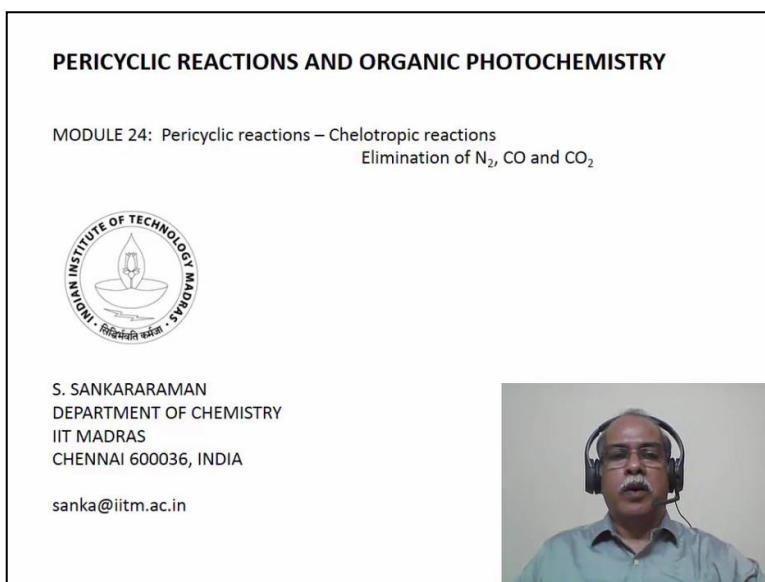


**Pericyclic Reactions and Organic Photochemistry**  
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**Module No. #05**  
**Lecture No. #24**  
**Pericyclic Reactions – Chelotropic reactions**  
**Elimination of N<sub>2</sub>, CO and CO<sub>2</sub>**

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**PERICYCLIC REACTIONS AND ORGANIC PHOTOCHEMISTRY**

MODULE 24: Pericyclic reactions – Chelotropic reactions  
Elimination of N<sub>2</sub>, CO and CO<sub>2</sub>

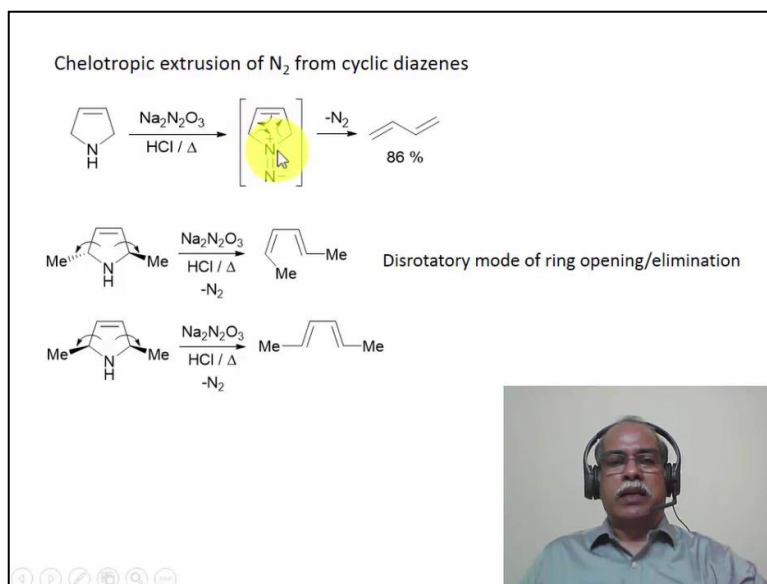


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Hello, welcome to Module 24 of the course on, Pericyclic Reactions and Organic Photochemistry. In the previous Module, that is in Module 23, we were looking at the Chelotropic reaction, and the Chelotropic elimination of Sulphur dioxide from Sulfolene kind of molecules. Now, in this module, we will continue with Chelotropic reactions. We will consider, elimination of neutral Nitrogen molecule, and Carbon monoxide molecule, in this particular module.

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Now, if you take a cyclic secondary Amine of this type here, and treat it with Angeli's salt,  $\text{Na}_2\text{N}_2\text{O}_3$  is called Angeli's salt. It is a source of  $\text{HNO}$ . And, that converts the secondary Amine, to a Cyclic Diazene, which is shown here. The Cyclic Diazene is an unstable compound. It is all ready to lose a molecule of Nitrogen, releasing Butadiene, in this particular case, at 86% yield. So, the elimination of the Nitrogen, from the Cyclic Diazene, is an example of a Chelotropic reaction. This Nitrogen is connected, simultaneously to these 2-Carbons.

So, the Carbon-Nitrogen bonds break, during the course of the Chelotropic reaction. Elimination of  $\text{N}_2$ , is the driving force, for this particular reaction. The elimination of  $\text{N}_2$ , from the Cyclic Diazene, is highly Diastereoselective in nature. In the sense that, if you take the Dimethyl derivative, which is a Trans derivative, and treat it with Angeli's salt, in acid, the cyclic Amine is converted into the corresponding Diazene, which is this Cyclic Diazene, which is this structure.

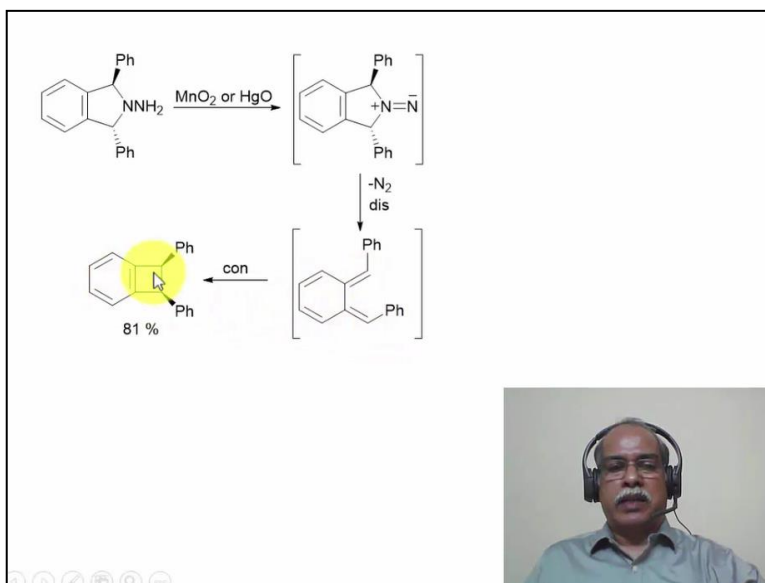
And, the reaction, undergoes a Disrotatory mode of ring opening, elimination of Nitrogen takes place, through a Disrotatory mode of ring opening reaction, to give the Hexa-2,4-Diene. The particular stereochemistry is that, this is the Z isomer, and this is a E isomer, in the Buta-Hexadiene, that is produced. This can be explained, by the Disrotatory twisting motion of the Carbon-Nitrogen bond, in this particular case, resulting in the formation of the Hexa-2,4-Diene, as the product.

If the opposite isomer is taken, namely the Cis Dimethyl derivative is taken, you are talking about [6,2,5]-Dimethyl Cyclic Diazene of this kind, resulting in the formation of again, a Hexa-2,4-Diene. Wherein, both the double bonds are E isomer, in this particular instance. This can also

be explained, by the Disrotatory mode of ring opening, elimination of the Nitrogen, under the Chelotropic condition, in a concerted manner, resulting in the formation of this product.

Remember, this reaction involves a 6-Electron elimination of the Nitrogen, involves a 6-Electron process, which is shown by the arrows, that are shown here, the 3-Arrows. The 2-Sigma bonds, which are the Carbon-Nitrogen Sigma bonds. And, this PI bond is involved, during the course of this Chelotropic elimination of nitrogen.

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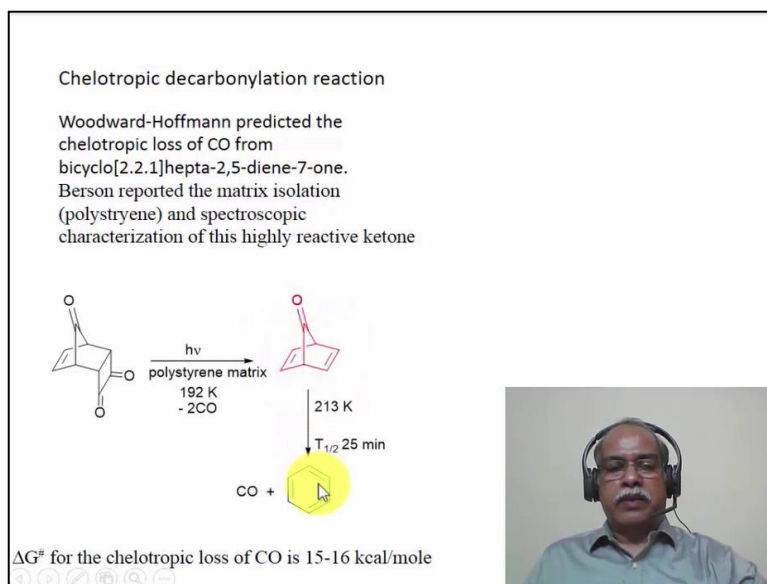
Now, Chelotropic elimination of Nitrogen has been shown, to be highly stereo selective, with this example also. Here, a Cyclic Hydrazine is taken. The Hydrazine is oxidized, either by Manganese dioxide, or by Mercuric oxide, resulting in the formation of the corresponding Cyclic Diazene. This Cyclic Diazene is unstable. Under the reaction conditions, it spontaneously eliminates Nitrogen. The Nitrogen-Nitrogen bond is the driving force, for all of this reaction, resulting in the formation of an Ortho Quinodimethide, in a stereoselective manner.

With, this stereochemistry being E, and this stereochemistry being Z, in the process of the Disrotatory ring opening elimination of this Nitrogen molecule, to give this particular product. In the absence of any trapping agent, this undergoes a conrotatory 4-Electron electrocyclic ring closure reaction, to give the Cis Diphenyl derivative of the Benzcyclobutene, in about 81 % yield. So, overall the first step is an oxidation step, to form the Cyclic Diazene, by the oxidation of the Hydrazine, to the corresponding Cyclic Diazene.

The second step, is the Chelotropic step. Elimination of Nitrogen, by breaking up the Carbon-Nitrogen bonds. These two Carbon-Nitrogen bonds are broken, resulting in the formation of

another reactive intermediate. This reactive intermediate again, this is a Pericyclic step, involving a conrotatory ring closure of the Exocyclic Diene of this kind, to produce the Benzenoid derivative, in this particular case. Benzocyclobutene derivative with the Cis Diphenyl derivative, is what is formed, in this reaction.

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Now, even before the experiments were performed, Woodward-Hoffmann predicted the Chelotropic loss of Carbon monoxide, from [2,2,1]-Bicyclic Heptadienone systems. A [2,2,1]-Hepta-2,5-Diene-7-One is this particular molecule, that is shown in the red color. This is not a stable molecule. Berson, first reported the characterization of this particular molecule, using spectroscopy, in a frozen matrix of Polystyrene, for example.

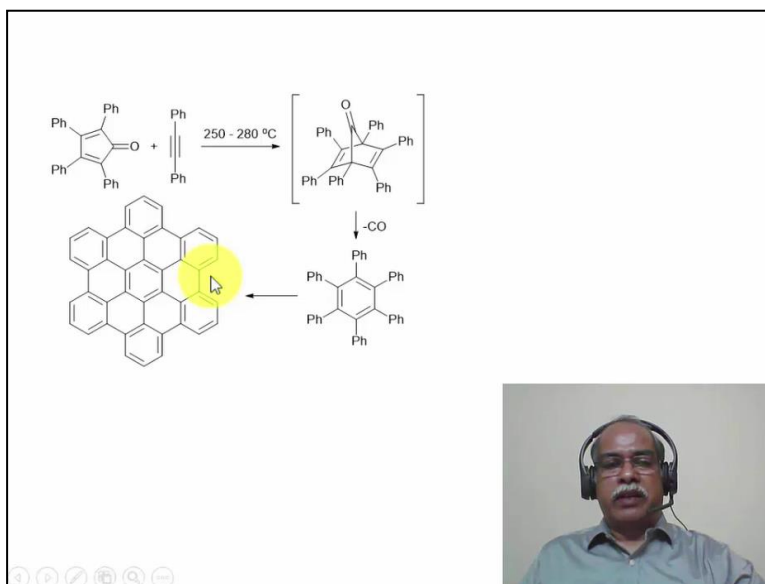
The spectroscopic characterization of this molecule is feasible, by the photolysis of this Tri-Ketone, which is a reasonably stable molecule. Under the photolytic condition, the Carbonyl N-PI-star excitation takes place, resulting in the formation of the loss of 2-Molecules of Carbon monoxide from this molecule, resulting in the formation of the desired Bicyclic Hepta-2,5-Diene-7-One, which is this molecule. This is taking place at 192° Kelvins, at this low temperature, in a polystyrene matrix.

The reason, it is done in the styrene matrix is, to make sure that, this is not reacting with itself, or with another of the molecule of the same species, for example. So, the high-dilution condition, and low temperature, is achievable using the polystyrene matrix. Once, this is characterized spectroscopically, it is warmed to 213 Kelvin, which results in the spontaneous decomposition of the elimination of Carbon monoxide from this molecule, to give Benzene.

The Delta G activation, free energy of activation, for the Chelotropic loss of Carbon monoxide, is about 15 to 16 kilocalories per mole for this molecule, to undergo this decomposition. This molecule is highly strained, because of the angle strain, that is associated with this particular bridged Carbonyl derivatives. There is one reason, it is very reactive.

The second reason is that, the loss of Carbon monoxide in a Chelotropic manner, concerted manner, results in the formation of a stable Aromatic compound like Benzene, in this particular case, for example. So, those are the two driving forces, which makes this molecule, a very reactive molecule. The Chelotropic elimination of Carbon dioxide is happening, around 213 Kelvin temperature, in this particular case.

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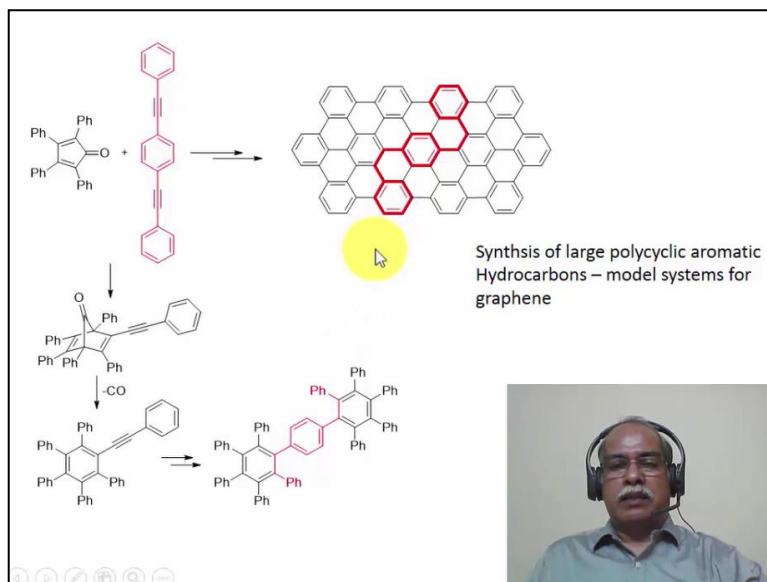
This particular reaction, has been put to use, in the organic synthesis. And, here is an example of synthesis of Hexabenzocoronene. This is Coronene molecule, synthesized by the Diels-Alder reaction, between the Tetraphenyl Cyclopentadienone, and Diphenyl Acetylene. This reaction takes place, at very high temperature, typically in refluxing Diphenyl Ether, is what is used as a solvent, for this reaction.

The 4+2 Cycloaddition product gives, these Hexaphenyl substituted [2,2,1]-Bicyclic Hepta-2,5-7-One is the product, that is formed. This as usual, like the previous example, this example. This is also unstable, under the reaction condition. It spontaneously eliminates Carbon monoxide, to give Hexaphenylbenzene. Hexaphenylbenzene is a precursor.

This can be oxidatively cyclized, in the presence of Ferric Chloride, Copper triflates, kind of oxidizing agent, to give the Coronene derivative, which is this particular Benzocoronene

derivative, which is shown in this particular structure, that is seen. This kind of molecules are interesting, because they are essentially a small segment of the Graphene molecule. Graphene is nothing but a, single sheet of graphite. So, this essentially represents as a model system, for studying Graphene.

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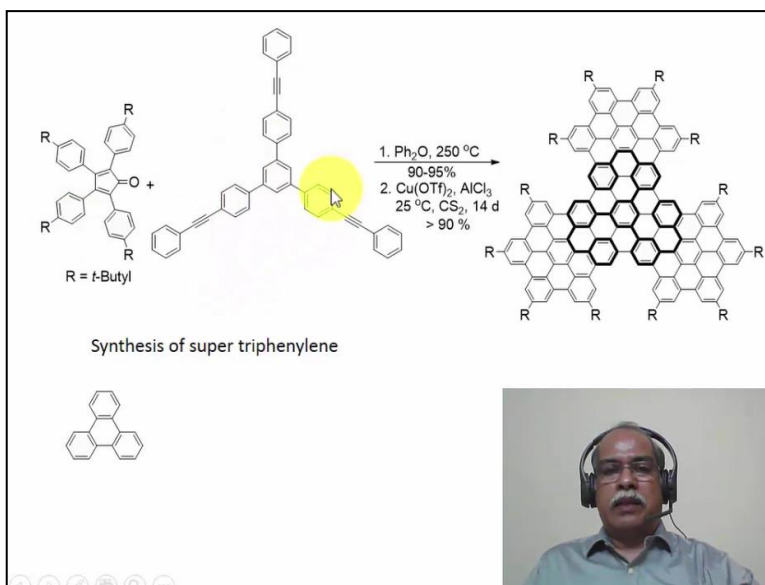
Larger systems are also known. One has to use the imagination, to identify the target molecule, and the corresponding precursors, to reach the target molecule. In this particular case, the Tetracyclone, which is Tetraphenyl Cyclopentadienone, is used as the Diene. And, this Diacetylene, is used as the Dienophile. A double Diels-Alder reaction, once with this triple-bond, another one with this triple-bond, results in the formation of a product, which is shown here.

The initial Diels-Alder reaction, with this triple-bond for example, results in the formation of the [2,2,1]-Bicyclic Ketones, which spontaneously undergoes the Carbon monoxide elimination, to give the Pentaphenyl substituted derivative, which still has a triple-bond. This triple-bond again, undergoes the same reaction, with the Tetracyclone molecule, resulting in the formation of the corresponding bridged bicyclic intermediate, which is not shown in this scheme, which undergoes spontaneously Carbon monoxide elimination, to give this particular product.

This product can be oxidatively cyclized, to this particular derivative, which is a synthesis of large polycyclic Aromatic Hydrocarbon model systems, for Graphene. This is one such fragment of the model system of Graphene, is what is represented here. Just for the sake of convenience, the Dienophile molecule, which is shaded in the red color, is clear that, originally this was a triple-bond, and this was also a triple-bond.

The black portion, essentially comes from the Tetracyclone unit, whereas the red portion, comes from the Diacetylene molecule, that is shown here. So, one has to perform an oxidative cyclisation, of all the Phenyl Ortho positions, to reach this particular molecule, from this particular molecule, which is a precursor. That scheme is not shown here. But, oxidative cyclization's are effectively carried out, using oxidants like Ferric chloride, Copper triflate, Copper (II) triflate, and so on.

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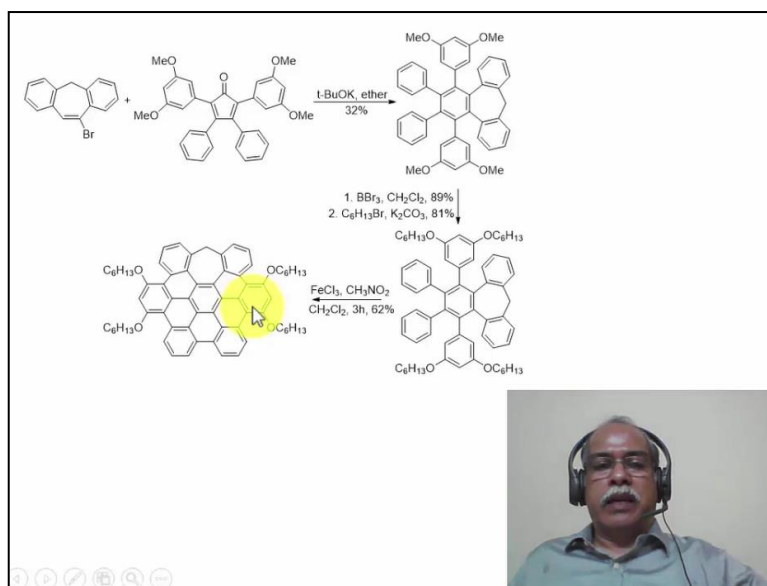


Here is another exotic example, of the same kind of chemistry. Tetracyclone, reacts with this Triacetylene molecule. And, the Triacetylene molecule, essentially produces the necessary Terphenyl substituted derivative, of the corresponding precursors to this particular molecule, which is oxidatively cyclized using Copper triflate, and Aluminium chloride, in Carbon Disulfide, which is non-nucleophilic solvent. In about 90% yield, this molecule is produced.

The Tertiary Butyl groups are, essentially incorporated. Because, this molecule is highly insoluble, in all the organic solvents, because of the fact that, this is a flat molecule. So, PI staking, results in poor solubility. Whereas, when you put Tertiary Butyl group in the periphery of the molecule, the solubility of this particular molecule, increases.

So, solution phase characterization of this molecule becomes easier, by means of doing the Tertiary Butyl substitution, in the starting material itself. This is known as the Super Triphenylene, for the simple reason that, this looks like a Triphenylene molecule, which is shown here, in the bottom. This is Triphenylene. This is essentially a Super Triphenylene, is what is represented by this particular structure.

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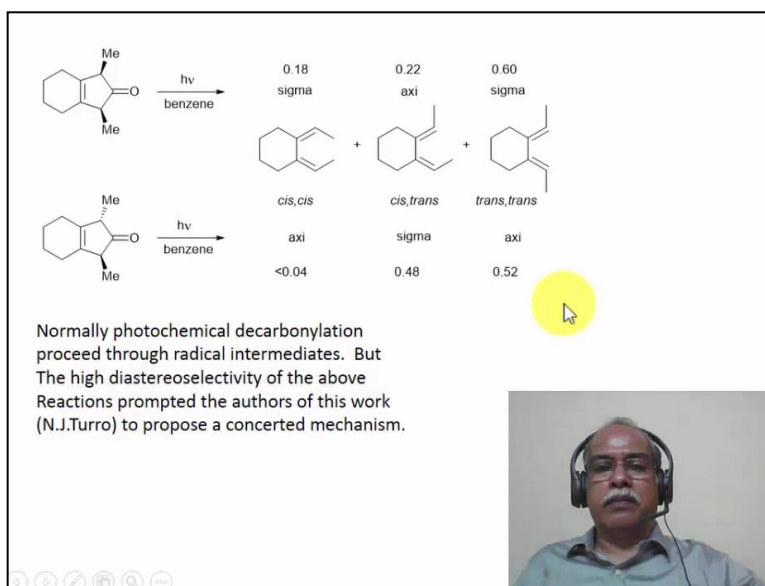
Here is another example of the same Cycloaddition reaction. In this particular instance, a 7-Membered ring is incorporated in the structure, to make the structure non-planar, with respect to the PI framework, that you have here. This is accomplished, by taking this particular Bromo compound. This Bromo compound, under the basic conditions of Tertiary Butoxide in Ether, undergoes Beta elimination process, to give the corresponding Cyclic Acetylene, which is a highly reactive molecule.

The Cyclic Acetylene, undergoes the Diels-Alder reaction, with the Tetracyclone, which is substituted with methoxy groups, to give this molecule. This is now de-methylated, using BBR<sub>3</sub>, in Dichloro Methane, in a yield of about 89%, followed by the free hydroxy, or the free phenolic groups, are now alkylated using Hexyl Bromine, normal Hexyl Bromide, and Potassium Carbonate, to give the corresponding Hexyl derivative. The Hexyl derivative is made essentially to make it more soluble, which is more soluble, compared to the methoxy derivative, in this particular instance.

Subsequence cyclisation, using oxidative cyclisation, using Ferric chloride, in Nitro Methane kind of a solvent, results in the formation of the fully conjugated system like this, with the CH<sub>2</sub>, which results in the 7-Membered ring, in this particular case. So, this is a non-planar version, of the Graphene kind of a molecule, where the conjugation is broken, in this particular place, because of the saturated Carbon, that is mentioned here.



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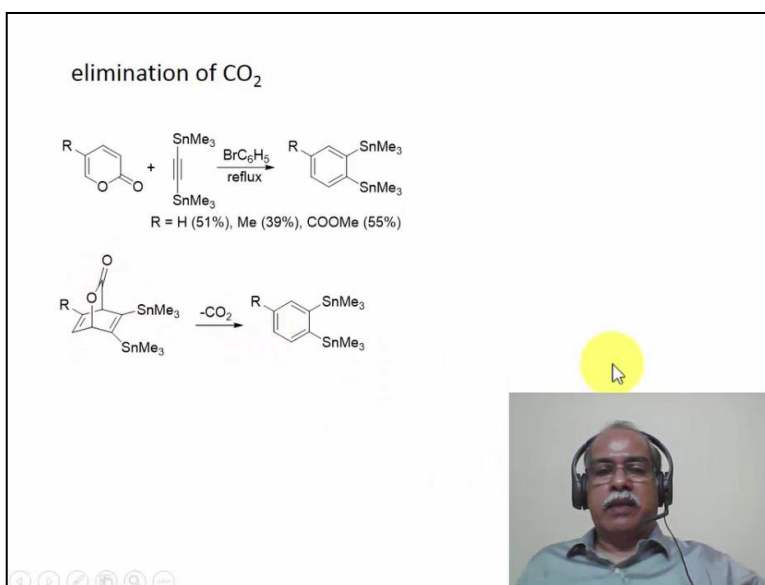
Now, first example of a Photochemical Decarbonylation, that is proceeding through a Pericyclic pathway, is shown by N J Turro. Normally, Photochemical Decarbonylation reactions proceed, through diradical intermediate. In other words, this will undergo an Alpha fragmentation, resulting in the formation of a radical species, here. Subsequent, Alpha fragmentation here also results in the formation of a radical species, resulting in a diradical kind of a species.

However, the high Diastereoselectivity, that is observed in the case of this two Diastereomeric Ketone, prompted the authors, to propose a concerted mechanism. The Axe symmetric, and Sigma symmetric, essentially means, this is a Linear kind of a Chelotropic reaction happening, and this is a Non-Linear kind of a Chelotropic reaction, that is happening. The Axe Sigma symmetric, and Axe symmetric, are another nomenclature, for the Linear Chelotropic, and the Non-Linear Chelotropic, respectively.

And, this product are essentially arising from the Linear, and Non-Linear Chelotropic reaction, of the elimination of Carbon monoxide, from this molecule. The numbers, that are given, are essentially the mole fraction of the product, that is formed. When the Cis isomer is fertilized, is predominantly gives the Trans-Trans isomer, as the major product.

Whereas, when the Trans isomer is fertilized, the Trans Dimethyl isomer is fertilized, it essentially gives the Cis Trans, as well as the Trans-Trans isomer, in almost equal amount. Whereas, the Cis-Cis product is formed, in a very small amount. So, the Diastereo selectivity, that is observed in the Diastereomeric career related starting material Ketones, is the reason, the Pericyclic pathway is proposed, for this kind of a Decarbonylation reaction.

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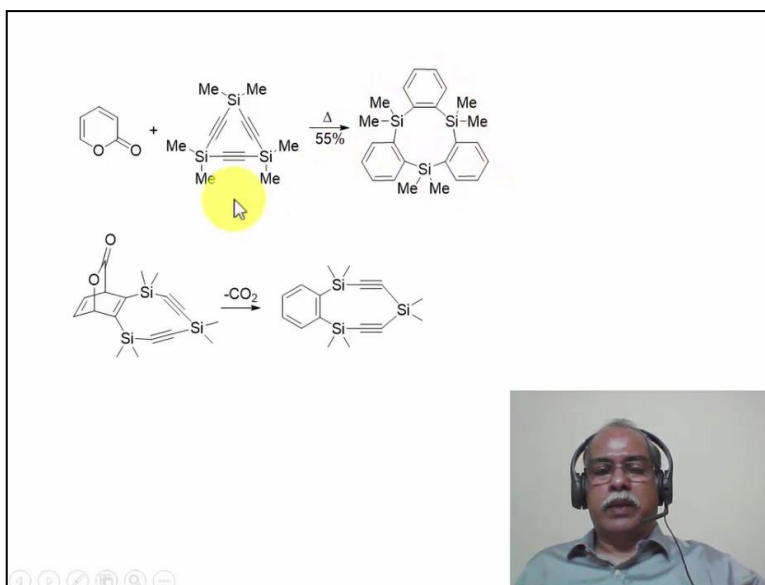
Now, as long as we are considering Carbon monoxide elimination, let us consider some reactions of Carbon dioxide elimination also. Carbon dioxide elimination happens, whenever you have a bridged Carbon dioxide kind of a precursor, that is being formed, in a diels alder reaction. The Diels-Alder reaction of Alpha-Pyrone, is a good example. When Alpha-Pyrone is reacted, with the Bis Trimethyl Tin Acetylene derivative, in reflexing Bromobenzene, as a solvent, for example.

The corresponding Ortho substituted Ditin derivative is formed, in good diels. Now, this reaction proceeds through a 4+2 Cycloaddition reaction, resulting in the formation of a constrained ring system, where the CO<sub>2</sub> is forming the bridging, in this [2,2,2]-Bicyclic system, for example. Elimination of Carbon dioxide, from this molecule, is not a Chelotropic process.

For the simple reason that, Carbon dioxide is not attached from the carbons. One of the Carbons, to 2-Carbons of this, or this Oxygen is not attached to the 2-Carbon. Remember, in the case of Chelotropic reaction, by definition, a Hetero Atom, or a Carbon Atom, has to be simultaneously attached to 2-Atoms. In other words, if this Carbon was attached to both this Carbons here, simultaneously by a Carbon-Carbon bond here, that would have been a Chelotropic process.

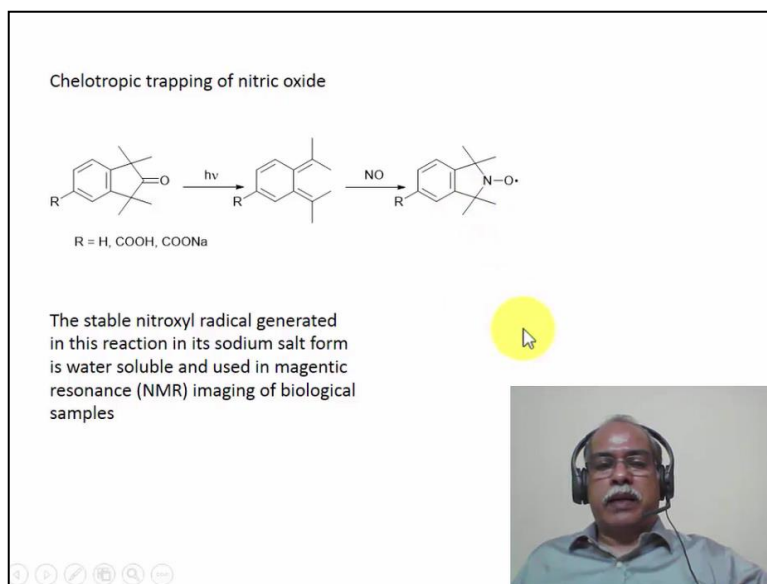
But, here the Carbon is attached to this Carbon, and another Oxygen is attached to this Carbon. So, this is not a Chelotropic process. Nevertheless, it is a very interesting and concerted mechanism, which it could be a Retro Cycloaddition kind of a reaction, is what is performed here. One can consider this as a Retro Diels-Alder reaction, with the release of Carbon dioxide, to produce the Ortho disubstituted derivative.

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This is synthetically useful, for making fancy molecules of this kind, by taking this Triacetylene, and performing the reaction with Alpha-Pyrone. The initially formed product, is this particular product, which undergoes the elimination of the Carbon dioxide, to give the Aromatic substrate. The two other Acetylenes, that are present in the system, also undergoes the similar reaction, by annullating the Aromatic ring, instead of the three Acetylene, the rings are annulated, by the Aromatic Benzene units, are annulated in this particular reaction.

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So, this is a very interesting reaction. I thought, I should mention here. Now, Chelotropic trapping of Nitric oxide, is known here. Although, the stereo chemical aspects of this, is not revealed here, this is an extremely useful reaction, for the following reason. Let us first consider, the reaction. The Ketone, which is an Alpha, Alpha, Alpha, Prime Alpha, Prime Tetra Substituted Ketone, is fertilized. This could undergo a Chelotropic Carbon monoxide elimination, or a stepwise Carbon monoxide elimination, we do not know.



Under Photochemical conditions, Carbon monoxide is eliminated, to give this intermediate. What is important is, the Chelotropic trapping of NO, is what is important here. Nitric oxide is chelotropically trapped by this Diene, to produce the corresponding Nitroxy radical. The Nitroxy radical is a quite stable compound. And, that is essentially, in the form of a Sodium salt. The R group is a Carboxylic acid group.

The Sodium salt of this Nitroxy radical is water-soluble. And, hence it is used in Magnetic Resonance Imaging of Biological samples. So, overall, this is probably, either a Chelotropic, or a Non-Chelotropic process, we are not serious about this particular reaction, here. However, the intermediate that is produced, is Chelotropically trapped by a NO<sub>2</sub>, to give the Nitroxy radical. The Nitroxy radical is useful in Biological systems, for the Imaging purpose using NMR as a technique.

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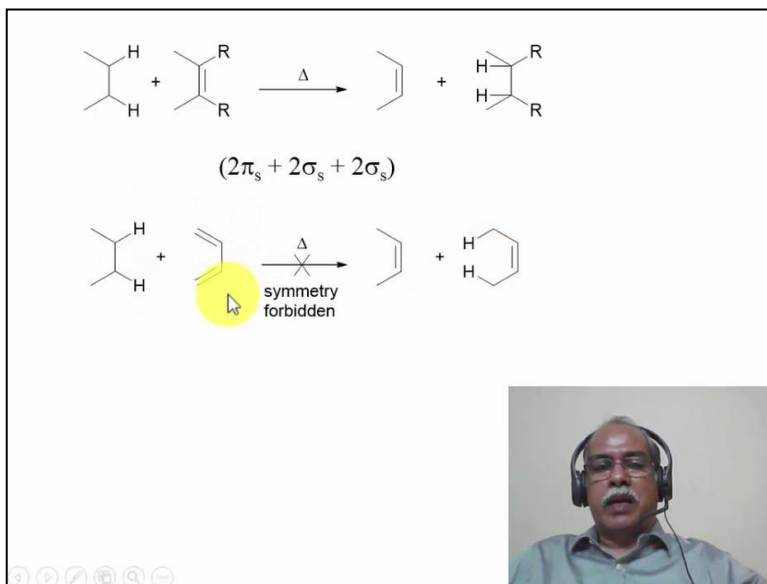
Group transfer reactions:

Transfer of groups and atoms in a concerted manner from one molecule to another molecule involving a cyclic transition states.

Now, let us go to the Group Transfer reaction. The definition of a Group Transfer reaction is that, the transfer of a Groups or Atoms, in a concerted manner, from one molecule to another molecule, involving a cyclic transition state, is what is known as the Group Transfer reaction.

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The simplest of Group Transfer reaction, is Hydrogen transfer reaction. If you take this example, the 2-Hydrogens, that are mentioned here, it is transferred onto this Olefin, in a manner, that it is concerted, so that, the 2-Hydrogens end up Cis with respect to each other, in the product also. So, overall, if you look at the process, this 2PI electrons are involved in this, because it gets

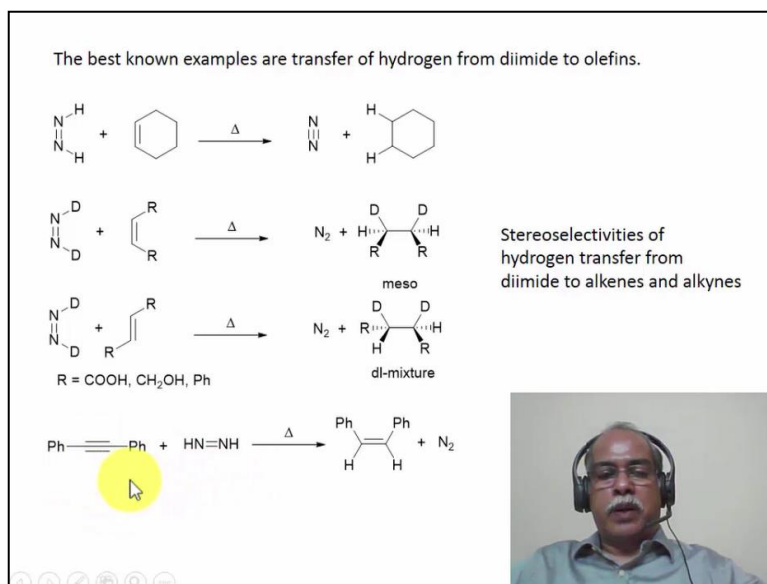
saturated. The 2-Sigma Electrons of the CH bond, the other 2-Sigma Electrons of the CH bonds, are also involved.

So, it is overall a 6-Electron system. It is a 6-Membered cyclic transition state, involving a 6-Electron system, under thermal condition. So, it should be allowed under Suprafacial-Suprafacial-Suprafacial manner. Overall, the selection rule, or the Woodward-Hoffmann rule, for the Hydrogen transfer, from the Cis Hydrogen transfer, from the starting material to this Olefin, is considered to be a 2PI Suprafacial-2-Sigma Suprafacial and 2-Sigma Suprafacial.

The 2PI, comes from this Olefin, here. This 2-Sigma, comes here from one of the CH bond. The other 2-Sigma, comes from the other CH bond. So, it is a cyclic transition, one can imagine, involving a 6-Electron process. Now, the [1,4] addition of the Hydrogen, which would involve, now an 8-Electron process. Two of the 2-Sigma + 2-Sigma, 4-Electrons, and this Butadiene 4PI electrons, overall 8-Electron system, under Suprafacial condition, is a symmetry forbidden reaction.

So, the [1,4] transfer of the 2-Hydrogens, to result in the formation of this particular Olefin, is a forbidden process, and it does not take place. At the best, what will happen in this reaction is, the Hydrogen will be transferred to the double bond, producing a Vinyl derivative. The Vinyl derivative can further undergo Hydrogen transfer reaction, to produce an Alkene, but not the Olefin, by the addition of the, [1,4] addition of the 2-Hydrogens, across this position. And, this position, with the reorganization of the PI system, to give this Olefin, that does not take place.

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The best-known examples of the Hydrogen transfer reactions, are from Diimide, which is this molecule. This Diimide can be produced, by the oxidation of Hydrazine, using Mercuric oxide kind of an oxidizing agent. MNO<sub>2</sub>, or Mercuric oxide kind of an oxidizing agent, oxidizes the Hydrazine to the Diimide. The Diimide, the 2-Hydrogens can be transferred onto the Olefin, resulting in the formation of the release of Nitrogen, and the corresponding hydrogenated product.

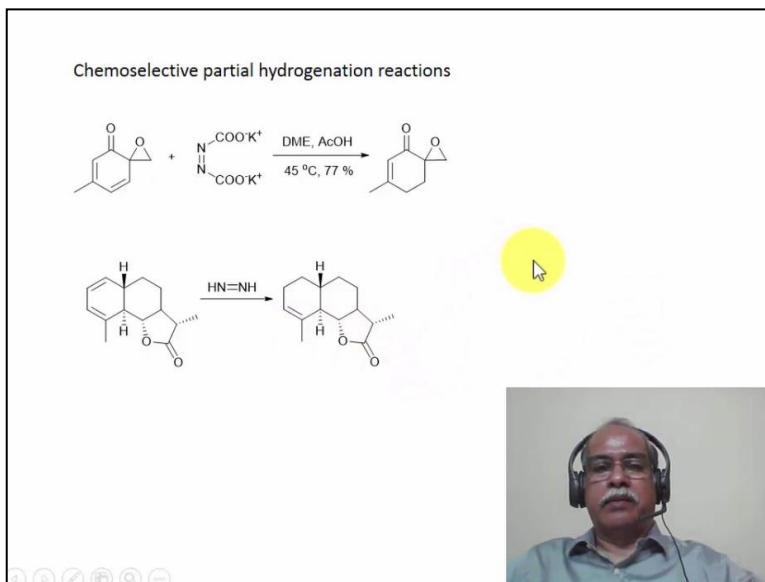
This is an example, of a Transfer Hydrogenation reaction. Now, the reaction is highly stereo selective, in the sense that, if you take a Cis Olefin, and if you label the Diimide using Deuterium, then the corresponding Erythro isomer, or the Meso isomer, is what is formed, during the course of the Hydrogen transfer. Because, a Deuterium transfer takes place, in a Syn manner. So, the Syn addition of the 2-Deuterium, is producing the corresponding Meso isomer, or the Erythro isomer, from the Cis compound.

The same reaction, when is carried out with the Trans compound of course, the Dual mixture, or the Threo isomer, is what is formed, with the release of Nitrogen. So, the reactions are highly stereoselective in nature, as far as this reaction proceed. So, they are probably proceeding through a 6-Membered cyclic transition state, in a concerted manner. The 2-Deuterium Atoms are transferred from the Diimide, which is Deutero-substituted Diimide, to give either the Meso, or a Diel, in a Stereoselective manner.

When the reaction is carried out with the Acetylene, then it produces the Cis Olefin. The Cis stilbene, is what is produced, from Diphenyl Acetylene, again indicating a Cis addition of the 2-

Hydrogens, probably taking place through a concerted Hydrogen transfer reaction, involving 6-Electron.

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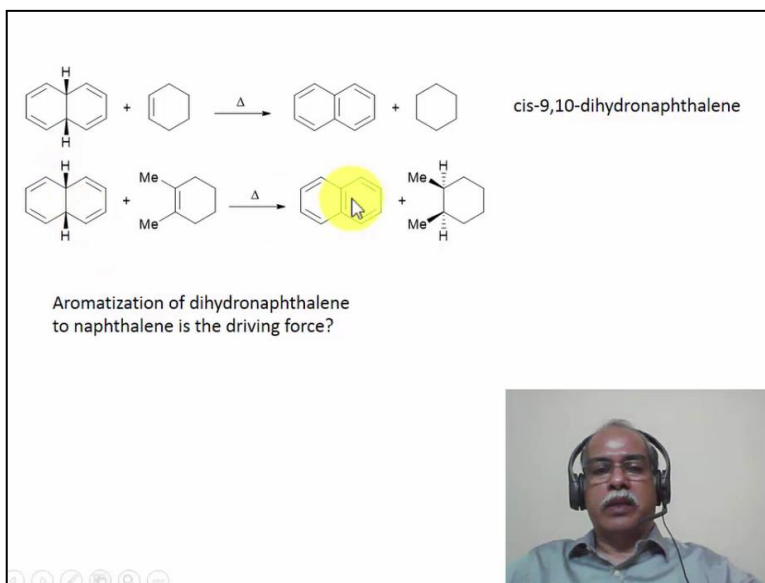
The reaction is also, highly Diastereoselective. If you look at this molecule, this is nothing but, Diaceto Dicarboxylic acid as the Potassium salt, is what is given here. When it is heated with acidic acid, Diimide is produced, by decarboxylation reaction. So, the Diimide is in-situ, generated in this particular unit. When the Diimide, that is in-situ generated, hydrogenates the electron rich double bond, and does not hydrogenate the electron poor double bond.

So, offering a chemo selective partial hydrogenation, of the remote double bond here, compare to this double bond here. Please bear in mind, the producible functional group, such as the Carbonyl functional group, and the Epoxy functional groups, are impacted this molecule. So, the reaction is not only chemo selective, with respect to the difference in the reactivity, between an electron deficient double bond, on an electron rich double bond.

It also distinguishes, the Carbonyl functional group, and the Epoxy functional group, which are left intact, during the course of the reaction. This particular Lactone, has a tri-substituted double bond, and a di-substituted double bond. Di-substituted double bond, is chemo selectively reduced by the Diimide molecule, to give this particular Olefin. Again, the Lactone ring is left intact, during the course of this particular reaction.

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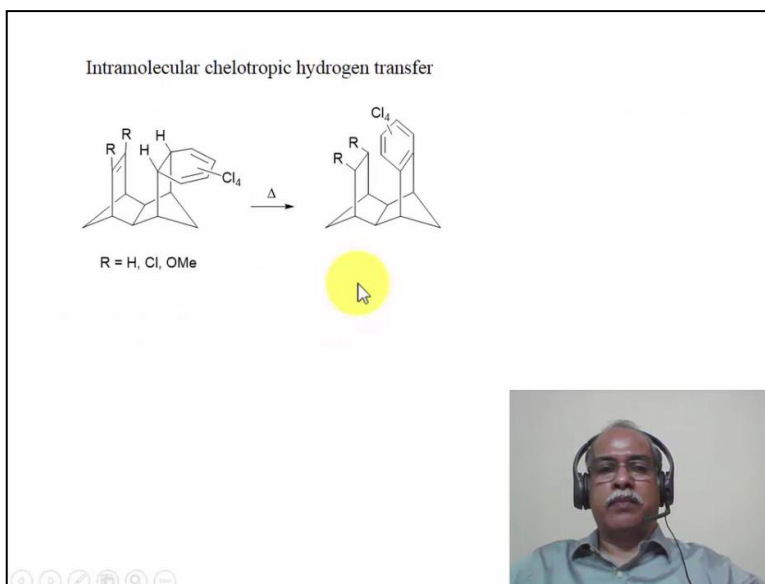




Here is an example of again, a Transfer Hydrogenation reaction involving, Cis [9,10]-Dihydronaphthalene. The 2-Hydrogens can be transferred onto the Cyclohexene, to produce Cyclohexene and Naphthalene. The driving force, for these two types of reaction, that are shown is probably, the Aromaticity that is gained, by the transfer of Hydrogen on to the Olefin, which is now reduced to the saturated compound.

So, it could very well be that, the Dihydro Aromatic derivative is aromatized, during the course of the reaction. And, that might be the driving force, for this particular reaction.

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Lastly, let us consider one exotic example of a Hydrogen transfer, which is an Intramolecular Hydrogen transfer reaction. This molecule is essentially, a very constrained geometry. You can see here that, this double bond, and this 2-Hydrogens, are essentially poking onto each other, that we are in close proximity, with respect to each other. And, that is a driving force for the transfer of this 2-Hydrogens, on to the double bond, resulting in the saturation of the double bond.

And, the Aromaticity of this particular ring, is gained in this process. This is a reaction, that was investigated during the course of the synthesis of Pagodane kind of a molecule, by Prince Bagh, the group of Prince Bagh in Germany. And, this is an example of an Intramolecular Chelotropic Hydrogen transfer reaction, that we have seen here. So, what we have seen in this module, is the elimination of Nitrogen from Cyclic Diazene molecules, which is considered to be highly Stereoselective in nature.

We saw examples of that. Chelotropic elimination of Carbon monoxide, something, that we see. And, look at the Carbon monoxide elimination, from the point of view of organic synthesis, particularly synthesizing large Polycyclic Aromatic Hydrocarbon molecules, which are model systems for the Graphene type of a molecule. Finally, we also looked at the Decarbonylation reaction, of the bridged [2,2,1]-Bicyclic Ketone, Carbon monoxide elimination, is what we saw. Finally, we saw, some Non-Chelotropic Carbon dioxide elimination also, because of the curiosity with respect to elimination of neutral molecule, that we have. I hope, you enjoyed this module. Thank you very much, for your kind attention.