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Some Hg(II) Complexes with Tetradentate Schiff Base Ligands

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Sixteen complexes of Hg(II) of the type $HgX_2 \cdot SB$ (where $X = Cl^-$ or NO_3^-; SB = tetradentate aromatic Schiff base ligands derived from diamines and glyoxal; 2,3-butanedione; acetylacetone and 1-phenylbutane-1, 3-dione) have been synthesised and characterised by elemental analysis, conductivity, IR and XPS data.

Although a number of Hg(II) complexes are known in the literature[1], Hg(II) complexes with aromatic Schiff base ligands are less known[2–7]. We report here the synthesis of some Hg(II) complexes with tetradentate ligands derived from ethylenediamine/phenylenediamine and glyoxal, 2, 3-butanedione, acetylacetone and 1-phenylbutane 1, 3-dione and their characterisation by physico-chemical and spectroscopic data.

The corresponding aromatic carbonyl compound (2 mmol) and the diamine (2 mmol) were mixed in methanol and refluxed for 3 h. TLC suggested complete conversion of the starting materials to the Schiff base. The HgX_2 (1 mmol) was then added and the mixture was refluxed for 3 h. The resulting solid was washed with methanol and air-dried.

Conductance measurements in DMF were made at room temperature using a Digisun Electronic conductivity bridge. Infrared spectra of ligands and complexes were recorded in nujol and KBr respectively on a Perkin-Elemer 457 spectrophotometer. The X-ray photoelectron spectra were recorded on a VG Scientific ESCA-3 MK(II) electron spectrometer. The AlK (1486.8 eV) was used for photoexcitation. The $Cu^2P_{3/2}$ (BE = 932.8 – 0.2) and $Au^4f_{7/2}$ (BE = 83.8 – 0.1 lines were used to calibrate the instrument and $Ag^3d_{5/2}$ (BE = 368.2) was used for cross checking[8]. All the spectra were recorded using the same spectrometer parameters of 50 eV pass energy and 4 mm slit-width. The reduced full-width at half maximum (FWHM) at $Au^4f_{7/2}$ (BE = 83.8 eV) level under these conditions was 1.2 eV. The usual least-square fitting procedure of determining peak position, line width and area was used.

Addition complexes of HgX_2 ($X = Cl^-, NO_3^-$) with the following tetradentate Schiff base ligands have been prepared: SB_1 = bis (glyoxal) ethylenediamine; SB_2 = bis (2,3-butanedione)ethylenediamine; SB_3 = bis (acetylacetone) ethylenediamine; SB_4 = bis (1-phenylbutance 1, 3-dione) ethylenediamine; SB_5 = bis (glyoxal) phenylenediamine; SB_6 = bis (2, 3-butanedione) phenylenediamine; SB_7 = bis (acetylacetone) phenylenediamine and SB_8 = bis (1, phenylbutance 1, 3-dione) phenylenediamine.

All these complexes are stable with high melting points and insoluble in common organic solvents except DMF and DMSO. Elemental analysis ($\pm 0.5\%$ for C, H and N) and molar conductance data (below than 60 ohm^{-1} cm^{-2} mol^{-1}) suggest that all complexes are non-electrolyte[9] with composition of $HgX_2 \cdot SB$.

The IR bands observed at 1610–1670 cm^{-1} of all the ligands and complexes are assigned to $\nu_{C=N}$ vibration[10]. The ligand $\nu_{C=N}$ bands around 1610 cm^{-1} in the ligands shifted to 1630–1760 cm^{-1} in the complexes due to an increase of bond order on coordination[11]. The bands at 420–390 and 255–210 cm^{-1} in the complexes may be assigned to ν_{Hg-N} and ν_{Hg-Cl}[12,13] respectively. The nitrato complexes showed a monodentate bonding mode of nitrate in the complexes[14].

The XPS spectra of $Hg^4P_{1/2,3/2}$ photoelectron peaks of HgX_2 (X = Cl, NO_3) and these prepared complexes have shown that Hg4p BE value in HgX_2 is higher than that (having same X) in $HgX_2 \cdot SB$ complexes. These XPS observations suggests that the Schiff base ligands are coordinated to metal ion[8]. Furthermore, the absence of multiple splitting in Hg3s photoelectron peak in all these prepared complexes, suggested the diamagnetic nature[8].

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