Elementary Electrochemistry
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Preparation of Primary Standard and Standardization of NaOH

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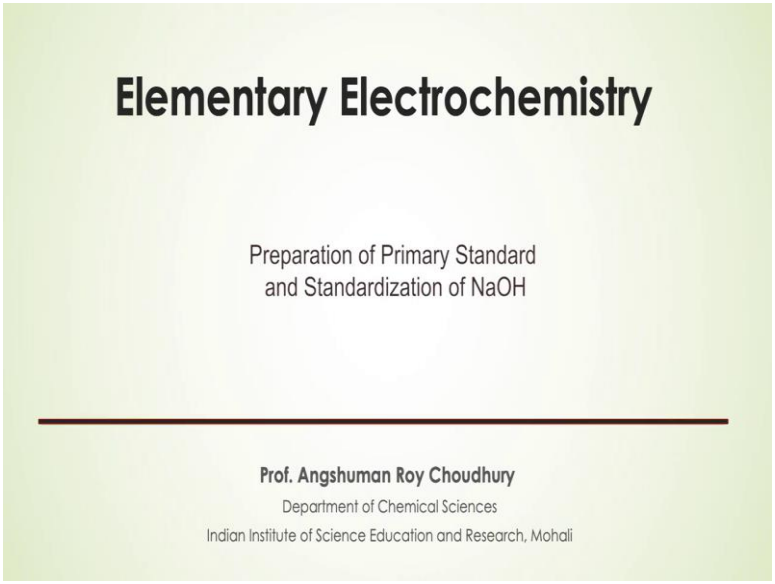

Welcome back to this course Elementary Electrochemistry. As we have discussed during the theory course, we will talk about some of the experiments related to the electrochemistry. And today in our first experiment, we will demonstrate the pH base, the titration that is Potentiometric titration of strong acid versus strong base, and then we will continue with weak acid with a strong base. And then that is hard experiment will be the titration of weak dibasic acid we will use oxalic acid as our dibasic acid and do a titration using NaOH.

So, as all of you probably know, because you have done some laboratory courses in your college to do any quantitative estimation one requests to prepare a primary standard solution. So, in these acid base titrations, we need to use oxalic acid as a primary standard. And I will first show you how one can very accurately prepare a solution of a primary standard. A primary standard is a compound which can be used for a long period of time in solution and the strength of that solution does not change over a period of time and the compound is very stable in solution and does not decompose easily.

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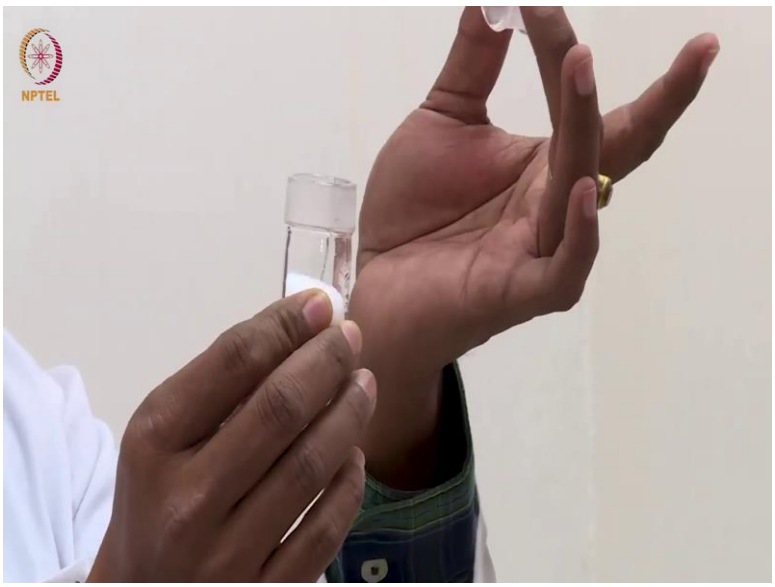

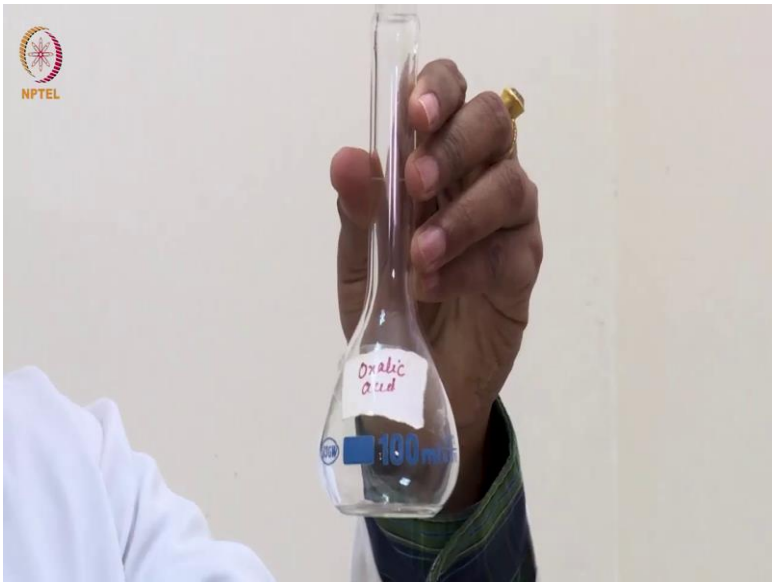
NPTEL
Oxalic
Acid
100 ml


NPTEL


NPTEL

So, for acid base titration, we use normally the oxalic acid dihydrate which is here in this bottle, this is a particular type of bottle we call it as the weighing bottle, you can see that we have the groove here and the lid and this comes in pair. So, when you have the bottle and the lid proper and then you take oxalic acid in this, we will use this weighing bottle to do the weighing.

To prepare a primary standard solution, you will need a volumetric flask which I have labelled as oxalic acid because this will be my primary standard oxalic acid solution. In addition to this glassware, we will use many different glass wares which I will show you one by one, we will use a 10 ml pipette to pipette out the solutions and we will use a pipette pump like this. To pipette out solutions, we will not use our mouth to be pipette out because that is not at all safe for anybody to be pipette out some solutions.

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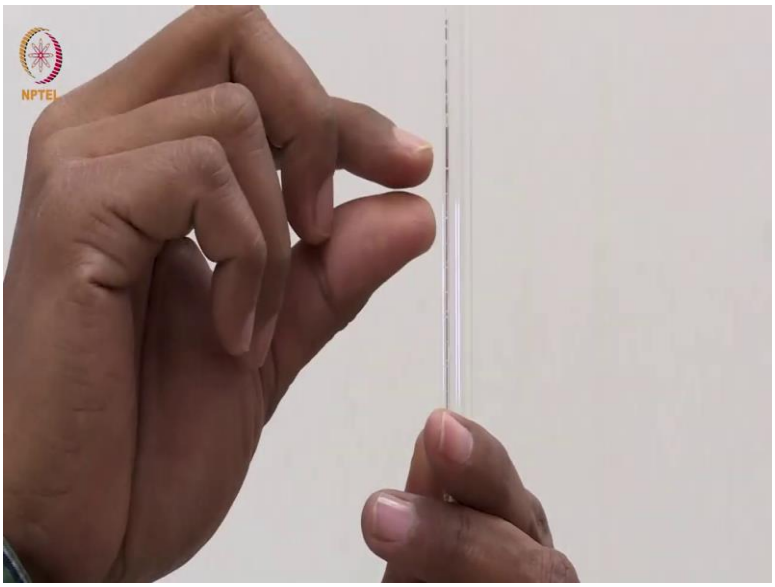


RANKEM
100ml

So, to do the titration, we will need a burette which I am showing here, this burette is a 50 ML burette, which you can see in your screen. And this burette measures 1 ml and that 1 ml is divided into 10 parts. So that is the smallest amount that can be disbursed from here is just point 1 ml. We will use this burette while doing the standardization experiment.

We have another burette here which is a 10 ml burette. Now, you can see the length of the burette is almost the same as that of the 50 ml burette. But this capacity is only 10 ml which essentially means the board size is very small in this particular burette. So, what we have is a 1 ml is measured in this wide region with a gap of 0.2 ml which is measured here and every smallest division in this burette measures 0.02 ml.

So, this is a very precision burette which we will be using for all our Potentiometric and conductometric titration experiments. Why is that because we need the reading of the pH or conductance value after a very, very small amount of addition of the titrant liquid which may be sodium hydroxide or maybe a silver nitrate solution.

In addition to that, we will lead to some conical flasks like this 250 ml conical flasks for titration or 100 ml conical flask for titration. We will need 100 ml beakers for titration of the solutions using potentiometer or the conductivity meter and then we will require glass funnel to prepare the primary standard solution and while transferring some liquid from one container to another.

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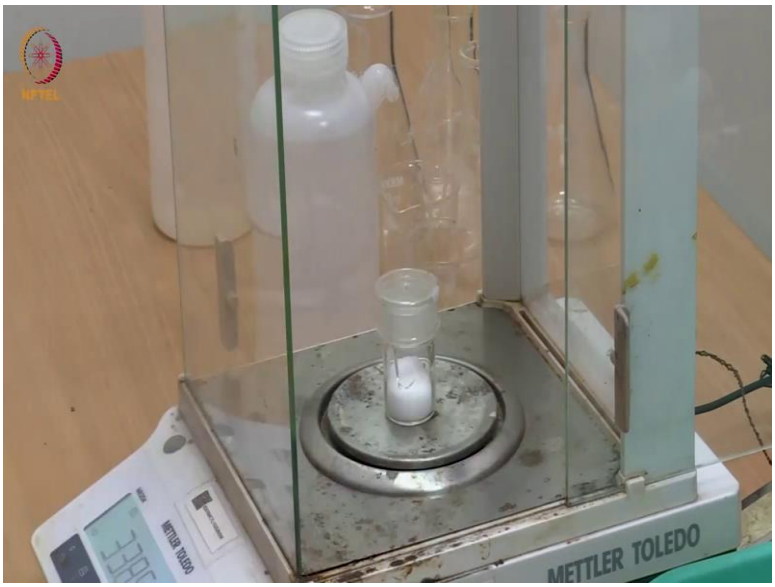

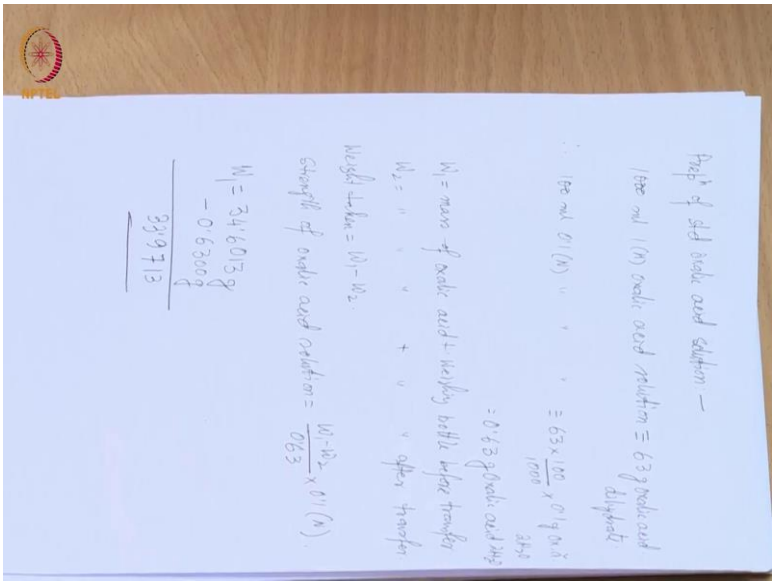

So, now, let me refresh you with what we have discussed in the class. So, when you try to prepare primary standard oxalic acid solution. So, to prepare 100 ml primary standard oxalic acid of approximately 0.1 normal strength, it will lead 0.63 grams of oxalic acid to be transferred in this volumetric flask. So, how to weigh that oxalic acid in this volumetric flask is a question. So, what one can do is take the volumetric flask with a funnel on top of it you take this weighing bottle with the oxalic acid in it and place it inside the balance and take the mass of this weighing bottle along with the oxalic acid before transferring.

So, when I am reading this value, I find that the mass of this combination, which I am calling it as W1 is equal to 34.6013 gram. Every time when you do an experiment, you should have a notebook to write down what you have observed. Now, what we need is to transfer exactly are approximately as close as possible to 0.63 grams of oxalic acid from this bottle. So, we

quickly do a subtraction of 0.6300 gram and we arrive at a number of 33.9713 grams of oxalic acid. So, when we transfer and put it back, we should reach a number which is 33.9713.

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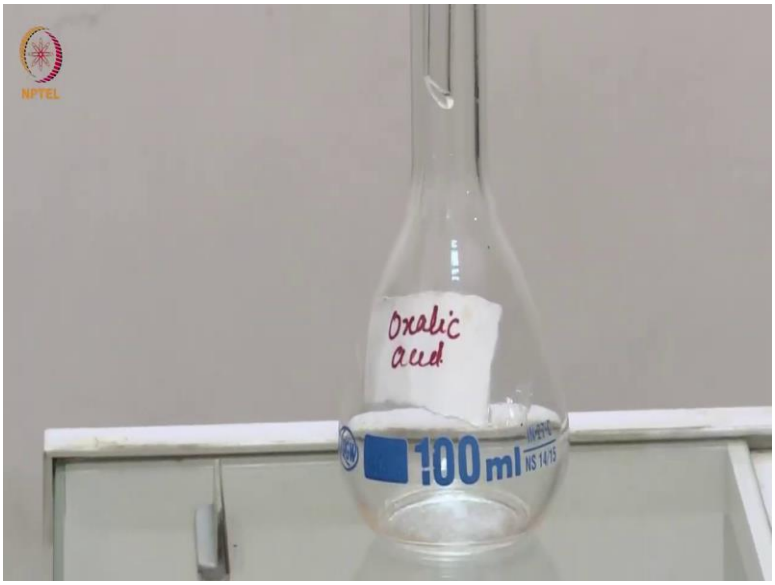
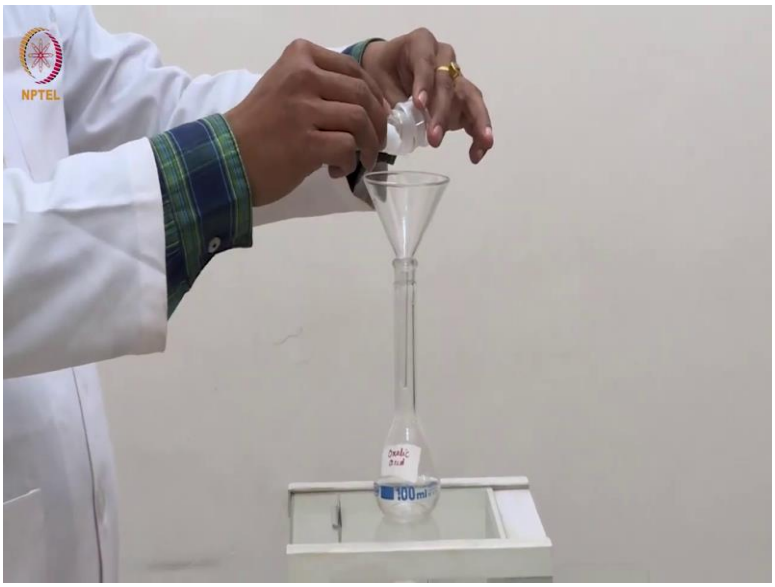
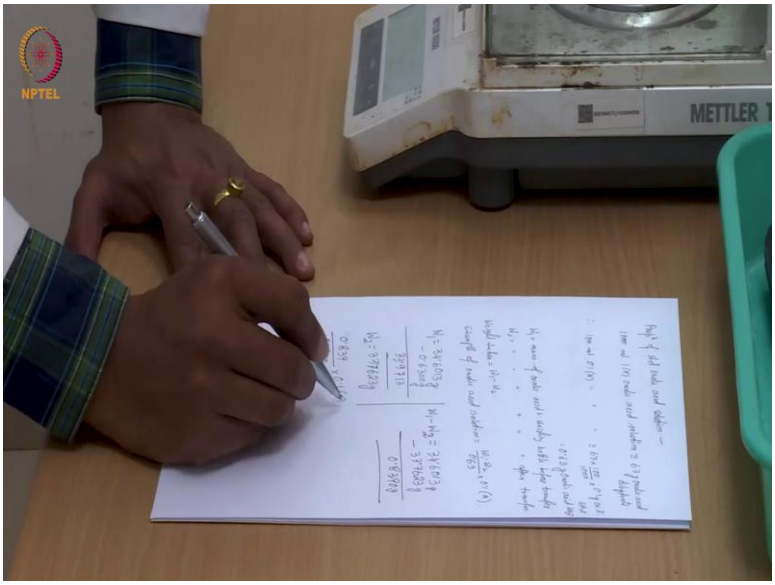



NPTEL
100ml


NPTEL
METTLER


NPTEL


NPTEL
100 ml


NPTEL


NPTEL
JSGW

So, I am now taking out this bottle from here and carefully trying to transfer a very small amount by not moving the lead completely away, but carefully transferring tiny amounts of oxalic acid in the volumetric flask and then taking it down and we will place it back to the balance and what I see here is it is reading 34.22. That means we have not transferred enough amount of oxalic acid.

So, we should again take it back and transfer a little more amount of oxalic acid. So, this volumetric flask carefully and cover it with the lid making sure that no drop or no particles falls outside the funnel and take it down in this one. So now what I see is that I have transferred a little more than what was required.

So, my final weight after transfer that is W2 turns out to be 33.7623 grams. So now, when I do this calculation, how much amount was transferred it is nothing but W1 minus W3 W2, which is the equal to 34.6013 minus 33.7623 grams. So, that difference in weight between W1 and W2 turns out to be 0.839 grams. So, what we have done is we have taken a little more than what was required. So, the strength of the solution that is now going to be prepared is nothing but the difference W2 1 minus W2 divided by 0.63, that is 0.839 divided by 0.630 into 0.1 normal oxalic acid.

So, again we need to do the calculation of this. So, this number turns out to be 0.1332 and the strength is 0.1332 normal. So, when I dissolved this oxalic acid in water and make up to the volume of 100 ml, the solution will be 0.1332 normal.

So, let us now first dissolve this using distilled water. So, first I am washing the funnel so that we do not leave any oxalic acid in this funnel, then one can remove the funnel from the volumetric flask, try to dissolve the amount of oxalic acid that I have taken using this water, we can add more water making sure that we do not overshoot the mark with which tells me that that is my 100 ml, by careful shaking, one should dissolve this oxalic acid completely before making up to the mark.

So, one should take care of complete dissolution before filling this volumetric flask. So now we can see that the acid has completely dissolved there is no floating particle. So now, I am going to fill a volumetric flask and you can see there is a mark here that mark on at this line indicates the 100 ml of this volumetric flask. So, we should fill this volumetric flask with water up to that mark carefully and should not overshoot. Now we should put a lid and then shake it upside down a couple of times so that the solution is homogeneous.

So, this is how one should prepare the primary standard. And what you see is the lower meniscus of this water is touching that mark. And now this is my primary standard oxalic acid. For the standardization experiment video, we have shown you how to prepare a primary standard solution of oxalic acid, which I am going to use in this experiment to standardize the solution of NaOH a sodium hydroxide solution which is approximately N by 10 in strength. So, after this, we will use this NaOH in the future experiments of Potentiometric titrations for strong acid HCl, weak acid, the acetic acid and the dibasic acid oxalic acid.

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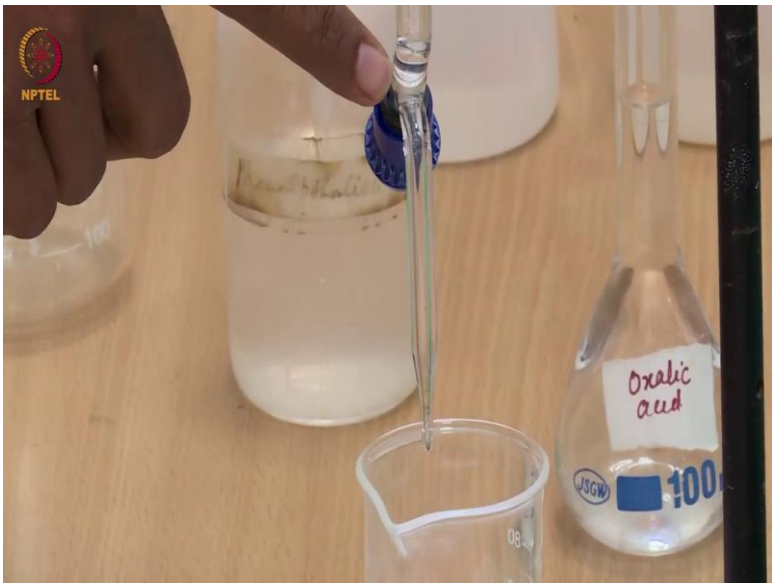
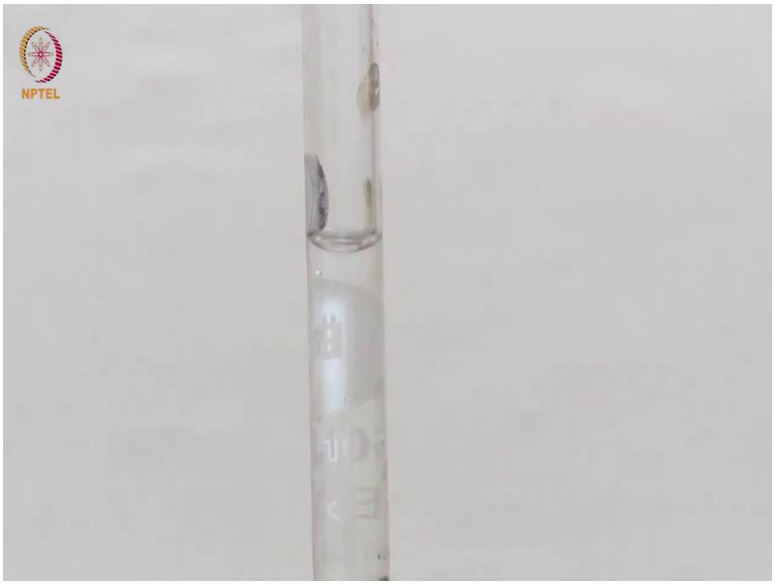
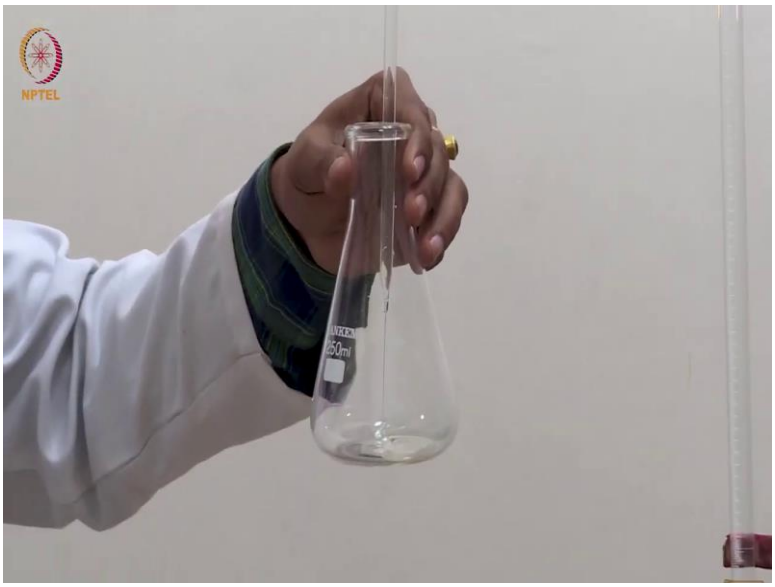

NPTEL
Oxalic acid


NPTEL


NPTEL



So, at the beginning we need to standardize this NaOH for our next set of experiments. So, we will do this standardization using a 250 ml conical, where we will be pipette out our oxalic acid and we will use this 50 ml burette which I have already mounted on this stand and clamp. We will Use this burette and fill it with sodium hydroxide and do the titration. Maybe it is better to keep a thing if there is a leakage it will fall on that only.

So, now we are going to fill this burette with N by 10 NaOH solution we are using a funnel to fill it carefully the bottom is closed. So, we should fill it beyond the 0 mark, because there will be an air gap at the bottom which has to be cleared after we completely fill it.

Now, you can see that NaOH is filled from this joint where we have the stopcock to the upper portion and the lower part is empty. So, we need to open the stopcock carefully to let the solution flow through and flow fast enough to fill that air gap completely. So, when the air

gap is removed, there is a continuous flow of NaOH from the burette, then we need to empty the burette only up to the point when you see a 0 mark at the top.

So, by careful opening of the bottom stopcock drop wise one can see that the apart level of NaOH is slowly coming down and we should bring it down and make sure that the burette is filled only up to the mark 0 with a very careful control on your hand with drop wise release of NaOH making it up to the mark that is 0.

So, now this burette is ready for titration. Now, I need to be pipette out 10 ml of oxalic acid from this volumetric flask to this conical for standardization. So, for that I am going to use this 10 ml pipette and these pipette pump and carefully take out the solution in this pipette and we should pipette out only up to the mark that indicates 10 ml in this, release it in this 250 ml volumetric flask, if you release the till the last drop false and just touch the tip at the bottom only once and we will see that a little amount of acid is still left at the tip. So, that much amount of acid is supposed to be left back in the pipette and not transferred by force to your conical flask for titration.

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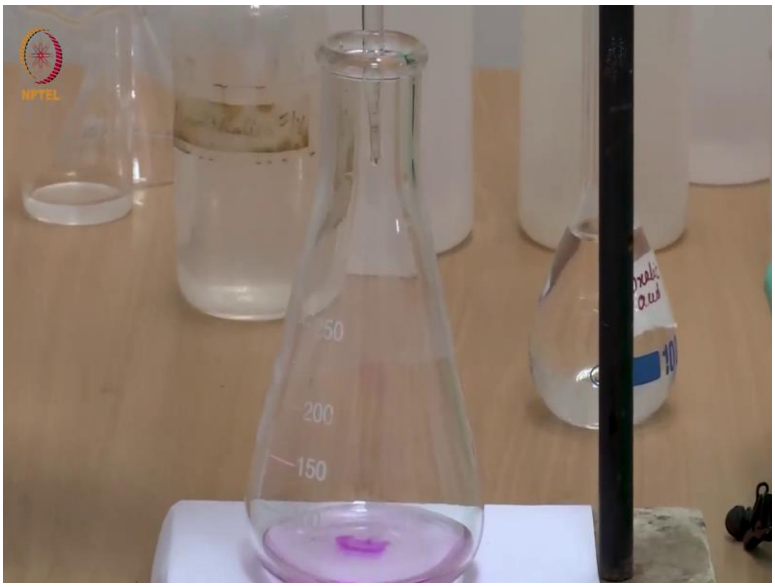
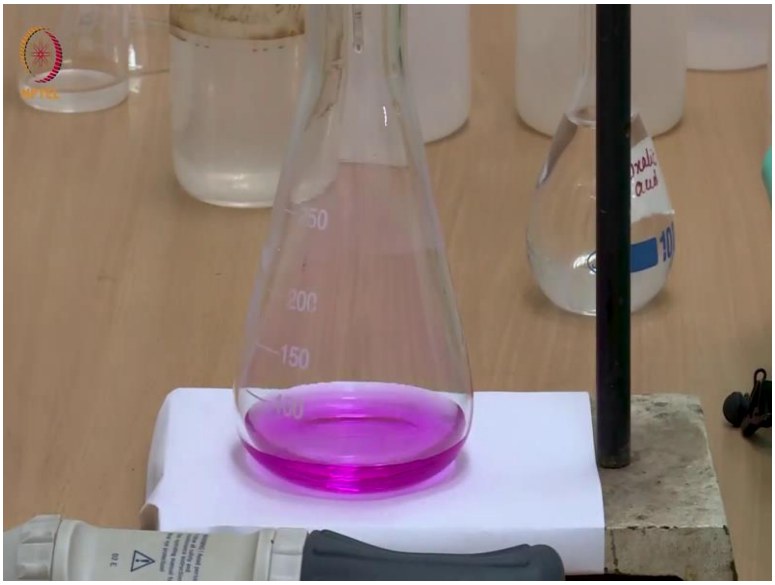
NPTEL
150
100


NPTEL


NPTEL





So, for this acid base titration using NaOH as the titrant and oxalic acid as a primary standard. We need to use phenolphthalein solution as indicator you know phenolphthalein is a colourless solution in acidic medium, but in alkaline medium, it will change its colour to a bright pink colour. So that will indicate the endpoint of this reaction.

Now, when I start adding from this NaOH solution drop wise you can see the appearance of faint pink colour in the solution and immediately disappears, can you see the colour faint pink colour comes and immediately disappears. So, we should do this titration till the pink colour persists, that will indicate the endpoint, you can see the appearance of faint pink colour and on shaking the colour disappears.

See, now on addition the pink colour is staying for a little longer time, which means we are close to the end point of this titration. Now, you will see it is taking much longer time than

before to completely decolorize. So, we are very, very close to the endpoint. See now with one drop, the colour has become pink, and it does not change on shaking or stirring, which indicates that we have reached the end point of this titration. So, now we need to take the burette reading accurately. What you can see here is the burette reading.

So, if I try to read carefully, this is 14.5. This reads 14.5 ml here, the lower meniscus is touching 14.5. So, we need to write down this reading, we started from 0, and the final rating is 14.5 ml. We should repeat this experiment at least three times to get concurrent readings and take the most likely reading that is coming out of those three or even maybe sometimes we do four or five times to get a concurrent reading. And one can try to add one more extra drop and see what happens to this colour. It will only darken but nothing else will happen.

So, your actual endpoint which we have noted is 14.5 and not more than that. So am I just carefully adding one extra drop, it will only intensify the colour and nothing else. So that previous point was my actual endpoint. And this is one drop of NaOH extra added. Once you take care of that you do not overshoot your endpoint because after you overshoot your endpoint, then on excess addition of NaOH. It will only intensify the colour and will give you erroneous reading. So, we should do this experiment at least three times to get concurrent reading.