

Silver(I) complexes of selenourea (^{13}C and ^{15}N labeled); characterization by ^{13}C , ^{15}N and ^{107}Ag NMR

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Received 15 December 2001; accepted 12 March 2002

Abstract

Silver(I) complexes of selenourea (Seu), $\text{Ag}(\text{Seu})\text{NO}_3$ and $\text{Ag}(\text{Seu})_2\text{NO}_3$ have been prepared and characterized by elemental analysis, IR and NMR (^1H , ^{13}C , ^{15}N and ^{107}Ag) spectroscopy. An upfield shift in ^{13}C NMR and downfield shifts in ^1H and ^{15}N NMR for selenourea resonances are consistent with the selenium coordination to Ag(I). In ^{107}Ag NMR, the AgNO_3 signal is deshielded by more than 600 ppm on its coordination to selenourea. © 2002 Published by Elsevier Science B.V.

Keywords: Silver(I); Complexes; Selenourea; NMR

1. Introduction

Selenourea, $[\text{SeC}(\text{NH}_2)_2]$ (Seu) has a high nucleophilicity, caused by the strong electron donating effect of the amino groups, which is comparable to that of thiourea [1]. Some metal complexes of Seu are already reported in the literature [2–4], but there is no known report describing the complexation of AgNO_3 with selenourea or other selenones. In this work we report the synthesis of the 1:1 and the 1:2 complexes of silver(I) with selenourea (10% ^{13}C and ^{15}N labeled) and their characterization by ^1H , ^{13}C , ^{15}N and ^{107}Ag NMR spectroscopy. Characterization of silver(I) complexes of such small ambidentate ligands would provide a basis for understanding and predicting the interaction with more complex selenone ligands. Recently, we reported the similar studies for silver(I) complexes of thiourea (Tu) [5].

2. Experimental

2.1. Preparation of the complexes

The complexes were prepared by mixing the solutions of Seu and AgNO_3 in acetonitrile in the molar ratios of

1:1 or 2:1 and stirring for 15 min. The resulting white precipitates were filtered and washed with acetone. After preparation the complexes were stored in refrigerator. Yield = 85%. Melting points; $\text{Ag}(\text{Seu})\text{NO}_3$ = decomposed at 108 °C, $\text{Ag}(\text{Seu})_2\text{NO}_3$ = 157–158 °C.

Anal. Found (Calc): C, 4.46 (4.10); H, 1.43 (1.38); N, 14.82 (14.35) for $\text{Ag}(\text{Seu})\text{NO}_3$ and C, 5.99 (5.77); H, 1.99 (1.94); N, 16.97 (16.84) for $\text{Ag}(\text{Seu})_2\text{NO}_3$.

2.2. Instrumentation

The solid-state IR spectra were recorded on a Perkin–Elmer FTIR 180 spectrophotometer using KBR pellets. All NMR measurements were carried out on a Jeol JNM-LA 500 NMR spectrophotometer at 297 K using 0.25 M solution of the complexes in DMSO-d_6 . Since both complexes give black deposits in solution after some time therefore, spectra were measured within 30–40 min. The ^{13}C NMR spectra were obtained at the frequency of 125.65 MHz with ^1H broadband decoupling and relative to TMS. The ^{15}N NMR spectrum were recorded at 50.55 MHz using $\text{NH}_4^{15}\text{NO}_3$ as an external reference, which lies at 375.11 ppm relative to pure CH_3NO_2 . The ^{107}Ag NMR was obtained at 20.13 MHz using 10 mm low frequency probe with 9.1 M aqueous AgNO_3 as an external reference. The spectral conditions were: 1.02 s acquisition time, 6.0 s delay time, 45° pulse angle and approximately 500 scans. The ^{77}Se

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