

A silver(I) complex with nalidixic acid hydrazide derivative

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Silver(I) complexes have shown to possess antibacterial activities.[1,2] The coordination of biologically active ligands to this metal may thus provide new antibacterial profiles in a seek for a synergist effect. Nalidixic acid (nx) is considered the precursor of the quinolones antibacterial series therefore making such compound and its derivatives potential active ligands to be coordinated to ions such as silver(I).[3] Moreover, the hydrazide derivatization of nx provide the possibility of new coordination sites to metal ions.

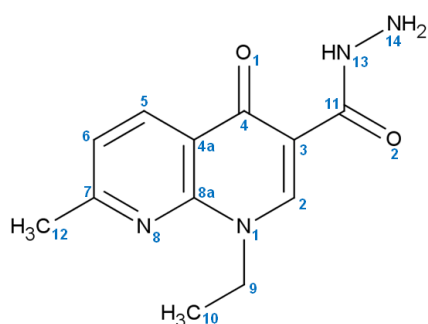


Figure 1. Hydrazide derivative of nx

This work presents the synthesis and characterization of a silver(I) complex with the hydrazide (hzd) derivative (Fig. 1; C₁₂H₁₄N₄O₂) of nx. The hydrazide, including the single crystal structure determination, was fully characterized and reported previously by us.[4] The complex was characterized by a set of chemical and spectroscopy analysis. Elemental analysis for the Ag(I) complex suggests a 1:1:1 Ag(I)/hzd/NO₃⁻ proportion. TG-DTA confirms the suggestion made by CHN analyses. ESI-QTOF-MS spectrum of the Ag(I) complex shows peaks at 353.0140 *m/z* and 599.1010 *m/z* corresponding to [Ag(C₁₂H₁₄N₄O₂)]⁺ and [Ag(C₁₂H₁₄N₄O₂)₂]⁺ ions, respectively. The IR spectrum of the Ag(I) complex presents the characteristic stretching bands of the -NH₂ moiety of the hydrazide, which reinforces the proposition that the ligand remains in its protonated form. The absence of a strong and sharp band in 1040-1100 cm⁻¹, which corresponds to uncoordinated nitrate, the broadening of the band at 1385 cm⁻¹ and the presence of a band at 1635 cm⁻¹ suggests that the nitrate ion is coordinated to the metal ion. No significant shifts were observed for the carbonyl bands. In the ¹H NMR spectrum of the Ag(I) complex (DMSO-d₆) it is possible to observe shifts of the hydrogen atoms bonded to N14 and the hydrogen bonded to N13, when compared to spectrum of hzd (MeOH-d₄). Due to the poor solubility of the Ag(I) complex the ¹³C NMR and ¹⁵N NMR spectra were performed in solid state (SS). The ¹³C SSNMR data suggest coordination by the carbonyl group (C11-O2). The ¹⁵N SSNMR presents a signal at 377.6ppm which corresponds to a coordinated nitrate ion. DFT studies are in progress.

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