

Solution and solid-state NMR studies of some cadmium–selenone complexes

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Abstract

Cadmium(II) complexes of Imidazolidine-2-selenone (ImSe) and its derivatives have been prepared with the general formula $\text{Cd}(\text{RImSe})_2\text{Cl}_2$ (where R = Me, Et, Pr, etc.). These complexes are characterized by elemental analysis, IR and NMR (^1H , ^{13}C , ^{77}Se and ^{113}Cd) spectroscopy. An upfield shift in C=Se resonance of selenones in ^{13}C NMR and in ^{77}Se and high-frequency shifts in N–H resonances in ^1H are consistent with the selenium coordination to Cd(II). The ^{77}Se nucleus in $\text{Cd}(\text{ImSe})_2\text{Cl}_2$ is shielded by 38 ppm on coordination, relative to the free ligand. The principal components of the ^{77}Se , ^{113}Cd and ^{13}C shielding tensors for the complexes were determined from solid-state NMR data. Large selenium chemical shift anisotropies were observed for these complexes.

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1. Introduction

The ^{113}Cd nucleus has received increasing attention as a spin 1/2 NMR probe for the study of the structural and/or functional role of metal(s) in metalloproteins. A number of research groups have been using ^{113}Cd NMR as a “spin-spy” in the study of zinc-containing proteins [1–3]. The correlation of ^{113}Cd chemical shift parameters, deduced from solid-state NMR, with the structure of bioinorganic complexes is a fruitful approach in investigation of metal centers present in proteins [1]. ^{113}Cd NMR spectroscopy is a sensitive probe of the cadmium coordination, allowing information about the chemical bonding, conformation and dynamics of molecules to be obtained.

Unlike solution chemical shifts, solid-state NMR results can be correlated directly with structural determinations. This technique has the advantage that it provides information about the three principal components of the chemical shift

tensor, as the isotropic average of the shielding tensors is not sufficiently discriminating [4,5]. From this analysis, we get information referring to the nature and disposition of coordinating ligands [6].

Stereochemical changes around cadmium have been analyzed by using chemical shift anisotropies deduced from NMR spectra. Environments in which ^{113}Cd is ligated by oxygen usually give rise to resonances between –100 and +150 ppm, coordination to nitrogen results in a downfield shift and resonances between +200 and +380 ppm [6]. Selenium-containing ligands, e.g., selenoates and selenones, are known to form stable complexes with class b metal ions, such as Au(I), because selenium is considered to be a soft Lewis base [7,8].

To the best of our knowledge, the ^{113}Cd ligated to selenones are not reported in the literature. In this paper, we are reporting the synthesis and characterization of the adducts of CdCl_2 with Imidazolidine-2-selenone (ImSe) and its derivatives. We also present high-resolution NMR spectroscopic data in solution as well as in the solid-state, from which the principal components of the shielding tensors were obtained. The cadmium environment in these compounds is important as they provide models

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