

Synthesis and characterization of mercury(II) complexes of selones: X-ray structures, CP MAS and solution NMR studies

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Abstract

Mercury(II) complexes of selones (L) having the general formulae, $[L_2HgCl_2]$, $[L_3HgCl]Cl$ and $[L_4Hg]Cl_2$ have been prepared and characterized by elemental analysis, IR and NMR (1H , ^{13}C , ^{15}N , ^{77}Se , ^{199}Hg) spectroscopy. A decrease in the IR frequency of the $>C=Se$ mode upon complexation is indicative of mercury(II) binding via a selone group. Upfield shifts in $>C=Se$ resonance of selones in ^{13}C and ^{77}Se NMR and downfield shifts in N–H resonances in 1H and ^{15}N NMR are consistent with the selenium coordination to mercury(II). The complex of dichloro-bis(*N-isopropyl*-imidazolidine-2-selone-S)mercury(II), has been characterized by X-ray crystal analysis. The principal components of the ^{77}Se and ^{199}Hg shielding tensors were determined from solid-state NMR data.
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1. Introduction

Selenium containing ligands e.g., selenolates and selones are known to form stable complexes with class b metal ions, such as gold(I) [1,2] and Hg(II) [3,4] because selenium is considered to be a soft Lewis base. Recent research has shown that mercury(II) is known to interact with selenium in the body resulting in a reduction of toxicity of both the metal ion and selenium [5,6]. Therefore, a systematic investigation of mercury complexation with selenium-containing ligands is important from a biological point of view. Although extensive research has been done on mercury(II) complexes of sulfur donating ligands [7–10], only limited reports are available about the coordination of selenium containing ligands [11,12]. We have been investigating the coordination chemistry of $>C=S$ and $>C=Se$ ligands with

d^{10} metal ions in an attempt to examine their mode of binding and to study their physical properties [7,13–17]. As an extension of our interest in the structural chemistry of metal–selenium interactions, the work on mercury(II) complexes of selones has been initiated [12]. The present report describes the synthesis of some mercury(II) complexes with a number of selones and their characterization by IR, ^{13}C , ^{15}N and ^{77}Se , ^{199}Hg NMR. The structures of the selones used in this study and their resonance assignments are described in Scheme 1.

2. Experimental

2.1. Chemicals

Selenourea and dimethylselenourea were obtained from Acros Organics. DMSO- d_6 and all solvents were obtained from Fluka–Aldrich Chemical Co., Germany. The selones were synthesized according to the procedure described in the literature [18,19]. Labeled selenourea was obtained from Isotec company, USA.

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