Spectroscopy 18 (2004) 113–119
IOS Press

Solid state NMR study of 1,3-imidazolidine-2-thione, 1,3-imidazolidine-2-selenone and some of their N-substituted derivatives

Mohamed I.M. Wazeer *, Anvarhusein A. Isab and Ali El-Rayyes
Department of Chemistry, King Fahd University of Petroleum and Minerals, Dhahran 31261, Saudi Arabia

Abstract. Solid-state NMR spectra were recorded for 1,3-imidazolidine-2-thione, 1,3-imidazolidine-2-selenone and some of their N-substituted derivatives. Spinning side-bands of thione and selenone carbons were analysed to yield chemical shift anisotropies for these carbons. The NMR spectrum of imidazolidine-2-thione (Imt) showed some evidence for the presence of thiol tautomer. Molecular computations were carried out for Imt and its N-methyl derivative to yield relative energies of various tautomers.

1. Introduction

There has been considerable interest in 1,3-imidazolidine-2-thiones (Imt), 1,3-imidazolidine-2-selenones (ImSe) and their derivatives, because of their ambidentate nature [1]. That is, they may coordinate to a metal through S (or Se), nitrogen or by various chelating modes. These penta-atomic ligands exist in a thiol $\rightleftharpoons$ thione equilibrium [2]. However, it has been established that the thione form dominates in the solid state of these complexes [3,4]. We have studied extensively the interaction of metal ions with Imt and its derivatives [5]. Most of the complexations with these ligands were carried out in solution and investigated by NMR spectroscopy. The X-ray structures of some of the complexes only show a specific unit for the ligand, i.e. thione form in the case of ligand and thiolate form in the case of complexation. This was explained as due to the weakening and elongation of the thione when bonded to metal, as demonstrated by IR [5].

In this study, we have carried out a solid state NMR studies of these penta-atomic ligands. Examination of 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 Imt–metal and ImSe–metal complexes, it is important to understand the shielding tensors for the uncoordinated ligand. Towards this end we present the results of solid state ^{13}C NMR for Imt and various related ligands and some of their selenium counterparts.

* Corresponding author.