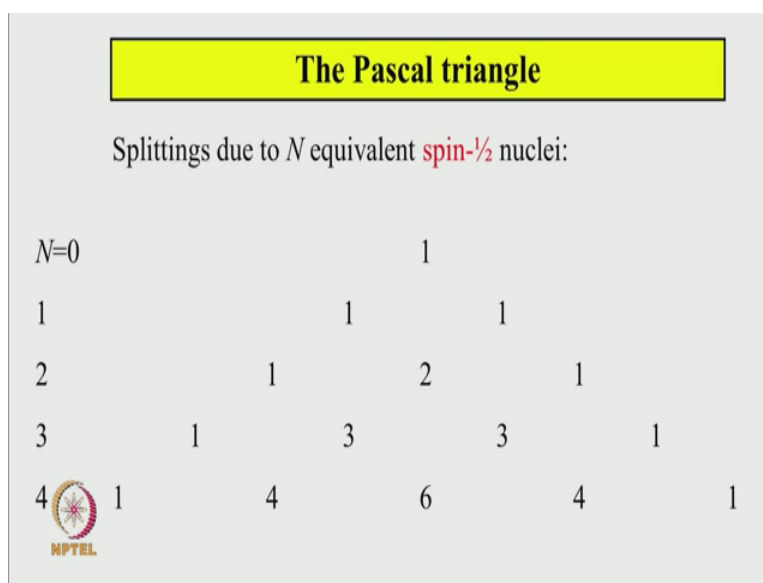


Principles and Applications of Electron Paramagnetic Resonance Spectroscopy
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Lecture - 04
Electron-Nuclear Hyperfine Interaction - II

Hello there. Last time we have seen how the EPS spectrum of any radical or other species can split into various hyperfine line; due to the interaction of electron magnetic moment with the nuclear magnetic moment, and we call them hyperfine splitting. We saw a lot of examples where the splitting comes from the spin half nuclei, particularly from proton. And we saw certain patterns that we can see in the spectrum when there are many equivalent spin up nuclear there.

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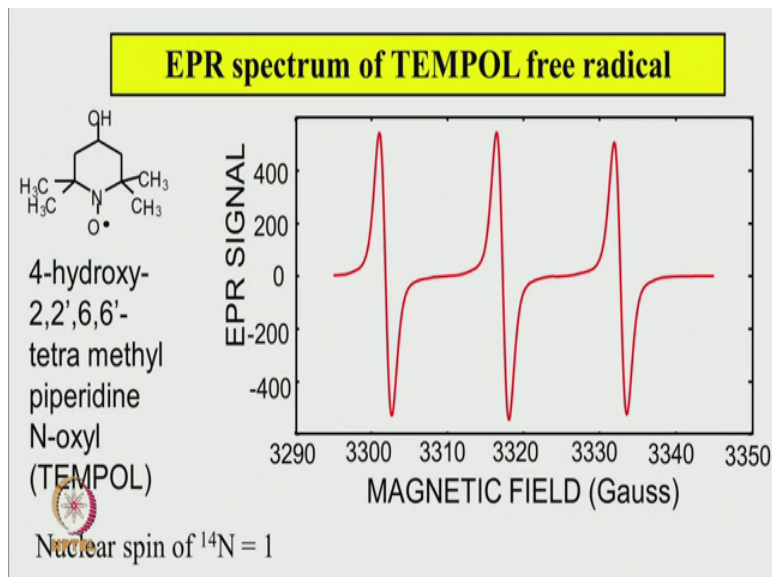


And we said that one can use the Pascal triangle to find out the number of lines and their relative intensities.

So, here we have this triangle again displayed here. So, when there is no nuclear spin, we get one line it is in the middle then we have one spin off nuclei displayed into 2. So, this much is the splitting due to the nuclear spin. So, you have got 2 nuclear spin, then again it split by the same amount here from this to this, this gap is same as this gap there. But the intensity ratio becomes one is to 2 is to 1. That way we can build up the various possibilities for any number of nuclear spins. Today we are going to generalize this idea

of many equivalent spin where the nuclear spin is more than half. Last time we have seen several examples of this kind. One example is here.

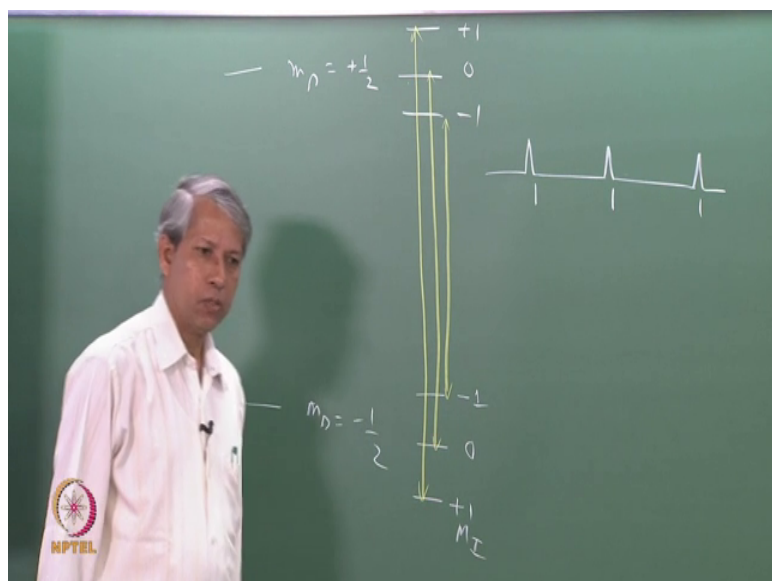
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That this free radical temporal.

Here the un pair electron interacts with nuclear spin of nitrogen, and we saw that it gives EPR spectrum this kind where 3 lines are equal intensity. Let us recapitulate how we arrived at that; first that the electron spin in a magnetic field splits into 2 because of the spin half m_s equal to minus half and plus half.

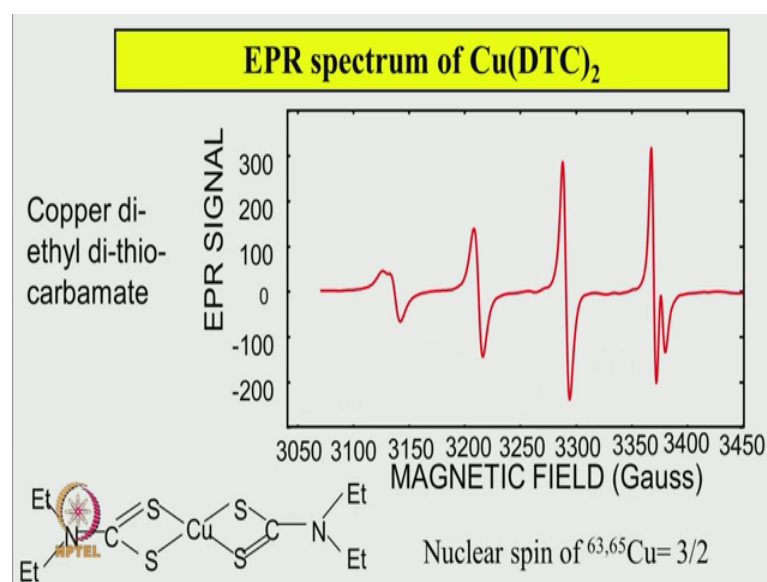
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Now this interacts with the nuclear spin of I equal to 1 or m_I equal to plus 1 0 and minus 1. These are the possible M_I values. Similarly the upper energy level is also split into 3, plus 1 0 to minus 1. And the transition involves the change of the electron spin from this to this without changing the nuclear spin.

So, we get 3 transitions of this kind. This so, since all this 3 levels are almost equally populated we get the spectrum with the intensity ratio 1 is to 1 is to 1, which are in this fashion 1 is to 1 is to 1. So, this gap is same as this gap which is a measure of the interaction of the nuclear spin with the electron spin all right. We also saw copper complex.

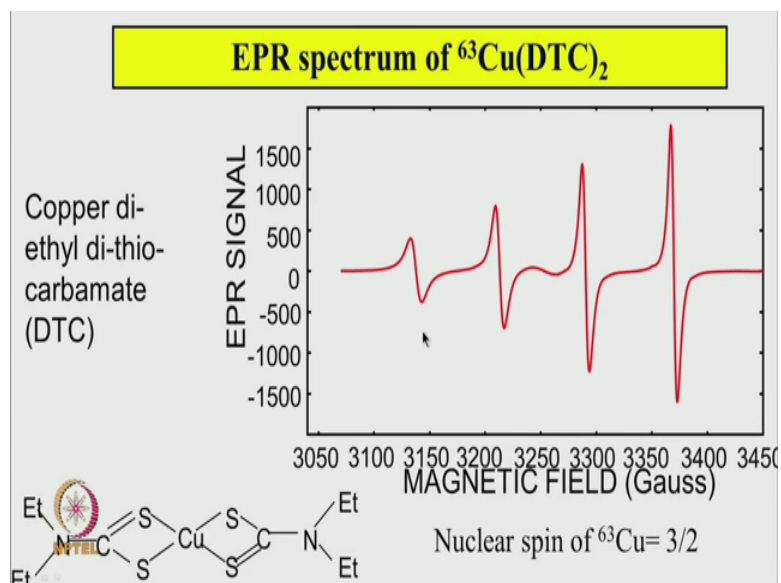
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Which is of course, a very complicated one if we see it. Now this spectrum comes from the naturally occurring copper complex.

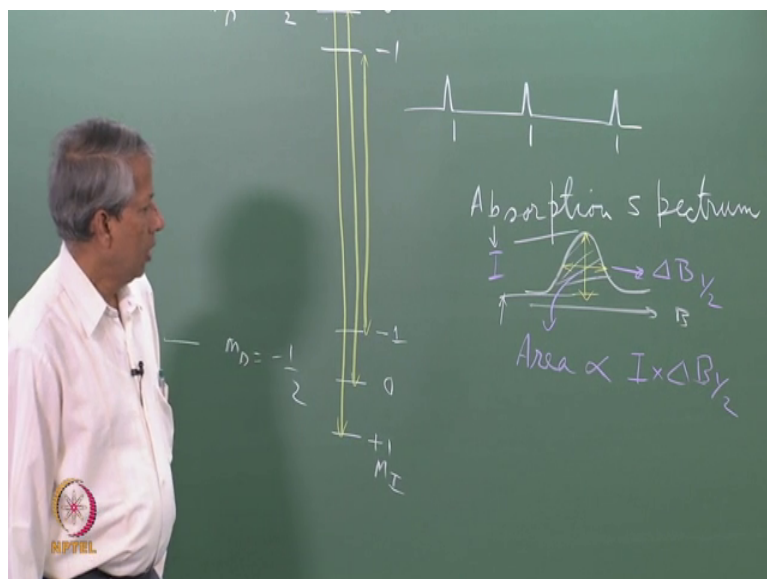
Now, naturally occurring copper as 2 types of isotopes copper 63. And 65. So, to understand the origin of this or other to simplify this spectrum.

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Let us get the look at the spectrum, where we have only one end it is isotope; that is, copper 63, and here is the spectrum. Of course, spectrum has become simpler than earlier, and what it shows is that the 4 lines of different intensity. By density if I mean the height of this one this height is bigger than this which is bigger than this and this is the least per same time if you notice carefully the widths are different.

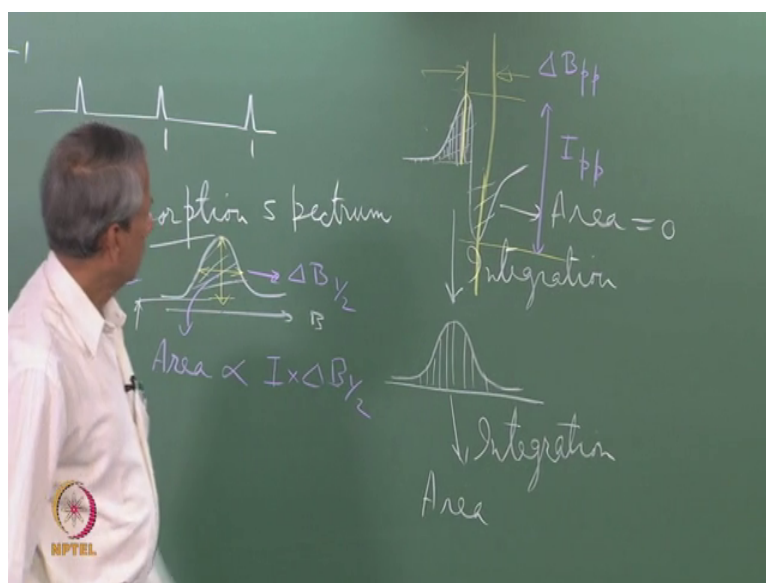
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So, one has to be careful in deciding what the intensity is.

So, for an absorption spectrum, which looks like this type of plane. This is the magnetic field, we normally say intensity is area of this. You know that is related to let us say if we take the whole width and a half intensity of this and measured this one the height of this one. So, this I will call $\Delta B_{1/2}$; that is, full width that half maximum of the intensity. And this one this let us call it I , then this area is proportional to I into so, that we all understand. But the EPS spectrum that appears here is in the form of derivative line.

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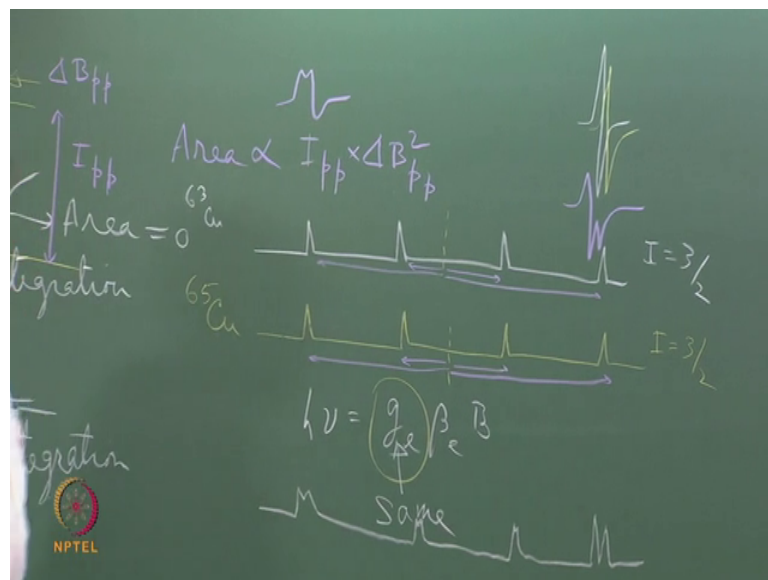
So, what can you say about this area? For derivative line the shape is of this kind. So, if you simply add this area, and this area this is going to give area will be equal to 0. So, that is of no use. We have to somehow therefore, relate this intensity to this type of intensity. So, what is done here is the first you convert the derivative signal to an absorptive signal of this kind by integration. That is start from this and keep on adding this area, various small strips taken then keep on adding this little small strips. Then continue to do this all the way to the extreme right. So, then it will give a absorption profile of this kind.

And then the area of this will be can be calculated in the similar fashion as this one, or one can actually do numerical integration this time. You get add all the little strips in then I will get the area of this one. So, further integration gives the area. Now one can go through this numerically with the advantage of computer this can be done very easily, but

one can also create a very good approximation to this in the similar way that we have done it here by measuring the full width or half maxima and this width a intensity here.

So, similarly we in a similar fashion we can define 2 quantities. One is this intensity let us call this peak to peak intensity. So, that is from the top of this to bottom of this, I will call it $I_{\text{peak to peak}}$. And in an analogous manner that the width that is used here, we can define a width from this to maybe release different color this to this. So, this can this one, this width from this peak position to this peak position, and I call it $\Delta b_{\text{peak to peak}}$.

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So, in this case the area of this after doing all the suitors are in 2 times integration, the area that we find here, will be this area will be proportional to this $I_{\text{peak to peak}}$ times the width that we have here $\Delta b_{\text{peak to peak}}$ square of that.

So, it is not the product of this and this the way it comes here, the reason is simple that we have to integrate it 2 times to get the area, first integration gives this second integration give this one. That is why the this comes in the form of product. So, because this area is now proportional to the width square. Even a small change in the width can really change the in height of the signal quite appreciably. Now that is seen here that width of this line is slightly broad, and that broader than this one this is broader than this one this is when broader that this one. So, this is the narrowest line. So, consequently the height is maximum here. So, if we can integrate all these areas either by computer or in

the absence of there we can have manually, find out the height of all these lines and also measure the width of this peak to.

And if we calculate it we will find that all 4 of them have exactly the same intensity. So that means, that the apartment intensity difference is not of any consequence, it is the intrinsically all 4 lines have the same intensity. So, why 4 lines coming now? This is easy to understand the nuclear spin of copper is 63. So, the way we have seen here the 3 by 2 is the nuclear spin. So, this will split into 4 lines. This will also split into 4 lines, and then I will get 4 transitions of a Korean density. And that is precisely seen here. So, why this lines have different width is something we will not try to understand now, but we will undersatand later. For the time being we notice that this is indeed a very special feature of this copper complex, when you compare all the spectrum that have seen for free radicals, where the line widths are essentially same for all hyperfine lines.

now to understand the this spectrum, now we try to see; let us look at this the various nuclear properties of these 2 isotopes.

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Properties of copper nucleus				
Isotope	Atomic mass	Natural abundance	Nuclear spin	Magnetic moment, β_N
^{63}Cu	62.929	69.17%	3/2	2.43
^{65}Cu	64.927	30.83%	3/2	2.54



Where is the difference? Copper 63 has about 69 percent natural abundance. 65 has 30 percent or 31 percent to be more accurate. Both of them have got the same nuclear spin, 3 by 2 by 3 by 2 here. So, what I have got in this spectrum is actually 2 types of complex. One has got copper 63 other is copper 65. So, both will therefore, split into 4 lines, because the spinoff this is let us say, this is for copper 63.

Now, copper 65 will give similarly, 4 line spectrum and because it is nuclear in also 3 by 3. What we have is a superposition of these 2. But when we super pose them will exactly overlap, just try to understand that first. Now center of the spectrum which is here, what is this for these 2 here will be the same for these 2 species, what is at the center of the spectrum? That comes from this relationship that. So, this value g factor or g value of this one. Say both the spectrum are recorded by in this at the same time, because sample had 63 as well as 65. So, they experience the same frequency. So, the difference can come if they have got different g factory. Now can g factor will different for these 2 species? Now g factor is an electronic property of the molecule with. It depends on how the electronic structure that the molecule has now why have I have got these 2 isotopes what is the difference only in the nuclei isotope, now when the copper nuclei is reason only heavy nuclei.

So, for such heavy nuclei usually the electronic wave function does not depend on the nuclear mass. Only if the nuclear mass is very light for example, hydrogen atom when you have got either it is proton or neutron the wave function can slightly be affected. But otherwise this difference is so, the mass is so high compared to the mass of electron, that this will have no effect on the electronic structure of this complex. So, the g factor will be same for both of them. In other words center of the spectrum will same for both the species, then what else can the difference be? Is the splitting go in to different we do see that splitting is different here, that if you see very carefully this side this small hump is there.

So, possible that it could come from overlap of 2 hyperfine line. Similarly, this could this; obviously, coming from to hyperfine line which are overlapping here though it is not very clearly seen only part of this line is seen here. So, it indicates that this splitting that we see here and here.

They are not quite the same, but only evidence is that which is some sort of difference here, but there overlapping here. So, can the hyperfine splitting constant will be different? Further we go back to the table of the properties of copper nuclei, you see that magnetic moment of copper 63 is 2.43 and for copper 65 is 2.54 indeed the magnetic moments are different copper 65 has slightly bigger magnetic moment than a copper 63; that means, the electron in the copper complex see s a little bigger magnetic field that is

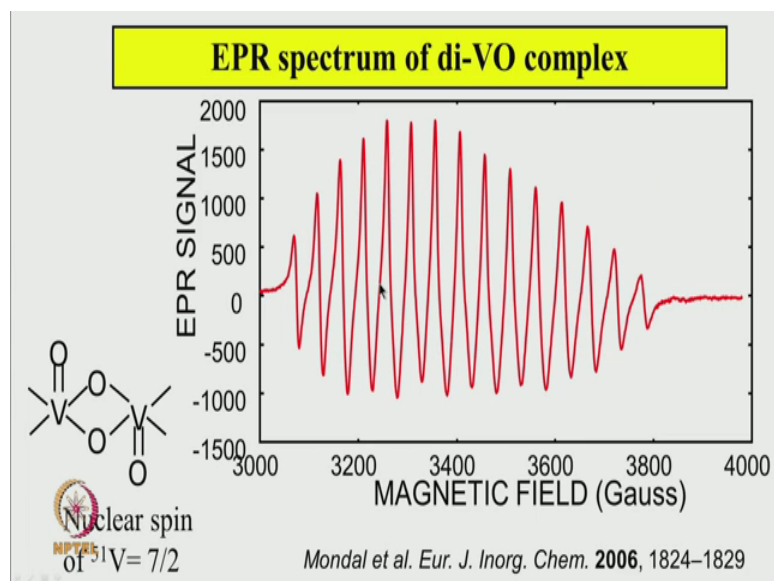
created by the 65 nuclei. So, so this splitting will be just a little bit bigger than this one. This will be slightly bigger than this one. Now how is it going to change?

So, I know this gap will be generally bigger than this gap. Similarly, from here this gap will be still bigger than the gap that is here. Same way the gap that is here will be little bit less than the gap that is here. And the gap between this to this will be much smaller than the gap that is here. So now, you can imagine, that if we now overlap this 2 I will see spectrum which may look like some sort of splitting here, and may be little bit splitting here, but more commonly here. And the top of that the width of this are not same across this from right to left.

The widths are more here and least here. So, what happens in the process? The resolution is such that is going to be such that this will be seen much more clearly than this one. And when you take the derivative we get a let us say here get one line which looks like this on this type of derivative seen you and other line this type of thing. So, there is a partial cancellation of this part of the spectrum with this part of the spectrum. So, net result is that we get spectrum which looks like this. This type here the lines are broad

So, what we see is small hump compared to and not. So, well visible line here. What is more? You see that copper 65 here has a 31 percent natural abundance. And 63 has which have 70 percent or say 69 percent. So, this intensity is correspondingly going to be contribution of this will be somewhat smaller. Well, by the exactly the same ratio. 30 percent intensity will come from here and 70 percent come from there. So, we will see we will have this smaller signal than this one. And that is exactly what we see here. So, we understand how this signal appears again the in with which changes gradually from the right most to the left most signal.

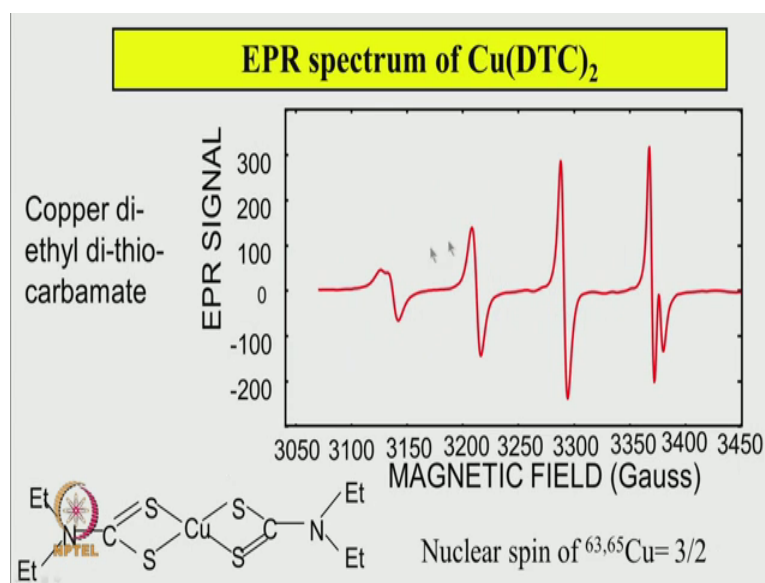
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The other example we saw earlier is this di vanadyl complex. This complex has two vanadyl centers here given here VO and VO there, and they are connected by this oxygen bridge. Either other part of the chemical conditions are shown here.

It is this 2 bonds here these 2 and there are a lot of things around this one which of which are of not much interest to us. So, all that is shown is that these 2 vanadyl centers the way they are bonded and form the complex they look completely equivalent. So, what sort of spectrum we expect from this 2 equivalent vanadyl complex? This is the spectrum that appears here. So, we see that again the widths are not the same.

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Unlike the copper complex; where the width increased gradually from right to left here the narrow lines are in the middle, and a broad lines are at the 2 sides are broader here and also here. So, that again will not try to understand origin of this right now we will postpone it to some other lecture. But write we will see the intensities are same or not. Here we both are similar argument that width and height together decide the intensity.

And for derivative line we use this formula we will see that intensity of these lines are not same. Intensity changes gradually from here become maximum here and goes down at this way. So, how does one account for this different intensities? So now, having learned so many different types of nuclear now it should not be difficult for us to generalize say this to this particular case for vanadyl complex.

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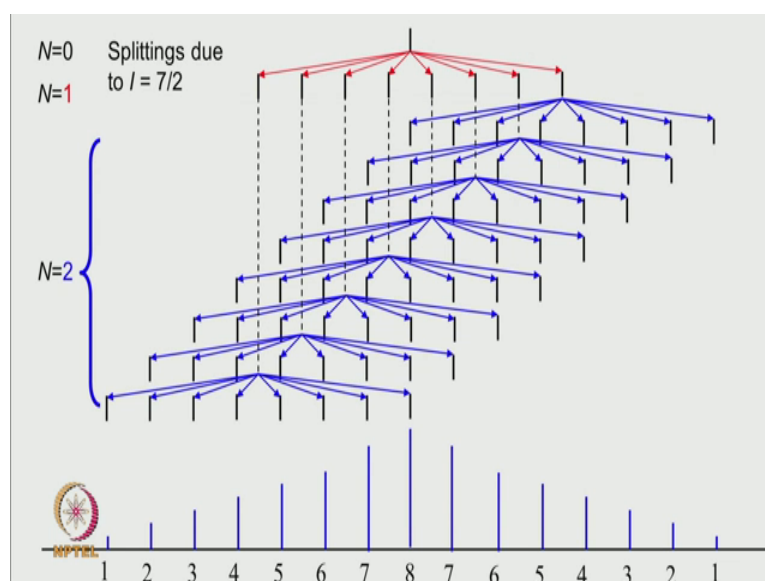
Properties of vanadium nucleus				
Isotope	Atomic mass	Natural abundance	Nuclear spin	Magnetic moment, β_N
^{50}V	49.947	0.250%	6	3.61
^{51}V	50.944	99.750%	$7/2$	5.83



For that we find out the magnetic properties of the vanadium nuclear see vanadyl complex is as the nuclear vanadium it has isotopes ^{50}V and ^{51}V . So, here the natural abundance is only 0.25 percent for ^{50}V . And 99.75 percent if ^{51}V so we can as well assume that this is absent there in.

Spectrum that we see here is coming from this isotope. And its nuclear spin is $7/2$. So, $7/2$ means how many lines I will get from all the disc that I have seen here? Typical 8 lines of equal intensity for only one vanadium.

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Then the second vanadium is going to split all the lines further, and I have shown it here. So, if this is the line position in the absence of any vanadium, then when I have got one such nucleus of vanadium whose nuclear spin is $\frac{7}{2}$. See this splits into say 8 lines this is the center. So, this gives such a way the this gap is equal to this gap and this equal to this gap. So, the 8 lines are split are same here. Now that is due to the first nucleus. When I have the second nucleus each of these lines are going to split further by the same amount and same number of lines. So, 8 lines comes from here. See the gap between this and this is same as gap between any of these line here.

Similarly, this line is split into again 8 lines that way all the other lines are shown here. So, what I see here therefore, is that at a given position more than one contribution comes. So, here therefore, count number of lines that I can see here, this intensity 1 by the 2 lines come there. So, this will be 2 that if you count all of them there will be 15 lines with intensity given here 1 2 3 4 5 6 7 8 7 6 5 4 3 2 1 and this is what is seen here. Though it is not obvious one, can actually integrate this various lines and come to this conclusion there. So, this shows that vanadyl complex when the 2 vanadyl layer which is perfectly (Refer Time: 23:01).

From the whatever we have learned here, the origin the way the various on line that appear there and how equivalent nuclei can produce different type of magnetic intensities.

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The Pascal-like triangle

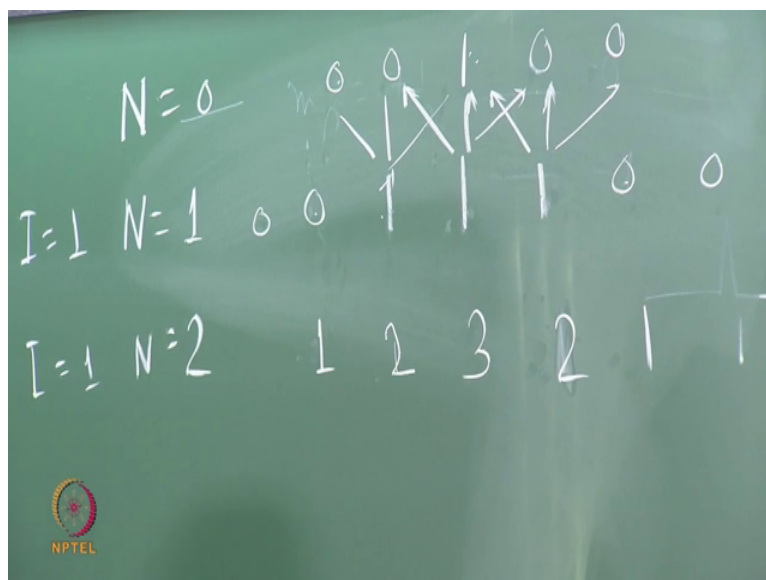
Splittings due to N equivalent **spin-1** nuclei:

$N=0$					1				
1				1	1	1			
2			1	2	3	2	1		
3		1	3	6	7	6	3	1	
4	1	4	10	16	19	16	10	4	1


NPTEL

So, can we generalize this Pascal triangle that we have seen for spin half system. We are trying to do that now. So, here the Pascal triangle is shown for spin one nucleus. Let us see how it is created start from line intensity one.

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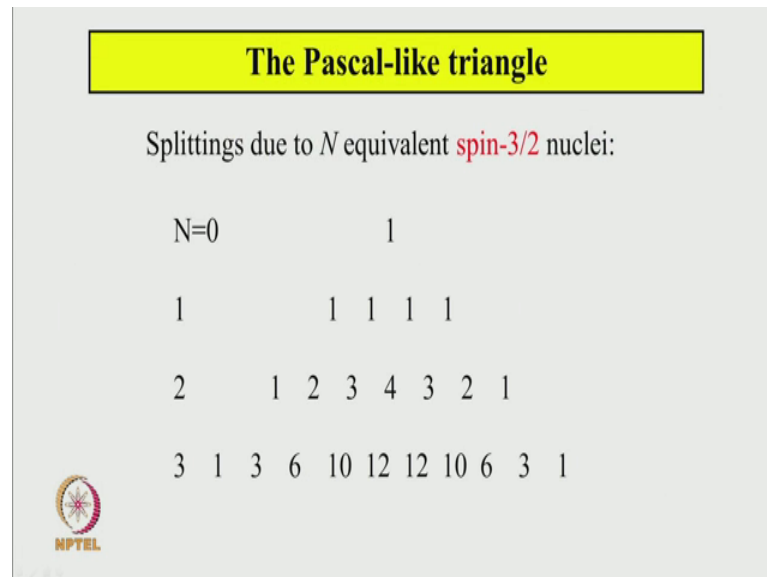
Where there is no nuclear hyperfine interaction. And that how is number of such nuclear 1 I is equal to so, then this will split into 3 lines with intensity 1 is to 1 is to 1, this can be found out by adding 3 numbers comes from this 3 that is appear in the top of that one directly above other is from the left other form the right.

So, here you can assume that 0 is here 0 there. So, this 0 0 1 gives one, and here I get this 3 lines from here and here and 0 is here then this gives one there. Similarly, this one I get 3 numbers from here, here and then I get 0 there, then I get 1 0 0 gives one that is fine 1 is to 1 is to one, that is what we have seen in case of temporal. Now I equal to 1, for n equal to 2. So, we follow same argument know that to get a number here I try to find out the numb the number which is directly on the left in this above and the right. So, I have got here 0 1 1 1 gives me 2, but here then immediately above is 0 0 1. So, this gives me one.

Then if we what is the intensity here we can expect, I get in value 1 1 1 is 3 similarly here 1 1 0 gives 1 1 0 0 gives sorry, this gives 2. So, it is 1. So, 1 2 3 2 1 is the relative intensities of spin 1 nuclear. And what is the 2 optimal present there. That way we can generalize this that every time it get number from here for example, I get add the

numbers which directly to the left and above and right 2 1 0 gives 3 that way the numbers are created there. So, this is not Pascal triangle, but very much like the Pascal triangle. We add 3 numbers one is one is left right and in the right directly above.

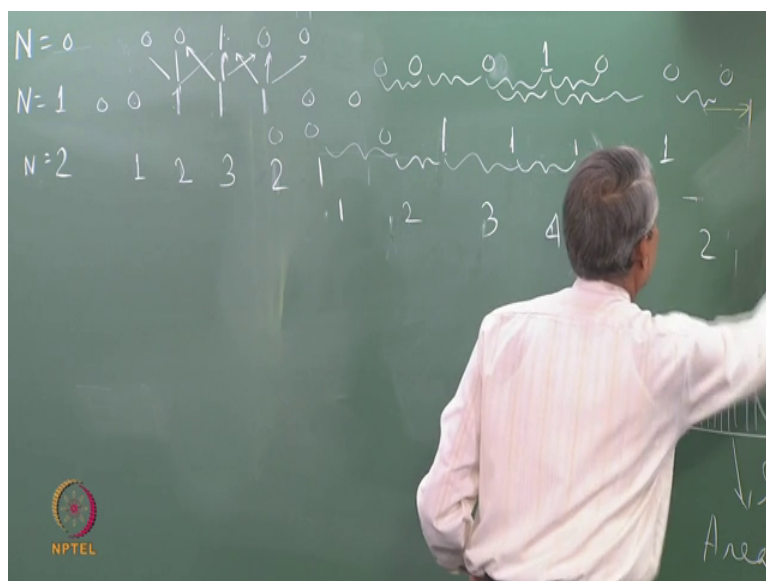
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Now, we have seen the copper spectrum where spin is 3 by 2.

Can we create a Pascal like triangle 3 by 2. Again the procedure is very similar, but not quite the same, and the triangle is shown here. When there is no hyperfine splitting that is the intensity one is here, I want one spin 3 by 2 splits the line then we have 4 transition. So, to get this 1 is to 1 is to 1 is to 1, all I do is the I add now 2 numbers from the left and the 2 numbers from the right, not it is let us see how it is done here now.

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I will get 4 transition here, here, here, here. So, assume 0 0 0 0. So, to get the intensity here I add 2 numbers from the left and 2 numbers from the right. So, this and this together gives a value 1. Now for similarly here I add this 2 numbers from left, and this 2 numbers from the right is gives me 1. Then to get the line here, I add 2 numbers from this left, and this 2 numbers from the right gives me 1. Similarly here I get I add this and it is it gives 1. So, this looks simple 1 1 1 1, but let us say do we understand the procedure correctly. So, for that we go down once more if you have 2 such nuclear of spin 3 by 2, then I will each of the lines will split into 4. So, there will be line here, a line here and for this line will be here, here.

Similarly, for here, here. So, to get the intensity here I have a this 0 0 0 I add this and this to get a value 1. That get the intensity here I add this and this. So, then I get a value this 2 to get the value here I add this 2 and this 2. So, that gives me 3. So, then further this will give me this, this and this, this, this is 4. Then this and this gives me 3 2 1. So, 1 2 3 4 3 2 1. That is the way line numbers are created here. See can check if really you can figure out how this numbers are found out. So, we see that Pascal triangle is a triangle which works for spin half system, but we can generalize this thing for other type of nuclei as well, we have generalize it to I equal to 1, then I equal to 3 by 2.

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Generalisation of hyperfine patterns

Nuclear spin quantum no. = i

No. of equivalent nuclei = N

Total Nuclear spin quantum no. $I = N \cdot i$

Components: $-I, -I+1, \dots, I-1, I$

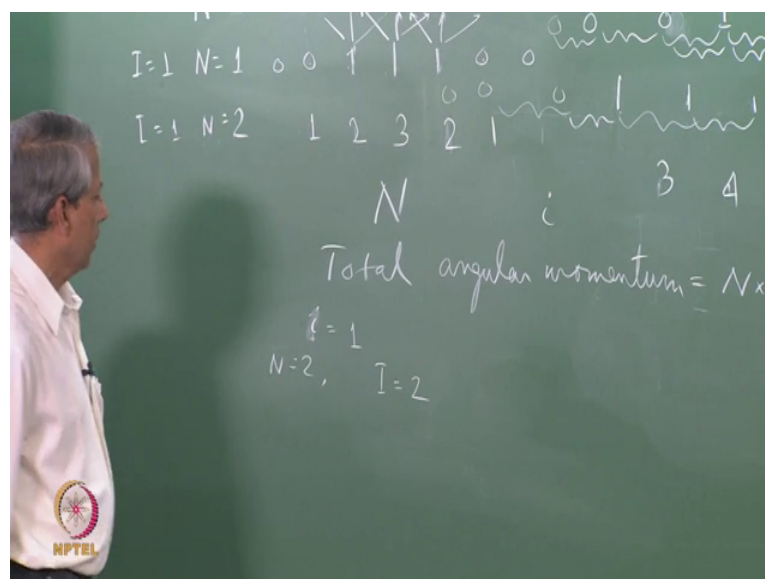
Total components = $2I+1$

Number of hyperfine lines: $2I+1$



Now, we are going to generalize this pattern that have seen here, and summaries what we have seen for various nuclei.

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$I=1, N=1$ 0 0 1 1 0 0
 $I=1, N=2$ 1 2 3 2 1 0 0 1 1 0 0

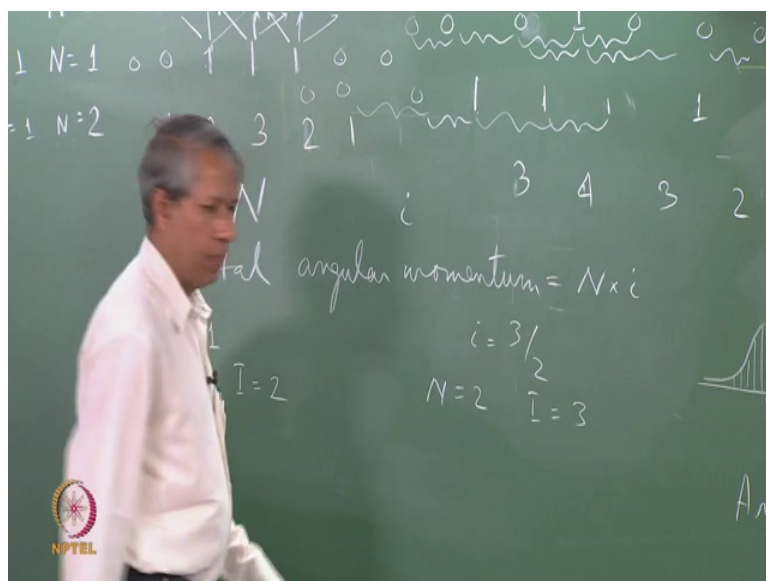
N	3	4
i		

 Total angular momentum = $N \cdot i$
 $i = 1$
 $N=2, I=2$



So, if there are let us say N nuclear spin. And with a individual spin angular momentum is let us say a little i , then the total angular momentum becomes N times i . Here for example, when we have got I equal one here. Now with this little i we change the notation this small i for in the each nuclear now when the 2 of them that there n equal to 2 , then my i becomes 2 .

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Similarly, when I equal to $3/2$, but n equal to 2 of them are there. So, then total I becomes 3 . So, then total nuclear spin quantum number becomes n times I . And each component will be minus I to plus I , changing in units of n . So, total components equal to therefore $2I + 1$. So, each of this produces a local magnetic field that the electron sees. So, there will be number of hyperfine lines given by this number $2I + 1$. Those many lines will be seen there.

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Generalisation of hyperfine patterns

If the total I is an integer, there will be a line at the centre.

If the total I is a half-integer, there will be no line at the centre.

Relative intensities: Pascal triangle for spin- $1/2$ nuclei

Analogous triangles for other nuclei



Now, if this total I happens to be an integer then there will be line at the center.

Why? Because then $2I + 1$ is an order number. So, odd number will give a line at the exact at the center, and if I is an half integer, then $2I + 1$ becomes an integer. So, there will not be line in center here for example, here 3 by 2 was there. So, there is no line at the center. And the relative intensities will be given to the Pascal triangle for spin half nuclei and analogously for other nuclei we can generate the triangle of similar kind. The summary is that all EPR spectra characterized by the basically 3 things. One is the line position.

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Summary

EPR spectra are characterized by:

- line position ($h\nu = g\beta_e B$)
- Linewidth
- Hyperfine splittings



Line position for any of the spectrum here is given by this relationship; that is, essentially given by the g value. And the width which also very important property we saw that for most of the organic nuclear the width are very similar.

But not necessarily true for transient complex copper complex or vanadyl complex is widths a different. And of course, the hyperfine splitting and how many of them are there and their relative intensities. These are the characteristics of EPR spectra.

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EPR or ESR?

Now is the time to decide whether *EPR* and *ESR* mean the same thing.

The spectrum arises due the magnetic moment of an electron.

Origin of the magnetic moment can be either due to the spin angular momentum or the orbital angular momentum of the electron, or both.



Now, have you learned this mattress it is time to decide that whether EPR or ESR mean the same thing or not. I mentioned in the first lecture that we must discuss this and come to an agreement after some time. It is time we did that.

You see that spectrum comes from the magnetic moment of the electron. Now magnetic moment can come either from the orbital motion of the electron or the spinning motion of the electron or it can come from both.

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EPR or ESR?

Spin angular momentum	$\vec{\mu}_s = -g_s \beta_e \vec{s}$	$g_s = 2.0023...$
Orbital angular momentum	$\vec{\mu}_l = -g_l \beta_e \vec{l}$	$g_l = 1$

Most organic free radicals have very little orbital angular momentum $\rightarrow$ *ESR* should be OK.



But for systems that have both angular momenta, then?

It is the relationship between the angular momentum and the orbital momentum. If this magnetic moment comes from the spin angular momentum, then this relation is $\mu_B g S$. S is the spin angular momentum. If it comes from the orbital motion of the electron, then orbital angular momentum of l this side and magnetic moment due to l relate very similar manner. Only difference is the value of g that is used for spin angular momentum is about 2.00 something.

These numbers are not of consequence to us. And the g value that is used for orbital motion is precisely equal to 1. So, that is what the difference is only difference will be quantity of and not quality qualitatively both are very similar. Most organic molecules have very little orbital angular momentum. So, in that case calling them ESR spectrum will be, but the system horizontal in which has both motion due to a s and l that is magnetic moment coming from the orbital motion and the spin motion what will you call them. Think about it.

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EPR or ESR?

It is also possible that a system has **no spin angular momentum** but has an orbital angular momentum.

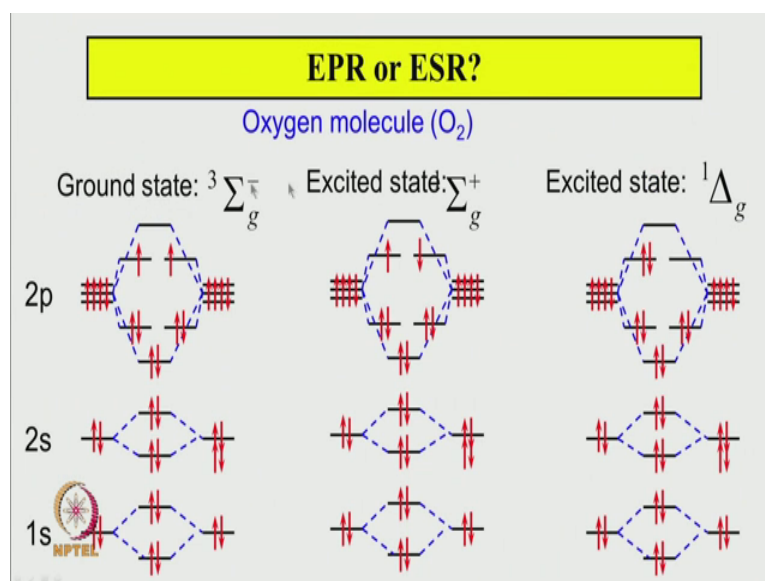
Then calling its paramagnetic resonance spectrum an **ESR** spectrum is simply wrong.



Now, it is also possible, that system has no spin angular momentum, but it has an orbital angular momentum has a magnetic momentum comes from that motion and you can get a corresponding parameter spectrum.

Then calling it an ESR spectrum, would be simply wrong. Because there is no spin motion present in the particular system.

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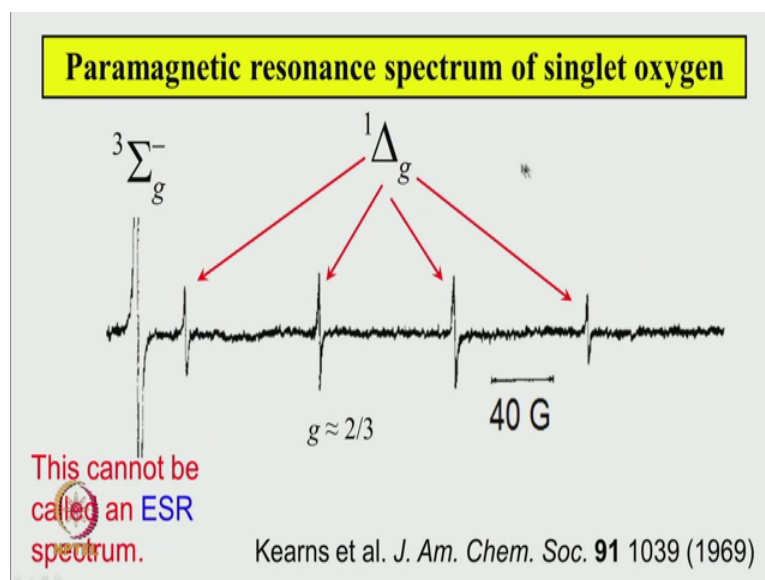


So, here in example. Oxygen molecule we are all familiar with, and usually its electronic configuration in the ground state is shown here. This $1s^2 2s^2 2p^4$ are the orbitals of the oxygen atom and there are 8 electrons there. And these middle horizontal lines are the molecular orbitals of the atoms. So, these electrons from one oxygen atom and electrons from the other oxygen atom produce this sort of electronic configuration (Refer Time: 35:03) and you must have red. In your MSE or may be under budget. Here important thing is that, these 2 electrons are unpaired here they are occupying 2 different π^* orbitals. So, that produces this triplet state of the oxygen molecule. So, oxygen molecule in the ground state is a paramagnetic species and one can actually see EPR spectrum.

Now if this oxygen molecule excited, it produces a singlet state, and it produces 2 types of singlet states. And the way it differs is you see here, these 2 spins are parallel. One of the excited states is that these 2 electrons become antiparallel now, but since these 2 occupy orbitals this can be a singlet state. And that singlet state has this sort of electronic configuration in this excited state, but it can have another excited state where these 2 electrons are promoted to stay in a one molecular orbital where naturally these spins are necessarily going to pair. So, this is a singlet state, but this π^* orbital gives rise to a net orbital angular momentum of λ which is equal to 2 gives rise to this Δ state.

So, this electronic state is $1\Delta_g$ has no spin angular momentum, but orbital angular momentum. And one can see it is if it is magnetic resonance spectrum which is shown here.

(Refer Slide Time: 36:45)



So, this singlet oxygen give rise to this 4 lines. And this spectrum also other lines which is present here which is very intense it actually comes from the ground state of oxygen triplet oxygen there.

So, you see that one can see the EPR spectrum of a molecule which has no spin angular momentum, but only pure orbital angular momentum. So, this cannot be called an ESR spectrum. By the way I must point out that this looks like hyperfine line that we have seen so far, but in oxygen molecule which is this nucleus has 16 this, also 16.

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So, this has no magnetic moment. This also no magnetic moment. So, these lines that appear here though they look like hyperfine line, but they are not. This is interesting that something else is seen here. These experiment was done in the gaseous phase. So, we take oxygen gas in the spectrometer and try to look at it is parameter resonance spectrum.

So, here the oxygen molecule will undergoes it is sort of rotational motion. A rotational motion are also angular momentum they can also give rise to a coupling of angular momentum to the orbital motion of that and that can produce such lines; which appears as if they are coming from sudden hyperfine interaction, but that is not so. Anyhow coming back to this the suddenly this cannot be called an ESR spectrum.

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EPR or ESR?

So electron **paramagnetic** resonance is a very general term.

Hence, we will continue to use term **electron paramagnetic resonance (EPR)** in these lectures.



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So, electron parametric resonance is a very general term now; which includes both parametric resonance coming from orbital motion as well as spin motion. So, we will continue to use the term electron parametric resonance in these lectures.

So, with this we come to an end of this discussion.