Introductory Organic Chemistry - II
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Indian Institute of Science Education and Research, Pune
Module 06
Lecture 44
Tutorial - 6

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Problem Set 6

Solutions

So, in this problem set, we are going to look at some of the questions related to reactions of enolates with specifically with respect to aldol reactions and so on.

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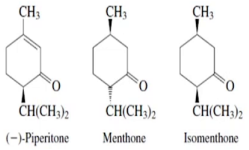
Consider the following compounds



Suggest reasonable explanations for each of the following observations:
(a) Optically active piperitone (α_D -32°) is converted to racemic piperitone on standing in a solution of sodium ethoxide in ethanol.
(b) Menthone is converted to a mixture of menthone and isomenthone on treatment with 90% sulfuric acid.

So, the first question here is about these three compounds, which are natural products. And so, the first question here is that piperitone is an optically active compound with a rotation of

minus 32 degrees. But when you put those in solution of sodium ethoxide in ethanol, you know it converts to a racemic piperitone, which is basically, you know, the rotation is 0 degrees. So, what is a proposed reasonable explanation for this? And then we will look at the second question after that.

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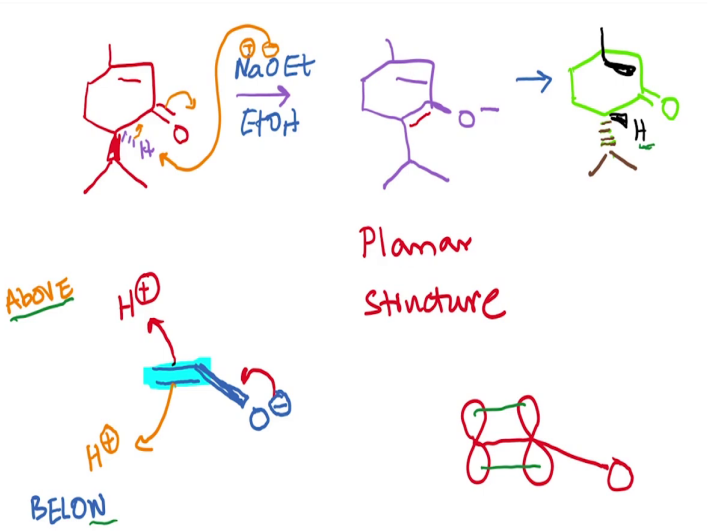

So, what we know is that, it is in the presence of base, this hydrogen which is alpha to this carbonyl can certainly get enolized. And so, when you have that situation where it undergoes enolization, it forms an enolate. So, when you look at an enolate, the enolate is a planar compound. So, you have two faces. So, if I have to draw the p orbitals, it is going to look like this, with the oxygen over here.

And so it is essentially a planar compound, just like an olefin. And so, you have two phases. So, if it reacts from above, the hydrogen would be above, that is this product. And if it reacts from below, that is the proton is added from the phase below, then you are going to get back the starting material. So, as you can see these two compounds are enantiomers of each other. And so, you will have the racemization that happens.

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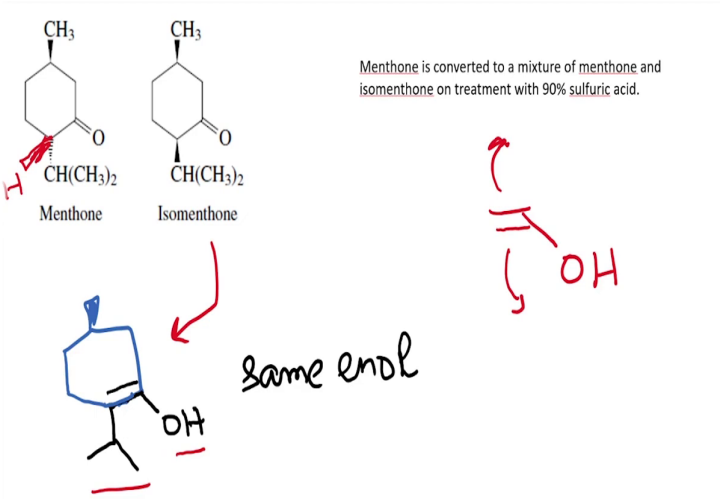

And the second part B, instead of base, you are doing the same thing in 90 percent sulphuric acid. So, now the question here is that you get a mixture of menthone and isomenthone with 90 percent sulfuric acid. So, if you look here, again menthone and isomenthone, let us say this hydrogen over here, which is facing up has to be deprotonated to form an enol. And so, if it forms the enol over here, then you get this enol.

And if you notice, Isomenthone will also give you the same enol. So, now, if you have a common intermediate, for this reaction, then the enol is anyway considerably less stable, or generally speaking, less stable compared to the keto form. So, when you have in the keto form, you are going to get back a mixture of menthone and isomenthone or you will have the same concept here, which is the enol is also a planar molecule. And it has two faces by which it can react.

And so, you are going to get a mixture of menthone and isomenthone when you treat it with 90 percent sulfuric acid. So, this is one of the problems that for example, in drug discovery people face, because when you have drugs with ketones in it, and if you have a chiral center, which is right next to it, then during metabolism, you will have relatively acidic, relatively basic compartments, and these compounds can racemize.

So, the problem with racemization is that you will have a 50 percent mixture or a 60:40 mixture or whatever. And each of those metabolites have to be individually sort of assessed, because there have been several cases of enantiomers having completely different biological

activities, and sometimes opposite activity that is that one of them is useful and the other one that can actually cause long term problems.

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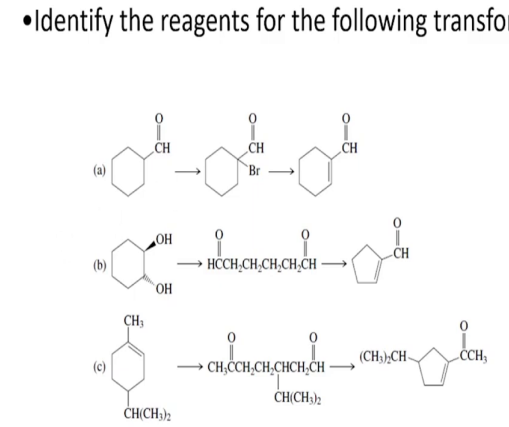

Now, let us move on to the next question, which is identify the reagents for the following transformations. So, the first question over here is if you take the ketone, and if you see here, there is alpha-Bromo ketone that is produced, which subsequently there seems to be a loss of HBr to give you the aldehyde. We will look at the other two shortly.

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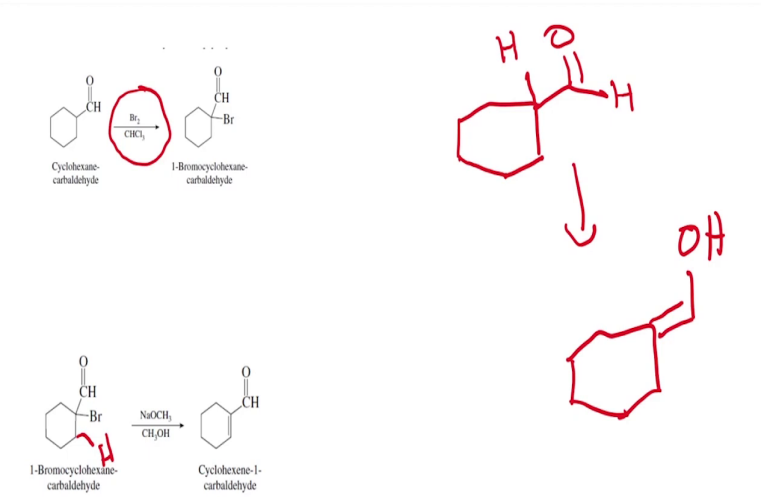

So, the first part of this question, what we can think about is the alpha hydrogen that is present. And we all know bromination can be achieved through Br_2 and chloroform. So, what

you can propose is that you have a enol and then this enol undergoes bromination to give you the product, so that is pretty straightforward. So, the reagents here are Br_2 and chloroform.

Now, coming to the next step, what we need to do here is an elimination reaction. And we could propose the standard elimination reaction conditions, which is sodium methoxide and methanol, or potassium tertiary-butoxide, and tertiary-butanol, and so on. And so, the mechanism here would be an E2 type mechanism, where you have the alpha hydrogen being pulled out by the base, and in the same step, the loss of bromide. Now, let us move to the next question.

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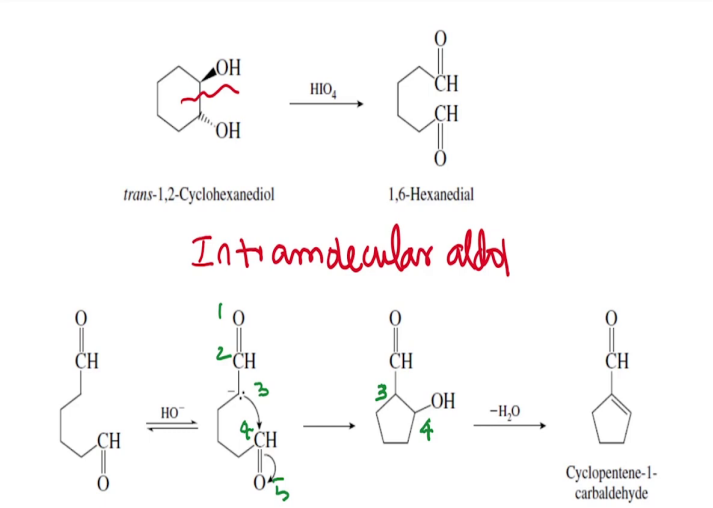

So, here, the question here is that you have a diol, and this diol we have studied in the past that this diol can be converted to the corresponding carbonyl compound by using Periodic acid, which can be HIO_4. And so, this is similar to the reaction that we might have studied earlier, which is called the ozonolysis, where you have a double bond, and you can convert it to the corresponding aldehyde or ketone and so on.

So, this is the standard conditions for breaking this the di-hydroxyl compound. And once you get this di-aldehyde, the next question here, or the next thing here is to convert it to the corresponding cyclopentene carbaldehyde. And as you can see here, this is a substrate for what is known as an intramolecular aldol, we have not looked at examples of this during the lecture, which is why it is important that all of you try and solve these problems and also pay attention during this problem-solving session.

So, here is an example of an intramolecular aldol reaction. So, you can sort of propose that this will form the enolate. So, this is actually the carbanion is shown here for just for convenience, but it would exist in the enolate form. And then there is an attack, an intermolecular attack that can happen. So, let me just number these carbons so that it is easy for us to follow.

So, this is 1, 2, 3, 4, 5. So, this is the numbering that we followed in the previous examples that we have used for aldol. And so, there is a bond between carbon 3, and carbon 4. And just to understand the size of the ring, so you will have 1, 2, 3, 4, 5, so you form a 5 membered ring over here, and then the rest of the chemistry is the same.

And now under basic conditions, you can understand that there would be an enolate that is produced, and the enolate could rearrange and give you this product. So, the reagents that you need to use here is basically base, you can use sodium hydroxide or potassium hydroxide, and that gives you the intramolecular aldol reaction to give you the product. Now, let us look at the next question.

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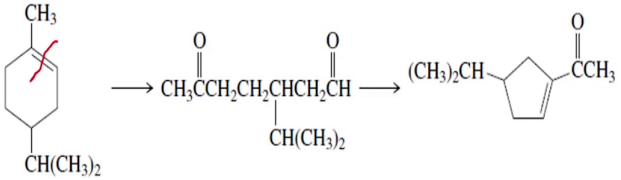

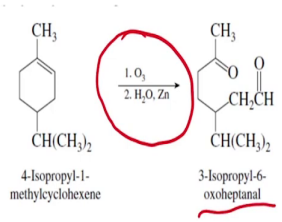

So, here you have cyclohexyl ring compound, and this ring compound is opened up, as you can see here. And so, like we discussed earlier, the one of those reactions that you guys have studied in the past is ozonolysis. And so, you can use ozonolysis to break this carbon-carbon double bond. And that gives you di-carbonyl compound as shown here.

So, what you guys need to do is, in order to be systematic in this question, you need to number the carbons and make sure that you follow the numbering very carefully. So, please go ahead and do that. And you will see that this is the product that is formed out of ozonolysis followed by reaction with the zinc.

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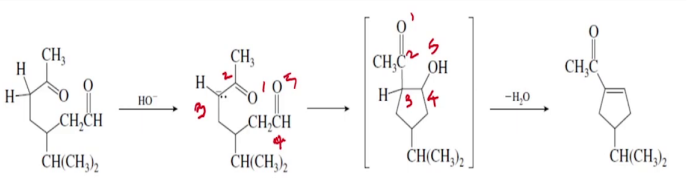

The next step, again, we are looking at an example of an intramolecular aldol reaction. And so, here you will get the formation of this enolate as shown here. So, you are going to have this keto form, which is going to get deprotonated and you will form an enolate as shown in this diagram. And the rest of the molecule remains the same. So, now, if you stick to the earlier numbering system, so this is 1, 2, 3, this is carbon 4, this is oxygen number 5.

So, thinking about the same 1, 2, 3, and this is 4, and this is 5. So, this correlates very well with our intramolecular reaction. And so, you have here the production of the corresponding 5-membered ring system upon elimination, you have a cyclopentene type of system that you form after elimination. So, this is a fairly straightforward question here.

So, later on in the problem set, we will look at now competition reactions and so on, for intramolecular aldol. And we can discuss certain examples of that here. So, therefore, the reagents that we would use for carrying out this transformation is, they are basically, number 1 would be ozone, and zinc, and the second one would be base. So, let us move on to the next problem.

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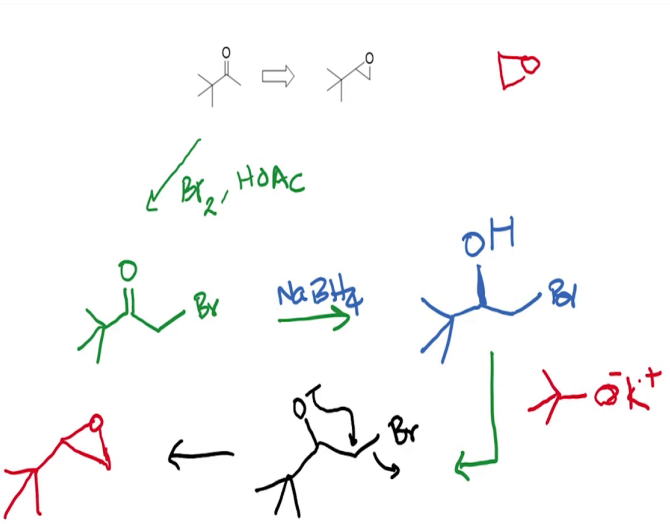

So, here, the question here is to convert this ketone here to the corresponding epoxide. I think many of you would know, the epoxide as a functional group, which is the 3-membered ring with an oxygen in it, and, of course, two carbons. So, the way we would approach this problem is that you can suggest that there could be a bromination of some sort, that could happen as the first step. And so, that bromination is something that we have already studied.

And now, in order to form the epoxide, the epoxide can be formed by an intramolecular ring closure, as long as this compound, this carbonyl is actually going to be an alcohol. And the way we would generate the alcohol is to do a reduction, we have already looked at this, sodium borohydride reduction is going to give you the alcohol. And maybe you would need some sort of a base, I am suggesting tertiary-butoxide, but you could have other bases as well.

And this is going to give you the O minus and the O minus can do an intramolecular attack and kick out and give you this epoxide. But the important point to note here is that the reaction, or that this alpha position that is alpha to this carbonyl has to be functionalized.

And so, one of the steps that we really need to propose is to have a functionalisation of this. And an important reaction for functionalisation of carbons, alpha to the carbonyl is bromination or halogenation. So, this is some reaction that we would come across again and again, during this course and perhaps in the next coming semesters.

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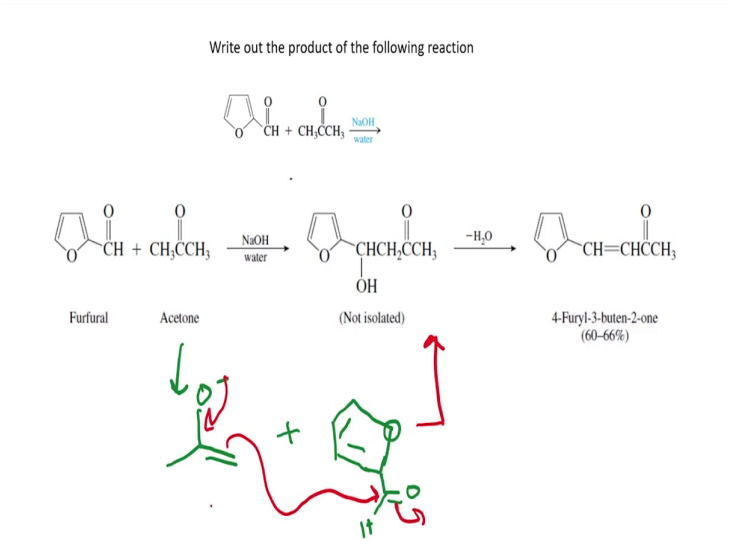

So, the next question is, what is the product, write out the product of the following reaction. So, here you have an aldehyde of furfural it is called and you have acetone in the presence of sodium hydroxide and water. So, normally aldehydes are more reactive when it comes to enolization, but this alpha position of this enolate there is no enolizable hydrogen in this aldehyde. So, therefore furfural would act as the electrophile in this case.

So, the enolate that is formed is the familiar enolate of acetone, you add sodium hydroxide and water you produce this enolate and then the enolate can react with the aldehyde over here and give you this product. And this product under these conditions is not isolated and straightaway loses water. Again, you know the mechanism for this, it is going to form the enolate and the enolate is going to undergo E1cB reaction to give you the eliminated product. And so, this product is isolated in about 60 to 66 percent yield.

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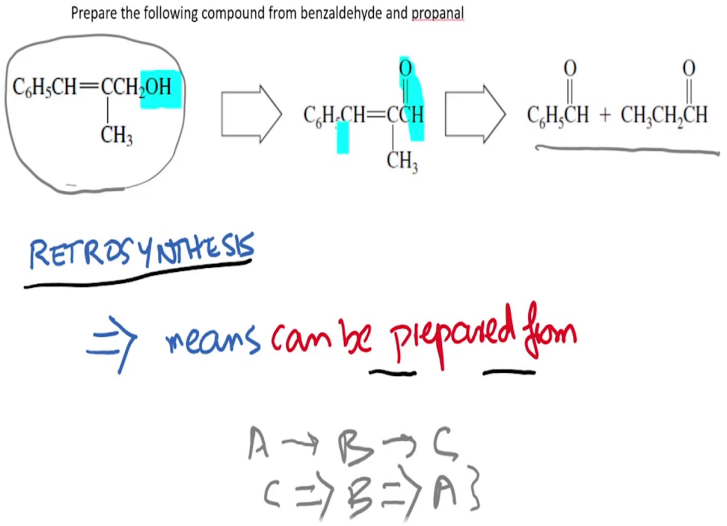

Now, to the next question. You need to prepare the following compound from benzaldehyde and propanal. So, here I would like to introduce the concept of retrosynthesis. So, retrosynthesis is basically, you know what you would propose the kind of transformations that you would want to propose, you write it in the reverse order. So, for example if a step, if in a particular synthesis scheme, you would write A goes to B goes to C.

And now, in the retrosynthesis, you would write it as C is derived from B, which in turn can be derived from A. So, this helps us sort of construct the synthesis in a linear fashion. And also, it helps us break down the synthesis into smaller parts. And this retrosynthesis is widely used in construction of complex natural products or complex compounds. And you will study this in the coming semesters. So, this retrosynthesis arrow means that it can be prepared from. So, the question here is that, so what they have asked you is to prepare this compound, and they have given you the starting materials.

So, what we can suggest is that this combination of benzaldehyde and propanal can give you, we already know that it can potentially undergo the aldol reaction to give you this kind of product which undergoes elimination to give you the olefin. And now, if I look at it, this is the aldehyde and this is the alcohol. And so, therefore, we can suggest that the alcohol can be produced from the aldehyde by a reduction reaction. Since the retrosynthesis is fairly straightforward, let us now move to the actual synthesis of the compound.

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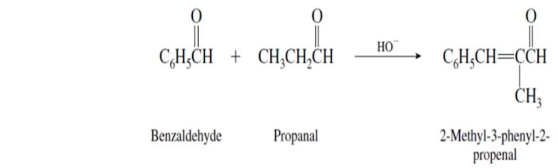

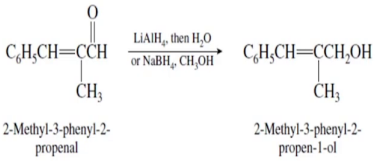

So, what we can do is, when we add benzaldehyde and propanal, this does not have any enolizable hydrogen. So, this is the enolate that is going to be produced, and then the enolate reacts with benzaldehyde to give you this product. And now to get from this aldehyde to the alcohol, you can propose Lithium Aluminium Hydride, of course, you need to work it up with water after that, and give you the product. Or you can also propose Sodium Borohydride and methanol to give you this product.

So, the important point to note here is that you need to be systematic, and you need to understand what are the bonds that are being broken? And what are the bonds that are potentially being formed.

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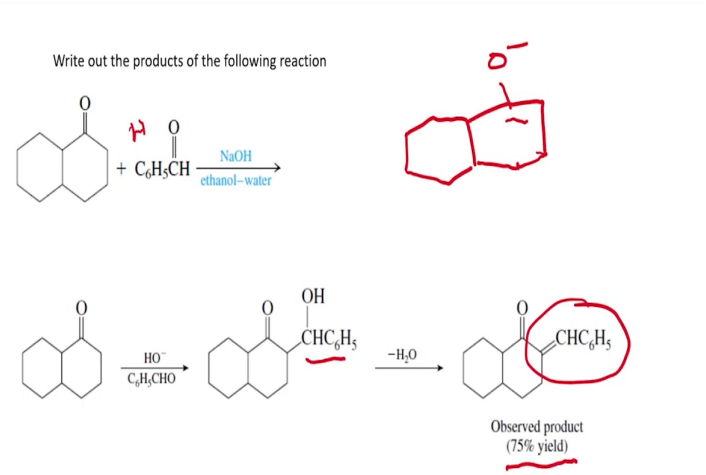

Now, to the next question. So, here, the question here is it you have been given this bicyclic compound, we have cyclohexane-cyclohexane with the ketone, and it is again reacted with benzaldehyde in the presence of sodium hydroxide. So, the first step is fairly straightforward but at the same time, there is a choice. So, let us look at choice number 1.

So, let us say this, hydrogen is enolized or this hydrogen is abstracted. So, it gives you the following enolate. And now, this enolate reacts with benzaldehyde, you get this product here, and this product, through E1cB reaction, you form the enolate here kicks out and gives you this product. And this product can be isolated in 75 percent yield. Now, you have a choice. And let us look at the choice in the next page.

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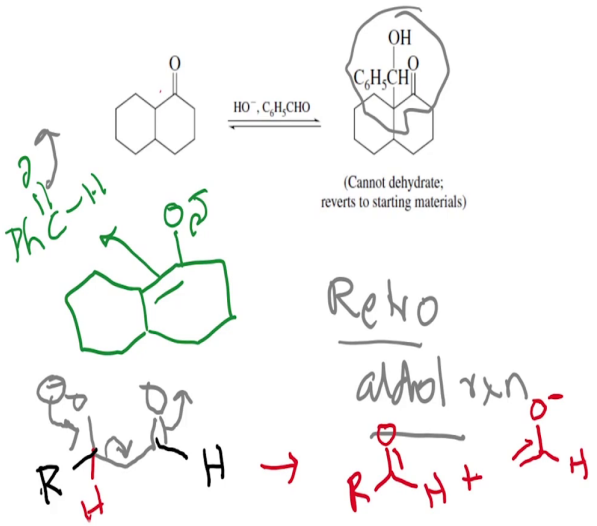

So, the choice here is that you can also produce the enolate on this side. So, you have the formation of this kind of an enolate, and if this enolate reacts with benzaldehyde, which can very easily occur. And so, what is going to happen is that it is going to give you this product over here. Now, the difference between this product and the other product is that there is no possibility of elimination over here.

And so, if there is no possibility of elimination, we have not looked at it in the class, but aldol reactions are actually reversible in some sense. And so, you can have what is known as a Retro Aldol reaction, that means that it is the reverse of the Aldol reaction. And the retro aldol reaction gives you back the aldehyde or ketone that we start with. So, very simple arrow pushing mechanism for the retro aldol reaction is as follows. So, you have O minus, so this is going to go here, this is going to move here, and this is going to move here.

So, these are the two fragments, so it gives you C double bond O, say this is R, and let us say this is hydrogen, so you would get the following products, which would be RCHO and we will get the enolate as the product. So, now, once this enolate is produced gives you back this enolate, the enolate can again pick up a proton and form the ketone, and the ketone will give you back the other enolate which is going to eventually give you the product that we want.

So, one of the important things to be noted here is that the experimental data shows that the product that is formed is about 70 to 75 percent yield. And therefore, this is something that we need to start getting familiar with is that we should start deriving explanations for the product that is formed. So, although this is indeed a distinct possibility, this appears to be a minor reaction.

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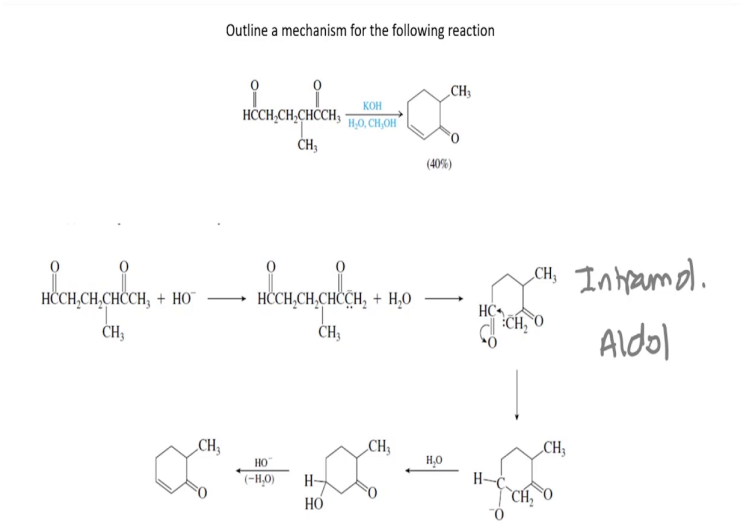

Now, moving on to the next question. So, this is again an example of an intramolecular aldol reaction. So, which we have looked at previously. So, again, it is very important for all of you to keep the numbering of carbons in the right order. So, we need to look at the product and figure out from the product for example, here you have a double bond and you have a ketone. So, this α, β-unsaturated system must have originated from the aldol reaction.

And so, from that, you need to start numbering things and figuring out how to do this. So, the proposed mechanism is as follows. You have the formation of the enolate as shown here, just for convenience sake, we are writing it as the carbanion. And now, this carbanion the enolate attacks this aldehyde over here and there is an example of an intramolecular aldol reaction.

And so, it forms a 6-membered ring. And subsequently this O minus can get protonated and form OH, and then loss of hydroxide ion is going to give you the product. So, I will let you guys work out the mechanism of this reaction, or figure out the exact numbering.

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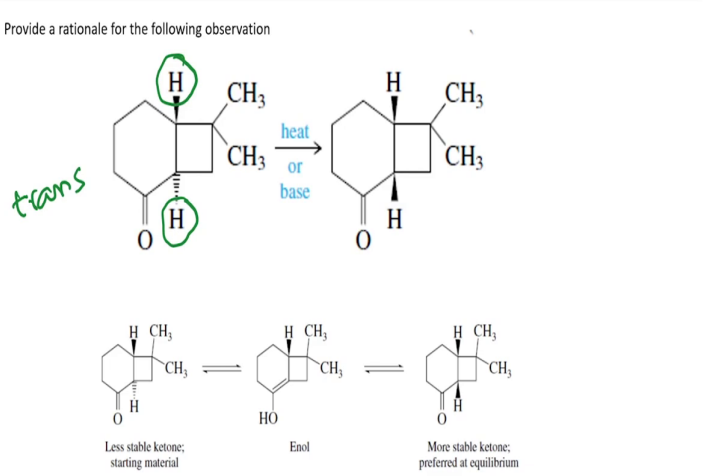

The next question, the question here is that provide a rationale for the following observation. So, when you take this bicyclic system, and if you either heat it, or you add a base, you get the other product, which is the cis product. So, the first thing to note here is that this is a trans ring fusion. And so, the trans ring fusion are very-very unstable. And so, therefore, they do not need a whole lot of impetus to form the cis ring fusion.

So, even if you just heat it up, what happens is that you can generate the enol and the enol can then pick up a proton or tautomerize to give you the ketone. And in doing so, it forms the more stable ketone in this equilibrium. So, this is what is sort of suggested that you will form the more stable ketone in the equilibrium. Now, you can do the same thing after adding a base.

So, the base will pick up this proton, and it will form the enolate. And the enolate, when it is going to give you back the ketone is going to give you the more stable ketone as shown here. So, enolization of the ketone yields an intermediate in which the stereochemical integrity of the intermediate is lost, this is exactly the way we would have suggested it. However, the difference here is that when you have a choice between an extremely stable and an unstable stereoisomer, it eventually leads to the formation of the most stable stereoisomer at equilibrium.

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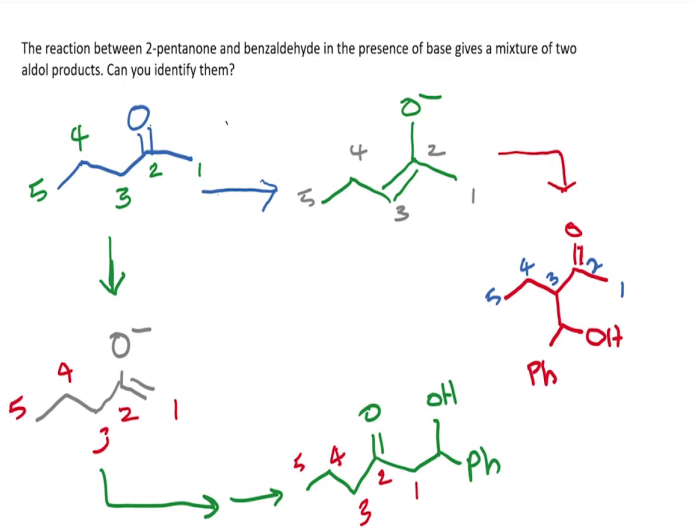

Now, let us move on to the next question. So, the reaction of 2-pentanone and benzaldehyde in the presence of a base gives a mixture of two aldol products, can you identify them? So let us look at the first possibility here. So, you have the enolate formation, this is 2-pentanone as shown here. And you can just number it, this is 2-pentanone. So, you can generate the enolate here between carbon 2 and carbon 3.

And once this reacts with benzaldehyde, you get this product as shown here. And so, this is one possibility. The other possibility is that you generate the enolate, as shown here, and this enolate can react and give you this product. So, this is the mixture of the two aldol products that are potentially produced during the reaction of 2-pentanone and benzaldehyde. And I might add here that the difference in stability between these two products is not that large, and therefore, it is not unreasonable to propose that it is a mixture of these two products.

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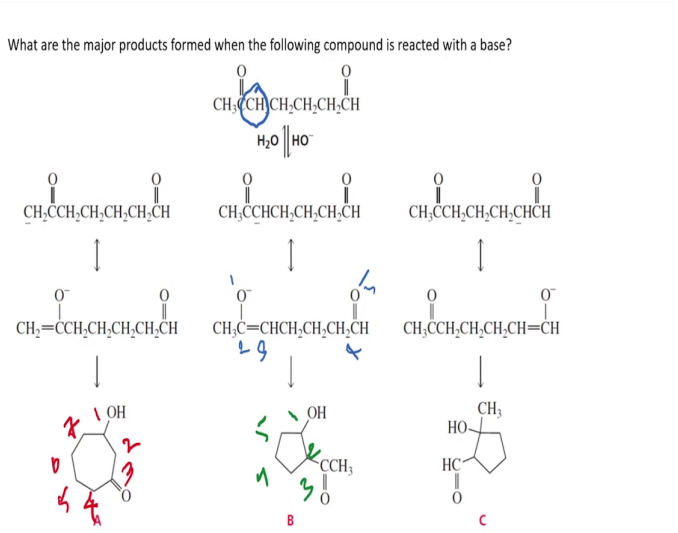

Now, to the next question, what are the major products which are formed when the following compound is reacted with the base. So, if you recall, in the previous examples, we looked at intramolecular aldol reaction. And so, here, we are just looking at this in a little bit of more detail. So, let us look at this systematically. The first reaction, first thing that we do here is to, from left to right, we can go and so the enolate that is going to be produced first would be this.

And this is the enolate that can be produced, and then the enolate can undergo an intramolecular aldol reaction, and it gives you this ring. So, let me just number this ring, so that we can understand this, this is 1, 2, 3, 4, 5, 6, 7. So, we have studied in the past that formation of larger ring systems beyond 6 are not highly favoured it can occur, but it is not highly favoured. So, nevertheless, let us keep that in mind. We move on to the next enolate which can be produced over here.

So, once this enolate is produced, this can also undergo an intramolecular reaction. And it gives you the following products if I number it 1, 2, 3, and this is 4 and 5. So, this is the reaction that occurs and it gives me a ring of the following size, let me just give you a sort of different numbering in terms of the ring numbering, this is 1, 2, 3, 4, and 5, and so this is going to give me 5-membered ring which is not unstable.

The next possibility is to form the enolate next to the aldehyde. And the aldehyde enolate can be produced in the following manner and it gives you this product, I will let you work out the

details of this mechanism by yourself. Now, to some comments on these products that are formed.

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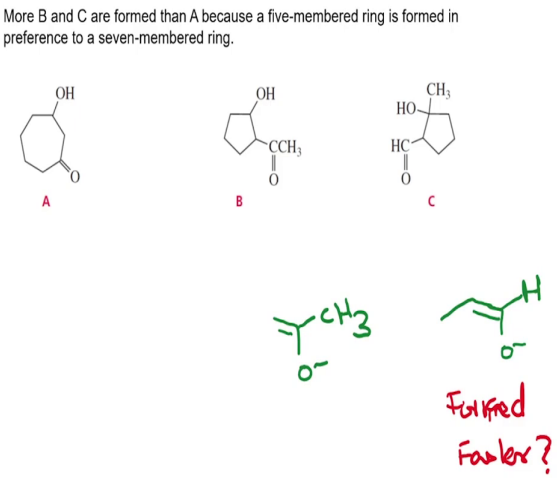

Now, because A is a 7-membered ring, we can suggest that B and C would be formed in preference to A and now between B and C what we can suggest is that the enolate from the aldehyde might be formed faster, because the aldehydes, generally speaking are more reactive when compared to ketones or the enolization is much faster.

So, if you have a choice, we may suggest that the enolate form from the aldehyde would be much more preferred. So, this is how we would solve this problem. But I cannot really comment on whether B is the major product, or C is the major product at this point. So, what we can say is that B and C maybe together might constitute the major products of this reaction.

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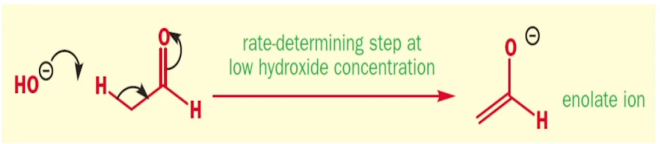
When the aldol reaction is carried out at high concentrations of hydroxide, the rate law is found to be:
Rate = k[aldehyde]^2[Base]
Provide a rationale for this observation



Not only is this step the most important; it is usually the rate-determining step. The rate expression for the aldol reaction at *low concentrations* of hydroxide is found experimentally to be

rate = $k_2[CH_3CHO] \times [HO^-]$

Though this is a proton transfer, which we normally expect to be fast, the proton is being removed from a carbon atom. Proton transfers to and from carbon atoms can be slow.

Let us move on to the next question. So, the reaction or the aldol reaction, when it is carried out at high concentrations of base, the rate law is actually,

$$\text{rate} = k\,[\text{aldehyde}]^2\,[\text{Base}].$$

So, the question here is provide a rationale for this observation. So, let us first look at the reaction under low base concentrations. And so, under these conditions, the enolate is formed.

And the enolate then reacts with the aldehyde and gives you the product. And so, experimentally, we observe that the rate is proportional to the concentration of aldehyde times the concentration of hydroxide ion. And so, this ends up being a first order reaction in the aldehyde as well as the base. So, from this, we suggest that the rate determining step is at low hydroxide concentration is the formation of enolate.

And this is quite reasonable to propose because there is still a difference in the pKa's of the aldehyde and water, so the aldehyde pKa is somewhere between 18, 20 and so on. And pKa of water is about 14-15. So, there is a few orders of magnitude difference in the equilibrium constant. And therefore, there is going to be some sort of challenge in generating the enolate ion.

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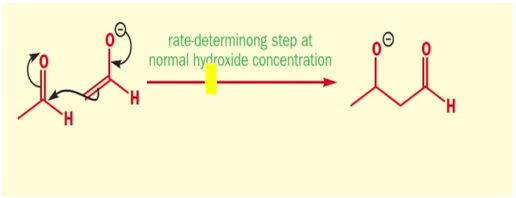

At higher hydroxide ion concentration, the rate expression becomes termolecular (k_3 expresses this) with the aldehyde concentration being squared.

$$\text{rate} = k_3[CH_3CHO]^2 \times [HO^-]$$

The mechanism does not, of course, involve three molecules colliding together. The rate-determining step has changed, and is now the second step.

But under the conditions where you have large or high concentration of the base, what is suggested, is that we have already looked at, it is a square of the aldehyde into the hydroxide ion concentration. So, at higher hydroxide ion concentration, the formation of the enolate is going to be really fast, because you have a very large concentration of the base. So, what it appears is that the next step, which is the reaction of the enolate with the aldehyde, might be the rate determining step.

So, clearly, you cannot have a termolecular reaction, that is we cannot have a situation where three molecules collide. And it becomes a rate determining step, because that is quite unlikely. However, it is more reasonable to propose that this step as shown here is the real determining step. And the formation of this the aldol oxide product, or the O minus product is the rate determining step.

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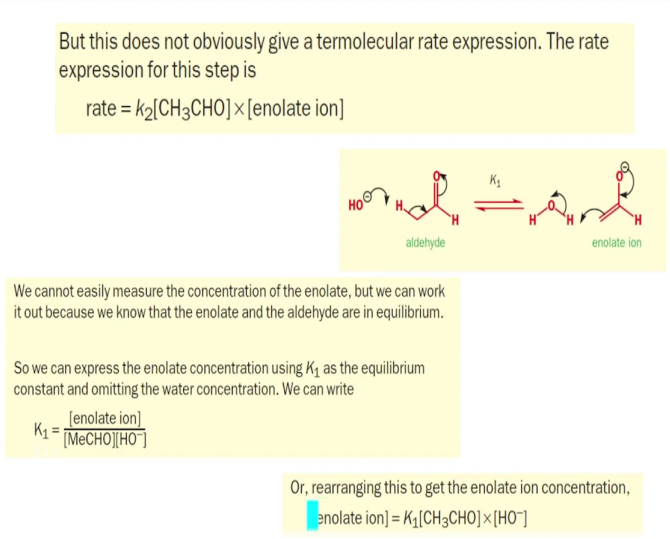

Now, when this kind of situation arises, how do we interpret a termolecular rate expression. So, what we can propose is that the reaction rate constant or the rate determining step depends on the concentration of the aldehyde and the concentration of the enolate. But since the concentration of enolate is we cannot estimate the concentration of the enolate, we now need to derive the concentration of enolate by using the information that we already know.

So, the enolate concentration is basically determined from this equilibrium constant, so where you have hydroxide ion attacking the aldehyde, and it forms water and the enolate. And so, this at high concentrations, would be an equilibrium reaction. And therefore, the equilibrium constant for this can be determined.

And so, the equilibrium constant here is nothing but the concentration of enolate divided by the concentration of the aldehyde times the concentration of hydroxide. So, now what we do is, in order to estimate the enolate concentration, we do a rearrangement. So, the rearrangement is shown here. So, the enolate ion concentration equals K equilibrium constant times aldehyde times hydroxide i.e.

$$[\text{enolate ion}] = K_1 [CH_3CHO [HO^-]$$

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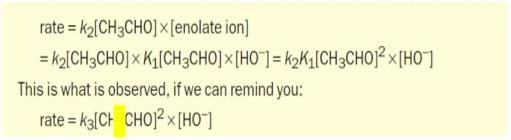

It just turns out that the 'termolecular rate constant' k3 is actually the product of an equilibrium constant K1 and a genuine bimolecular rate constant k2 such that k3 = K1 × k2

Now, if I plugin this reaction this enolate ion into the concentration of enolate ion into the equation, what I see here is that the rate becomes k_2 times concentration of aldehyde multiplied by the concentration of enolate. And the enolate concentration is nothing but K_1 times concentration of aldehyde times hydroxide ion. So, since you have two units, two sort of instances of acetaldehyde concentration, you end up with a square term here and hydroxide ion.

So, this together can merge into the k_3, and this is going to be the rate law. So clearly, the termolecular rate constant is nothing but it is an indication that reaction of an intermediate is actually the rate determining step and it is not very common, but it has been observed in several important examples that you can have a termolecular rate constant that is the rate of the reaction depends on the square of a particular reagent and also to the power of one for the other reagent.

So, here you have the aldehyde square term and hydroxide is to the power of 1. And so, the interpretation from this is that it is the one of the intermediates that is reacting in the rate determining step. And now, in order to derive the concentration of intermediate you may have to have a square term in the rate law.

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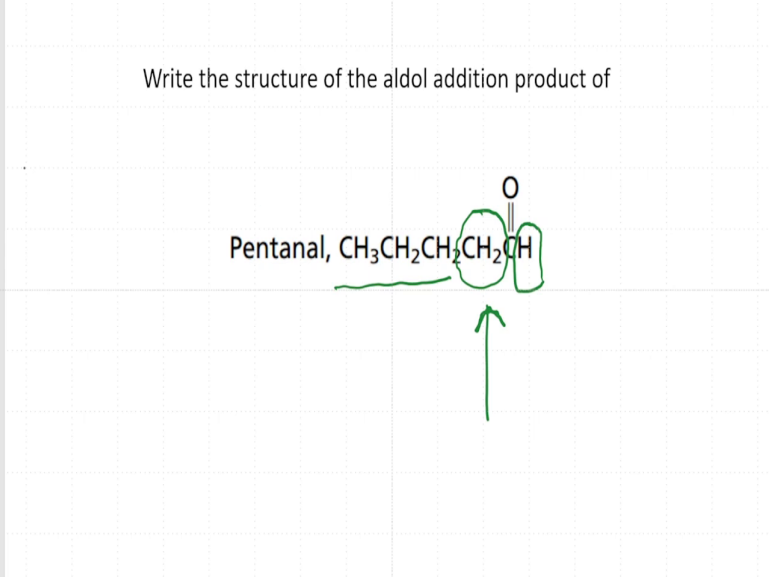

Write the structure of the aldol addition product of Pentanal. So, in this question, we would assume that Pentanal is exposed to some kind of a base such as maybe potassium hydroxide or sodium hydroxide or something like that. And then we need to figure out what the product of the aldol addition is. So, the first thing to look at in such a question is to see where the active methylene group is.

So, here for example, you have a CH_2 which is right adjacent to a carbonyl and you none of the other sort of hydrogens are ionisable. And of course, you have an aldehyde here, so which is also not ionisable. So, therefore, this is the most important CH_2 for us to consider.

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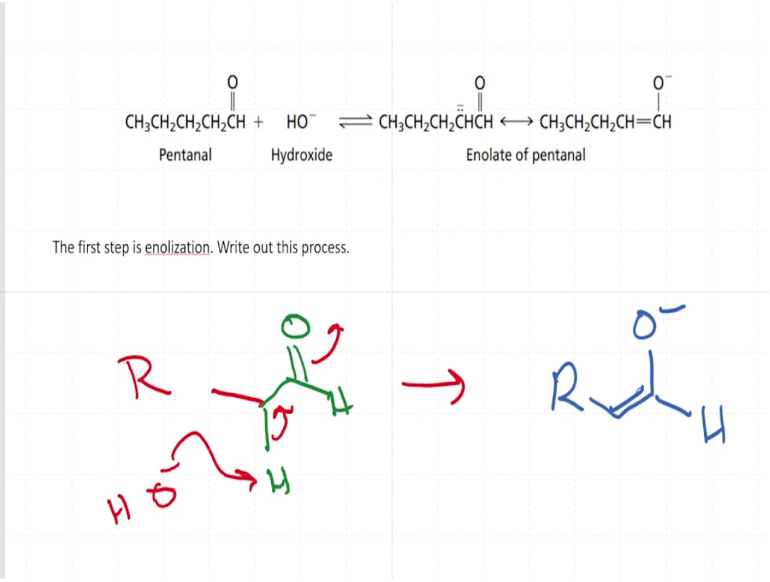

So, if you assume that the CH_2 can undergo enolization in the presence of Pentanal, then the first product that is going to be formed is the enolate. And which of course, exists in the resonance form with the carbanion. And so, that would be the first step of the reaction. So, in order to draw that out, so if I assume that this is the aldehyde and here is a hydrogen.

So, you have R here and you have hydroxide attacking here and producing to give you the enolate. So, this is something that we are fairly familiar with. Now, what we have done in this process is we have generated the nucleophile. And if there is no electrophile, then it is going to form the enolate. But if there is an electrophile, which is Pentanal itself, then the aldol reaction can occur. So, this is a self-aldol reaction.

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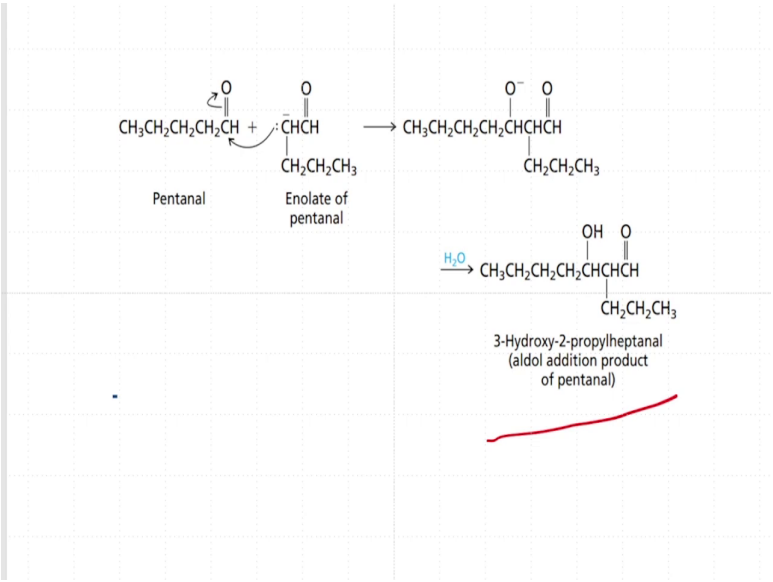

So, you have the enolate of Pentanal can attack the carbonyl of another Pentanal, and then produce this kind of an intermediate. And then this intermediate can then pick up a proton to give you 3-hydroxy-2-propylheptanal. So, this product here is the aldol addition product of Pentanal. But the story does not end here.

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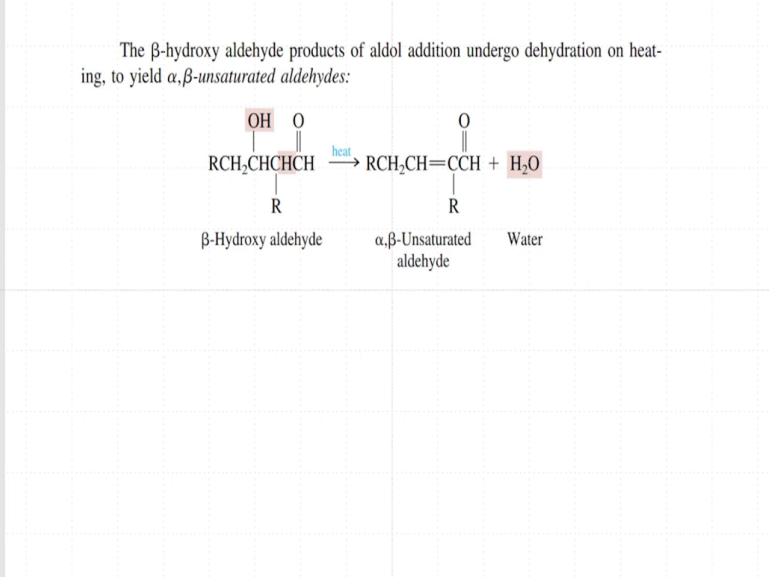

So, what invariably ends up happening is that these β-hydroxy aldehydes have a tendency to lose water and produce the olefin. And so, what is fairly well known and we have discussed this previously as well, is that there is a dehydration reaction, which can give you an α, β-unsaturated aldehyde.

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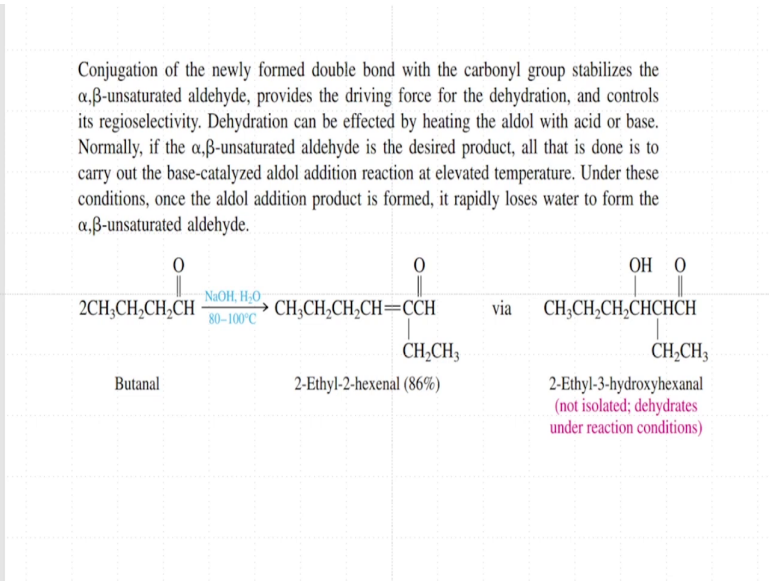

So therefore, what we would expect is that, under experimental conditions, if you take butanal, and let us say you add sodium hydroxide and water and you heat it up to about 80, 100 degrees, then the yield of the α, β-unsaturated aldehyde, or ketone, in this case is quite good, it is about 80-86 percent.

So, if you want to make the actual aldol product, it is a little difficult to make it because the stability that is achieved by conversion of the alcohol to the olefin is fairly good. And already it is in a positive direction. And so, what invariably ends up happening is that the aldol product, once it is formed, it rapidly loses water to form the α, β-unsaturated aldehyde.

So, you would need to really sort of do some special reaction conditions to produce, if you want to make this hydroxy carbonyl compounds. Otherwise, the normally the product that is formed is actually the Ethyl-2-hexenal.