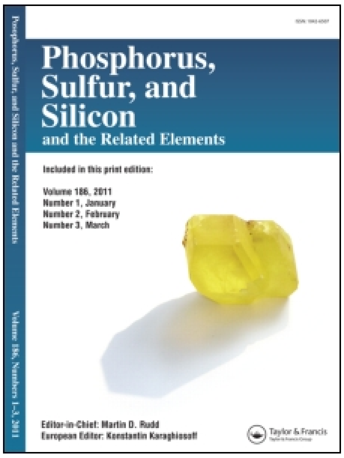

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Preparation and Spectral Investigation of Bis[N(2-methyl-phenyl) 4-Nitro-thiobenzamidato] mercury(II), Bis[N(2-methoxy-phenyl) 4-Nitro-thiobenzamidato]mercury(II), and Bis[N(2-chloro-phenyl) 4-Nitro-thiobenzamidato]mercury(II) Complexes

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Preparation and Spectral Investigation of Bis[N(2-methyl-phenyl) 4-Nitro-thiobenzamidato]-mercury(II), Bis[N(2-methoxy-phenyl) 4-Nitro-thiobenzamidato]mercury(II), and Bis[N(2-chloro-phenyl) 4-Nitro-thiobenzamidato]mercury(II) Complexes

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Several preparative routes to bis[N(substituted-phenyl) 4-nitro-thiobenzamidato] mercury(II) complexes are presented, including the reaction of mercury(II) oxide, fluoride, chloride, bromide, cyanide, acetate, and nitrate with N(substituted-phenyl) 4-nitro-thiobenzamide derivatives. ^{1}H-NMR, Raman, and IR measurements confirmed the complexation of mercury to sulphur.

Keywords Mercury compounds; mercury(II)complexes; thioamide; thiobenzamide

INTRODUCTION

Current interest in the coordination chemistry of mercury complexes containing ligands bearing sulphur atoms is related to mercury-cysteine thionato interactions in toxicological behavior of this metal,[1–2] in detoxification of mercury by metallothioneins,[3] in a DNA-binding protein[4] and in mercury reductase and related proteins.[5] The sustained interest in the coordination chemistry of mercury with sulphur-containing ligands is related to the environmental consequences of the high toxicity of the metal to living systems.[6–11] Consequently,

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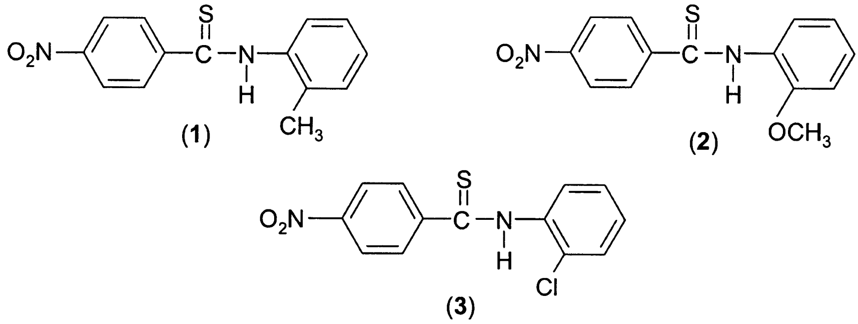
a number of attempts have been made to explore the coordination chemistry of mercury(II) with sulphur-containing ligands such as phenyl thiolate and heterocyclic thiones (e.g. pyrimidine-2-thione and imidazoline-2-(OH)-thione derivatives).[12–35] Thiolate ligands have a high affinity for Hg(II) to produce various complexes, such as $Hg(SR)_2$, $[Hg(SR)_3]^-$, and $[Hg(SR)_2Cl_2]^{2-}$. The common coordination form for the thiolate complexes is a linear di-coordination with a short Hg—S distance.[36–40] Mercury(II) ions interact with many biological molecules through coordination with deprotonated thiol, imidazole, disulfide, thioether, amino, or carboxylate groups and its interactions in model molecules and in proteins is well known.[41] In previous articles, we have reported the thermal and photochemical reactions of mercury compounds.[42–45] Recently we reported the preparation and application of bis(2-mercaptobenzoxazolato)mercury(II), $[Hg(MBO)_2]$ and bis(2-pyridinethiolato) mercury(II), $[Hg(PT)_2]$ complexes as carriers for thiocyanate selective electrode.[46–47] In this article, we describe the preparation and spectroscopic studies of three thiobenzamide complexes of mercury(II); bis[N(2-methyl-phenyl) 4-nitro-thiobenzamidato] mercury(II) $[Hg(Nmpntb)_2]$ (**4**); and bis[N(2-methoxy-phenyl) 4-nitro-thiobenzamidato] mercury(II) $[Hg(Nmtpntb)_2]$ (**5**); and bis[N(2-chloro-phenyl) 4-nitro-thiobenzamidato]mercury(II) $[Hg(Ncpntb)_2]$ (**6**) derived from the reaction (Scheme 1) of HgX_2 with N(2-methyl-phenyl)4-nitro-thiobenzamide (**1**), N(2-methoxy-phenyl) 4-nitro-thiobenzamide (**2**), N(2-chloro-phenyl) 4-nitro-thiobenzamide (**3**).[48–49]



SCHEME 1

RESULTS AND DISCUSSION

Bis[N(2-methyl-phenyl) 4-nitro-thiobenzamidato] mercury(II) [Hg-$(Nmpntb)_2$], bis[N(2-methoxy-phenyl) 4-nitro-thiobenzamidato] mercury(II) $[Hg(Nmtpntb)_2]$ and bis[N(2-Chloro-phenyl) 4-nitro-thiobenzamidato]mercury(II) $[Hg(Ncpntb)_2]$ complexes were readily prepared in

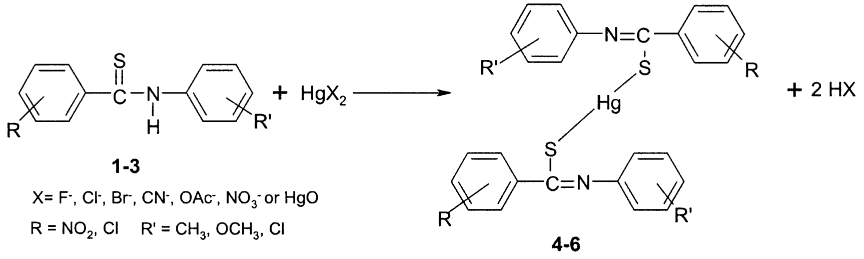
TABLE I Preparation Method, Reaction Times and Yields of 4–6

Complex no.	Thiobenzamides	Mercury(II) salts	Time (min)	Yield (%)[a]
4	NmpntbH **1**	a: HgO	130	95
		b: HgF_2	90	70
		c: $HgCl_2$	5	89
		d: $HgBr_2$	15	85
		e: $Hg(CN)_2$	310	80
		f: $Hg(OAc)_2$	2	74
		g: $Hg(NO3)_2$	105	62
5	NmtpntbH **2**	a: HgO	330	94
6	NcpntbH **3**	a: HgO	90	87
		b: HgF_2	210	30
		c: $HgCl_2$	5	72
		d: $HgBr_2$	120	58
		e: $Hg(CN)_2$	165	83
		f: $Hg(OAc)_2$	3	64
		g: $Hg(NO3)_2$	120	25

[a] Isolated yield.

good yield by the addition of equimolar quantities of HgX_2 ($X = F^-$, Cl^-, Br^-, CN^-, NO_3^-, OAc^-) and HgO to the ligands in $CHCl_3$ (Table I, Scheme 2). The most common preparation is achieved by the reaction of HgO. Other starting compounds can be used, such as HgF_2, $HgCl_2$, $HgBr_2$, $Hg(CN)_2$, $Hg(OAc)_2$, and $Hg(NO_3)_2$. The reaction of one equivilant HgO with 2 equivilants of thiobenzamides proceeded at lower yields and longer reaction times (1–2 days). Irradiation of the reaction mixture by 400 W high pressure mercury lamp enhanced the rate by 30%. No reaction was observed with photolysis of thiobenzamides in the absence of acetonitrils solution of HgO. In reaction of thiobenzamides with one equivilant of $Hg(NO_3)_2$ in chloroform, the isolated products were 30–54% benzoamide and 25–62% 1:2 complex. The reaction of thiobenzamide with $Hg(OAc)_2$ produced 1:2 complex in a few minutes and the complex decomposed to imides in solution immediately. The same reactions were proceeded in the presence of a catalytic amount of triethylamine as an axillary base and 1:2 complexes were formed at longer reaction time, which decomposed to imides.[50] The reaction of N-phenyl-N-methyl-4-nitro-thiobenzamide did not proceed with mercury(II) compound in various solvents (e.g. acetonitrile, chloroform, dichloromethane, and tetrahydrofuran) even at prolonged times, and under photochemical conditions. The complexes **4–6** melt in the range of 129–212.6°C whereas the thiobenzamide analogues melt at 122–152°C. The complexes are white to pale yellow, whereas the parent thiobenzamides are yellow to orange.

All three ligands are monodentate and coordination is expected to occur to mercury via sulfur. In order to establish the mode of coordination in thiobenzamide complexes, we have examined their Raman spectra, as well as the ^{1}H-NMR spectra of the 1:2 complexes.



SCHEME 2

The ^{1}H-NMR data for all three complexes, together with those for the free ligands, recorded in $CDCl_3$ solutions are listed in (Table II). It would be expected that the phenyl group nearest to sulfur would be the most sensitive to coordination. In these complexes, the absence of the N—H shift at 8.88–9.35 ppm in the ^{1}H-NMR spectra of **4–6**, (c.f., the presence of N—H shift at 8.88–9.35 in the free thiobenzamides) clearly indicates that ligands are coordinated via the thiobenzamide sulfur in all three complexes.

Characteristic infarared absorption bands and Raman bands are summarized in Tables III and IV, respectively. Shift to lower frequency in the thioamide(IV) band as well as in the (C=S) absorption are associated with sulphur donation in which C=S upon donation gains some C—S character. In these complexes the absence of the ν(NH) bands at 3313–3334 cm^{-1}, the presence of the ν(NH) bands at 3313–3334 cm^{-1}, as well as the characteristic thiobenzamide bands clearly indicate that the ligands (NmpntbH, NmtpntbH, and NcpntbH) are coordinated via

TABLE II ^{1}H-NMR Data (δ in ppm in $CDCl_3$) for Three Thiobenzamide Complexes of Mercury(II) and Their Corresponding Free Ligands

	N—H	*o*-tb	*o*-C_6H_4	*m*-tb	*m*-C_6H_4	*p*-C_6H_4	CH_3/OCH_3
NmpntbH **1**	8.88	8.33	7.59	8.06	7.36	7.36	2.4
[Hg(Nmpntb)$_2$]**4**	—	8.34	6.41	8.24	7.35	7.21	2.02
NmtpntbH **2**	9.1	8.3	7.22	7.9	7.1	6.9	3.92
[Hg(Nmtpntb)$_2$] **5**	—	8.4	6.24	8	7.1	6.8	3.79
NcpntbH **3**	9.35	8.3	7.6	8.1	7.21–7.40	7.6	—
[Hg(Ncpntb)$_2$] **6**	—	8.36	6.26	8.27	7.26–7.46	6.8	—

TABLE III Major Infarared Bands (cm^{-1}) and Their Assignments[a] for Thiobenzamide Complexes of Mercury(II) and Their Corresponding Free Ligands

	υ(NH)	Thioamide bands[b]			
		I	II	III	IV
NmpntbH **1**	3313 m	1580 w	1227 m	994 m	730 w, 670 w
[Hg(Nmpntb)$_2$] **4**	—	1585 vs	1181 m	920 m	740 w, 616 m
NmtpntbH **2**	3334 w	1599 m	1200 m	929 m	736 m, 641 w
[Hg(Nmpntb)$_2$] **5**	—	1595 vs	1187 m	929s	740 m, 617 m
NcpntbH **3**	3328 m	1595 m	1207 m	930 w	732 w, 665 w
[Hg(Ncpntb)$_2$] **6**	—	1591 vs	1185 m	923 vs	743 w, 616 m

[a] s, strong; vs, very strong; m, medium; w, weak. υ, stretching frequency; υ_s, symmetric stretching; υ_{as}, asymmetric (or antisymmetric) stretching; δ, bending.

[b] Thioamide-type mode are described as follows: thioamide I = υ(C—N) + δ(CH); thioamide II = υ(C—N) + δ(CH) + υ(C=S), thioamide III = υ(C—N) + υ(C=S), thioamide IV = υ_s(C—S) + υ_{as}(C—S).

sulphur in all three mercury complexes. The most significant change was seen for the thiobenzamide bands especially the thioamide(IV) band. This has shifted to the lower frequency of the band.[51–52] Complementary information is obtained by assignment of Raman vibrational spectral features of the complexes (Table IV). Structural information can be obtained for complexes in the low frequency range (below 700 cm^{-1}). The skeletal vibrations, as before, can readily be identified by comparison with the pure ligand spectrum. The remaining spectral features are due to the complexation, i.e. ν(Hg—S).

Mass spectra have been recorded for NmpntbH ligand and [Hg(Nmpntb)$_2$] complex. In Free ligands species are fully confirmed by a molecular ion $(M—H)^+$ peak of m/z 272 and peak $(M+2)^+$ attributed

TABLE IV Raman Data for Three Thiobenzamide Complexes of Mercury(II) and Their Corresponding Free Ligands

	Sk	Sk	Sk	Sk*	Sk*	
NmpntbH **1**	644 m	413 m	358 m	283s	219 m	
[Hg(Nmpntb)$_2$] **4**	694 s	437 w	381s	281 m	—	171.6, 90.6, 57.8
NmtpntbH **2**	594 w	408 w	303 w	—	219 m	
[Hg(Nmpntb)$_2$] **5**	627 m	—	332 m	—	—	84.7, 69.4, 56.3
NcpntbH **3**	640 m	474 m	345 m	276 m	218 m	
[Hg(Ncpntb)$_2$] **6**	675 m	—	372s	271 m	—	165.8, 90.6, 65.5, 46.2

Sk, skeletal bands of the ligand; Sk*, changes in the electronic structure due to complexation.

to the isotopic pattern of sulphur. The most intense peaks at m/z 257, 105, 156, 167, and 91 are attributed to $(M{-}CH_3)^+$, $(NC_6H_4CH_3)^+$, $(NO_2C_6H_5SH)^+$, $(NO_2C_6H_4)^+$, and $(C_6H_4CH_3)^+$ with abundances 49, 56, 100, 89, and 73, respectively. The most intense peaks for complexes are attributed to $(NO_2C_6H_5CNC_6H_5CH_3)^+$, $(C_6H_5CH_3)^+$, and the isotopic pattern of mercury with abundances 65, 100, and 8–20%, respectively. The presence of the peak at m/z 239 in complex with high abundance clearly indicates that the ligand is coordinated to mercury via thiobenzamide sulphur atom.

CONCLUSION

We have shown that the preparation of three thiobenzamide complexes of mercury(II) bis[N(2-methyl-phenyl) 4-nitro-thiobenzamidato] mercury(II) [Hg(Nmpntb)$_2$] (**4**), bis[N(2-methoxy-phenyl) 4-nitro-thiobenzamidato] mercury(II) [Hg(Nmtpntb)$_2$] (**5**), and bis[N(2-chloro-phenyl) 4-nitro-thiobenzamidato]mercury(II) [Hg(Ncpntb)$_2$] (**6**), from the reaction of mercury(II) oxide, fluoride, chloride, bromide, cyanide, acetate, and nitrate with N(2-methyl-phenyl)4-nitro-thiobenzamide (**1**), N(2-methoxy-phenyl) 4-nitro-thiobenzamide (**2**), N(2-chloro-phenyl) 4-nitro-thiobenzamide (**3**) proceeded from good to high yields at a relatively short reaction time under thermal or photochemical conditions. ^{1}H-NMR, Raman, and IR measurements confirmed the formation of mercury to sulphur bonds.

EXPERIMENTAL

N(2-methyl-phenyl) 4-nitro-thiobenzamide NmpntbH (**1**), N(2-methoxy-phenyl) 4-nitro-thiobenzamide NmtpntbH (**2**); N(2-Chloro-phenyl) 4-nitro-thiobenzamide NcpntbH (**3**), as ligands were prepared according to the reported procedure[48–49] and mercury(II) inorganic compounds were commercially available and were used without further purification.

Microanalysis was carried out by Elemental Micro-Analysis 2400 CHN/O Analyzer Perkin Elmer, UK. Mercury was analyzed using Shimadzu AA-680 Atomic Absorption. The IR spectra of the ligands and complexes were taken from KBr disks in the range 4000–400 cm^{-1} with a Shimadzu 430 spectrometer. Raman spectra were collected employing a 1800 back scattering geometry and a Bomem MB-154 Fourier Transform Raman spectrometer, which was equipped with a ZnSe beam splitter and TE cooled InGaAs detector. Rayleigh filtering was afforded by two sets of 2 Holographic technology filters. Laser power at the samples was 40 MW. The ^{1}H-NMR spectra were obtained as solutions in

$CDCl_3$ with a Bruker AC 250 spectrometer. Mass spectra were acquired using a Micromass platform spectrometer, EI-mode at 70 ev.

Preparation of Complexes

Preparation of Bis[N(2-methyl-phenyl) 4-nitro-thiobenzamidato]mercury(II), [Hg(Nmpntb)$_2$]

One (1.00) mmol of mercury (II) oxide (0.216 g) (excess) was added to a solution of N(2-methyl-phenyl) 4-nitro-thiobenzamide (0.273 g, 1.00 mmol) at ambient temperature in chloroform (35 mL) for 130 m with stirring. The completion of the reaction was followed by thin layer chromatography (tlc) CCl_4/CH_3OH, 15:1 as eluent. The mixture was filtered through celite or BaS to remove unreacted mercury(II) compounds. The pale yellow crystals which formed by slow evaporization was separated and recrystallised from chloroform as fine pale yellow crystals and dried in vacuo.

Yield: 95%. Anal. Calc. for $C_{28}H_{22}N_4O_4S_2Hg$: C, 45.28; H, 2.96; N, 7.55; Hg, 26.95. Found: C, 45.12; H, 3.12; N, 7.35; Hg, 27.08%. M.p. 193–195°C.

Preparation of Bis[N(2-methoxy-phenyl) 4-Nitro-thiobenzamidato]mercury(II), [Hg(Nmtpntb)$_2$]

The same method of preparation was used as for the [Hg(Nmpntb)$_2$] using N(2-methoxy-phenyl) 4-nitro-thiobenzamide (0.288 g, 1.00 mmol) in chloroform (45 mL, 330 min) and the complex recrystallised from CH_2Cl_2 as pale yellow crystals.

Yield: 94%. Anal. Calc. for $C_{28}H_{22}N_4O_6S_2Hg$: C, 43.41; H, 2.84; N, 7.24; Hg, 25.84. Found: C, 43.56; H, 2.67; N, 7.39; Hg, 25.97%. M.p. 129–131°C.

Preparation of Bis[N(2-chloro-phenyl) 4-Nitro-thiobenzamidato]mercury(II), [Hg(Ncpntb)$_2$]

The same method of preparation was used as for the [Hg(Nmpntb)$_2$] using N(2-chloro-phenyl) 4-nitro-thiobenzamide (0.2925 g, 1.00 mmol) in chloroform (50 mL, 90 m) and the complex recrystallised from $CHCl_3$ as yellow crystals.

Yield: 87%. Anal. Calc. for $C_{26}H_{16}N_4O_4S_2Cl_2Hg$: C, 39.84; H, 2.04; N, 7.15; Hg, 25.5. Found: C, 39.55; H, 1.97; N, 7.02; Hg, 25.75%. M.p. 210–212.6°C.

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