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Short Communication

Solid-state NMR studies of 1,3-imidazolidine-2-selenone and some related compounds

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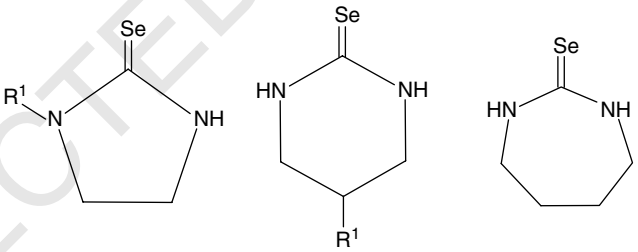
Solid-state cross-polarization magic angle spinning ^{13}C, ^{77}Se and ^{15}N NMR spectra were recorded for a 1,3-imidazolidine-2-selenone, its N-substituted derivatives and some related compounds. The spinning sideband manifold intensities were used to obtain principal values of ^{13}C and ^{77}Se chemical shift tensors. Large selenium chemical shift anisotropies were observed for these selenones. Copyright © 2003 John Wiley & Sons, Ltd.

KEYWORDS: solid-state NMR; ^{13}C NMR; ^{77}Se NMR; ^{15}N NMR; cross-polarization magic angle spinning; 1,3-imidazolidine-2-selenone; 1,3-diazinane-2-selenone; 1,3-diazepine-2-selenone; chemical shift anisotropy

INTRODUCTION

In recent years, solid-state NMR has become a very powerful method for studying the structure and dynamics of biological molecules.[1] Crystalline peptides and amino acids often serve as model compounds to search for the relationship between molecular structure, dynamic properties and spectroscopic data.[2,3] It is reported that Se-glutathione peroxidase is the only mammalian selenoprotein with known catalytic activity.[4] This protein has selenocysteine as its binding site.[5] Because of the significance of selenocysteine, this amino acid has recently been termed the twenty-first amino acid.[6] Although ^{77}Se is an additional probe for structural studies, less attention has been paid to the ^{77}Se NMR of biologically important ligands, except for recent studies of ^{77}Se NMR of solid-state selenomethionine[7] and 92% Se-enriched l,l-selenocystine in D_2O.[8]

We are interested in sulfur- and selenium-containing biologically important model compound ligands, such as imidazolidine-2-thione (Imt) and imidazolidine-2-selenone (ImSe) (**1**) and their derivatives 1,3-Diazinane-2-selenone (**2**), 1,3-diazepine-2-selenone (**3**). So far we have reported the binding of these molecules with gold(I), silver (I) metal ions, etc., in solution.[9–11] We have recently reported the ^{13}C NMR solid-state chemistry of Imt ligands and a few ImSe ligands.[12] Solid-state NMR of powder samples provides principal components of the chemical shift tensors, which potentially contain a wealth of structural information. Before embarking on a solid-state NMR study of ImSe–metal complexes, it is important to understand the shielding tensors of the uncoordinated ligands. Towards this end, we present have the results of solid-state ^{13}C, ^{77}Se and ^{15}N NMR studies of ImSe and related ligands of general structure **1, 2** and **3**.



1 **2** **3**

RESULTS AND DISCUSSION

The chemical shift tensors, anisotropies and asymmetry factors for the C═Se carbon of the compounds studied are shown in Table 1. The shielding tensor component 3 is defined as the one furthest from the isotropic value and component 2 is the one closest to the isotropic value. When component 2 is less shielded than the isotropic value, the anisotropy will be positive, as observed. There seems to be only a minor effect on the tensors of substitution on the nitrogen of the ImSe moiety. The carbon peaks of compound **1d** showed doublets (Fig. 1), indicating the possible presence of two non-equivalent molecules (see below) in the unit cell. The splitting observed cannot be attributed to the second-order quadrupolar effects due to the nitrogen nuclei as this was not observed in any of the other related molecules. Moreover, at the high field (11.74 T) used in our experiments, this effect is expected to be very small. The second order due to two equivalent nitrogens should result in a 4:4:1 triplet

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