CCDC 695970 and 695971 contain the supplementary crystallographic data for **2** and **3**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2008.10.035.

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