Polyhedron 44 (2012) 138–142

Contents lists available at SciVerse ScienceDirect

Polyhedron

journal homepage: www.elsevier.com/locate/poly

Synthesis, characterization and crystal structures of Hg^{II} complexes with asymmetric ortho-functionalized 1,3-bis(aryl)triazenide ligands

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ARTICLE INFO

Article history:
Received 28 March 2012
Accepted 19 June 2012
Available online 28 June 2012

Keywords:
Mercury(II)
Triazene
X-ray crystal structure
π···π Stacking
C–H···π interaction

ABSTRACT

The synthesis of two $[Hg(L1)_2]$ and $[Hg(L2)_2]$ complexes using asymmetric triazenes as ligands are reported. The triazene ligands are substituted with cyano and chloride groups in the *ortho* positions of the aryl rings (where HL1 and HL2 are 1-(2-ethoxyphenyl)-3-(2-cyanophenyl)triazene and 1-(2-ethoxyphenyl)-3-(2-chlorophenyl) triazene, respectively). These complexes were prepared by the reaction of corresponding triazenes with $Hg(NO_3)_2$ and were characterized by FT-IR, NMR, elemental and single crystal X-ray analyses. Both triazene ligands were found to deprotonate on coordination and act as tridentate chelating ligands forming distorted N_4O_2 octahedral geometry around Hg^{II} atoms. Hydrogen bonds, π···π and C–H···π stacking interactions help to the stabilization of the resulted frameworks.

1. Introduction

Hydrogen-bonding and other types of non-covalent interactions like π···π and C–H···π stacking interactions play an important role in the building of supramolecular systems [1,2]. Aryl triazenes have been studied over several years for their interesting structural, anticancer, and reactivity properties [3–10]. Triazene compounds characterized by having a diazoamino group (–N=NNH–) commonly adopt a *trans* configuration in the solid state. As ligands, the N=NNH moieties can show different types of coordination in metal complexes. They can be monodentate, (N1,N3)-chelating towards one metal atom or (N1,N3)-bridging over two metal atoms [11], toward wide variety of metal transition complexes [12,13]. In these compounds secondary bonds, or interactions, such as hydrogen bonds and metal·π-aryl interactions, can play an important role in their structural stability [14,15]. In recent years, we have been involved in the complexation of transition metal ions with several triazene compounds as ligands. Starting from different bis(diaryl) symmetric and asymmetric-substituted triazenide as ligand. Recently, we have shown that several complexes of Hg^{II} have a remarkable ability to self-assemble in different manners through metal–η^2–η^2-arene π-interactions and non-classical C–H···O hydrogen bonding. We have previously reported the synthesis of [1,3-bis(2-methoxyphenyl)]triazene [16], [1,3-bis(2-ethoxyphenyl)]triazene [17] and [1,3-bis(2-cyanophenyl)]triazene [18] molecules that can act as ligands. Also, we have published the Hg(II) complexes with [1,3-bis(2-methoxyphenyl)]triazene by using $HgCl_2$ [19], $HgBr_2$ [20], $Hg(CH_3COO)_2$ and $Hg(SCN)_2$ [21] salts as starting materials. More recently, a Hg(II) complex with [1,3-bis(2-ethoxyphenyl)]triazene as ligand is reported in which $Hg(NO_3)_2$ is used as starting salt [22]. To investigate the effect of the substituted derivatives on coordination behavior of the triazene ligands, we have introduced ortho-, meta- and para-bis(phenyltriazene)benzenes (and substituted derivatives), with two triazene groups in one molecule as ligands. In this work we studied the complexation of 1-(2-ethoxyphenyl)-3-(2-chlorophenyl)triazene and 1-(2-ethoxyphenyl)-3-(2-cyanophenyl)triazene) with nitrate salt of Hg^{II} ion in aqueous solution, in order to investigate the stoichiometry of the resulting mercury(II) complexes.

2. Experimental

2.1. Materials and physical techniques

All chemicals were of analytical grade and were used as commercially obtained without further purification. IR spectra in the frequency range of 4000–400 cm^{-1} were recorded using

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0277-5387/$ - see front matter
http://dx.doi.org/10.1016/j.poly.2012.06.045

Perkin–Elmer RXI spectrometer using KBr disks. Elemental analysis was carried out using a Perkin–Elmer 2400(II) CHNS/O analyzer. Melting points were measured on a Barnstead Electrothermal 9200 apparatus.

2.1.1. Synthesis of [(EtOPhNHNNPhCN)], [HL1]

A 100 ml flask was charged with 10 g of ice and 15 ml of water and then cooled to 273 K in an ice-bath. To this was added 2 mmol (0.24 g) of 2-aminobenzonitrile and 2 mmol of hydrochloric acid (36.5%) and 2 ml of water. To this solution was then added a solution containing $NaNO_2$ (0.32 g) in 3 ml of water during a 15 min period. The *p*H of the solution was then adjusted at 6 by adding a solution containing 1 g of sodium acetate in 10 ml of water. After mixing for 15 min, the obtained solution was added to a solution of 2 mmol (0.26 ml) of *o*-phenetidine and 2 ml of methanol and 4 ml of water. After mixing for 24 h, the yellow-orange precipitate was filtered off and dried. Our attempt to crystallize this compound was unsuccessful. m.p.: 121–123 °C. IR (KBr): $\nu(\text{cm}^{-1})$: 3331, 2223, 1602, 1513, 1478, 1470, 1437 , 1313, 1275, 1231, 1119, 1040, 927, 770, 735, 603. ^1H NMR (300 MHz, d_6-DMSO): 1.38 (3H, CH_3), 4.13 (2H, CH_2), 6.90–7.85 (8H, aromatic), 12 (1H, NH). ^{13}C NMR (300 MHz, d_6-DMSO): 14.5, 64.0, 102.2–145.5 ppm. Elemental *Anal.* calc. for $C_{15}H_{14}N_4O$: C, 67.66; H, 5.26; N, 21.05. Found: C, 67.38; H, 4.26; N, 20.65%.

*2.1.2. Synthesis of [(EtOPhNHNNPhCl)] [HL2], (**1**)*

This ligand was also prepared by the same method as described above with this difference that *o*-chlorobenzenamine (0.1 mol, 10.54 ml) was used as starting material. Also, recrystallization was performed by *n*-hexane. m.p.: 73–75 °C. IR (KBr): $\nu(\text{cm}^{-1})$: 3336, 3067, 1600, 1530, 1467, 1437, 1410 , 1306, 1259, 1212, 1119, 1045, 1035, 746, 732, 719. ^1H NMR (300 MHz, d_6-DMSO): 1.38 (3H, CH_3), 4.12 (2H, CH_2), 6.92–7.61 (8H, aromatic), 11.72 (1H, NH). ^{13}C NMR (300 MHz, d_6-DMSO): 14.7, 64.1, 112.8–146.5 ppm. Elemental *Anal.* calc. for $C_{14}H_{14}N_3OCl$: C, 60.98; H, 5.12; N, 15.24. Found: C, 61.04; H, 5.0; N, 15.29%.

*2.1.3. Synthesis of [Hg^{II}(EtOPhNNNPhCN)$_2$], [Hg(L1)$_2$], (**2**)*

The complex was prepared by mixing 0.27 g (1 mmol) of [1-(2-ethoxyphenyl)-3-(2-cyanophenyl)triazene) in 10 ml of anhydrous methanol with 0.27 g (1 mmol) of mercury(II) nitrate in 10 ml of anhydrous methanol. After being mixed for an hour, a yellow precipitate was obtained. The resultant precipitate after filtration and washing was dissolved in THF. Yellow needle-like crystals suitable for X-ray analysis of the complex (**2**) were obtained by slow evaporation of the solvent in 2 weeks.

$C_{30}H_{26}HgN_8O_2$ (731.18); m.p.: 219–221 °C. IR (KBr): $\nu(\text{cm}^{-1})$: 3069, 2977 , 2220, 1589, 1494, 1477, 1372, 1333, 1291, 1275, 1218, 1161, 1120, 1038, 918, 743, 734, 672. ^1H NMR (300 MHz, d_6-DMSO): 1.16 (3H, CH_3), 4.10 (2H, CH_2), 7.00–7.81 (8H, aromatic). ^{13}C NMR 300 MHz, d_6-DMSO): 14.0, 64.5, 104.8–151.4 ppm. Elemental *Anal.* calc. for $C_{30}H_{26}HgN_8O_2$: C, 49.31; H, 3.56; N, 15.34. Found: C, 49.60; H, 3.38; N, 15.26%.

*2.1.4. Synthesis of [Hg^{II}(EtOPhNNNPhCl)$_2$], [Hg(L2)$_2$], (**3**)*

To a yellow solution prepared by dissolving 0.29 g (1 mmol) of 1-(2-ethoxyphenyl)-3-(2-chlorophenyl)triazene in 40 ml of anhydrous methanol, 0.16 g (0.5 mmol) of mercury(II) nitrate dissolved in 5 ml of anhydrous methanol were added. After 1/2 h stirring, an orange precipitate was formed. It was then filtered off, washed with methanol and dried in vacuum. The solid was dissolved in 20 ml THF. Orange-red plate like crystals suitable for X-ray analysis of the complex, were obtained by slow evaporation of the solvent within a week. IR (KBr): $\nu(\text{cm}^{-1})$: 2881, 1584, 1493, 1470, 1456, 1386, 1286, 1263, 1235, 121, 1196, 1121, 1035, 918, 759, 741, 735. ^1H NMR (300 MHz, d_6-DMSO): 1.17 (3H, CH_3), 4.06 (2H, CH_2), 6.99–7.73 (8H, aromatic). ^{13}C NMR (300 MHz, d_6-DMSO): 14.1, 64.3, 113.0–150.5 ppm. Elemental *Anal.* calc. for $C_{28}H_{26}Cl_2HgN_6O_2$: C, 44.84; H, 3.49; N, 11.2. Found: C, 45.18; H, 3.51; N, 11.12%.

3. Crystallography

Crystallography measurements were made using a Bruker APEX2 CCD diffractometer for compounds (2) and (3), and also

Table 1
Crystal data for the compounds (**1**)–(**3**).

	1	**2**	**3**
Empirical formula	$C_{14}H_{14}ClN_3O$	$C_{30}H_{26}HgN_8O_2$	$C_{28}H_{26}Cl_2HgN_6O_2$
Formula weight	275.73	731.18	750.04
T (K)	298	293	293
λ (Å)	0.71073	0.71073	0.71073
Crystal system	monoclinic	triclinic	monoclinic
Space group	$P2_1/c$	$P\bar{1}$	$C2/c$
Unit cell dimensions			
a (Å)	14.688 (3)	7.9472 (3)	22.497 (4)
b (Å)	4.4064 (9)	8.6812 (3)	9.049 (2)
c (Å)	22.337 (8)	20.7136 (7)	14.746 (3)
α (Å)		91.245 (2)	
β (Å)	111.46 (2)	92.970 (2)	108.89 (2)
γ (Å)		91.811 (2)	
V (Å^3)	1345.5 (6)	1426.03 (9)	2840.2 (10)
Z	4	2	4
D_{calc} (Mg m^{-3})	1.361	1.703	1.754
Absorption coefficient (mm^{-1})	0.279	4.439	5.646
F(000)	576	716	1464
θ Range for data collection (°)	1.96–29.22	0.98–30.06	2.68–30.30
Index ranges	$-20 \leqslant h \leqslant 20, -6 \leqslant k \leqslant 5, -28 \leqslant l \leqslant 30$	$-11 \leqslant h \leqslant 11, -12 \leqslant k \leqslant 12, -29 \leqslant l \leqslant 29$	$-31 \leqslant h \leqslant 31, -12 \leqslant k \leqslant 12, -20 \leqslant l \leqslant 20$
Reflections collected/unique (R_{int})	13923/3595 (0.0905)	57106/8309 (0.052)	58699/4223 (0.031)
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	3595 / 0 / 177	8309 / 0 / 370	4223 / 0 / 177
Goodness-of-fit on F^2	1.134	1.14	1.019
Final $R[I > 2\sigma(I)]$	$R1 = 0.0742$, $wR2 = 0.1335$	$R1 = 0.0654$, $wR2 = 0.1665$	$R1 = 0.0242$, $wR2 = 0.0718$
R indices (all data)	$R1 = 0.1346$, $wR2 = 0.1553$	R1 = 0.0748, wR2 = 0.1702	R1 = 0.0273, $wR2 = 0.0737$
Largest differences in peak and hole (e A^{-3})	0.231 and −0.184	2.89 and −4.32	0.89 and −1.36

STOE IPDS 2T diffractometer for compound (1). [23]. The intensity data were collected 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 [24]. The molecular structure plots were prepared using Platon [25] and mercury [26]. 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 details of data collection, refinement and crystal data are summarized in Table 1.

4. Results and discussion

The molecular structure of *[(EtOPhNHN = NPhCl)],[HL2],* (**1**) is presented in Fig. 1. Crystal data for compound (**1**) is given in Table 1. Selected bond distances and angels are listed in Table 2. This molecule crystallized in $P2_1/c$ space group with four molecules per unit cell. As it can be seen, the molecule adopts *trans* configuration with respect to the (–N=N–) bond. In the FT-IR spectra, the band at 3336 cm^{-1} is responsible for N—H bond stretching. The N—N and N=N bond stretching modes appear at 1119 and 1437 cm^{-1}, respectively. The peak at 1231 cm^{-1} is assigned to the stretching of C—O bond. In ^{1}H NMR spectra, the peak at 11.72 ppm can be assigned to the presence of NH group. In addition, two aromatic rings are twisted with respect to each other and the dihedral angel between two aromatic rings is 17.81(14)°. Also, the N1—N2 and N2—N3 bond distances are 1.328(3) Å and 1.263(3) Å, which indicated the presence of single and double bond characters, respectively. The bond distances and bond angles in the title compound are in agreement with the corresponding bond distances and bond angles reported for a closely related structure, 3-(2-ethoxyphenyl)-1-(3-nitrophenyl)triaz-1-ene [27]. There is also weak π···π stacking interactions between parallel phenyl rings that stack the molecules in the *b* direction and the mean centroid–centroid distance is 4.4 Å. So, in the lattice of this compound, the molecules are stacked along the crystallographic *b*-axis to form infinite chains. In addition, a distinct intermolecular N1—H1···O1 hydrogen bond with D···A distance of 2.618(3) Å is present in this compound (see Table 3).

Compound (**2**), *[Hg^{II}(EtOPhNNNPhCN)$_2$], [Hg(L1)$_2$],* was prepared by reacting the corresponding ligand with Hg(NO$_3$)$_2$ salt. However, we could not obtain suitable single crystals of the HL1 ligand itself. The molecular structure of the compound (**2**) is shown in Fig. 2, with ellipsoids drawn at 30% probability level. The molecule crystallized in $P\bar{1}$ space group with two molecules per unit cell. The central atom is surrounded by two L1 ligands through six atoms, [Hg—N1 = 2.101(7) Å, Hg—N3 = 2.670(7) Å, Hg—N4 = 2.096(8) Å, Hg—N6 = 2.664(8) Å] and [Hg—O1 = 2.654(7) Å, Hg—O2 = 2.704(7) Å], in which the N_4O_2 set can be considered as a distorted octahedral (Fig. 3). As found before, in ^{1}H NMR spectra, the peak for NH group of free ligand is disappeared *via* deprotonation prior to complexation. Hg—O and Hg—N bond distances are in a range which is in good agreement with distances reported for similar compounds [22]. Another interesting feature of the compound (**2**), is the presence of π···π and C–H···π interactions. These π···π interactions

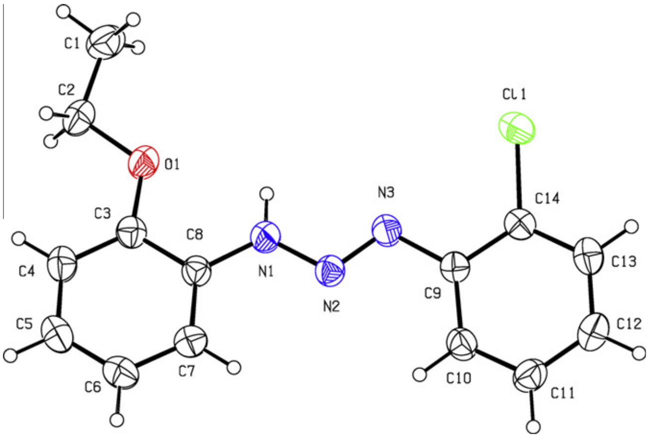


Fig. 1. ORTEP diagram of the structural unit of (**1**) with the atomic numbering scheme. Thermal ellipsoids are drawn at 30% probability level.

Table 2
Selected bond lengths and angles for compounds (**1**)–(**3**).

*Compound (**1**)*			
N1—N2	1.328 (3)	N3—C9	1.417 (3)
N2—N3	1.263 (3)	N1—C8	1.397 (3)
O1—C3	1.375 (3)	Cl1—C14	1.737 (3)
N3—N2—N1	112.9 (2)	N2—N3—C9	112.2 (2)
N2—N1—C8	121.3 (2)	C3—O1—C2	117.7 (2)
*Compound (**2**)*			
Hg—N1	2.101 (7)	Hg—N4	2.096 (8)
Hg—N3	2.670 (7)	Hg—N6	2.664 (8)
Hg—O1	2.654 (7)	Hg—O2	2.704 (7)
*Compound (**3**)*			
Hg1—N1	2.066 (3)	N1^i—Hg1—N1	176.19 (11)
Hg1—N3	2.754 (3)		
Hg1—O1	2.723 (3)		

Symmetry code: (i) $-x$, y, $-z + 1/2$.

Table 3
Hydrogen bond geometries for compounds (**1**)–(**3**).

D–H···A	D–H (Å)	H···A(Å)	D···A(Å)	<D–H···A
*Compound (**1**)*				
N1–H1···O1	0.83(3)	2.28(3)	2.618(3)	105(3)
*Compound (**2**)*				
C11–H11···N8 $^{\#1}$	0.93	2.48	3.337(15)	153
C29–H29B···N8 $^{\#2}$	0.97	2.60	3.337(17)	137
*Compound (**3**)*				
C4–H4···N2 $^{\#3}$	0.93	2.56	3.419(6)	153

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

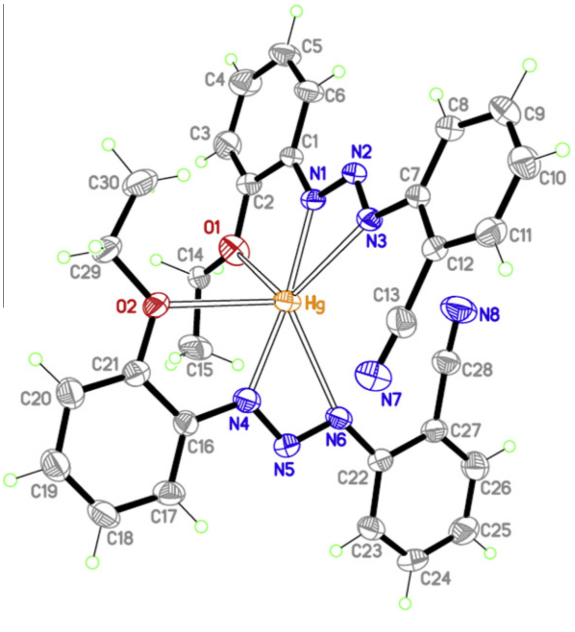


Fig. 2. ORTEP diagram of the structural unit of (**2**) with the atomic numbering scheme. Thermal ellipsoids are drawn at 30% probability level.

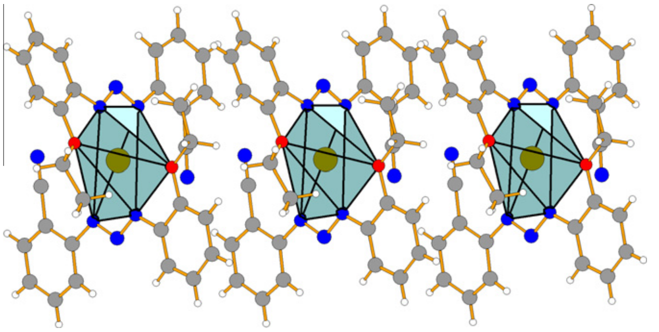


Fig. 3. Octahedral polyhedrons around the Hg^{II} atoms in the compound (**2**).

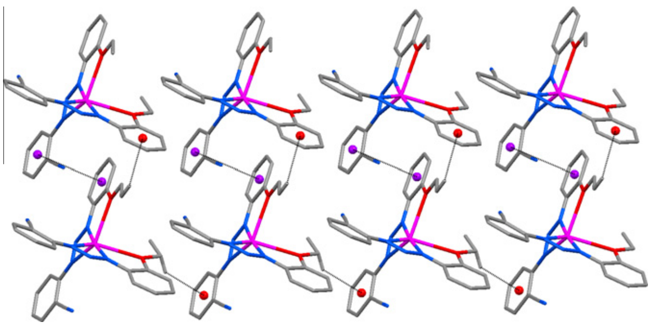


Fig. 4. Presence of $\pi\cdots\pi$ and C–H$\cdots\pi$ interactions between *Cg*1—*Cg*2 with centroid–centroid distance of 3.824(6) Å and C—H$\cdots\pi$ edge-to-face interactions between CH groups with H$\cdots\pi$ distance of 2.78 Å for C15—H15A$\cdots$*Cg*3 and 2.83 Å for C29—H29A$\cdots$*Cg*2, [*Cg*1, *Cg*2 *Cg*3 are centers of C1/C6, C7/C12 and C16/C21 rings, respectively].

are exist between *Cg*1—*Cg*2 with centroid–centroid distance of 3.824(6) Å, in which *Cg*1 and *Cg*2 are centers of C1/C6 and C7/C12 rings, respectively. Also, C—H$\cdots\pi$ edge-to-face interactions are present between CH groups with H$\cdots\pi$ distance of 2.78 Å for C15—H15A$\cdots$*Cg*3 (1 + *x*, *y*, *z*) and 2.83 Å for C29—H29A$\cdots$*Cg*2 (*x*, 1 + *y*, *z*), [Cg3 is centroid for C16/C21 ring]. These non-covalent interactions bring together the fragments in which layers of $[Hg(L1)_2]$ complexes forms (Fig. 4).

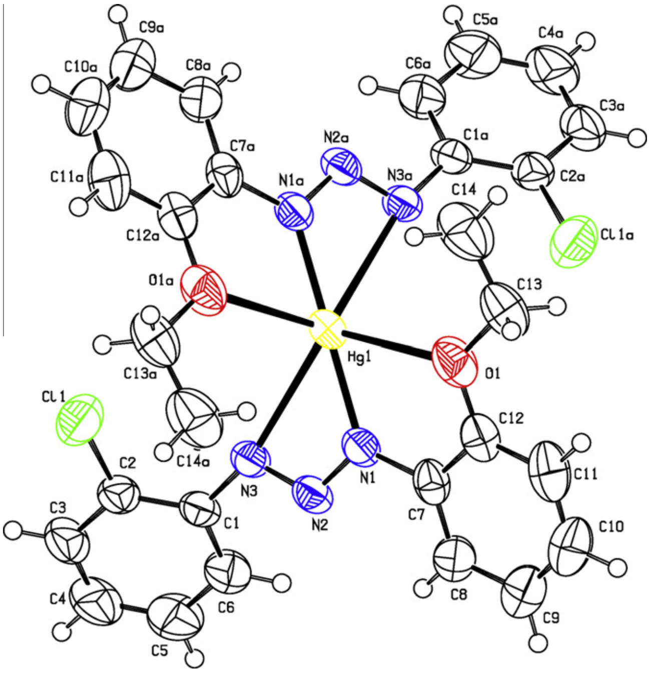
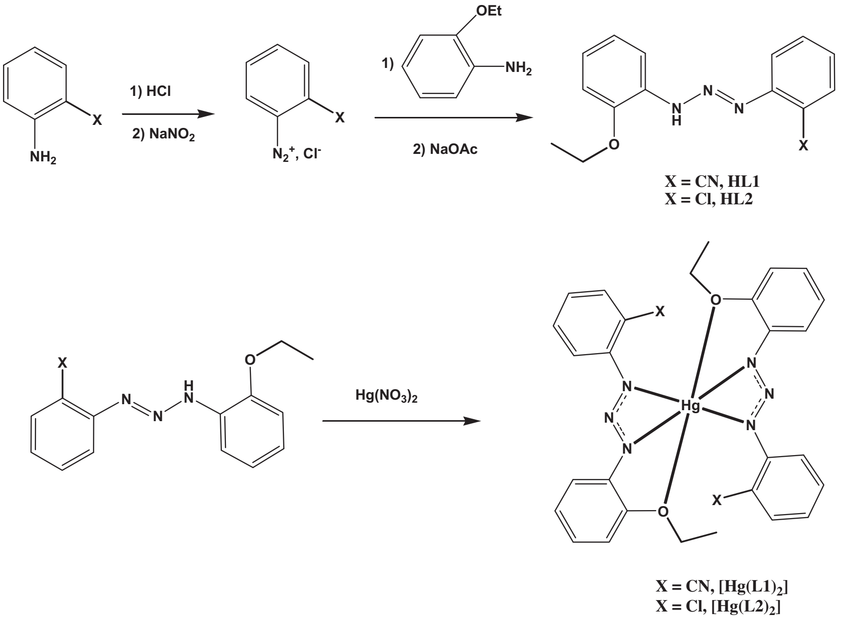


Fig. 5. ORTEP diagram of the structural unit of (**3**) with the atomic numbering scheme. Thermal ellipsoids are drawn at 50% probability level.

Compound (**3**), *$[Hg^{II}(EtOPhNNNPhCl)_2]$, $[Hg(L2)_2]$*, was also prepared by reacting the corresponding ligand with Hg^{II} nitrate and its preparation procedure is outlined in Scheme 1. The molecular structure of the compound (**3**) is presented in Fig. 5. Crystal data for complex (**3**) is also given in Table 1. The molecule crystallized in *C*2/*c* space group with four molecules per unit cell. As it can be seen, the Hg^{II} atom is coordinated by two interlocked triazenide ligands and the central Hg^{II} atom is six-coordinated by two tridentate L2 ligands through nitrogen [Hg1—N1 = 2.066(3), Hg1—N3 = 2.754(3)] and oxygen [Hg1—O1 = 2.723(3)] atoms. By comparison of ^{1}H NMR spectra of the complex (**3**) with that of the free ligand, it is found that the signal for N—H (at 11.72 ppm) is



Scheme 1. Synthesis procedure for preparation of compounds.

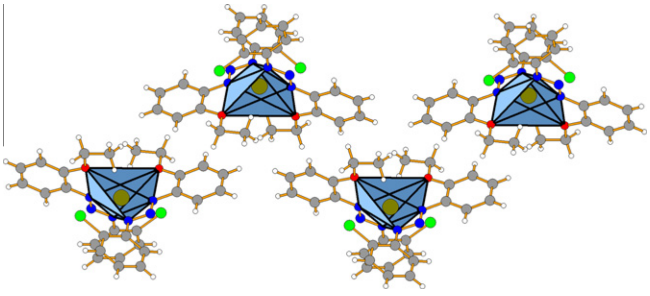


Fig. 6. Octahedral polyhedrons around the Hg^{II} atoms in the compound (**3**).

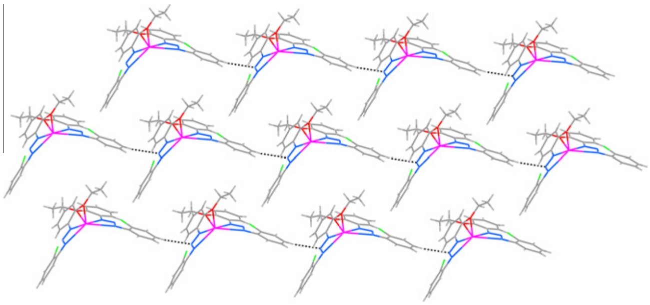


Fig. 7. Non-classic C—H···N hydrogen bonds (dashed lines) with D···A distance of 3.419(6) Å connect the monomeric fragments into finite chains in a wave-like shape in compound (**3**).

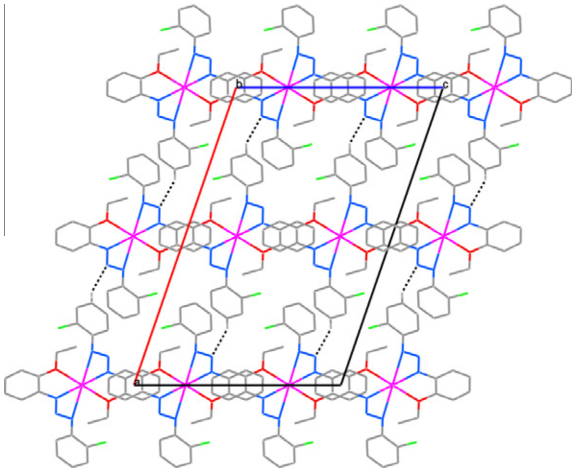


Fig. 8. Unit cell packing diagram of the compound (**3**) viewed down *c* axis. C—H···N hydrogen bonds are shown by dashed lines.

disappeared which indicates deprotonation of the NH group due complexation. The N1—N2 and N2—N bond distances are 1.304(4) and 1.271(4) Å, respectively which indicates the formation of resonance structure. Hg–O bonds are relatively weaker interaction. Hg—O and Hg—N interactions with longer bond distances have been reported in the literature [28]. The N1—Hg1—N1^{i} bond angel is 176.19(11)°, which is deviated from linearity, and the coordinating N_4O_2 set can be described as distorted octahedral (Fig. 6). In addition, the presence of non-classic C4—H4···N2 hydrogen bonds with D···A distance of 3.419(6) Å connect the monomeric fragments into finite chains in a wave-like shape, in which a weaker $\pi\cdots\pi$ stacking interactions help to the stabilization of the resulted structure (See Fig. 7 and Table 3). The unit cell packing diagram of the compound (**3**) is illustrated in Fig. 8.

Appendix A. Supplementary data

CCDC 859577, 859582 and 871223 contains the supplementary crystallographic data for compounds (**1**) to (**3**), respectively. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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