



# Synthesis of silver(I) complexes of thiones and their characterization by $^{13}\text{C}$ , $^{15}\text{N}$ and $^{107}\text{Ag}$ NMR spectroscopy

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## Abstract

Silver(I) complexes of thiones (L),  $[\text{LAgNO}_3]$  and  $[\text{AgL}_2]\text{NO}_3$  have been prepared and characterized by elemental analysis, IR and NMR ( $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{15}\text{N}$  and  $^{107}\text{Ag}$ ) spectroscopy. An upfield shift in the C=S resonance of thiones in  $^{13}\text{C}$  NMR and downfield shifts in N–H resonances in  $^1\text{H}$  and  $^{15}\text{N}$  NMR are consistent with the sulfur coordination to silver(I). In  $^{107}\text{Ag}$  NMR, the  $\text{AgNO}_3$  signal is deshielded by 300–500 ppm on its coordination to thiones. Greater upfield shifts in  $^{13}\text{C}$  NMR are observed for  $[\text{LAgNO}_3]$  compared with  $[\text{AgL}_2]\text{NO}_3$  complexes, whereas the opposite trend is observed for  $^1\text{H}$ ,  $^{15}\text{N}$  and  $^{107}\text{Ag}$  chemical shifts. © 2002 Elsevier Science Ltd. All rights reserved.

**Keywords:** Silver(I) complexes; Thiones; NMR studies

## 1. Introduction

Complexes of heterocyclic thiones such as imidazolidine-2-thione (Imt), diazinane-2-thione (Diaz) and their derivatives with transition metals are of interest in bioinorganic chemistry because of the search for simple model compounds for metal proteins [1,2]. In view of this, Cu(I) [3], Ag(I) [4,5], Au(I) [6–8], Hg(II) [9] and Cd(II) [10] complexes with thiones have been widely studied in recent years. In our previous work on silver-thione complexes, we reported the synthesis and spectroscopic characterization of various  $[\text{LAgNO}_3]$  (where L = Imt, Diaz and their derivatives) [4] and  $\text{Ag}(\text{Tu})_x\text{NO}_3$  (Tu = thiourea and  $x = 1–4$ ) complexes [11]. The present report describes the synthesis of silver(I) complexes of the stoichiometry,  $[\text{AgL}_2]\text{NO}_3$  for a series of thiones and their characterization by  $^{13}\text{C}$ ,  $^{15}\text{N}$  and  $^{107}\text{Ag}$  NMR spectroscopy. The results of  $^{15}\text{N}$  and  $^{107}\text{Ag}$  NMR for  $[\text{LAgNO}_3]$  complexes are also presented. Among  $[\text{LAgNO}_3]$  complexes,  $[\text{DmTuAgNO}_3]$  and  $[\text{DiapAgNO}_3]$  were not reported earlier. The main goal of the study is to provide a data

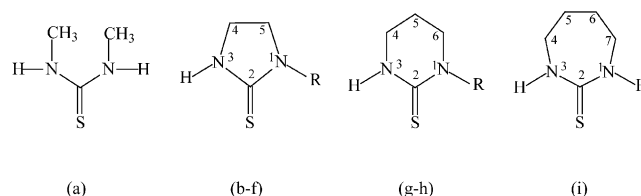
base of  $^{13}\text{C}$  and  $^{107}\text{Ag}$  NMR spectra for silver(I) complexes of thiones.

The structures of the thiones used in this study are described in Scheme 1.

## 2. Experimental

### 2.1. Chemicals

*N,N'*-dimethylthiourea, methanol, acetone, acetonitrile and  $\text{DMSO}-d_6$  were obtained from Fluka Chemical Co. The thione ligands were synthesized according to



Scheme 1. (a) *N,N'*-dimethylthiourea (DmTu); (b) R = H; imidazolidine-2-thione (Imt); (c) R = CH<sub>3</sub>; *N*-methylimidazolidine-2-thione (MeImt); (d) R = C<sub>2</sub>H<sub>5</sub>; *N*-ethylimidazolidine-2-thione (EtImt); (e) R = C<sub>3</sub>H<sub>7</sub>; *N*-propylimidazolidine-2-thione (PrImt); (f) R = *i*-C<sub>3</sub>H<sub>7</sub>; *N*-(*i*-propyl)imidazolidine-2-thione (*i*-PrImt); (g) R = H; 1,3-diazinane-2-thione (Diaz); (h) R = C<sub>2</sub>H<sub>5</sub>; *N*-ethyl-1,3-diazinane-2-thione (EtDiaz); (i) 1,3-diazipane-2-thione (Diap).

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