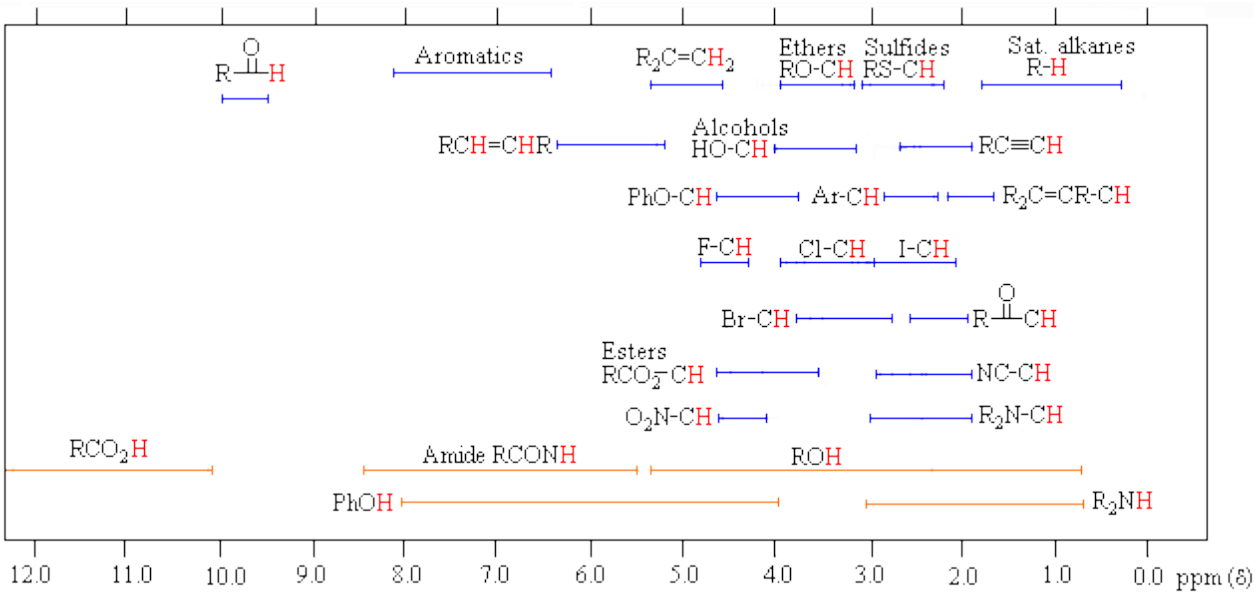
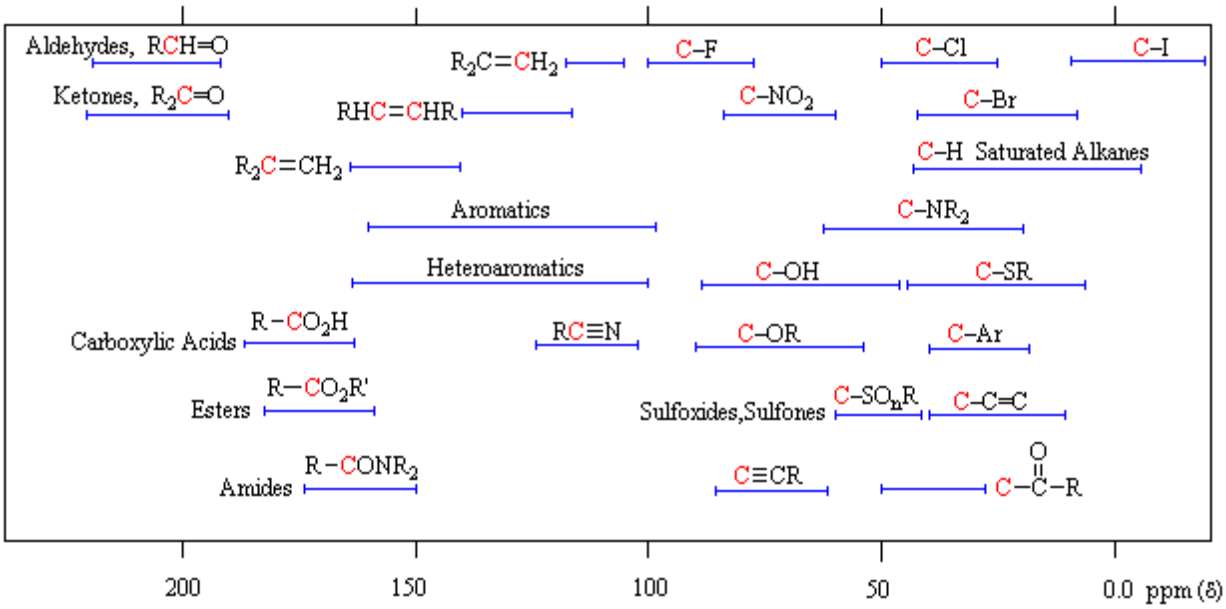
I. NMR is used to characterize many compounds and materials. The most common atom studied with NMR spectroscopy is the proton NMR. Carbon NMR is also used but is less sensitive.

Here are the typical tables that are used to guide the chemist in the rationalization of the chemical shift observed in proton and carbon NMR.

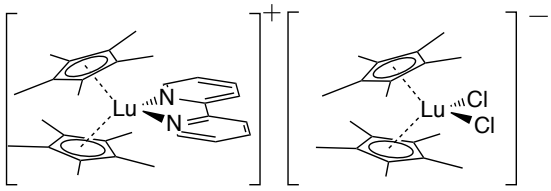




1. The scale is given in ppm. What does this mean ?
2. The spectral window is about 12 ppm for the protons and more than 200 ppm for the carbons. Do you have an explanation for this ?
3. How is choosen the zero value ?
4. Can you explain the difference in chemical shift for the alcanes protons and the aromatic protons ?
5. Same question for the chemical shift of the halogenoalcanes.
6. How would you distinguish a benzene carbon chemical shift and a pyrdine carbon chemical shift ?

II. We will consider the following lanthanide complex:

$[Cp^*_2Lu(bipy)][Cp^*_2LuCl_2]$

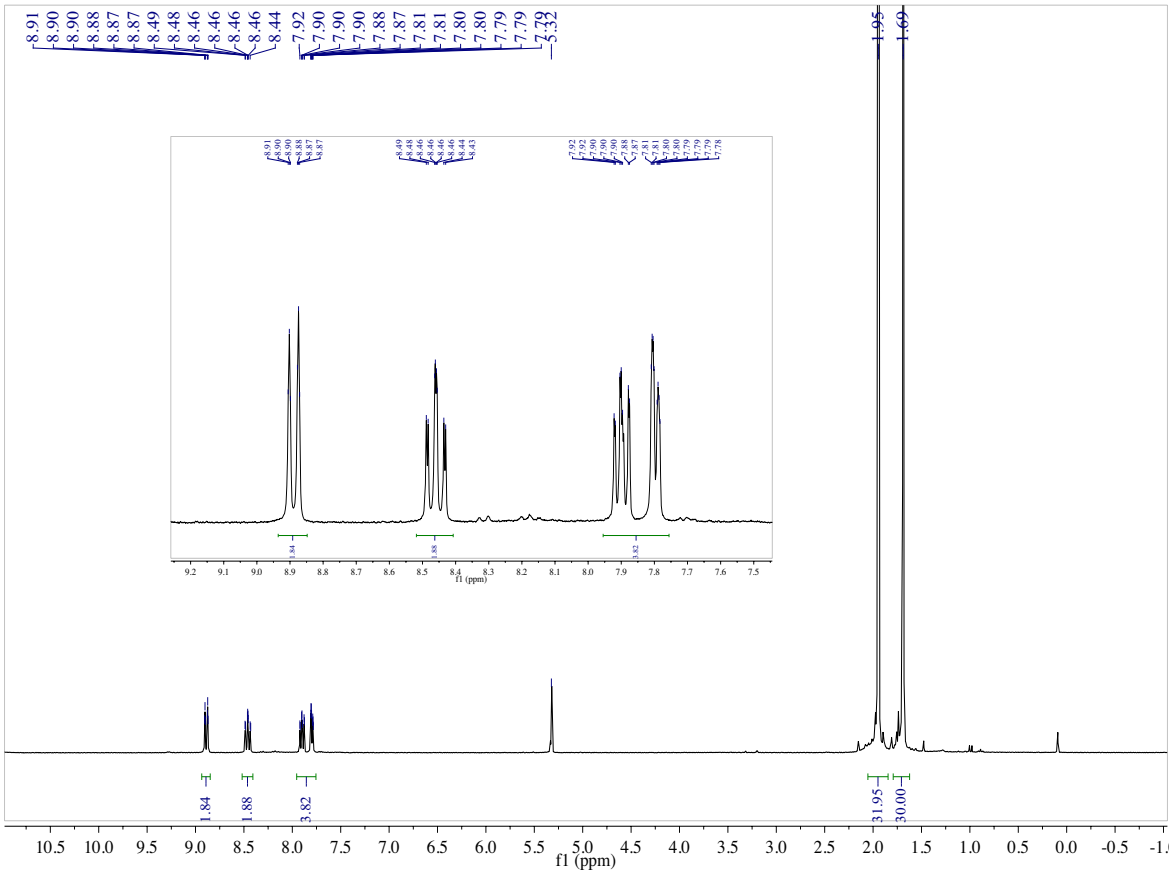


1. What is the Cp* ligand?
2. What bipy stands for?

Lutecium is a lanthanide element named after Lutèce (Lutetia), former name of Paris. It atom is number is 71 and it is the last lanthanide element.

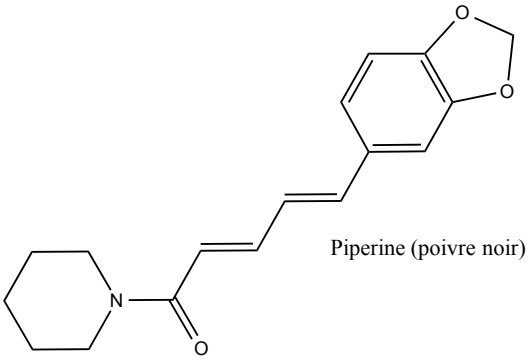
We will try to construct and analyze its NMR proton spectrum.

3. How many proton resonances would you have expected for the complex? Explain the origin of each.
4. How many peaks would you expect for each resonance. Draw a scheme.
5. Compare your scheme and the measured spectrum below.
6. In the Spectrum, a small resonance is present around zero ppm. Do you have an idea of what it can be?
7. The resonance at 5.32 ppm is attributed to the solvent but we made the experiment in a deuteurated solvent. Why do we observe a signal when recording a proton spectrum?
8. The integrations for each resonance are given in the spectrum beow. Some of the resonances are not quite close to integer number. Can you propose an explanation for this?

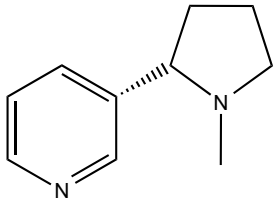


III. 4 different molecules are represented. For each of them :

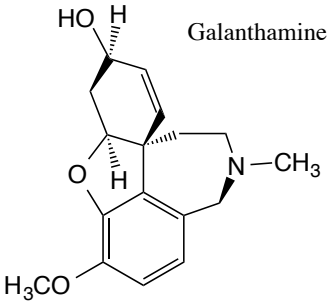
1. Give the number of expected resonnances.
2. Discuss the multiplicity and the integration for each resonance.
3. Give an estimation of the chemical shift.
4. Compare with the given spectra, attribute each spectrum to each molecule and discuss the specificities.



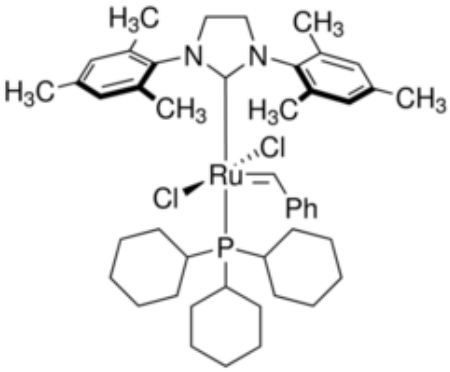
Piperine (poivre noir)



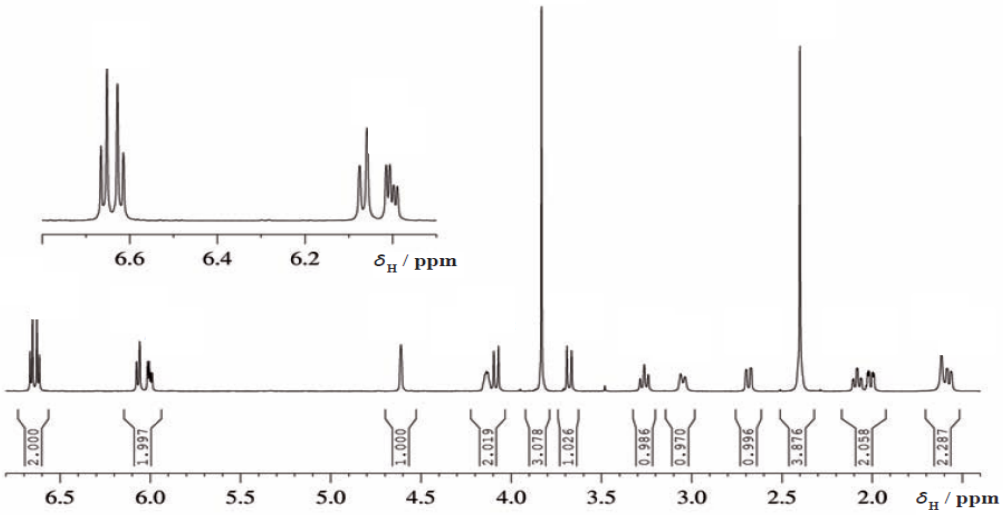
Nicotine (Tabac)



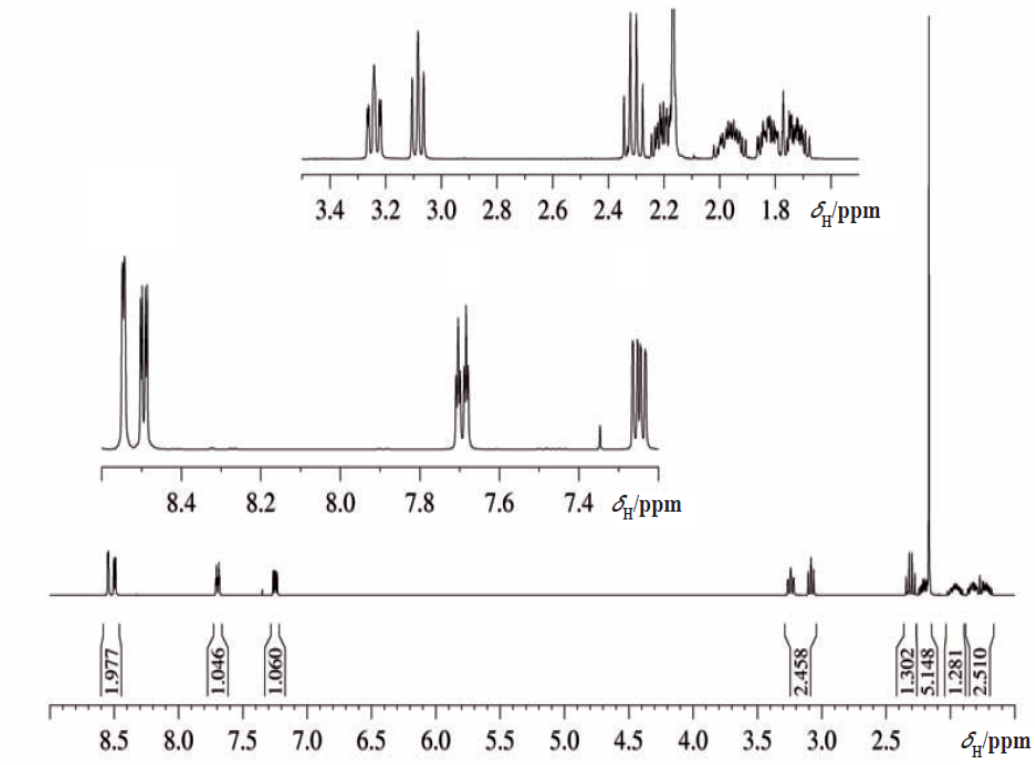
Galanthamine



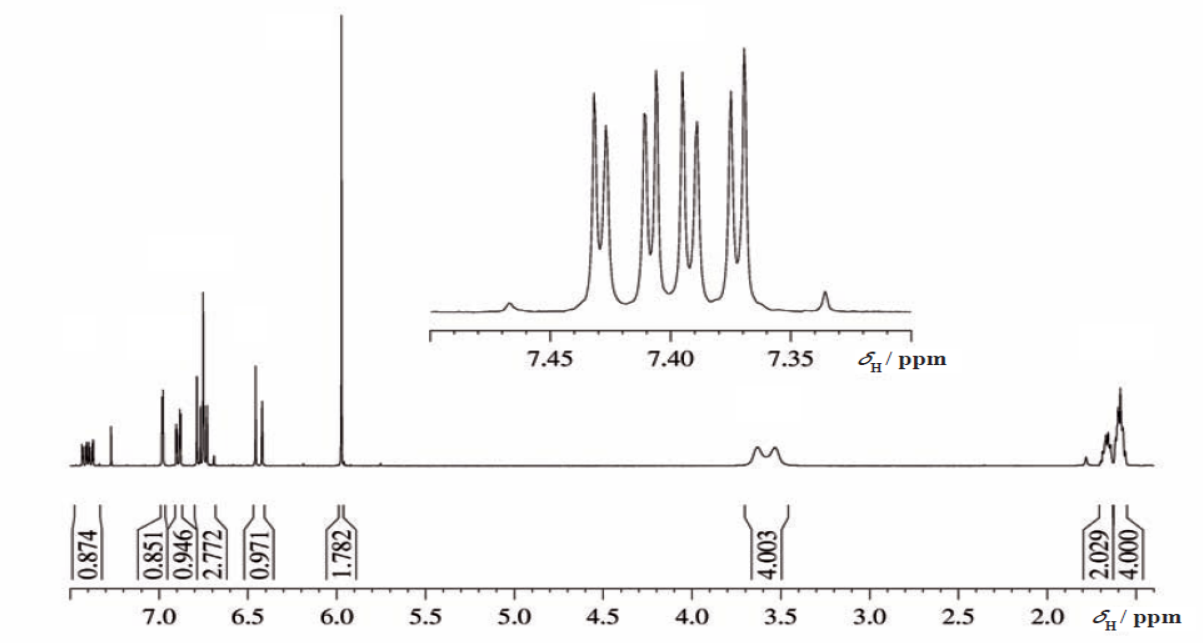
Spectrum A



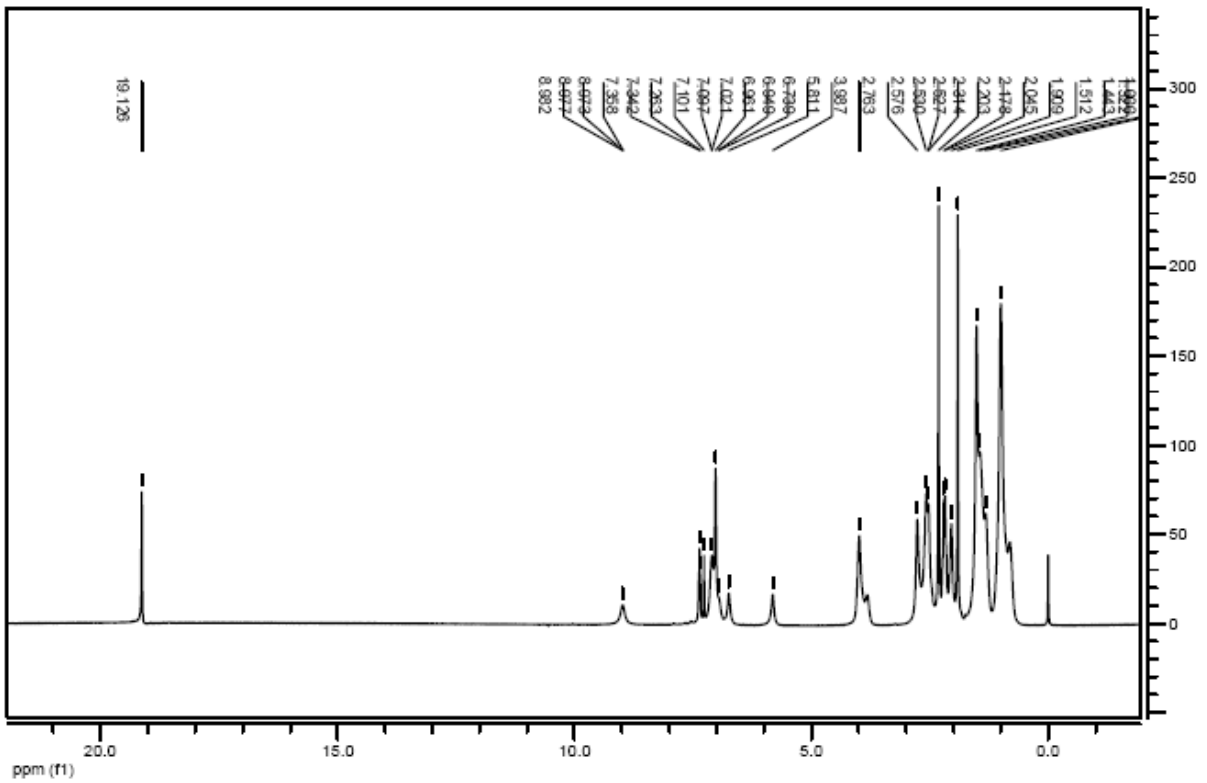
Spectrum B



Spectrum C

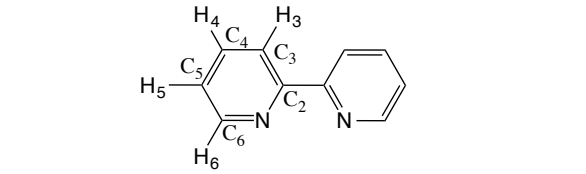


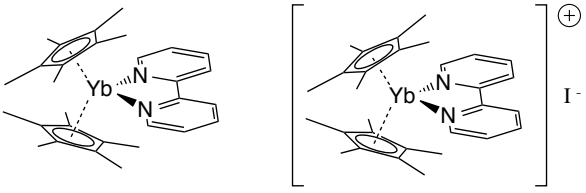
Spectrum D



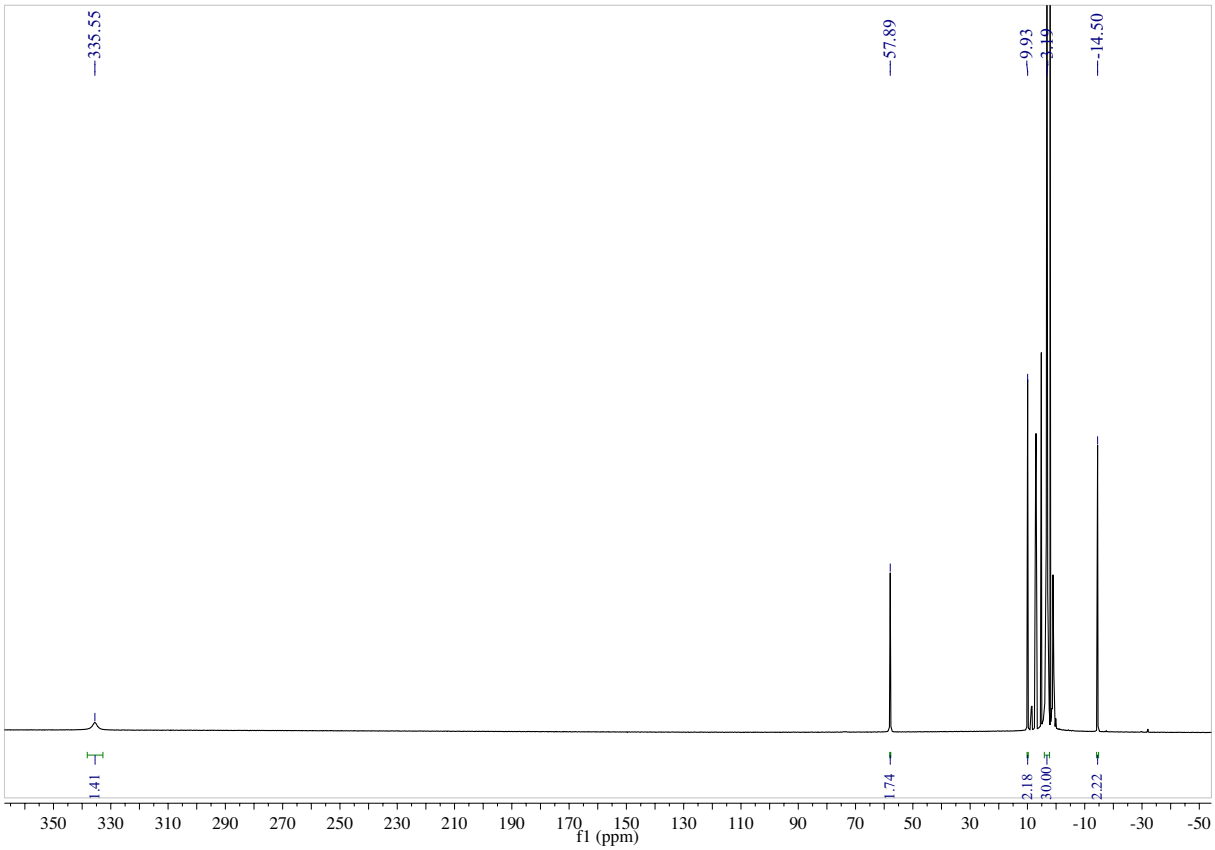
IV. The compound **1** has the following ^{1}H NMR spectrum (see below).

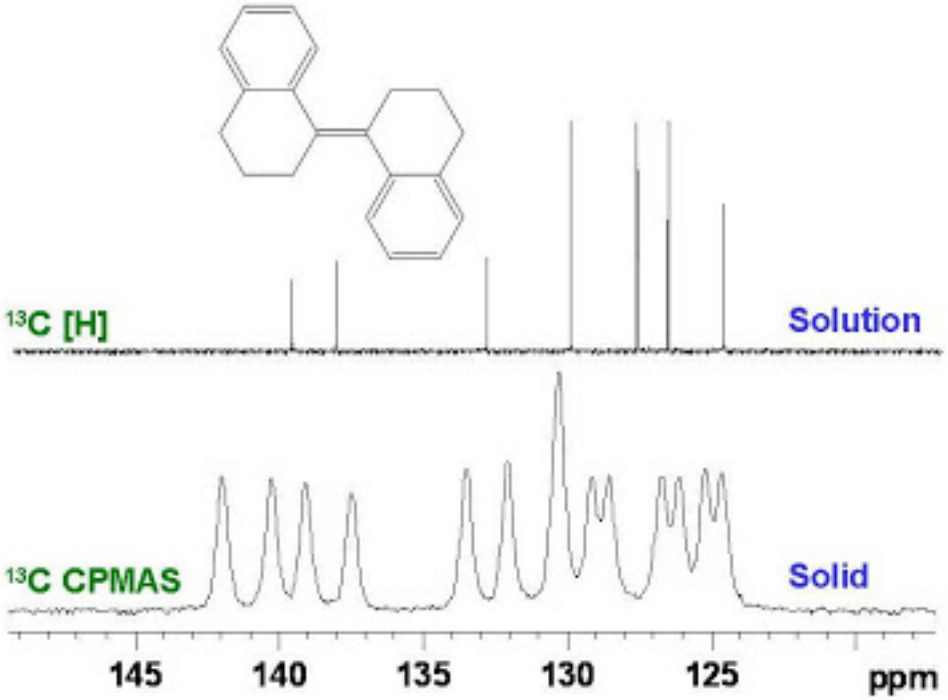
1. How many resonances would you have expected ?
2. How many do you see ?
3. What seams wrong with this spectrum ?
4. Are the tables of exercice I of any use ?
5. Could you find any explanation ?





1 **2**



V. The figure below represents the ^{13}C spectrum recorded in solution and in the solid-state for the same molecule.

1. How many resonnances would you have expected with both methods ?
2. Comment on the differences observed and propose an explanation.