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Synthesis, Structure, and Solution Study of a Mercury(II) Complex with the Ligand [1-(2-Methoxyphenyl)-3-(4-chlorophenyl)]triazene

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Keywords: Mercury; Triazene; X-ray diffraction; Solution study; C–H···π interaction

Abstract. The title ligand, [1-(2-methoxyphenyl)-3-(4-chlorophenyl)]triazene, HL (1), was prepared. In a reaction with $Hg(NO_3)_2$ it forms the complex $[Hg(C_{26}H_{22}Cl_2N_6O_2)]$, [Hg**L**$_2$] (**2**). Both compounds were characterized by means of X-ray crystallography, CHN analysis, FT-IR, ^{1}H NMR, and ^{13}C NMR spectroscopy. In the structure of compound **1**, two independent fragments are present in the unit cell. They exhibit *trans* arrangement about the –N=N– double bond. The dihedral angles between two benzene rings in both fragments are 4.36 and 18.79 Å, respectively. Non-classic C–H···N hydrogen bonding and C–H···π interactions form a layer structure along the crystallographic *ab* plane [110]. In compound **2**, the Hg^{II} atom is hexacoordinated by two tridentate [1-(2-methoxyphenyl)-3-(4-chlorophenyl)]triazenide ligands through a N_2O_2 set. In addition, in the structure of **2**, monomeric complexes are connected to each other by C–H···π stacking interactions, resulting in a 2D architecture. These C–H···π edge-to-face interactions are present with H···π distances of 3.156 and 3.027 Å. The results of studies of the stoichiometry and formation of complex **2** in methanol solution were found to support its solid state stoichiometry.

1 Introduction

Triazene compounds having a diazoamino group (–N=NNH–) commonly adopt a *trans* configuration in the ground state.[1] Transition-metal complexes containing 1,3-diaryltriazenide ligands have been extensively studied because of the versatility of their coordination, which yield a variety of coordination compounds with large structural diversity.[2,3] Triazenes and anionic triazenide ligands show different types of coordination in metal complexes like monodentately, (N1,N3)-chelating towards one metal atom or (N1,N3)-bridging over two metal atoms,[4] showing a remarkable ability to support the stereochemical requisites of a wide variety of metal transition complexes.[5,6] In these compounds secondary bonds or interactions such as hydrogen bonds and metal π-aryl interactions can play important roles in the structural stability.[7–11] In this regard, organic ligands play an important role in adjusting the architectures of the resulting metal complexes, so proper selection of ligands is the key issue in the construction of supramolecular networks.[12–14] However, the crystal engineering with desired topologies and specific properties still remains a difficult challenge since a variety of factors influence the self-assembly processes such as coordination arrangement and oxidation state of the metal ions,[15–17] metal-to-ligand ratio,[18] nature of the ligands,[19] solvents,[20] and/or counter-anions,[21] or even the reaction temperature and pH value.[22,23]

We have previously reported the synthesis of 1,3-bis(2-methoxyphenyl)]triazene,[24] [1,3-bis(2-ethoxyphenyl)]triazene,[25] and [1,3-bis(2-cyanophenyl)]triazene[26] molecules that can act as ligands. Additionally, we have published the Hg^{II} complexes with [1,3-bis(2-methoxyphenyl)]triazene by using $HgCl_2$,[27] $HgBr_2$,[28] $Hg(CH_3COO)_2$, and $Hg(SCN)_2$ salts as starting materials.[29] Moreover, Ag^I and Cd^{II} complexes with this ligand are known.[30,31] Just recently, a Hg^{II} complex with [1,3-bis(2-ethoxyphenyl)]triazene as ligand has been reported, in which $HgCl_2$ has been used as starting salt.[32] From recent structural studies, it was argued that counter-anions play an important role in determining the solid state lattices of these compounds. Moreover, we are performing the synthesis and spectroscopic study of some new asymmetric ligands and their complexes with transition metal ions in our laboratory. In continuation with our previous work, we herein report the synthesis, characterization, and molecular structure of a new asymmetric triazene [1-(2-methoxyphenyl)-3-(4-chlorophenyl)]triazene, H**L** (**1**), and its Hg^{II} complex $[Hg(C_{26}H_{22}Cl_2N_6O_2)]$, [Hg**L**$_2$], (**2**) in methanol as solvent.

2 Experimental Section

2.1 Materials and Physical Techniques

All chemicals were of analytical grade and were used as commercially obtained without further purification. IR spectra were recorded with a Perkin-Elmer RXI spectrometer using KBr disks. Elemental analysis was carried out with a Perkin-Elmer 2400(II) CHNS/O analyzer. Melting points were measured with a Barnstead Electrothermal 9200 appa-

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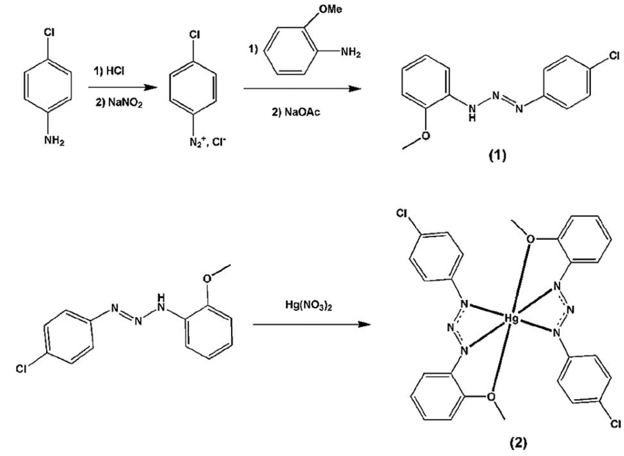
ratus. UV/Vis spectra were recorded with a Perkin-Elmer Lambda 25 spectrophotometer, using two matched 10 mm quartz cells. Crystallographic measurements were made at 296 K with a Bruker APEX II CCD area detector diffractometer. The intensity data were collected within the range 2.2–27.5 using graphite monochromated Mo-K_α radiation (λ = 0.71073 Å). The structure has been solved by direct methods and refined by full-matrix least-squares techniques on F^2 using SHELXTL.[33] The molecular structure plots were prepared using ORTEP[34] and mercury.[35] Refinement of F^2 was made against all reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt), etc. and is not relevant to the choice of reflections for refinement. R factors based on F^2 are statistically about twice as large as those based on F, and R factors based on all data are even larger.

2.2 Synthesis of [1-(2-Methoxyphenyl)-3-(4-chlorophenyl)]triazene (1)

The reaction pathway is shown in Scheme 1. *p*-Chloroaniline (6.36 g, 0.05 mol) and HCl (4.68 g, 0.13 mol, d = 1.18 g·ml^{-1}) were put in a 1000 mL flask in an ice bath. To the obtained solution, a solution of sodium nitrite (4.14 g in 25 mL H_2O) was added dropwise. Afterwards, a diluted solution of *o*-anizidine (6.15 g, 0.05 mol) in methanol (10 mL) was added to the above mixture. The pH of the solution was adjusted at about 7–8 by addition of a solution of sodium acetate (14.76 g, 0.18 mol) in H_2O (14.76 g). The solution was stirred for about 1 h, yielding a brown precipitate. It was filtered off and dried in vacuo. After dissolving in *n*-hexane and recrystallization, brown crystals of the title ligand were obtained. M.p. 68 °C. Elemental analysis for $C_{13}H_{12}ClN_3O$: calcd. C 59.80, H 4.59, N 16.01 %; found C 59.65, H 4.59, N 16.06 %. **^{1}H NMR** (300 MHz, [D$_6$]DMSO): δ = 3.82 (3 *H*, CH_3), 6.92–7.52 (8 *H*, aromatic) and 12.69 for NH group. **^{13}C NMR** (100 MHz, DMSO): δ = 55.5 (O–CH_3), 111.8–153.6 (C atoms of aromatic rings) ppm. **FT-IR** (KBr): $\tilde{\nu}$ = 3430, 3335, 2107, 1610, 1543, 1461, 1256, 1214, 1002, 822 cm^{-1}.



Scheme 1.

2.3 Synthesis of $[Hg^{II}(C_{13}H_{11}ClN_3O)_2]$, HgL_2 (2)

To a yellow solution prepared by dissolving [1-(2-methoxyphenyl)-3-(4-chlorophenyl)]triazene (0.261 g, 1 mmol) in anhydrous methanol (20 mL), mercury(II) nitrate (0.159 g, 2 mmol) dissolved in anhydrous methanol (20 mL) was added. After stirring for 30 min, a yellow precipitate was formed. It was filtered off, washed with methanol and dried in vacuo. The solid was dissolved in DMF (20 mL). The orange-colored crystals of the complex suitable for X-ray analysis was obtained by slow evaporation of the solvent in a week. M.p. 216–218 °C. Elemental analysis for $C_{26}H_{22}HgCl_2N_6O_4$ (713.15): calcd. C 47.15; H 3.92; N 11.77; found: C 47.13; H 3.69; N 11.72 %. **FT-IR** (KBr): $\tilde{\nu}$ = the N–H band is suppressed; 1020 [s, m (O–C)], 1287 cm^{-1} [s, (NNN)], a mean value with respect to the N–N absorptions in the free ligand (average bond order) cm^{-1}. **^{1}H NMR** (300 MHz, [D$_6$]DMSO): δ = 3.72 (3 *H*, CH_3), 6.95–7.67 (16 *H*, aromatic) ppm.

Crystallographic data (excluding structure factors) for the structures in this paper have been deposited with the Cambridge Crystallographic Data Centre, CCDC, 12 Union Road, Cambridge CB21EZ, UK. Copies of the data can be obtained free of charge on quoting the depository numbers CCDC-812066 and CCDC-856173 (Fax: +44-1223-336-033; E-Mail: deposit@ccdc.cam.ac.uk, http://www.ccdc.cam.ac.uk).

3 Results and Discussion

3.1 Structure Descriptions

Crystal data and experimental conditions for compounds **1** and **2** are given in Table 1. Selected bond lengths and angles are listed in Table 2. The molecular structure of compound **1** is shown in Figure 1, thermal ellipsoids are drawn at 50 % probability level. The title compound crystallizes in space group $P2_1/n$ with six molecules per unit cell. The unit cell parameters are: a = 20.5443(4) Å, b = 6.00030(10) Å, c = 10.4847(10) Å, and β = 109.7980(10)°. The final R value was 0.038 based on 4708 reflections. The hydrogen bonding arrangements are given in Table 3. The title compound exhibits a *trans* arrangement about the N2=N3 and N2A=N3A double bonds in the solid state, and individual molecules are nearly planar. The dihedral angles in both fragments between two benzene rings are 4.36° and 18.79°, respectively.

On the other hand, the C7–N1–N2–N3, C8–N3–N2–N1, C7A–N1A–N2A–N3A, and C8A–N3A–N2A–N1A torsion angles are 173.60(12)°, –6.3(2)°, 5.4(1)°, and 1.8 (3)°, respectively. The N1–N2, N2–N3, N1A–N2A, and N2A–N3A bond lengths are 1.3297(17) Å, 1.2592(16) Å, 1.3307(18) Å, and 1.2560(17) Å, respectively, which indicate the presence of distinct single and double bonds between nitrogen atoms and are in good agreement with the reported data for N=N bond lengths. In the lattice of the title ligand, the monomeric [*R*–NH–N=N–*R*] moieties are linked to pairs through non-classical C–H···N hydrogen bonds with C13A···N3 = 3.578(2) Å and C13A–H8···N3 = 174.01°. Also intramolecular classical N–H···O hydrogen bonds with N1···O1 = 2.5872(17) Å and N1A···O1A = 2.5917(17) Å are present in the crystal structure. The resulted dimeric units are connected with one another by C–H···π stacking interactions, resulting in the 2D architecture. These C–H···π stacking interactions are present between CH groups and aromatic rings with H···π distances of 2.82 Å for C3–H22···*Cg*1 (3/2–*x*, –1/2+*y*, 1/2–*z*), in which *Cg*1 is the centroid for the C2–C7 ring (see Figure 2). The unit cell packing diagram of the title compound is presented at Figure 3.

The molecular structure of compound **2** is shown in Figure 4. Thermal ellipsoids are drawn at 50 % probability level.

Table 1. Crystal data for compounds **1** and **2**.

	1	**2**
Empirical formula	$C_{17.33}H_{16}Cl_{1.33}N_4O_{1.33}$	$\mathrm{C26H22Cl_2HgN_6O_2}$
Formula weight	348.94	721.99
Temperature	296	296
Wavelength	0.71073	0.71073
Crystal system	monoclinic	monoclinic
Space group	$P2_1/n$	$P2_1/c$
Unit cell dimensions		
	a = 20.5443(4) Å	a = 19.2781(16) Å
	b = 6.00030(10) Å	b = 10.5275(9) Å
	β = 109.7980(10)°	β = 98.637(4)°
	c = 22.1483(4) Å	c = 13.2783(12) Å
Volume /Å^3	2568.89(8)	2664.3(4)
Z	6	4
Density (calculated) /Mg·m^{-3}	1.353	1.8
Absorption coefficient (mm^{-1})	0.29	6.013
F(000)	1088	1400
θ Range for data collection	2.8 to 27.5 deg	2.2 to 27.2 deg
Index ranges	$-27 \le h \le 27$	$-24 \le h \le 24$
	$-7 \le k \le 7$	$-13 \le k \le 13$
	$-29 \le l \le 29$	$-16 \le l \le 16$
Reflections collected/unique	133163/4708	85027/4951
	[R(int) = 0.026]	[R(int) = 0.035]
Absorption correction	multi-scan, Sheldrick, G. M. (2004)	multi-scan, Sheldrick, G. M. (2004)
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	4708/ 0 / 336	4951 / 0 / 337
Goodness-of-fit on F^2	1.02	1.07
Final $R[I > 2\sigma(I)]$	R_1 = 0.038 wR_2 = 0.10	R_1 = 0.017, wR_2 = 0.036
R indices (all data)	R_1 = 0.05, wR_2 = 0.12	R_1 = 0.025, wR_2 = 0.039
Largest differences in peak and hole /e·A^{-3}	0.21 and –0.2	0.52 and –0.58

Table 2. Selected bond lengths /Å and angles /° for compounds **1** and **2**.

1			
O1–C2	1.3692(17)	O1A–C2A	1.3715(18)
O1–C1	1.4174(19)	O1A–C1A	1.4158(19)
N1–N2	1.3297(17)	N1A–N2A	1.3307(18)
N3–N2	1.2592(16)	N2A–N3A	1.2560(17)
N3–N2–N1	111.44(12)	N3A–N2A–N1A	111.53(12)
2			
Hg–N3	2.0686(19)	N2–N1	1.273(3)
Hg–N6	2.0764(19)	N2–N3	1.322(3)
Hg–O2	2.6731(18)	N4–N5	1.271(3)
Hg–O1	2.6751(18)	N6–N5	1.322(3)
N3–Hg–N6	178.53(7)	N3–Hg–O1	66.70 (6)
N3–Hg–O2	112.07(7)	N6–Hg–O1	114.66(7)
N6–Hg–O2	66.68(7)	O2–Hg–O1	131.31(7)
N1–N2–N3	111.72(19)	N4–N5–N6	112.12(19)

The title complex crystallized in space group $P2_1/c$ with four molecules per unit cell. The unit cell parameters are: a = 19.2781(16) Å, b = 10.5275(9) Å, c = 13.2783(12) Å, and β = 98.637(4)°. The final R value was 0.017 based on 4951 reflections. The Hg^{II} atom is coordinated by two symmetrically independent triazenide ions. Each triazenide ion is coordinated to central atom through two nitrogen atoms [Hg1–N1 = 2.757(2) Å and Hg1–N3 = 2.0686(19) Å] for one ligand, and [Hg1–N4 = 2.766(2) Å and Hg1–N6 = 2.0764(19) Å] for the other one, and one oxygen atom [Hg1–O1 = 2.6751(18) Å, and Hg1–O2 = 2.6731(18) Å], which are relatively weaker interaction. Hg–O and Hg–N bonds with longer distance have been reported in the literature.[36,37] In the lattice of compound **2**, the monomeric [Hg(RNNNR)$_2$] moieties are linked to parallel chain through C–H···π stacking interactions (Figure 5). These C–H···π edge-to-face interactions are present between CH group of methoxy with aromatic rings with H···π distances of 3.156 Å and 3.027 Å for C24–H16···*Cg2* (x, 1/2–y, 1/2+z) and C26–H45C···*Cg2* [*Cg2* = C7–C12], respectively.

3.2 Solution Studies

In a typical procedure, the ligand solution (2.0 mL, 5.0 × 10^{-5} M) in MeOH was placed in the spectrophotometer cell and

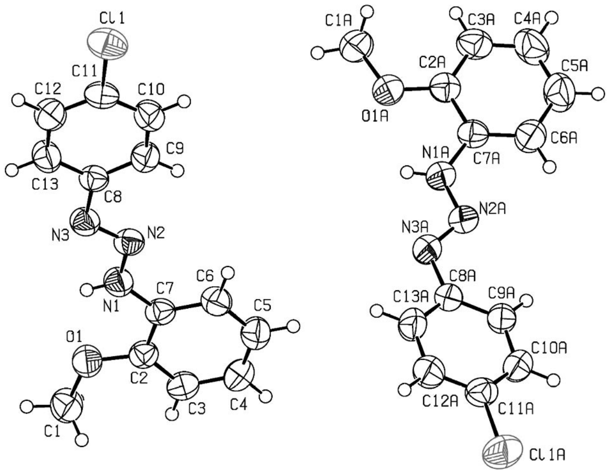


Figure 1. ORTEP diagram of the structural unit of the ligand **1** with the atomic numbering scheme. Thermal ellipsoids are drawn at 50 % probability level.

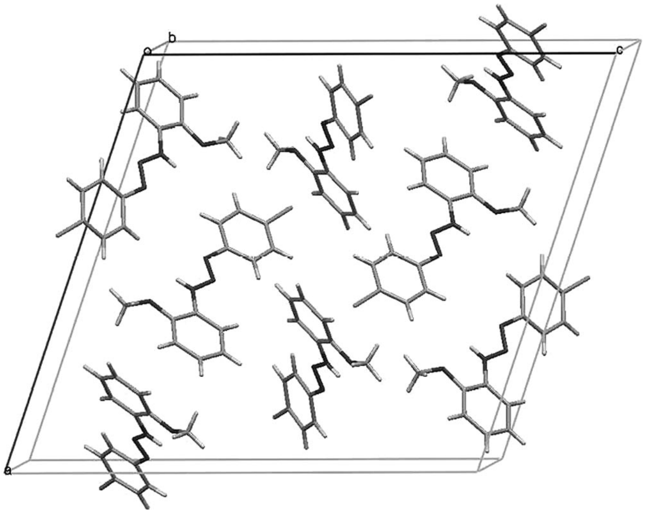


Figure 3. Unit-cell packing diagram of **1** viewed down *b* axis.

Table 3. Hydrogen bonds and C–H···π interactions arrangement /Å,° for compounds **1** and **2**[a)].

D–H···*A*	*d*(*D*–H)	*d*(H···*A*)	*d*(*D*···*A*)	<(*D*–H···*A*)
1				
N1A–H55···O1A	0.82	2.251	2.5917(17)	105.3
N1–H56···O1	0.80	2.249	2.5872(17)	106.3
C3–H22···*Cg*1 #1	0.93	2.820	3.5779(17)	139
2				
C24–H16···*Cg2* #2	0.93	3.156	4.019(3)	155.3
C26–H45C···*Cg2* #2	0.96	3.027	3.737(3)	132

a) *Cg*1 and *Cg*2 are the centers of the C2–C7 and C7–C12 rings, respectively. #1 (3/2–*x*, –1/2+*y*, 1/2–*z*); #2 (*x*, 1/2–*y*, 1/2+*z*).

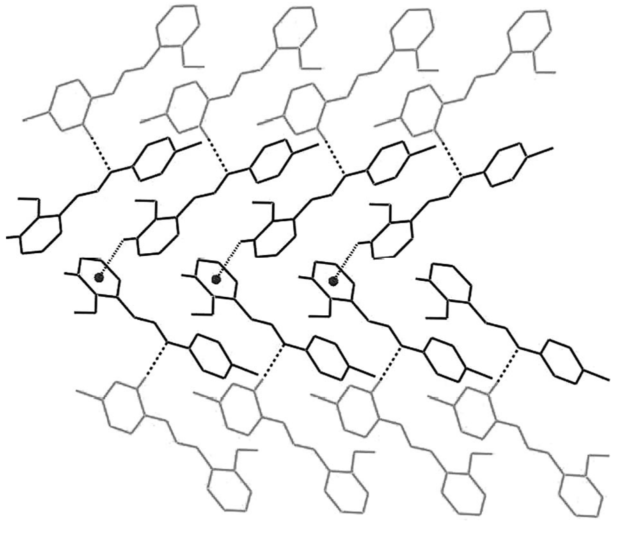


Figure 2. View of supramolecular network formed by C–H···N and C–H···π interactions in **1**.

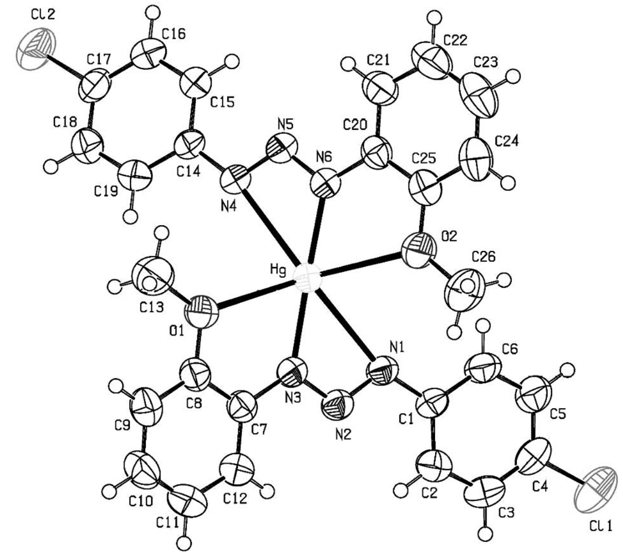


Figure 4. ORTEP diagram of the structural unit of complex **2** with the atomic numbering scheme. Thermal ellipsoids are drawn at 50 % probability level.

the absorbance of the solution was measured. Afterwards, a known amount of the concentrated solution of mercury(II) nitrate in MeOH (5.0×10^{-3} M) was added in a stepwise manner using a 10 µL Hamilton syringe. The absorbance spectrum of the solution was recorded after each addition. The mercury(II) nitrate solution was continuously added until the desired metal to ligand mole ratio was achieved. The electronic absorption spectra of the ligand in the presence of increasing concentration of mercury(II) nitrate at room temperature is shown in Figure 6. The resulting absorbance (at 366 nm) against [Hg^{2+}]/

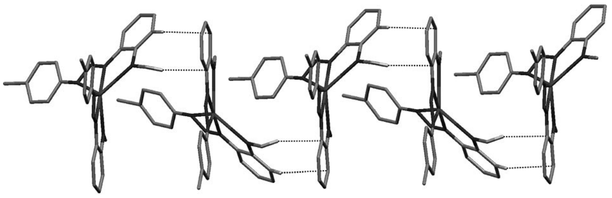


Figure 5. C–H···π stacking interactions between two [HgL$_2$] moieties in **2**.

[HL] mole ratio plot reveals distinct inflection points at a metal-to-ligand molar ratio of about 0.5 and 1 emphasizing the formation of 1:2 and 1:1 complexes. For evaluation of the conditional formation constants, the mole ratio data obtained by the physicochemical method employed were fitted to the previously reported equations,[38,39] using a non-linear least-squares curve fitting program KINFIT.[40] The conditional formation constants were evaluated as log $K_1 = 4.78 \pm 0.003$ and log $K_2 = 4.85 \pm 0.001$ for the formation of 1:2 and 1:1 complexes, respectively. For the confirmation of the obtained results, the complex formation was also investigated by ^{1}H NMR spectroscopy. In this procedure, the ligand (0.5 mL, 0.005 M) was dissolved in [D$_6$]DMSO and its spectra was recorded. Subsequently, a certain amount of the concentrated solution of mercury(II) nitrate in [D$_6$]DMSO (5.0×10^{-2} M) was added in a stepwise manner using a 10 µL Hamilton syringe. The ^{1}H NMR spectra of the solution were recorded after each addition. The mercury(II) nitrate solution was continuously added until the desired metal to ligand mole ratio was achieved. Figure 7 shows the changes in the ^{1}H NMR spectra patterns. Distinct chemical shifts at a metal-to-ligand molar ratio of about 0.5 and 1 confirm the formation of 1:2 and 1:1 complexes. The resulting spectra clearly indicated that the sharp singlet of O–Me protons observed at $\delta = 3.82$ ppm for ligand, shifted up-field upon addition of Hg^{2+} ions to [Hg^{2+}]/[HL] = 0.5 and shifted down-field upon addition of Hg^{2+} ions to metal-to-ligand molar ratio of about 1.

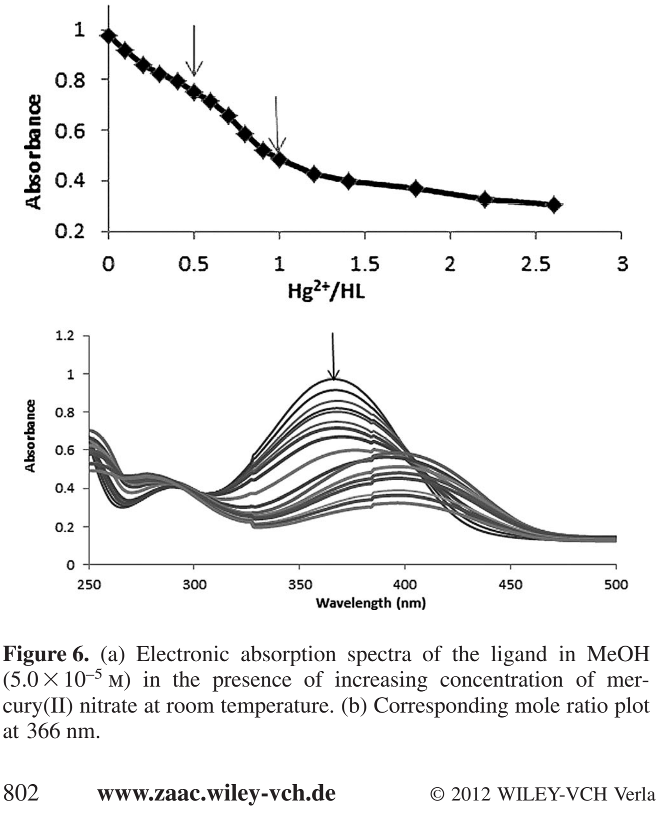


Figure 6. (a) Electronic absorption spectra of the ligand in MeOH (5.0×10^{-5} M) in the presence of increasing concentration of mercury(II) nitrate at room temperature. (b) Corresponding mole ratio plot at 366 nm.

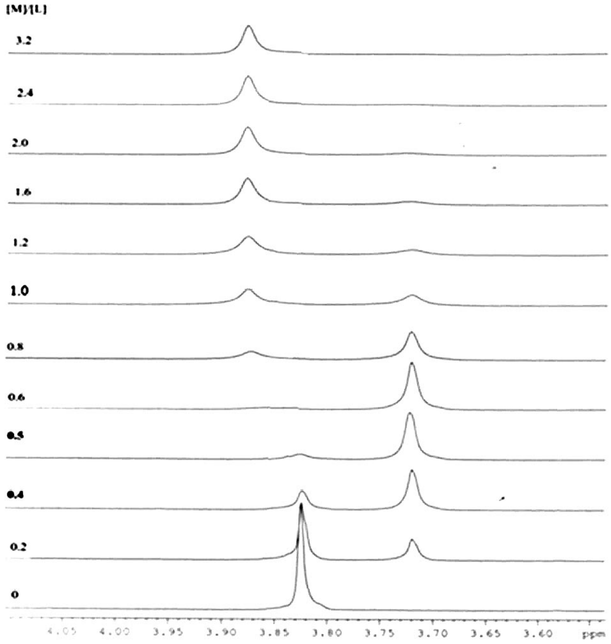


Figure 7. NMR spectra of the ligand [HL] in (C$_4$D$_8$O) (5.0×10^{-3} M) in the presence of increasing concentration of mercury(II) nitrate at room temperature.

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