Elementary Electrochemistry
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Potentiometric Titration of Dibasic Acid with Strong Base

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Welcome back to the experimental sessions for the course elementary electrochemistry. In the previous videos, we have demonstrated two experiments of Potentiometric titrations. One using strong acid and strong base that was with HCl and NaOH. And the second one was a weak acid and a strong base that is acetic acid verses NaOH.

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NPTEL
oxalic acid


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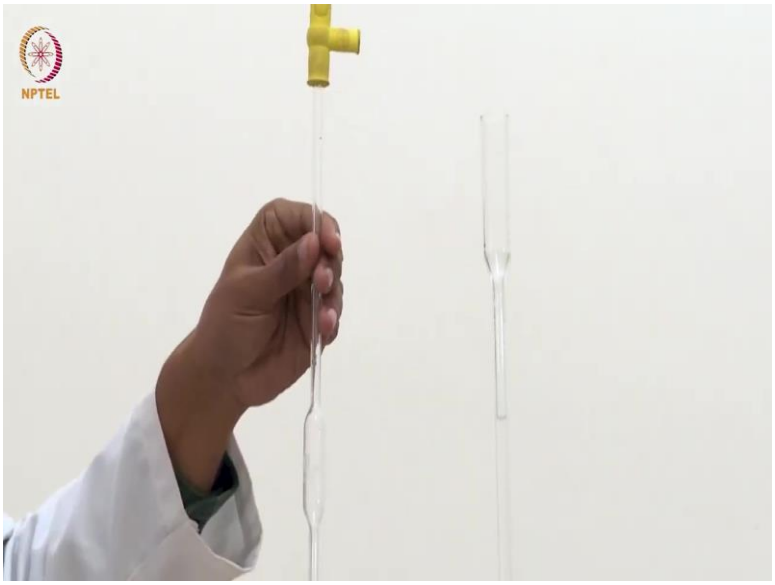
100 ml
oxalic acid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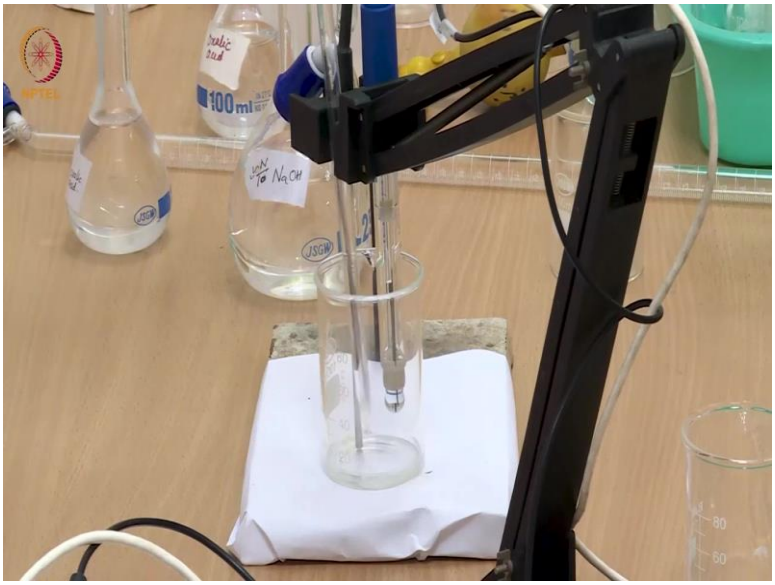
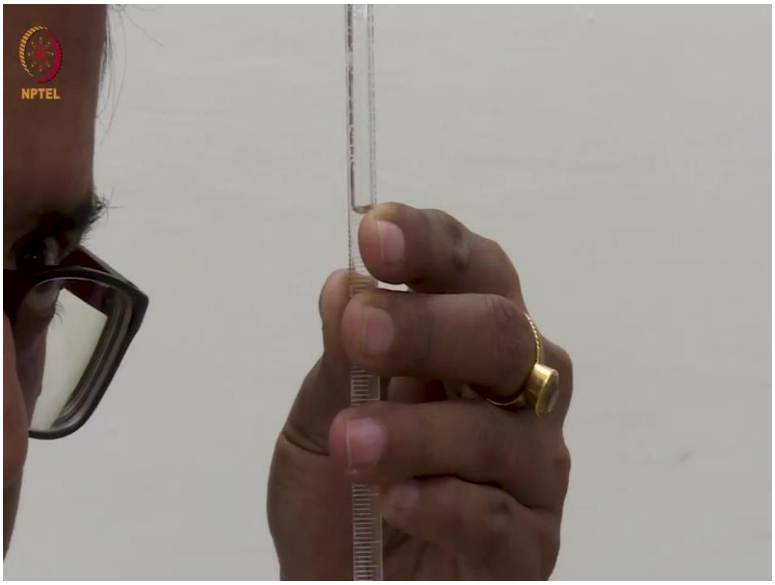

100 ml
NaOH


100 ml


LABINDIA
pH/COND METER
PICO+
RESET
CAL
SCAN
PRINT
MODE
ENTER



So, now in this third experiment, I am going to use a weak dibasic acid, which is here, this is oxalic acid dihydrate approximately N by 100 strengths. As usual, this is a very dilute solution of approximate and by 100 concentrations. And I am going to titrate this using the standardized N by 10 Approximately N by 10 NaOH solution. So, this NaOH solution is 10 times more concentrated with respect to that oxalic acid.

So, what do we expect here is that oxalic acid when it reacts with NaOH it is two carboxylic acids will deprotonate stepwise. So, we should get two minute jumps in my Potentiometric titration graph, when we plot the volume of NaOH versus pH. So, to start we need to use 10 ml of pipette with a pipette pump, I will use these 10 ml pipette to pipette out 10 ml of this approximately N by 100 oxalic acid solutions and transfer it to the beaker in which we will do the titration and we have the pH electrode inserted in that beaker.

As we will see that this tunnel of acid will not be sufficient enough to beat the electorate completely in within the solution. So, after transferring this acid, we will have to add about another 20 to 25 ml of water to make the entire electrode dip the tip of the electrode deep inside the solution for measurement of pH we first try to lower it as much as possible So, that we need to add a minimum quantity of water the electrodes should start reading the pH and should be displayed in the meter as you can probably see in the inset.

So, with just addition about 20 ml of water the electrode is completely deep and we are ready to start the titration process. So, before addition of any NaOH that is at 0 volume of NaOH the pH is 2.70. Now, I am going to start adding NaOH slowly add in a step up 0.02 ml as

before and we will shake the solution stir the solution and homogenize it before taking the reading.

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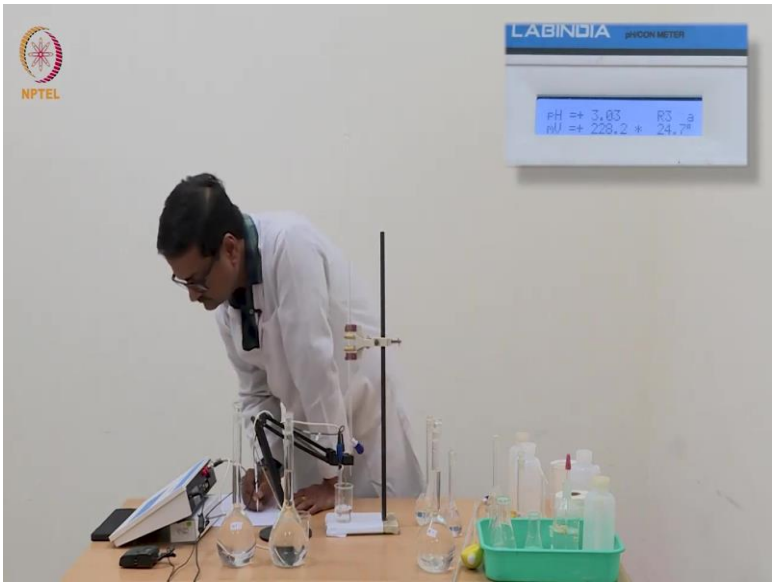
LABINDIA
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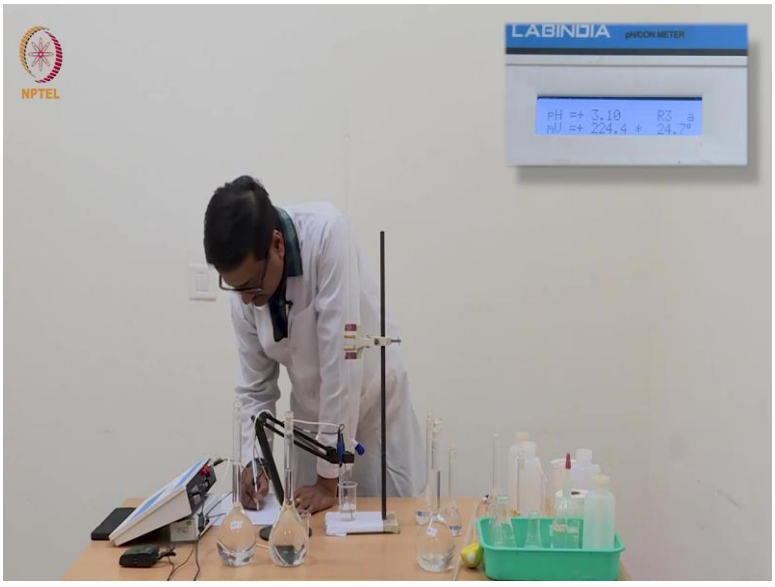
mV =+ 228.2 *
24.7°


LABINDIA
pH =+ 3.10
mV =+ 224.4 *
24.7°

So, after addition of 0.02 ml of NaOH the pH has not changed much it remains that 2.68 So, we should continue this addition as before which we have already done for strong acid and strong base and then for weak acid and strong base. So, with the addition of 0.04 ml of NaOH now the pH has slightly increased to 2.83. So, we will continue to add as before 0.02 ml further of.

Now, we have added up to 0.1 ml of NaOH and the pH has changed to 2.87. So, it is slowly increasing as we expect. We have been adding 0.02 ml each step and now we have reached 0.2 ml and the pH has increased to 2.94. So, we will again continue addition of 0.2 ml 0.02 ml at a time and keep taking readings till we start see a significant increase in pH.

Now, we have reached 0.34 ml and we can see that pH has increased to 3.10. So, the increase is continuous and in a regular manner. So, we have now added 0.4 ml of NaOH and second the mixture it has reached equilibrium and we can see that there is an increase up to 3.15. So, these readings may not look significant for you when you are doing the experiment, but when you plot the Delta pH versus volume of NaOH we will see that there is a point which we are approaching.

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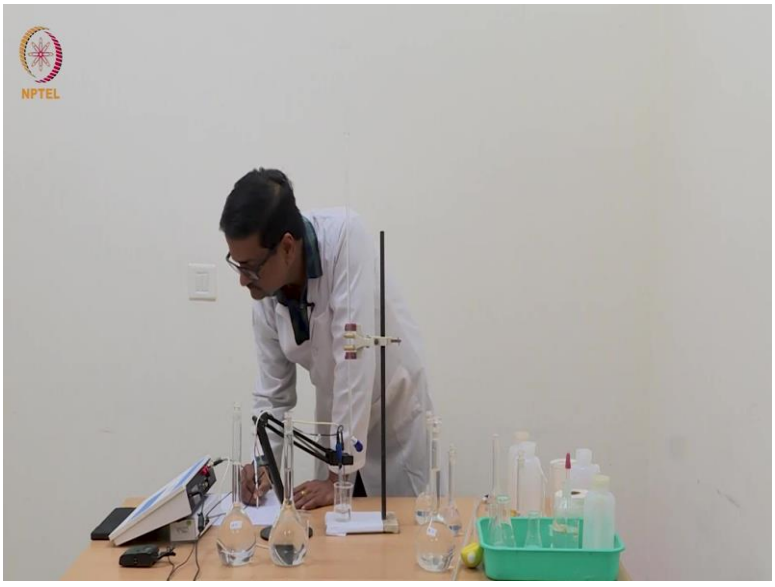


LABINDIA
pH/CON METER
PICO+
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mV =+ 215.9 *
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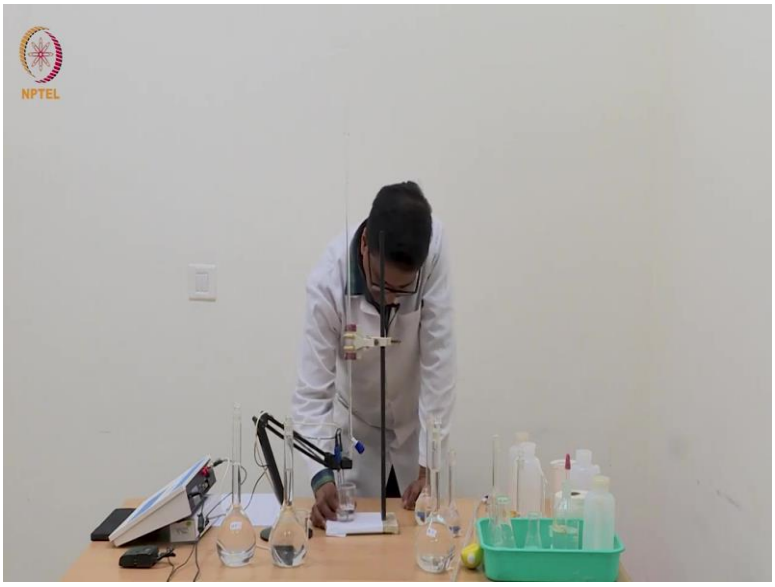
RESET
CAL
FWD TEMPCO
SCAN
PRINT
MODE
ENTER
CE BACK
GLP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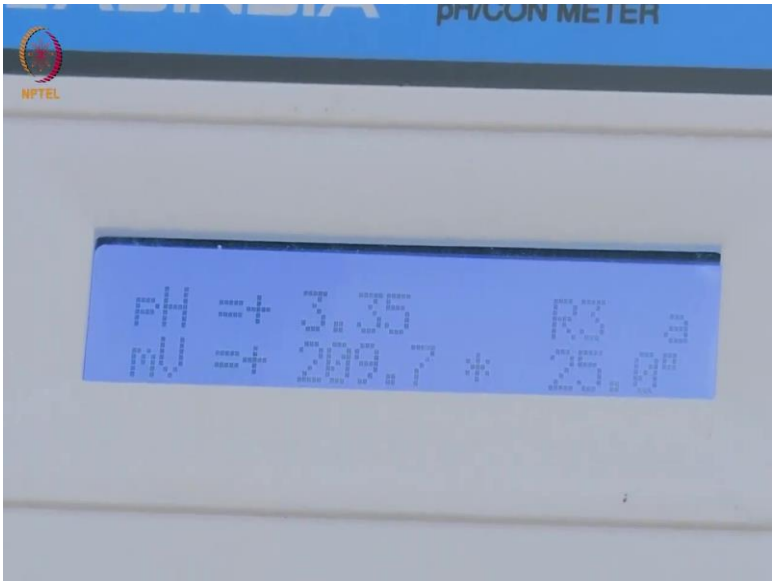
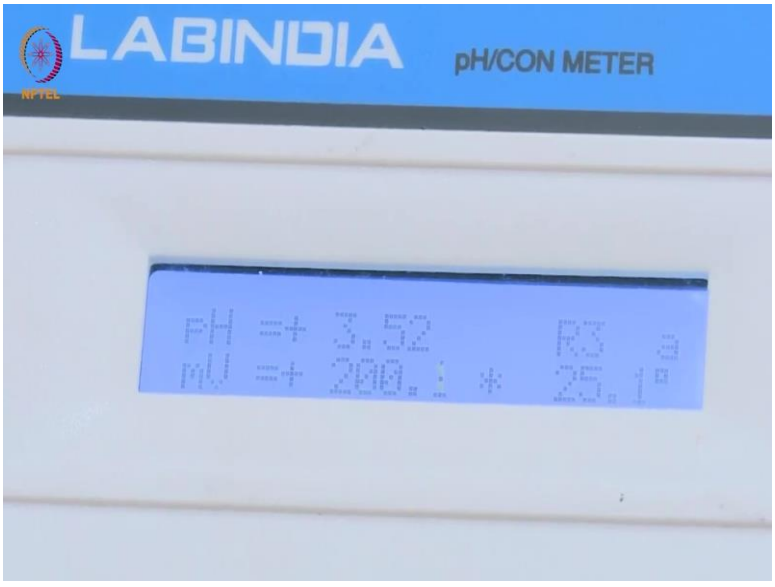
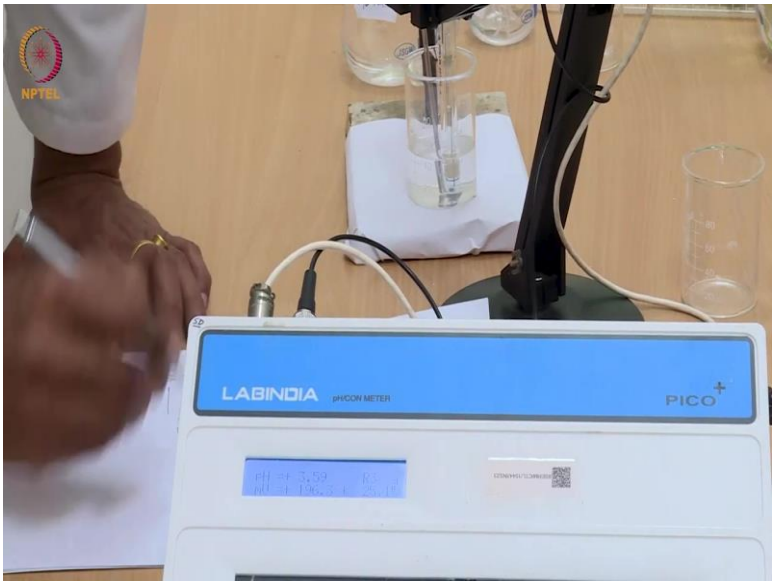

pH/CON METER
NPTEL


LABINDIA
pH/CON METER


LABINDIA
pH/CON METER



Now, which will indicate the half equivalence point that is the first part of oxalic acid that is you have two carboxylic acid groups one carboxylic acid is completely deprotonated to mono oxalate. So, at that point the pH will increase more than the previous readings. So, with the to note down that reading that very carefully. Now, you can see that we have reached 0.5 ml which is about the halfway I am not 100 percent Sure, but this could be defined from where there will be a certain jump in pH because we are now very close to the neutralization of fast carboxylate and for carboxylic acid of the oxalic acid.

So, the pH is now 3.29 now we have added another 0.04 ml this time and we have reached 0.54 ml and pH is 3.35. So, the increase is like 0.05, 0.04 in pH that is happening almost constantly now, I have reached 0.60 ml and the pH has now jumped to 3.44. So, what you will see is that there is a jump in pH by 0.09 which is important when we plot delta pH versus volume of NaOH these differences will become evident.

So, now we have reached 0.64 ml and pH is 3.53 which is again a jump of about 0.9 which indicates that we are close to the half neutralization point we have added another 0.04 ml so, we have reached 0.68 ml and the pH is 3.59. So, what you see here is there was a small region where the Delta pH increased from 0 initially by 0.04 and then 0.06 and then 0.09 and now again the increase is just about 0.07 and with the next addition I presume that it will be again less than 0.07 which will then indicate that we have just crossed the half neutralization point.

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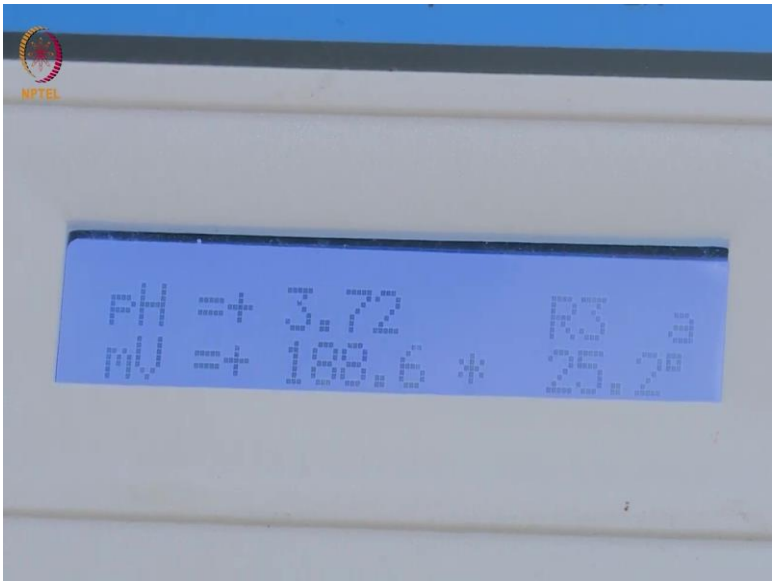
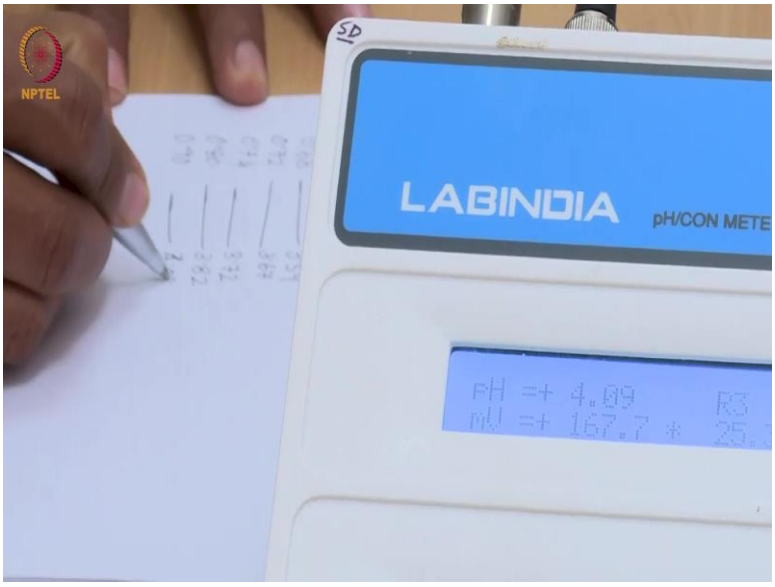
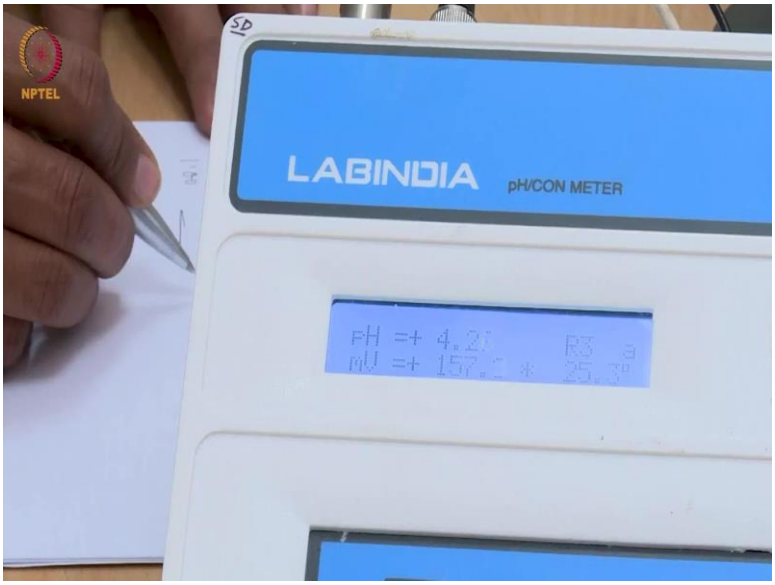



NPTEL


NPTEL
JSGW


NPTEL
SD
LABINDIA
pH/CON METER
pH =+ 3.82
mV =+ 182.8 *
25.3°


LABINDIA
pH/CON METE
pH =+ 4.09
mV =+ 167.7



So, now we are at 0.72 and the pH has changed to 3.67 which is like 0.08 in terms of pH. Now, we have reached 0.74 and pH is 3.72. So, we have just re crossed the half equivalence point and then we should continue as usual till we see a jump above pH 7, which will indicate the complete neutralization point. Now we have reached a point of 0.90 ml that is now the pH is 4.09 where we can see that again the pH is starting to increase because we are now nearing the complete equivalence point.

So, we have added up to 1 ml now and with 1 ml it is 4.27. Now we have reached a point of 1.20 ml and the pH that you can see is 4.85 the accuracy of this experiment depends on how close your readings are with respect to the amount of NaOH added. So, we should always add 0.02 ml in each step. So, that our delta pH versus delta V plot is done very accurately and one can determine the end point or the equivalence point accurately.

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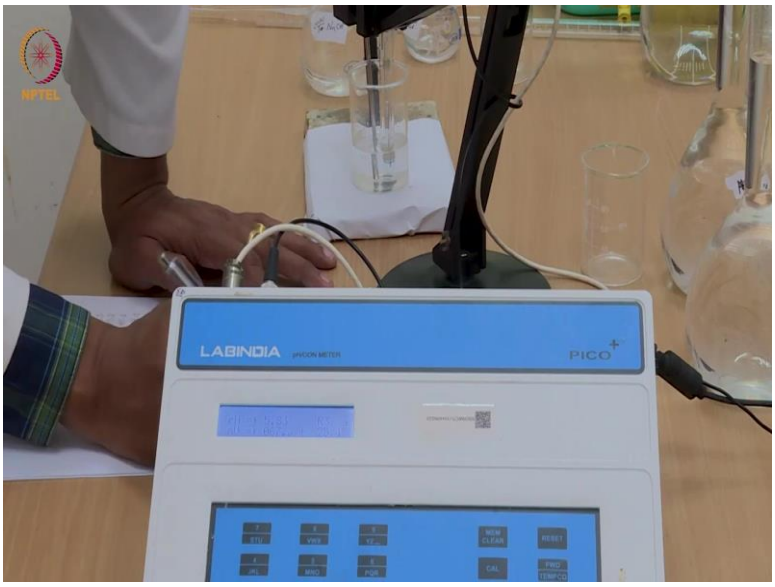

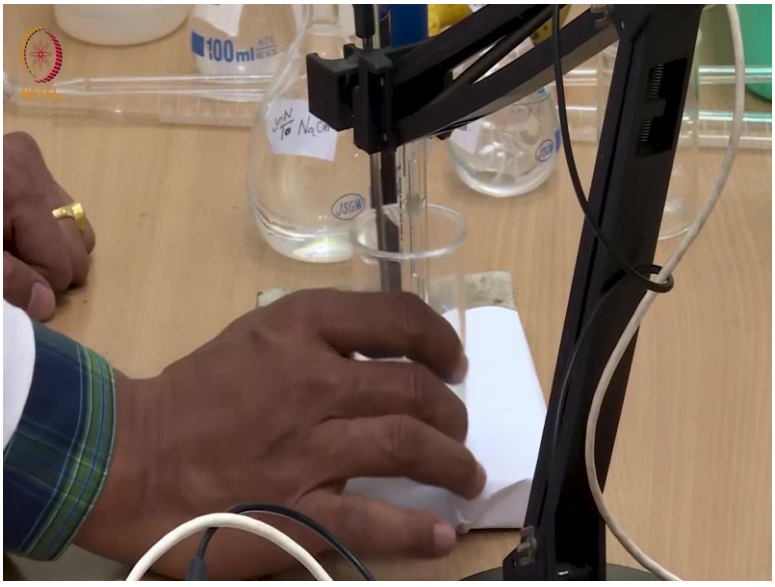


LABINDIA
pH/CON METER
pH =+ 6.53
mV =+ 027.0 *
25.5°


pH =+ 6.73
mV =+ 015.8 *
25.6°

Now, by stepwise addition, I have reached a point where I have added 1.30 ml and the pH reads 5.38 which now indicates that the S type of curve has started going upwards. So, to draw a curve very nicely, one should have readings with very small valid difference in the volume of NaOH added that is, we should now use again 0.2 ml steps at a time. So, with just addition of 0.02 ml of this NaOH which is 10 times stronger has increased the pH from 5.38 to 5.83.

So, we continue addition of 0.02 ml each time and mix it well. So, that the solution is homogeneous and the pH that we read is the pH of the solution and not a local pH. So, what you can see is that now, we have 0.02 ml edition of N by 10 NaOH the pH that is increasing is increasing in the order of 0.4 earlier reading was 5.83 and now it is 6.23. 1.38 ml is now added and the pH is still increasing because it has not homogenized yet.

So, we should start it well for a longer period of time to homogenize so, that the pH meter can read the correct pH. So, 6.69 it is still increasing it is going very close to 7 which essentially indicates that now we are very close to the equivalence point 6.73 should be our reading. So, now I am guessing that with one more drop of NaOH which will take us to 1.4 ml we will have is jump in pH and the end point will be reached.

So, with 1.4 ml of NaOH added our pH has now crossed the mark of 7. So, it is now 7.08. Remember that the actual end point will be calculated when then we plot Delta pH versus volume of NaOH and where there will be a peak in that plot that will indicate the endpoint. So, here by looking at these readings.

I can guess that the endpoint will be somewhere between 1.34 and 1.38 because the jump there is about point 0.5 ml point the jump of pH is about 0.5 and in the previous case The jump was 0.4 and then again from 7, 6.73 to 7.08. The jump is about 0.3. So, the increase that is the delta pH versus V NaOH the increased delta pH is reducing now. So, somewhere in this region we will have the end point.

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NPTEL


NPTEL


NPTEL


NPTEL
pH =+ 9.46
mV =- 145.9 *
25.7°


NPTEL


NPTEL
pH =+ 9.86
mV =- 169.4 *
25.7°


pH =+10.39

So, we will when we plot we will be able to see it better. So, by continuing addition of 0.04 ml in each step, now, I have reached 1.5 ml and we can see that we have crossed pH 7 and reached a value of 8.22 it is still increasing still homogenizing the solution. So, we should stir it well. So, that the solution becomes homogeneous quickly and the pH meter can read the correct pH.

We take this value as 8.55 and then continue adding more NaOH till it reaches above 10. I have reached 1.54 ml and pH is 9.4644, 9.46. So, we continue for another 3 or 4 readings I am sure with that we will reach above 10. So, now we have added 1.58 ml and pH has now reached 9.86. So, it is going very close to 10. And you see that the difference in the consecutive readings is now decreasing slowly.

Now, we have reached 1.60 ml and pH has just crossed 10. So I think we can do 2 or 3 more readings and then stop this experiment because now we have reached a situation where towards the top, the pH is now slowly stabilizing at about 10. So, with these data points we will be able to draw a nice S like type curve. So, now, in 2 steps I have added 1.64 and then 1.68 ml of N by 10 NaOH and I have reached the pH of 10.33.

So, if you look at the consecutive last few readings the increase is now very small. So, with one more reading with 1.7 ml we will end this titration and then go back to the bench and write draw the curve for this experiment. So, now, I have added just 0.02 ml of NaOH to end this titration and the pH is 10.39. So, what we see is the increase towards the end has now again reduced back to a difference of 0.05 or 0.06 ml 0.05 or 0.06 in pH and that is where one

should stop this titration and use this entire data to plot the volume of NaOH versus pH and also Delta pH versus volume of NaOH.

And see how the S type of curve comes for 3 different acids. Once you try, actually try to plot these data in the same graph paper and compare them quantitatively together and see how the shape of that S curve is different from strong acid strong base to weak acid strong base to a weak dibasic acid to a strong base. So, I will discuss these during the theory class with the plots that we will generate from these data and we will have a complete discussion after the experiment is over today.

So, with this we end first part of our experimental discussion using Potentiometric titration. This will be followed by the calculations graphs of these experiments. And we will conclude the week with those data. And in the next week, we will have experiments next time we will have the experiments related to conductometric and conductometric titrations. Thank you.