Lab exercise 3: NMR

For this lab rapport, we were only required to give an adequate response to question 4.

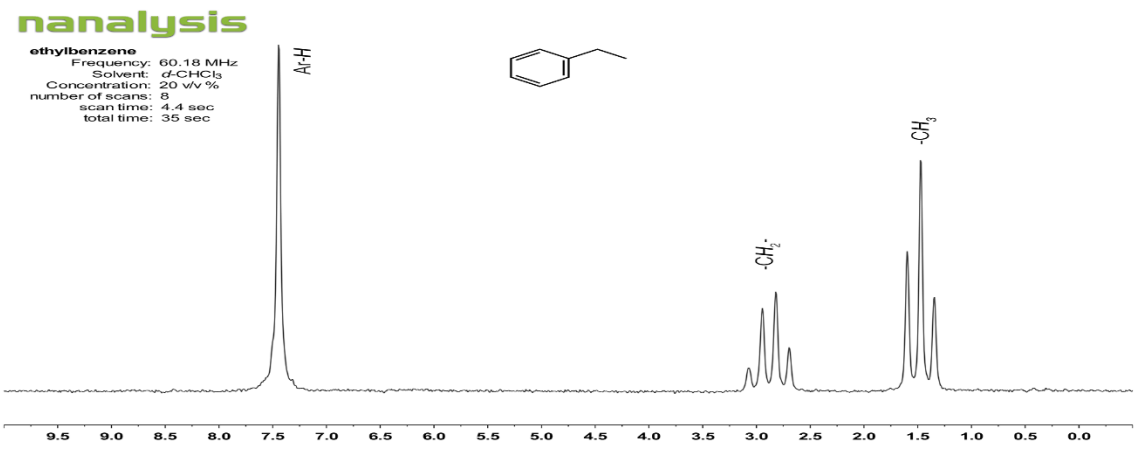
a) Superconductive magnets (used in this demonstration) are developed for high field strengths and large sample volume. Shimming coils are used to improve the homogeneity in the magnetic fields of the superconductive magnets. These are small electric coils places around the probe. By adjusting the current in these shimming coils, corrective magnetic fields can be set up and the homogeneity in the magnet can be improved. So what does the homogeneity in magnetic fields mean? It basically means that you have a magnetic field over the sample with no variation. If the strength of the magnetic fields varies by some percentage over a NMR sample, the resonance frequency will vary by the same percentage, which will smear the NMR lines and possibly distort their shape.

   The way the effect of shimming can be measured is by looking at the half value width of the resonance lines in the NMR spectrum. The resonance lines width increases with in homogeneities in the magnetic field.

b) Liquid helium is use as a coolant for the superconducting magnets. In order for superconductors to be efficient, they must be kept below their critical temperature. Nitrogen is used for the cooling of helium.

   If you stop refilling helium and nitrogen, the superconducting magnet warms up and no longer has zero resistance. With an increase in resistance, it follows that an increase in thermal energy is inevitable. There's quite a lot of energy stored in the current of the superconductor that has to go somewhere. This leads to a process called "quenching", which could be potentially quite dangerous.

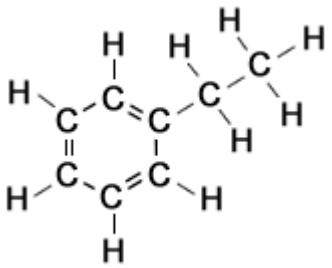
c)



The graph above is the NMR-spectrum for the ethylbenzene that was demonstrated in the lab-exercise. We had two rules to consider when we evaluate the spectrum evaluated. These were:

- The splitting of a protons absorption is due to interaction with neighbouring protons and the multiplicity of the splitting is determined by the number of neighbouring protons
- Doublets have the intensity ratio 1:1, triplets have 1:2:1.

So first we have to look at the structural formula of ethylbenzene:



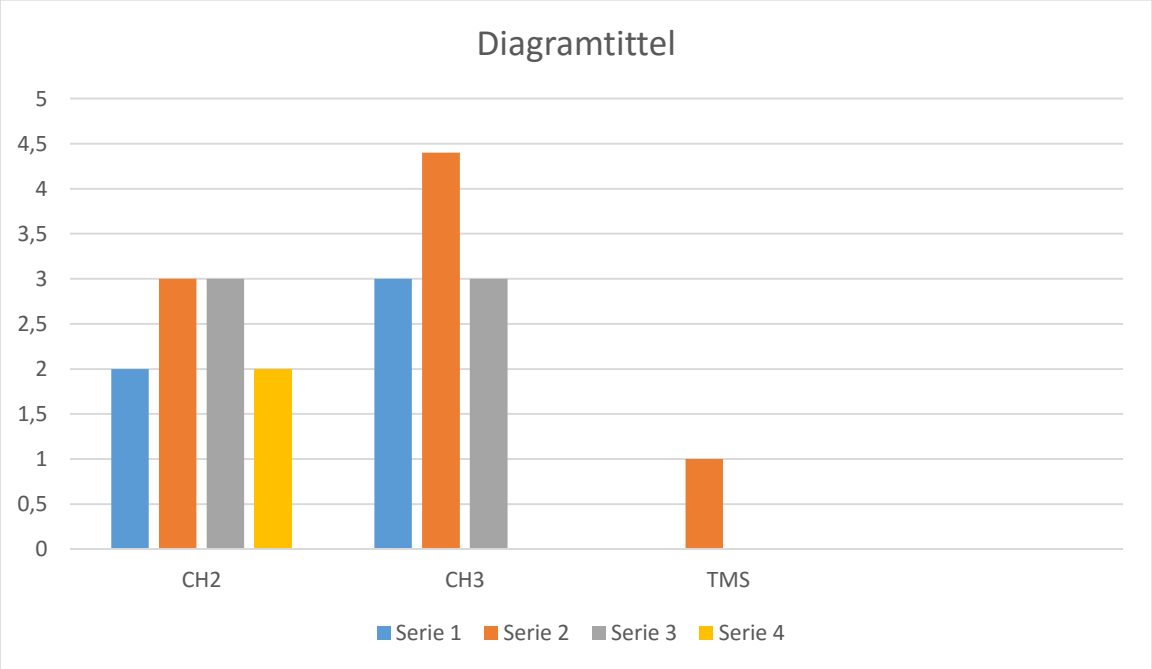
We can see that from the first peak that we only have one long peak with no multiplicity. This means that it has no next door protons (hydrogens) to consider and this gives that this spectre is for the aromatic part of the organic molecule. We look at the rest of the molecule, the next part of the molecule is CH2 which has 3 hydrogen-neighbours. That means that it should have 4 peaks in the NMR-spectrum. The last part that could have a peak is the methyl part of the molecule (meaning: CH3). CH3 is neighbour to CH2, meaning that it has 2+1 peaks since it has two protons. This observation gives that the 4 peaks in NMR spectrum belongs to CH2 and the 3 peaks belongs to CH3, as given in the figure above.

If we use a stronger magnetic field, it will lead to an amplification in the difference in resonance frequency.

d) I have given the peaks their corresponding numbers from 0 ppm and upwards.
Peak 1: TMS, Peak 2: CH3, Peak 3: DMSO, Peak 4: CH2, Peak 5: OH. The methylene group have greater chemical shift because the protection of protons is weaker for CH2 than CH3.
For the diluted ethanol, the ethanol molecules are not hydrogen-bonded to each other and there is little exchange among the OH-protons between molecules allowing for the observation of J coupling. This gives it high proton protection and thereby lower chemical shift. With regards to the temperature and the critical shift, we already know

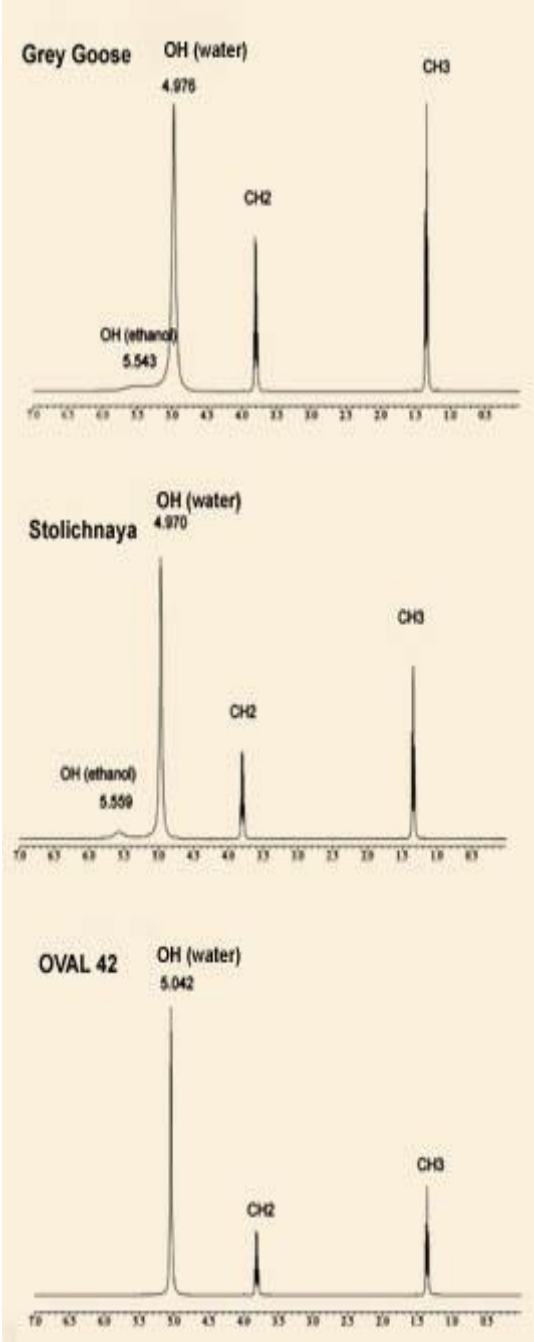
from previous questions that there is a correlation between temperature and magnetic field. We also know that there is a correlation between magnetic field and critical shift. To put it in a different way: In chemical shift, the difference in frequency compared to the reference is proportional to the amplitude of the B0 magnetic field, that again depends on the temperature.

e) I chose to draw it in word (hopefully this procedure will be accepted, since the ppm levels have already been given in the lab-text (CH2= 3.6ppm and CH3 = 1.5 ppm) and TMS is the reference-point at ppm =0).



With regards to the changing of hydroxyl group with other atoms like Fluor and Iodine, there not be much change since they do not contain any protons.

f) It would not have the straight up peak that goes almost to infinity (meaning peak 3) on the graph with DMSO. That is specifically for DMSO as solvent. If you mix water and ethanol, you get for example Vodka. Vodka is a mixture of ethanol and water. If you vary the amount of water, you get different brands of Vodka and you get different NMR-spectrums as seen in the figure below:



As we can see, increasing the water amount gives higher peaks for OH in the NMR-spectrum.