

Video « Ion pair extraction – titration »

Time	Text
00 :09	As you have seen in the previous video, I made the mixture necessary for the titration of the lidocaine hydrochloride by the DOSS solution which is in this Erlenmeyer. Again, you can distinguish two phases, the dichloromethane phase being under the aqueous phase because dichloromethane is denser than water. You can also observe that the mixed indicator gives a green colour to the mixture.
00 :32	There is a stirred bar in this mixture in order to perform a good stirring which is necessary for a good titration in a biphasic media.
00 :46	The DOSS solution is placed in this burette. It will be progressively added to the mixture with the lidocaine hydrochloride.
01 :00	First, let's start the stirring. It needs to be sufficiently strong to make a little emulsion, as you can see here, but not too strong to avoid any splashes. Alright, this speed is suitable, so the DOSS adding can begin.
01 :28	It is always necessary to calculate a theoretical endpoint first. For this titration, I have calculated an equivalent volume around 6 ml, obviously by making the hypothesis that lidocaine hydrochloride is pure. As a remind, the lidocaine hydrochloride is a raw material, not a galenic form.
01 :47	As I am far from the endpoint, I can start by adding the DOSS solution a little bit faster, for example 0.5 ml by 0.5 ml.
02 :07	As you can observe here, the emulsion is stronger, which is completely normal because the DOSS is a surfactant and so, it will help in the formation of this emulsion.
02 :22	The adding keeps going.
02 :32	The expected switch is purple, it means that the indicator will switch from green to purple at the endpoint. At that time, I have added around 3 ml.
02 :44	Now I have added 4 ml, the emulsion is stronger.
02 :50	Here we go, I have almost added 5 ml. So now, as I am close to the endpoint, I will perform a drop by drop adding, which is a little bit tricky.
03 :08	Obviously, you must be very cautious and well observe the mixture. As soon as the mixture colour switch into purple, you must stop the addition.
03 :21	As you can see, the mixture starts to become purple. So now, you need to stop the adding and let the mixing go on. In that way, the DOSS can partition between the two phases or, to be more accurate, the hydrochloride salt linked to the DOSS can go into the dichloromethane phase.
03 :42	And now, you can observe the expected purple colour. Indeed, I have added 6 ml of DOSS solution.
03 :50	So, this is how to perform this titration with the DOSS solution. I am advising you to stop the stirring and to wait a bit in order to see if the purple is persistent. Indeed, because of the emulsion, some interface phenomenon could happen.
04 :06	Here, you can see that the purple colour is intense and stable. So, the titration is over.
04 :13	Now, you just have to make the calculations in order to find if your raw material is conformed to the pharmacopeia and so, to calculate its purity.