3rd International Microscale Chemistry Symposium

Workshop:

Microscale Analytical Chemistry with Locally Produced Low Cost Equipment.

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1) Introduction (power-point presentation) 30 min.

-Microtitrimetric analysis (v<1 mL)
-micropotentiometry: titrimetry and microbiosensors
-microconductometry
-microchronoamperometry
-microphotocolorimetry

2) Experiments

a) Calibration plot of a solid state micro-pH sensor: W°|H_2O||Cu°. NaOH normalization plot.

b) Sodium Chloride- Silver nitrate micropotentiometric tiration without salt bridge.

c) Microconductimetric titration of aspirin in tablets.

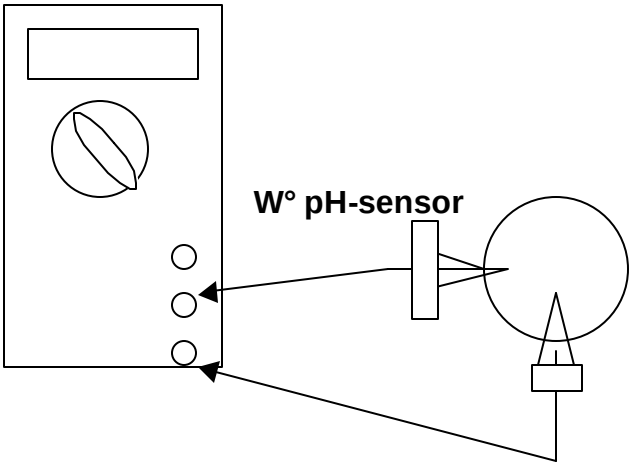
d) Microphotocolorimetric calibration plot for Cu(II). Absorbance = pT = f(conc.)

EXPERIMENTS

a) Calibration plot of a solid-state micro-pH-sensor.

Experimental:

- Fill the 1 mL microburet with 0.1 M NaOH as shown in the previous video.
- Pour in the potentiometric cell a 0.5 mL aliquot of 0.1 mol/L potassium monoacid phtalate . Add a microstirrer bar.
- Add enough water in order to cover microelectrodes.
  Connect microelectrodes to voltmeter as shown below:



Ref. Electrode: Cu°|H_2O||

Add NaOH solution from the burette in small increments of 20 microliters (0.02mL) stirring well.

Measure the potential value after each addition of titrant.
Find the straight line equation that relates E and pH using the following data:

NaOH added	pH	E (V)
0.0 mL	4.0	
1.0 mL	12.0	

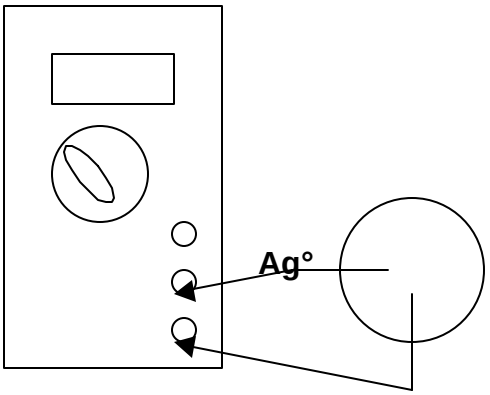
Plot the graph of pH versus volume of NaOH added.
Conclude.

b) Sodium Chloride- Silver nitrate micropotentiometric tiration without salt bridge.

Experimental:

- Fill the 1 mL microburet with 0.1 M $AgNO_3$.
- Pour in the potentiometric cell a 0.5 mL aliquot of 0.1 mol/L NaCL.
- Add enough water in order to cover microelectrodes.

Connect microelectrodes to voltmeter as shown below:



Ref. Electrode: $Cu°|H_2O||$

Add $AgNO_3$ solution from the burette in small increments of 20 microliters (0.02mL) stirring well.
Measure the potential value after each addition of titrant.

Plot the graph of E versus volume of $AgNO_3$ added.
Conclude.

c) Microconductimetric titration of aspirin in tablets

Experimental:

- Fill the 1 mL microburet with 0.1 M NaOH.
- Pour in the conductimetric cell a 0.5 mL aliquot of a dispersed tablet in 10 mL alcohol water.
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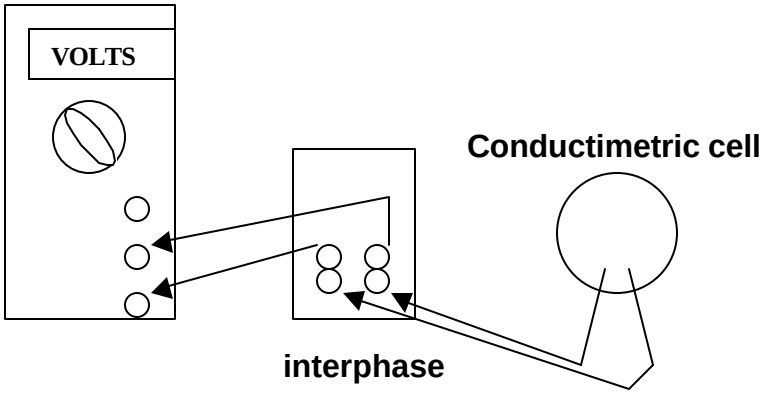
- Add enough water in order to cover microelectrodes.

Connect microelectrodes to voltmeter and to the conductimetric interphase as shown below:



Add NaOH solution from the burette in small increments of 20 microliters (0.02mL) stirring well.
Measure the potential value after each addition of titrant.

Plot the graph of E versus volume of NaOH added.
Conclude in terms of content of aspirin found in one tablet.

d) Microphotocolorimetric calibration plot for Cu(II).
Absorbance = pT = f(conc.)

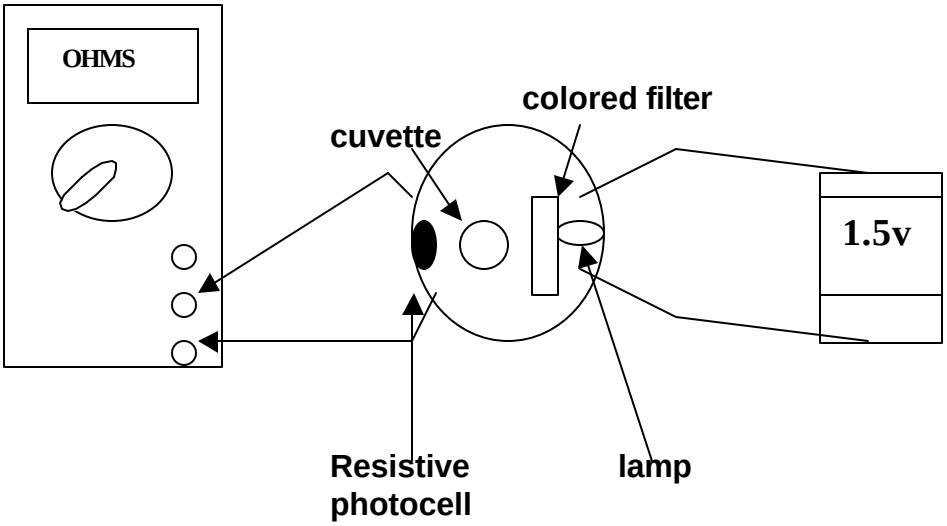
Experimental

Preparation of calibration plot. In 1.5 mL Eppendorf tubes make the mixtures indicated below*:

Tube	**$CuSO_4$ 0.1 mol/L**	**NH_3 conc.**
0	0.00	1.00
1	0.04	0.96
2	0.06	0.94
3	0.08	0.92
4	0.1	0.90

*Aliquots can be added with an insulin syringe .

Connect the resistive photodetector to the ohmmeter and the little lamp to the 1.5 DCV power supply:



Measure the resistence of the photodetector with the lamp off (base line response: R_r). Covering the microphotocolorimeter.
Pour in the red solution as colored filter.
Fill the cuvette with blank solution (tube 0) and turn the lamp on. Measure the resistence (R_0). Covering the microphotocolorimeter.
Pour out the blank solution with a syringe.
Rinse the cuvette two times with the most diluted solution (tube 1). Fill the cuvette with this solution. Measure the resistence (R_i).
Repeat the above procedure with the rest of the standard solutions.

Calculate the absorbance, pT=-logT, according to:

$$A = pT = -\log (R_i - R_r / R_0 - R_r)$$

Plot pT against concentration of copper (II) to obtain the calibration plot.

Conclude according to the linearity obtained.