Advanced NMR Techniques in Solution and Solid-State
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Module-08
Multiplicity Pattern and Analysis of NMR Spectra -II
Lecture – 08

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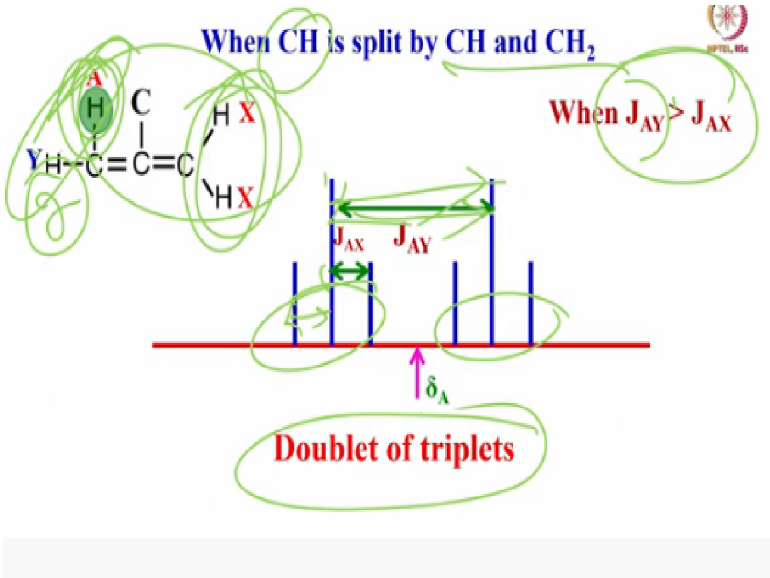

Welcome all of you, for this course on advanced NMR technique in solution and solid state NMR. In the last couple of classes, we were discussing various internal interaction parameters of NMR, the chemical shifts and coupling constants. In the scalar couplings, we discuss a lot of things, we discussed how the coupling pattern arises, what is the mechanism of coupling and what happens to the multiplicity pattern when several spins are coupled among themselves when they are chemical inequivalent, what happens if 1 spin is coupled to chemical equivalent other spins, etc.

How does the multiplicity come and we understood what is a triplet, quartet, how does triplet comes? How does quartet come, and how in the case of triplets, even though it appears as 3 lines of intensity ratio 1 is to 2 is to 1, we know it contains 4 peaks, because the centre 2 lines will overlap. It is because, if I consider the 4 possible spin orientations of 2 spins, alpha alpha and beta beta are the 2 outer peaks and the central peak is because of alpha beta and beta alpha, both are equally probable and they are overlapped.

That is one way of understanding and actually I showed you by diagrammatically how the central peak is getting overlaped and then we start getting triplet. Similarly, we also saw quartet and we also understand what is the pentet, what is the septet what is the sextet everything. All those things we understood; quite a bit we discussed. And we understood the multiplicity pattern which were called as doublet, triplet of triplet, triplet of doublets and various things we were trying to do.

When we were trying to understand several multiplicity patterns, we came to a point and tried to find out when one spin is coupled to different spins, which are chemical equivalent, or chemical inequivalent. How does, this multiplicity comes? We took an example like this, where we were seeing what happens when CH proton is coupled to CH and CH2. And I am concentrating on proton A, it is the CH proton, when it is coupled to CH and CH2, I use the condition that JAY is larger than JAX. So, what is going to happen? Since I am concentrating on proton A, proton A when it is coupled to single proton CH proton, it will become a doublet. It is going to give a doublet because there are only 2 possible spin states for this proton. And then each component of this doublet, it will further split into triplet because of 2 equivalent protons of CH2.

So, the pattern what we are going to get is like this, a large doublet and each of the doublet is split into triplet like this. And this large separation,n this large doublet gives your coupling between proton A and proton Y, and the separation between these 2 adjacent lines of the triplet gives you the coupling between proton A and proton X. So, this is the pattern we are going to expect for this type of proton, when it is coupled to CH and CH2 like this.

And what do you call this pattern? This pattern is called doublet of triplets. Why not triplet of doublets? That is a different question. You always start with the one which is the largest coupling. Larger coupling is AY which is this one. So, this is the larger coupling; which gives rise to a doublet and then each line becomes a triplet. So, this pattern is called doublet of triplets.

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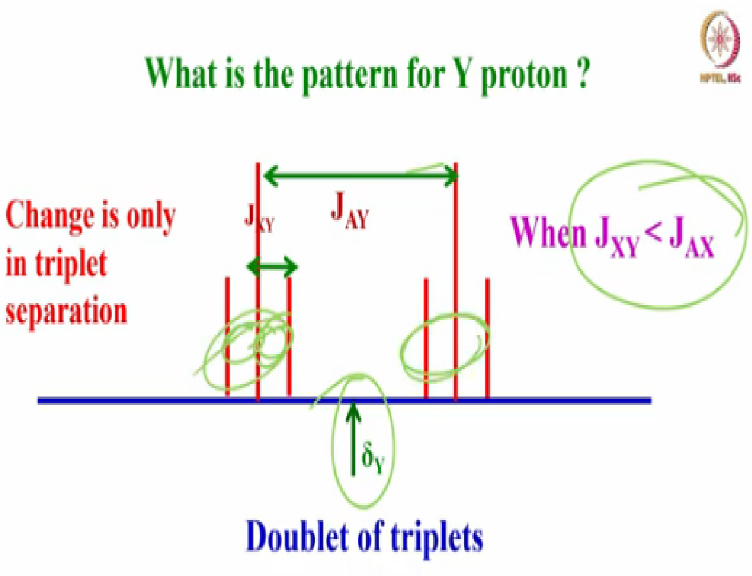

So now, the question is, what is the pattern for Y instead of A, if I observe Y, what is going to happen? See, now again, I consider the situation JXY is smaller than JAX, this is the chemical shift of Y. Now again, this is a large doublet because Y is coupled to proton A, that coupling remains same because it is not changed. But now, we also see the coupling of Y with proton X. And again, it is going to be doublet of triplet.

What is the difference? The change is only the separation here; that is all because this is a large coupling. That is Y coupled to X is quite small, they are far away separated. And this is the large coupling, this is very small coupling, similar to the pattern what you saw for proton A when you observed, it is also a doublet of triplet. The only thing is the triplets are pretty smaller here compared to the previous one.

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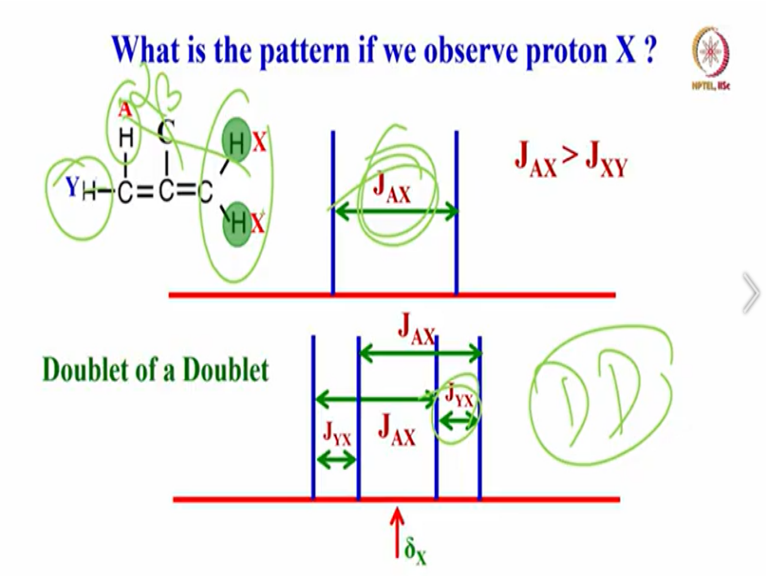

Now I am going to see what will happen, if you observe the proton X. Proton X is the CH2 group. So, far I was observing protons which are coupled to proton and then equivalent groups, CH2 group. Now, I directly observe CH2 protons, what is going to happen? What is the pattern if you observe proton X? Now, proton X is going to split by proton A into doublet; because this proton has only 2 possibilities; alpha and beta.

So, assuming this is larger than this one, what is going to happen is first it is going to become a large doublet, because of the coupling between A and X. The separation gives me JAX. Now, each line of this doublet is going to be split into another doublet because of this proton and this coupling is smaller. So, this is going to be large coupling which is this one; this separation is going to give you XY coupling. So, this is why this pattern is called doublet of a doublet. In this case, larger coupling and smaller coupling both are doublets and it is called doublet of doublets.

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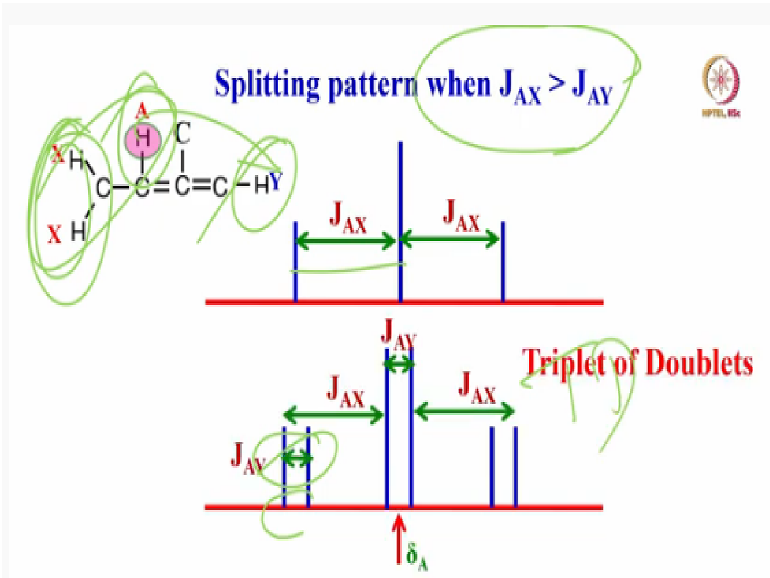

Now, consider another example of a molecule like this. I am concentrating on proton A which is highlighted in red colour. Now, I assume the condition JAX coupling between proton A and CH2 protons is larger than AY coupling. Now, how does the pattern come? First, A is going to be a triplet because of this CH2. And this separation gives you JAX coupling; and each line of this triplet is going to be split into a doublet because of this proton.

So, this is the pattern we are going to get, this doublet separation gives you JAY and this last separation gives you JAX. And this pattern, as I said you always start with a larger coupling, larger coupling is a triplet and a smaller coupling is a doublet; this is called triplet of doublets. This pattern is called triplet of doublets.

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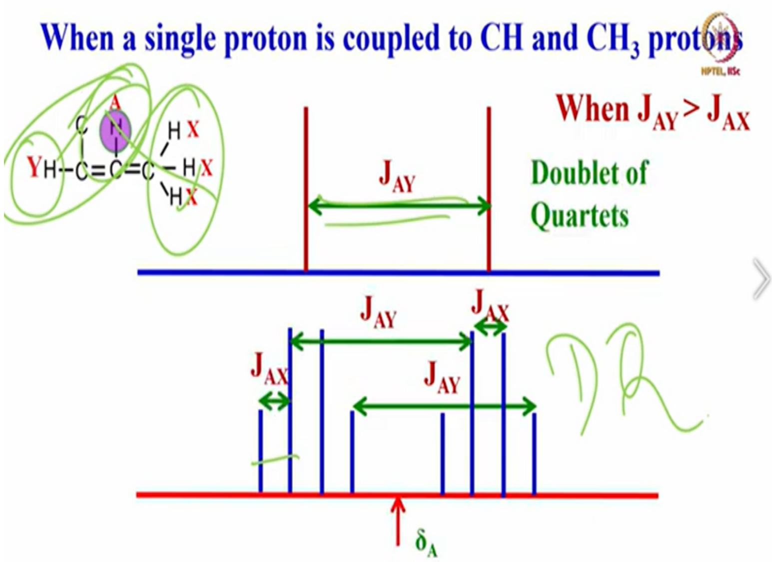

So, you can start discussing a lot of things, what happens when a single proton is coupled to CH and CH3 now, same logic you can extend. Now we are looking at the proton A. Again, JAY is assumed to be larger than JAX, when JAY is larger, it has to be a doublet and this is a large coupling. A will split into a doublet because of this proton. Now, each line of this doublet is split into quartet because of the CH3 group; and this coupling is much smaller; this is the AX coupling, this is AY coupling.

And as I said, this being a larger coupling with a doublet, this is called doublet of quartets, we call it as doublet of quartets.

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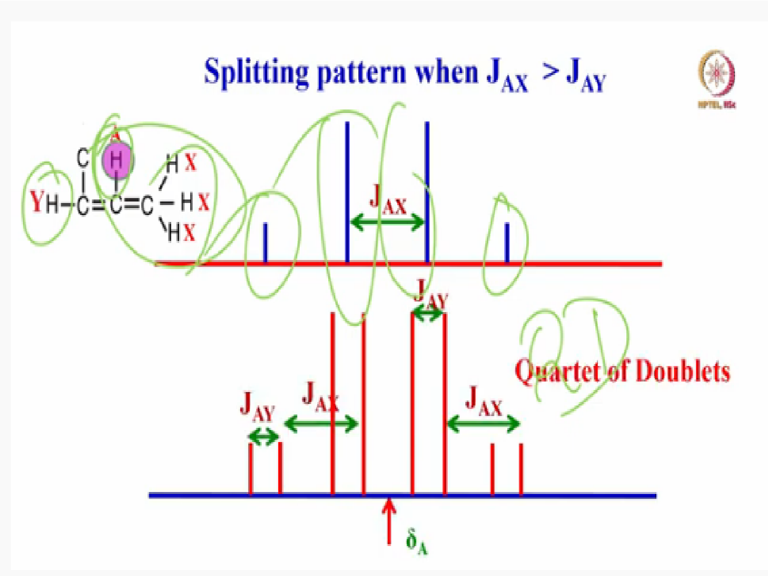

Now, you can also think of a situation what happens if AX is larger than AY. The quartet coupling between the proton A and CH3 group is larger, then this will be quartet because of the CH3 coupling, A will be quartet. Now, what will happen when there is a smaller coupling because of this proton. As a consequence, each line of this quartet is going to become a doublet and this doublet coupling is smaller in strength, because, this coupling, I assumed to be smaller.

So, what do you call this pattern? It is called quartets of doublet. Larger coupling is due to quartet and smaller coupling is due to the doublet. This pattern is called quartet of doublets.

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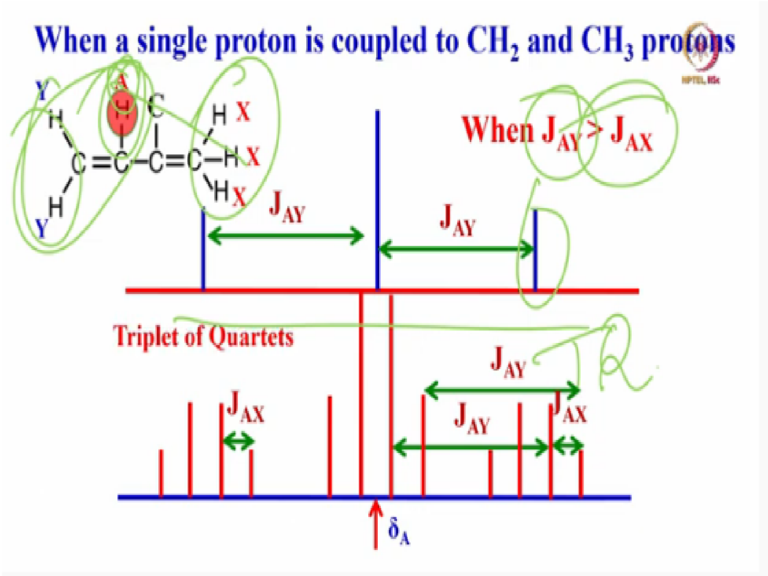

So, like this we can start talking lot of things and we could think of, for example, what happens if the CH2 group is coupled to CH3. Again you can consider a varieties of possible combinations, what happens when JAY is larger than JAX or JAX is larger than JAY, the pattern changes. Now I assume that JAY is larger than JAX. JAY is, if we consider the proton A which is highlighted in red, because of CH2, this proton A will become a triplet; that we have been discussing all along.

So, this is a triplet we are going to see. Now I invoke the coupling with a proton A and proton X, that is CH3 group. What will happen now? Each line of this triplet is going to become a quartet. And this quartet coupling is smaller. I have written, you know a stick plot like this. What do you call this pattern? Triplet is larger than the Quartet. So, we call it as triplet of quartets.

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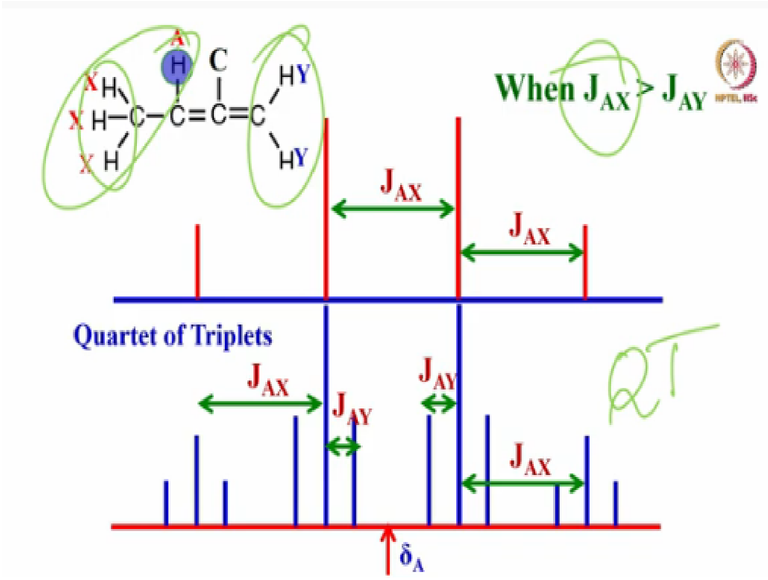

This is the pattern you have to call it as triplet of quartets. Of course, we can start talking what happens if AX is larger than AY, then quartet is going to be larger, if this coupling is larger, because that is a CH3 proton, 3 chemically equivalent protons. You split this proton A into a quartet and each layer of the quartet is going to be split into a triplet, because of CH2 group. Now as a consequence, you are going to get quartet of triplets. Remember this nomenclature difference.

Earlier it was triplet of quartets, now, it is quartet of triplets, because the larger coupling is quartet. This is the way you have to start naming the multiplicity pattern in NMR.

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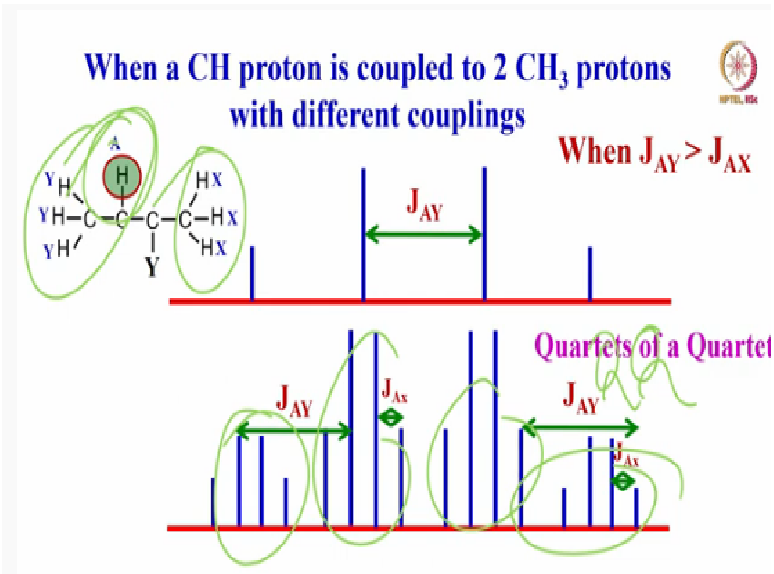

When you want to analyse the spectrum, this is what you should understand. Now, of course, you could think of varieties of possibilities. And these are I am giving you common examples, because you come across such type of patterns in the routine analysis of your spectrum. So now, you look at this one, the proton A.

Now it is coupled to 2 CH3 groups of different coupling strengths, what pattern we should expect, one of them is going to be a quartet because of this, but larger coupling. Now, each line of this quartet has to be further quartet because of another CH3 group. So, you are going to get a pattern like this, each line is going to be a quartet. What do you call this pattern? It is called Quartets of a Quartet. Larger coupling is also quartet. Smaller coupling is also quartet. So, this pattern, you should call it as Quartets of Quartet.

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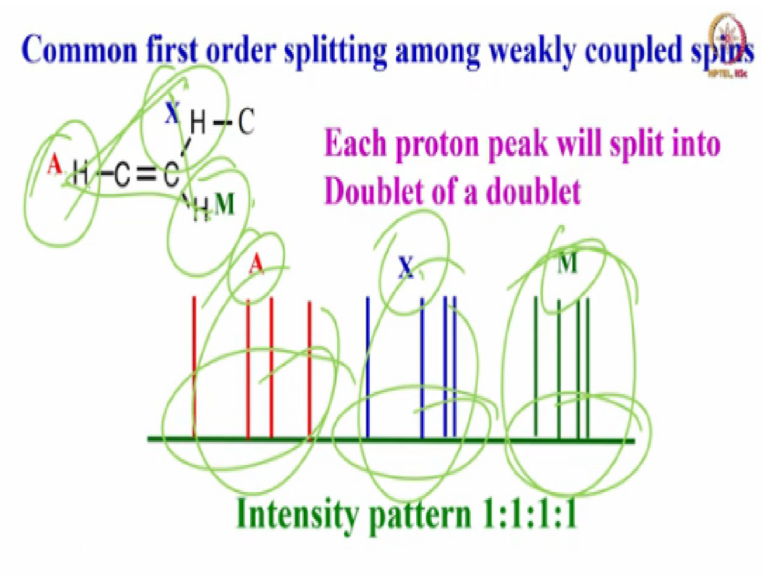

So, this is how you start naming. Now if you look at many of the moleculed like this, you will come across chemically equivalent spins, chemically non-equivalent spins will be there, varieties of combinations you can think of. Consider a situation we have proton A, proton M, proton X. All are chemically inequivalent. Then what is the type of pattern you are going to get?

Depending upon there are 3 types of coupling you can think of, coupling between A and X, A and M, M and X. Depending upon which coupling is larger and which coupling is smaller, the patterns can change, at the site of the nucleus you are observing. Now, if I see the proton

A, proton A experiences coupling with proton X and proton M, then it is going to be doublet of doublet. Similarly, the proton X is going to couple with A and couple with M.

So, it is going to be doublet of doublet. Similarly, M also doublet of doublet. So, this is a pattern called doublet of doublets only. Each proton will split into doublets of a doublet. Now see, you cannot talk for the entire spectrum, I am telling for each group is going to be doublet of doublet, and intensity pattern here is 1 is to1 is to1 is to 1. Unlike in the previous examples, they are chemically equivalent spins, intensity pattern obeyed Pascal's triangle, which is nothing but the; coefficients of binomial expansion, which I explained yesterday. So, as a result, here, in the non-equivalent case, the intensity pattern is always equal 1 is to1 is to1 is to 1. Now one proton is coupled to more than 2 different protons or groups of protons.

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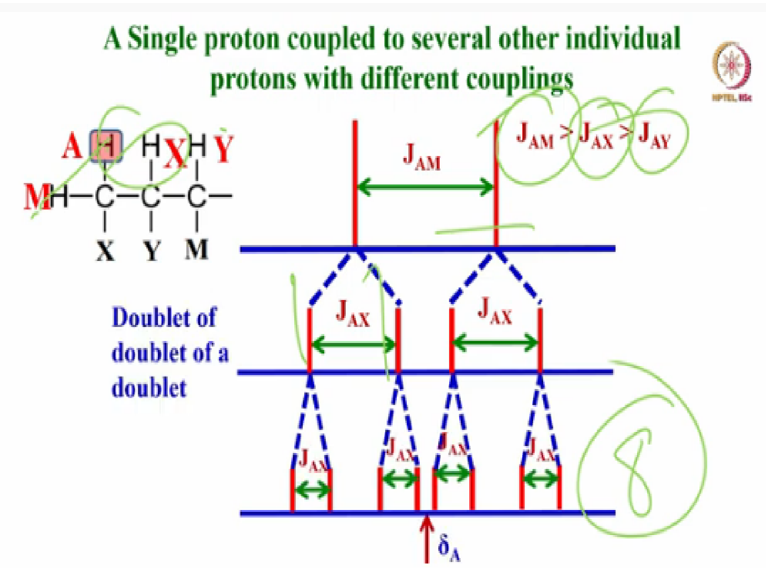

You can come across varieties of problems like this. Now I have one proton coupled to 3 different protons. I do not need to explain for you, now with the condition I have given you, JAM is larger than JAX larger than JAY. Now, one proton experience coupling with 3 independent chemically inequivalent protons. Remember, all the 3 protons which are coupled to proton A here are typically inequivalent. So, what is the type of pattern we should expect? Larger coupling because of M it is going to be a doublet. Another coupling because of X is going to be doublet of doublet and then another coupling because of Y it is going to be doublet of doublets of doublet of doublets. So, it is going to be 8 lines pattern. This is what we understood, when you analyse the splitting patterns, how you get in the case of chemical inequivalent spins, we have what is called to the power of N, that you must be remembering.

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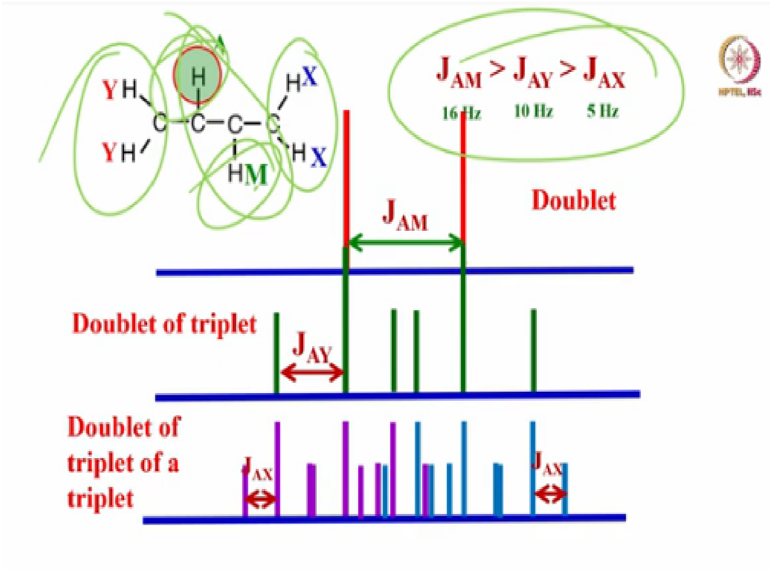

So, this is the pattern we are going to get for different cases. So, you can also think of what happens if the situation is like this, when this is coupled to CH2, CH and CH2 like this. Now it is not one or two protons, it can couple with three different protons. Remember, as I told you, one spin can experience coupled with number of different spins simultaneously; as a consequence, with the condition that I have adopted here. For a hypothetical molecule I have chosen, I said JAM is larger; it means M is a single proton, it is a doublet because this one proton it coupled to this proton, splits into a doublet; and then what will happen next? Very easy to understand, there are two equivalent CH2 groups. So, each component of a doublet is going to be triplets like this. Now, another CH2 coupled to this will be triplet of triplets; each component is the triplet is further split into triplets. So, what is the pattern you are going to get now? This is not just doublet of doublet. It is doublet of triplets of a triplet, it is doublet of triplets of triplet.

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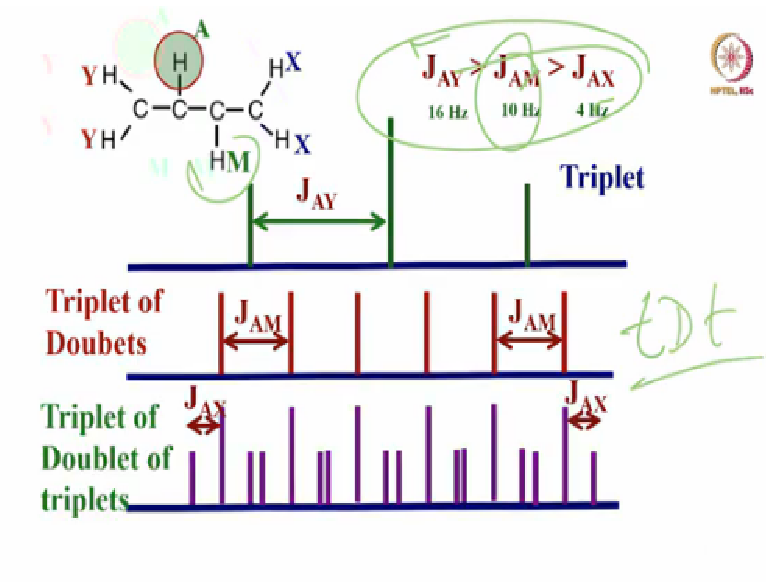

This is a pattern you are going to get. So, now we can see if the situation gets reversed. If this coupling is larger than this one and then this one, now the triplet is going to be there first because of JAY coupling and then M will split each of them into a doublet because this second one is larger I have chosen. So, this is going to be a triplet of doublets and then each line of it this doublet is further going to split into the triplet. So, this is a pattern we are going to get. Then what do we call this pattern, larger is triplet, then doublet, then triplet. So, this pattern is called triplet of doublet of triplets.

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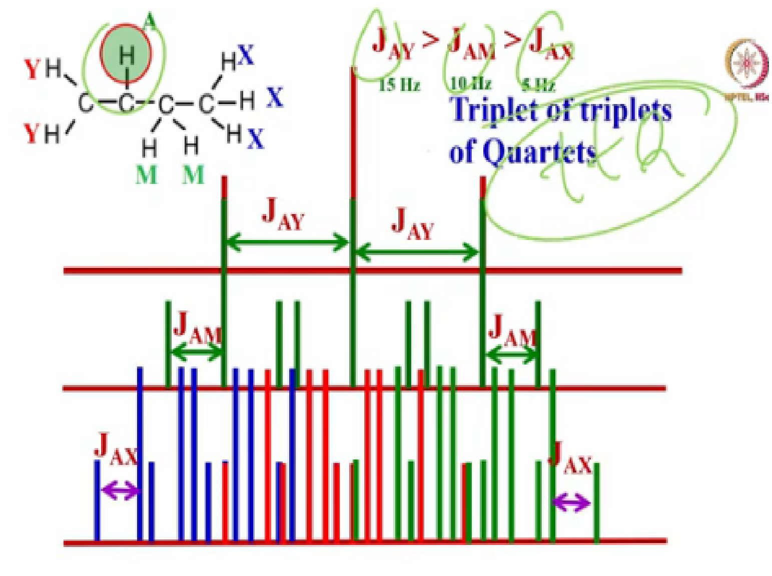

Very easy to understand; so, you can start doing this. I do not want to give any more examples. I think with the number of examples that I have already given you, you must have got the hang of the type of pattern that we are expecting. How to interpret the pattern based on the chemically equivalent and inequivalent groups that are coupled to a proton that you are detecting.

So, now in this case A is being detected, again, I am using the condition this is larger this than this; then this; now it is going to be 2 triplets and quartets. So, it is firstly going to be a triplet, and again another triplet and each line is a triplet is going to be a quartet like this. So, what you are going to get is triplet of triplet of a quartet, this is the pattern.

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Nomenclature of Splitting patterns in NMR

Splitting pattern is at the site of Active proton coupled to other protons

Abbreviation	Splitting Pattern	Largest Coupling	Second Largest Coupling	Smallest Coupling
dd	Doublet of doublet	doublet	doublet	
dt	Doublet of triplet	doublet	triplet	
td	Triplet of doublet	triplet	doublet	
tq	Triplet of quartet	triplet	quartet	
qt	Quartet of triplet	quartet	triplet	
dq	Doublet of quartet	doublet	quartet	
qd	Quartet of doublet	quartet	doublet	
ddq	Doublet of doublet of quartet	doublet	doublet	Quartet
dtq	Doublet of triplet of quartet	doublet	triplet	Quartet
tdt	Triplet of doublet of triplet	triplet	doublet	Triplet
ttd	Triplet of triplet of doublet	triplet	Triplet	Doublet
tdq	Triplet of doublet of quartet	triplet	doublet	quartet

So, finally, you are remember one thing; this is the table which you must remember, the nomenclature of splitting patterns in NMR is like this. If it is dd, if I say 2 doublets, it is called doublet of doublet, larger coupling is doublet, smaller coupling is also a doublet. If I consider dt I call it a dt, doublet of triplets. What do you mean by that? Larger coupling is doublet and smaller coupling is triplet.

Instead of dt I call it td, that means triplet of doublet, larger coupling is triplet, smaller coupling is doublet. Same thing if there are a number of other protons are coupled; ddq I call it Doublet of doublet of quartets. First this larger coupling is doublet, second larger coupling is doublet and then is a quartet. This is the way you have to understand the splitting pattern of 1 or 2 protons which you are observing, when it is coupled to n number of other protons, which are either chemically equivalent or chemically non-equivalent. This is the way you have to understand.

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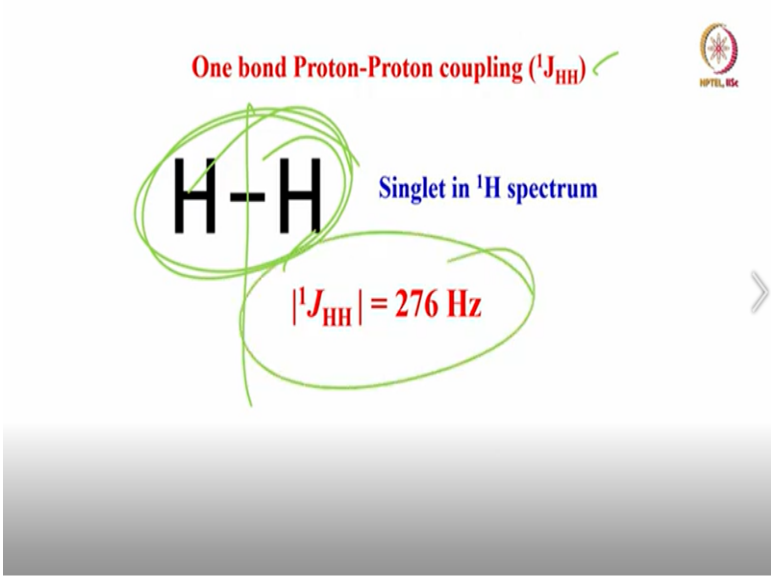

So, with this multiplicity pattern we understood, I just want to tell you some idea about the strengths of the coupling. So far, we may have we said splitting is there; coupling we can measure. But what is the strength of coupling? approximately the 1 bond coupling is always larger than 2 bond coupling, 2 bond coupling, which is always larger than 3 bond coupling. It is a general trend, need not always be true; many times that can be deviations, but general trend is one1 bond coupling is larger.

This is hydrogen molecule the two protons are coupled to each other, but it is chemically equivalent. In principle, you should not see the coupling because as I said, there is an exception for 2nI + 1 rule. But nevertheless there is a way we can find out coupling by using deuterium substitution, etc. which I do not want to discuss now. In the previous course, this was discussed in length. Remember, we have a coupling here; one bond coupling is very large in the case of protons.

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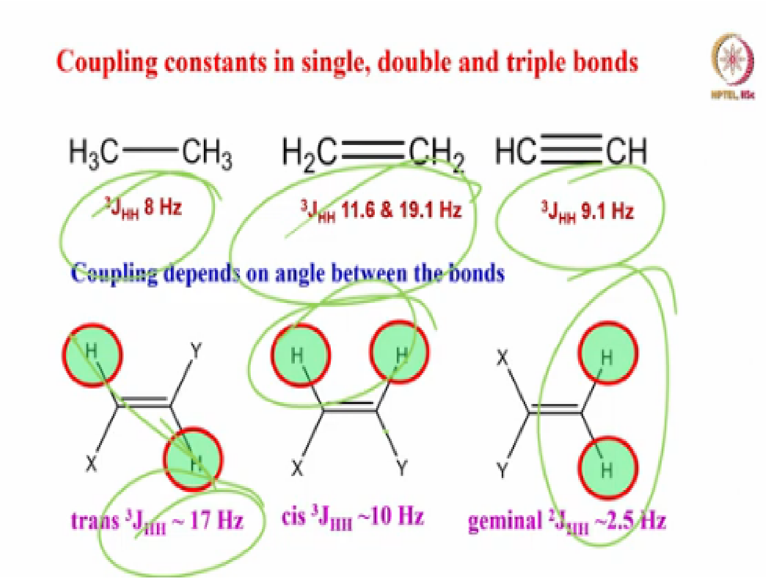

Then most of the time you come across 2 bonds and 3 bonds like this. We see in molecules like this, three bond couping will be 8 hertz and 2 bond will be of the order of 10 to 12 Hertz and 3 bond is of this order; in case of a triple bond, especially in the ethylenic substituted molecules like this. For example, with cis and trans substitution, the trans coupling is very large compared to cis coupling and this is quite large compared to geminal coupling.

Please remember this point trans coupling is larger than cis coupling, this is larger than geminal coupling. This pattern you should remember because whenever you have to analyse the proton spectrum, invariably you will come across such patterns where you have to use this knowledge to analyse the spectrum.

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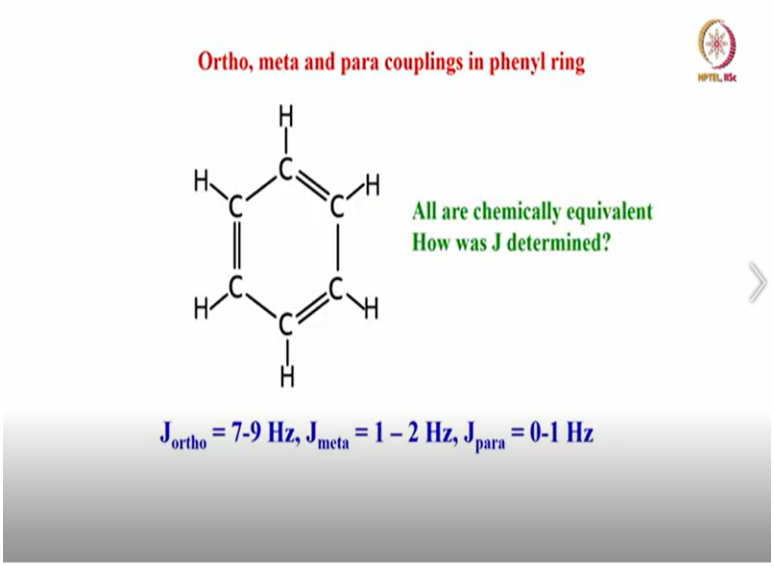

Similarly, if you look at the benzene molecule, as I told you, all the 6 protons here are chemically equivalent and each proton experiences ortho coupling, meta coupling and para coupling. Similarly, this proton experience ortho, meta and para. So, each proton experiences 3 different types of couplings. All are protons are chemically equivalent, but then this is called magnetically equivalent with 3 different couplings.

Though you will not see couplings in the spectrum you will see only a single peak, then how do you get the couplings? There is a way to do it, which again I do not want to discuss now, but we know that in such aromatic systems, the ortho coupling is quite large, it is of the order of 8 Hertz. meta coupling is 1 to 2 and para is between 0 to 1 hertz. So, this is the knowledge which I wanted to tell you.

And with this I have more or less given a lot of information, I have given you about the coupling patterns; spin spin coupling, We have discussed 2 important NMR interaction parameters. But the internal interaction parameters, I discussed at length, a lot about chemical shift, I discuss lot about coupling patterns how we get? how do we interpret? everything, we use this and analyse the spectrum later.

When you go to analysis of the spectrum, this pattern ofcourse, I have taken the example for the proton, but it did not always be proton, for any of the nucleus this method of interpretation remains the same. Whether you get triplet here in proton, you can also get in carbon, when this carbon is coupled to 2 equivalent protons like that. So, triplet, multiplicity, quartet, singlet, doublet everything, the concept remains same and whichever the nucleus you are going to observe.

So, what we will do is; we will now start analyzing spectra of some different nuclei. That will give you basically an idea as how to go ahead and analyse the spectrum. See, in fact, I wanted to tell you, now on, before getting the spectrum, we apply radio frequency pulses at different directions, and get the signal collect and do the Fourier transformation. I want to talk to you about how the evolution takes place, evolution of magnetization, I wanted to talk to you about the pulse phase, receiver phase etc.

Since, for the benefit of the people who have not taken the previous course, I will give you the analysis of the spectrum now. And then afterwards, we will talk something more about Fourier transformation, evolution of the couplings and chemically shift basically, all those things, advanced topics will take up soon. So, now I am what I am going to do is, I will stop the discussion about couplings, we will straight away go to the analysis of the spectrum, we will start analysis the 1D spectrum. I will not introduce 2D now.

I will come to 2D later, it will take couple of more classes to introduce 2D and other things. But we will analyse the 1D spectrum of some of the nuclei, right from proton NMR, which is commonly employed; carbon 13 NMR, and then various, some exotic nuclei with some examples. We will start and then try to understand how to interpret the spectrum. This is very much essential, when you want to apply your knowledge of NMR analysis for molecules that you have synthesised. What do we get if we analyse different spectra? That is the first question.

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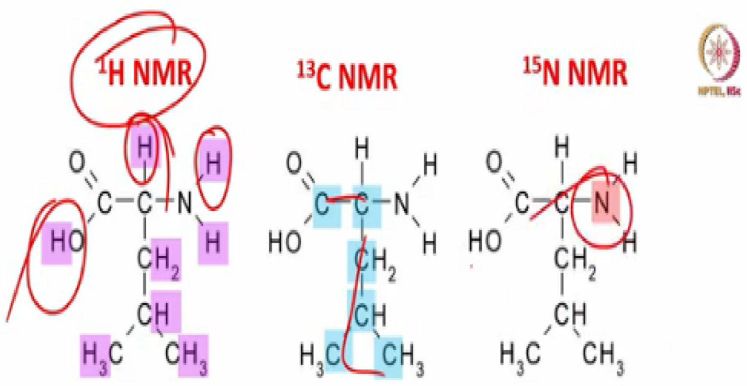

If I want to take the proton NMR. If I take the proton spectra, what am I going to get? See, for example, in a molecule like this, I will get information about the protons, I can make assignment of the peak and get all information with the different types of protons that are present here, different functional groups, as far the protons are concerned. If I go for carbon 13 NMR, then I will know how the carbons are situated here. Which carbon is bonded to which carbon like that, if we go to nitrogen 15 NMR, for example, there is only one nitrogen, I will get information about nitrogen. So, different nuclei if you study, if you take the NMR spectrum of different nuclei, then what you are going to see? different types of spectra are going to give more information about the particular nuclei which is present in your molecule. So, I will take the analysis of first order spectra, some representative examples, first I will tell you how we can synthesize the spectrum based on the family tree diagram which I explained to you earlier.

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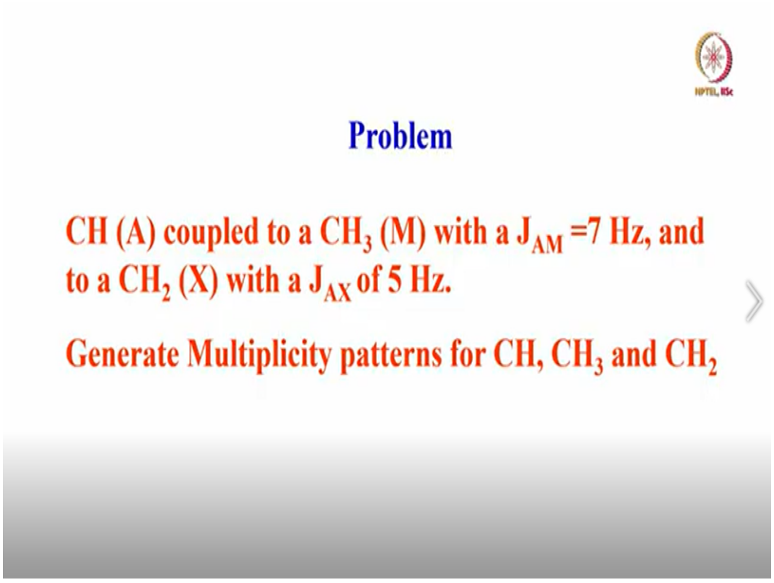

I will take a problem like this. Let us say there is a proton A, CH proton single proton is attached to carbon which is coupled to CH3 group with a coupling constant of 7 Hertz. And CH is also coupled to CH2 with a coupling constant of 5 Hertz. Now generate the multiplicity pattern for CH proton, CH3 and CH2; all the 3; we can separately do it. Now this information sufficient. There are 3 different couplings, you must generate the multiplicity pattern, we can simulate the spectrum theoretically.

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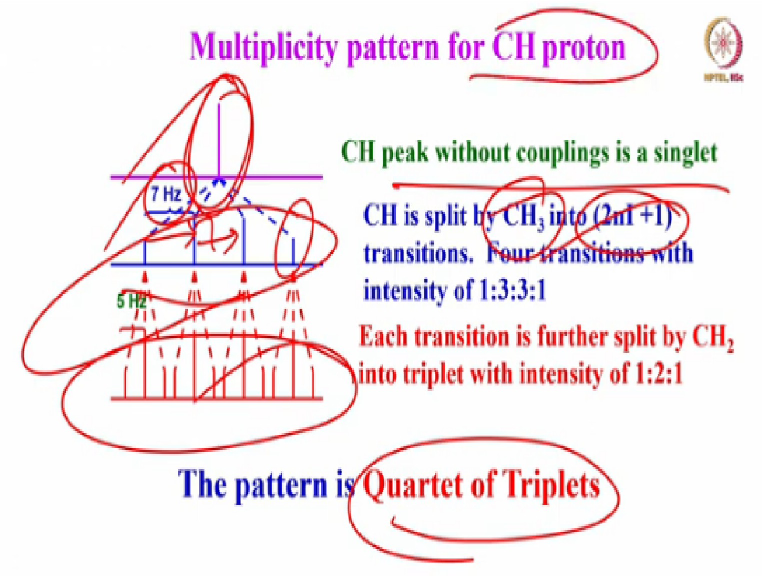

What we will do is we will start with the multiplicity pattern for a CH proton, it is simple. Assume there is no coupling to proton. What is this pattern you are going to get? CH proton without any couplings should be a singlet. Let us start simulating assuming there is no coupling. Now I am going to bring in the coupling of CH to CH3; this is a larger coupling of 7 Hertz. First largest coupling, you can think of. It is coupled to CH3 group. What will happen when CH3 is coupled to CH? I explained to you CH is going to become a quartet. Clearly it became quartet. Now we can generate it; we can see the separation; we can say it is 7 hertz, because that is a coupling information given to you. Use this 2nI+1 rule and generate the multiplicity you get a quartet. This is also coupled to CH2. That coupling is 5 Hertz I said. So, each line is the quartet become a triplet. So, what do you call this pattern? Quartet of triplets.

So, you can generate this type of spectrum of these molecules, this is what we understood. So, this is the typical example we saw for this molecule, you can simulate for the CH proton.

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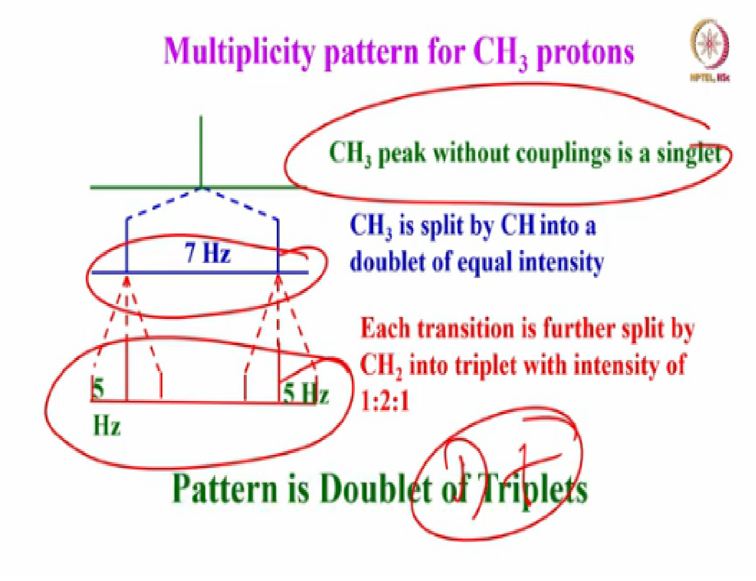

Now, what happens if I take the CH3 proton, I am observing CH3, again CH3 without coupling is a singlet. Now, with this coupling to CH it is going to be a doublet. Remember, when I was observing CH it was a quartet. Now, it is going to be a doublet. And this doublet is further split by CH2, each line is going to be a triplet. So, it means you are going to get doublet of triplet for CH3, remember, for CH you got a different pattern for CH you got doublet of triplets.

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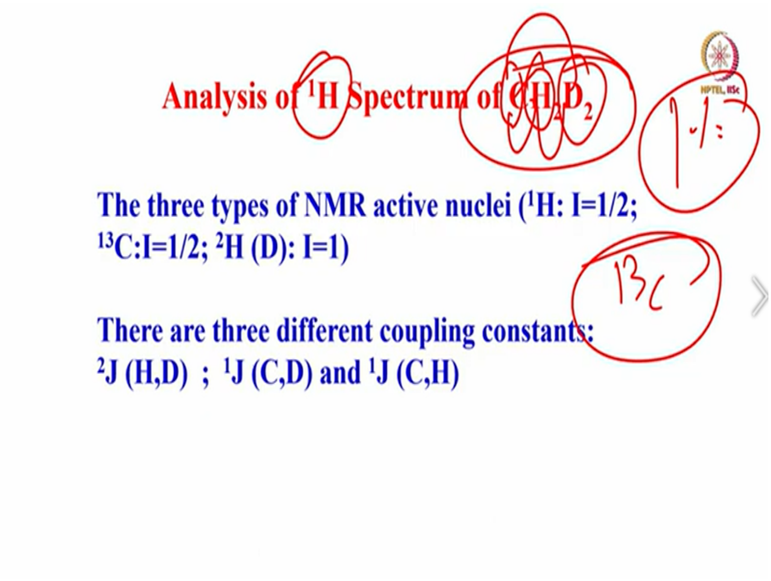

So, this way you can start under analysing this. Now we will try to simulate the spectrum for simple molecules CH2D2. It is a methane, deuterium substituted 2 protons are replaced by deuterium. Now analyse the 1H spectrum for this. What is the type of spectrum you are going to get for this? There are 3 types of NMR active nuclei present in this molecule, 1 is carbon

13 that is spin half, proton spin half, deuterium spin 1, there are 3 nuclei and you can think of 3 different types of coupling.

There can be coupling between C and H, there can be coupling between H and D, there can be a coupling between C and D also, that is also possible. So, all the 3 types of couplings you can think of, but I will give you a condition carbon 13 is in natural abundance. Natural abundance is only 1%. So, generally when there is 1% abundance, we see small intensity peaks on either side of the main peak, we call them satellites. We discussed again in the earlier course. So, remember, I want to consider carbon 13 in the natural abundance, you should get only satellites.

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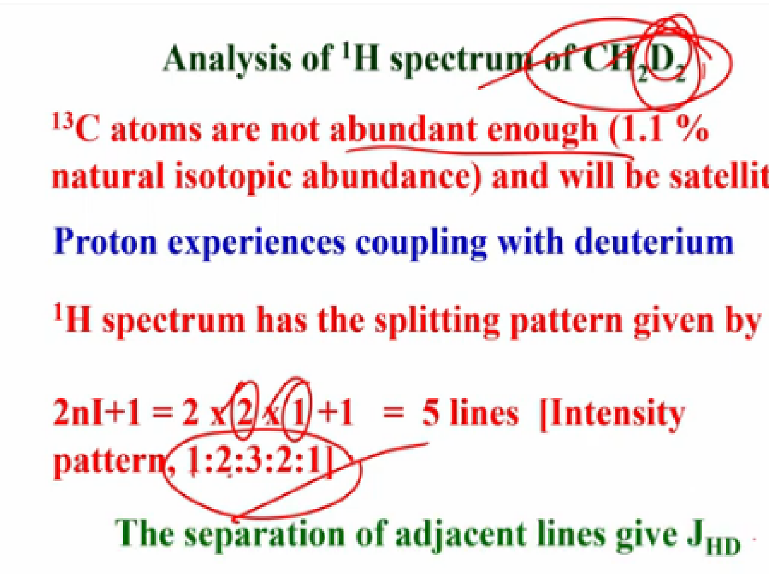

Now, let us analyse the 1H spectrum of CH2D2; what is going to happen? If we look at the carbon-13 atoms which are not abundant, we will ignore it because they are satellites. Right now, we do not worry about it, and we consider only to deuterium. Then what is the pattern you expect? The pattern we expect, it is simply put the 2nI + 1, deuterium spin is 1, there are 2 deuterium's which are coupled to proton. So, 2 into 2 into 1 + 1. So, you are going to get 5 lines pattern and the intensity is 1 is to 2 is to 3 is to 2 is to 1. Very simple. If you take this molecule, take the protons spectrum you are going to get a pentet of intensity 1:2:3:2:1.

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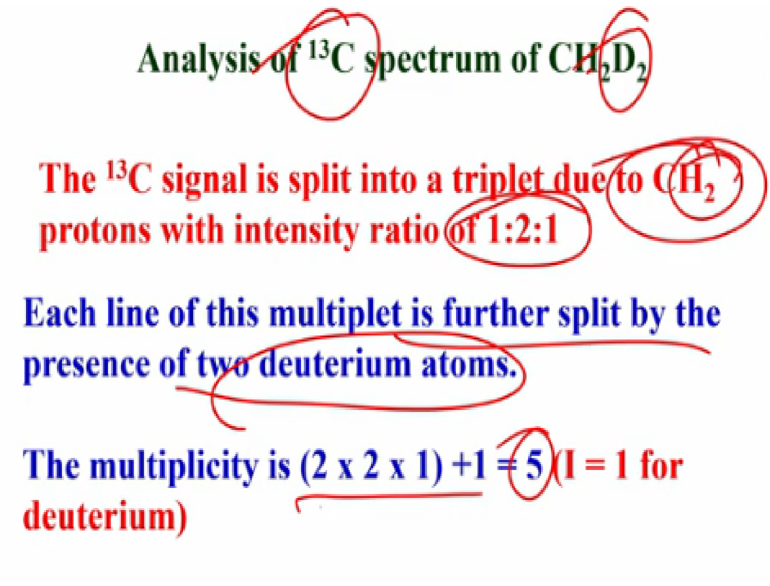

Now what happens if I look at the carbon 13 spectrum, but remember carbon 13 when I am observing proton is a satellite, but when I am directly detecting carbon 13, I do not worry about satellites. Then these two are abundant. So, they are directly coupled to that, I am directly detecting carbon 13. Now, what will happen that spectrum? Carbon 13 is split into triplet because of CH2. What is the intensity pattern? see carbon coupled to chemically equivalent protons, with intensity is 1 is to 2 is to 1. Now, each line of the multipler is split by the presence of 2 deuterium atoms. Spin is 1. Use this 2nI + 1 rule again and now you are going to get 5 lines. So, each line of the triplet of carbon 13 is going to be a pentet. So, we get a triplet with each line will be pentet of the intensity pattern 1:2:3:2:1.

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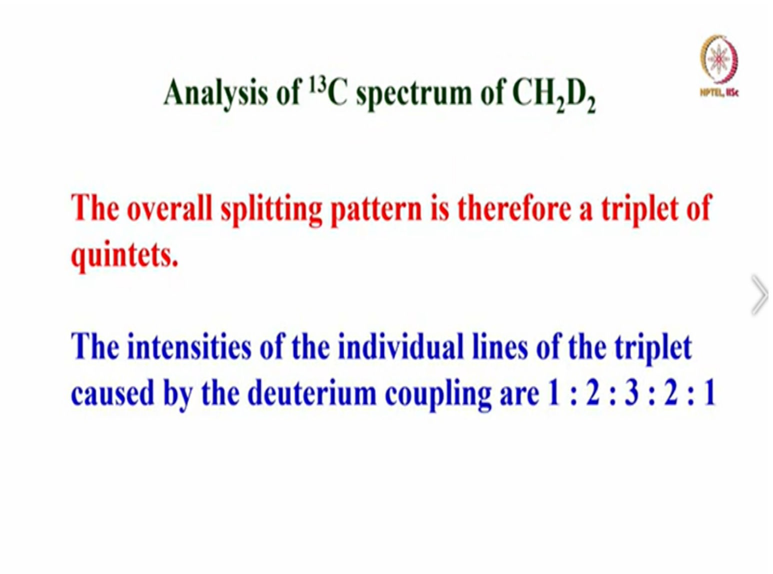

So, this is how each line is going to be a pentet, and then each line of the triplet is going to be pentet like this.

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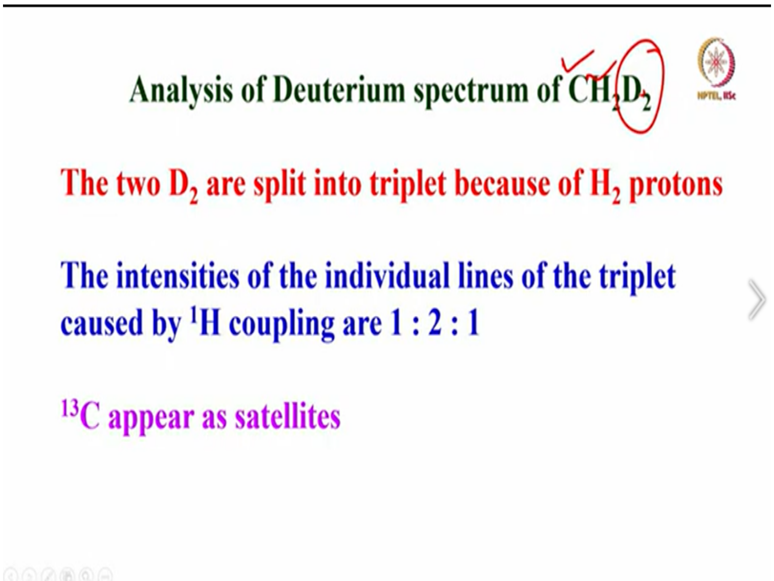

Now what happens if I see the deuterium now, this is another possibility. I saw carbon 13, I saw proton, I agree. What about deuterium then. The deuterium will split into a triplet because of CH2 protons, equalent protons. This coupled to carbon 13, yes, it does couple to carbon 13. But carbon 13 coupled peaks appear as satellites. Since I said this is in natural abundance right now, we do not worry about it.

So, it is small intensity peaks which if you ignore, there is a coupling, but still if you ignore for the deuterium spectrum, you are going to get a triplet of 1: 2 :1. Supposing you also look at the carbon-13 satellites also. Then either side of the triplet, you are going to get 3 more lines which is 3 satellite lines which are split into again triplet because of this. So, this is how you understand the spectrum of it.

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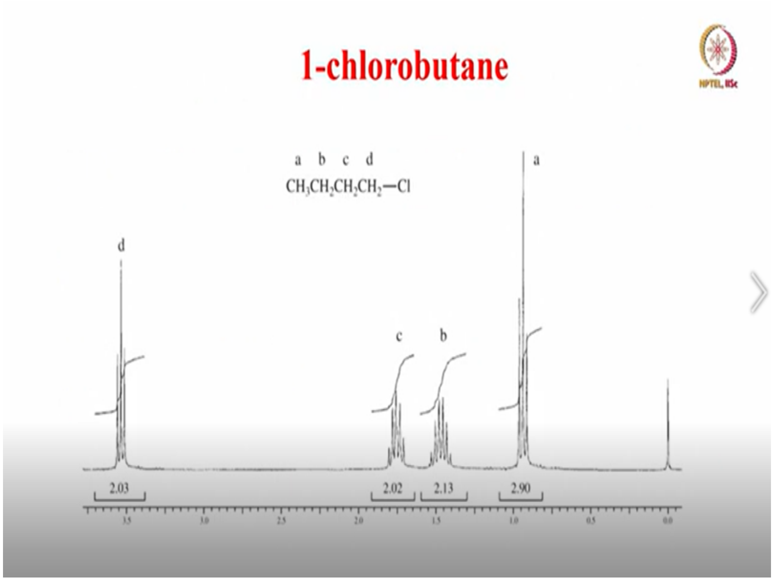

So, now I think the time is up now. What I am going to do is I will stop now, what we understood today is, I told you a lot about the coupling, multiplicity pattern, strengths of couplings, how do we generate different types of coupling patterns? what is the nomenclature for the pattern, whether it is doublet, triplet, triplet of doublet, doublet of triplet, quartet of triplet; all those things we understood. Depending upon the strength of the coupling, you can name it; and it is applicable to every type of nuclei, not necessarily proton.

And then we simulated the spectrum. We know how to simulate the spectrum based on the family tree approach, whether one proton is coupled to chemically inequivalent protons or chemically equivalent protons; depending on that, we can generate the multiplicity pattern. So, we understood simple molecule CH2D2, how the proton spectrum comes, how the deuterium spectrum comes, how the carbon spectrum comes, when it is coupled to spin half nucleus and also spin 1 nucleus.

So, these are all the interesting things, and we synthesized the spectrum; understood by using the knowledge of multiplicity pattern. That is why I said, this knowledge is very much useful in the analysis spectrum. So, now, what we will do is, we will stop now. We will come back and then continue with the analysis of few more spectra of protons; and other nuclei. I stop here. Thank you.