Enaminone analysis by ^{1}H NMR spectroscopy

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Introduction

Over the last 30 years, no new antibiotic molecule has been discovered. This fact is worrying because bacteria are more and more resistant, especially in hospitals. In order to solve this problem, the Multimaterial and Interfaces Laboratory of the Claude Bernard University is doing research on the synthesis of metallo-antibiotics complexes.
As part of this programme, I synthesized an enaminone from sulfamethazine, an antibiotic; (called L5), to increase its affinity with metal ions. Once the synthesis was finished, I determined the purity of the synthetic product.

Experimental method

First, I ran the UV-visible spectrum of L5 and the antibiotic synthesized enamione with a Perkin Elmer Lambda XLS Spectrometer. Figure 1 presents these spectra, where : Concentration: 0.25 $mol.L^{-1}$, Absorbance < 0.8 (detection limit), Wavelength range : 200-800 nm

On the spectra, we can see different peak absorption for these 2 molecules but we cannot be sure if the synthetic product is not a mixture of L5 and the enaminone because my synthetic product absorbs close to 268 nm, the characteristic peak of L5. That is why I realized a ^{1}H NMR spectrum of my synthetic product, where : Solvent : DMSO, Scans: 8

Figure 2 presents this spectrum and Figure 3 HEL5H and L5 structures.

Results

Assignment peaks:

Protons	δ	Coupling constant	Integral
^{1}Ha	5.93 ppm	J=12.6 Hz(d)	I=1.03
^{1}Hb	8.443 ppm	J=12 Hz (dd)	I=0.85
^{1}Hc	7.61 ppm	J=8.7 Hz(dd)	I=0.63
^{1}Hd	7.96 ppm	J=8.7 Hz (d)	I=2.04
^{1}He	7.405 ppm	J=9Hz (d)	I=1.76
^{1}Hf	11.16 ppm	J=12.9Hz (d)	I=0.90
^{1}Hg	2.25 ppm	(s)	I=6.22
^{1}Hh	6.76 ppm	(s)	I=1.14

Peaks at 3.33 ppm and 2.5 correspond to water and DMSO present in these NMR tubes. Nevertheless, we can see another peak at 11.55 ppm. This chemical shift, which is significant, certainly correspond to a proton in an amine functional group. The antibiotic used to synthesize the enaminone has an anime functional group, from which one can conclude that this is not a complete synthesis.

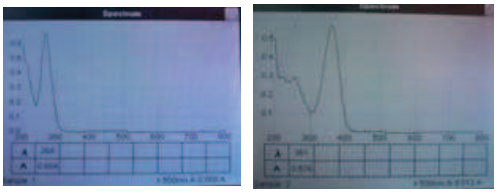

Figure 1 : *UV-visible spectra of L5 and the synthesize énaminone, between 200 and 800 nm*

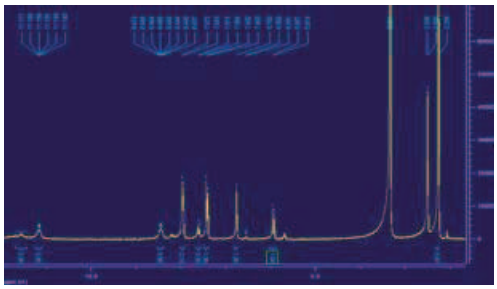

Figure 2 : *NRM 1D Spectrum of enaminone synthesize*

Conclusion

To conclude, the integral of the peak corresponding to NH_2 is 0.46. So 1 proton in the antibiotic has an integral of 0.23 and 1 proton in HEL5H has an integral of 1.
1,0+0.23=1.23 wheils corresponds to 100 % of the molecules.

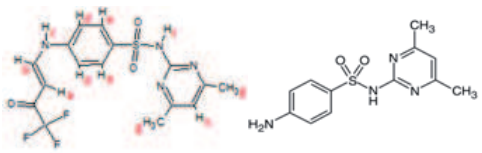

Figure 3 : *HEL5H and L5 structure*

1/1.23=81% 0.23/1.23=19%

The combination is a compound of 81.3% of HEL5H and 18.7% of L5.