TITRATION OF AN ACID MIXTURE
INSTRUCTOR RESOURCES

The CCLI Initiative

Learning Objectives

The objectives of this experiment are to . . .

- analyze an acid mixture by titration with NaOH solution.
- use the ***MicroLAB*** interface to gather and store titration data.
- use the ***MicroLAB*** spreadsheet to graph the titration curve and accurately determine the endpoints.

Procedure Overview

- a solution containing both HCl and H_3PO_4 is titrated using standardized NaOH solution.
- the titration is followed using a pH electrode attached to the ***MicroLAB*** interface.
- NaOH volumes are read from the buret and manually entered into the spreadsheet from the keyboard. This allows four significant figure volume data for the experiment.
- titration and first derivative curves are constructed and the two acid concentrations are determined from the inflection points.

Name ______________________________ Section ________________ Date _____________

TITRATION OF AN ACID MIXTURE

Report Sheet

Molarity of Standardized NaOH ___________________________

	First Equivalence Point	**Second Equivalence Point**
Final volume (ml)	______________________	______________________
Initial volume (ml)	______________________	______________________
Buret correction (ml)	______________________	______________________
Total volume NaOH delivered (ml)	______________________	______________________

Name ______________________________ Section ________________ Date _____________

TITRATION OF AN ACID MIXTURE

Report Sheet (page 2)

Calculations

1. Calculate the number of moles of H_3PO_4 present in the 25 ml aliquot of unknown. Show your work.

2. Calculate the molarity of H_3PO_4 in the unknown. Show your work.

3. Calculate the number of moles of HCl present in the 25 ml aliquot of unknown. Show your work.

4. Calculate the molarity of HCl in the unknown. Show your work.

Name ______________________________ Section ________________ Date _____________

TITRATION OF AN ACID MIXTURE

Questions/Problems

1. Calculate the pH of a solution containing 0.10 *M* HCl and 0.10 *M* acetic acid ($HC_2H_3O_2$, K_a = 1.8×10^{-5}).

2. Calculate the pH of a solution containing 1.0×10^{-3} *M* HCl and 2.0 *M* formic acid (HCOOH, K_a = 1.8×10^{-4}).

3. Consider the titration of an unknown mixture of HCl and H_3PO_4. A 50.0 ml sample of this mixture required 38.50 ml of 0.100 *M* NaOH to reach the first equivalence point and a total of 53.80 ml of 0.100 *M* NaOH to reach the second equivalence point. Calculate the original concentrations of HCl and H_3PO_4 in the mixture.

TITRATION OF AN ACID MIXTURE

Tips and Traps

1. A standardized NaOH solution (~0.1 *M*) is needed. Students can either prepare and standardize the base solution or it can be provided directly.

2. Be sure students record a pH reading before any NaOH is added.

3. Students need not start the titration at a buret reading of 0.00 ml. Have students enter the actual volume reading from the buret when requested. Then let the spreadsheet correct for not starting at the 0.00 ml mark by setting Column C = Column A + buret reading at the start of the titration. Column C then contains the actual volume of NaOH delivered at each point.

4. The above necessitates modifying graphing procedures and spreadsheet set up as follows:
   a. to graph the titration curve plot Column B (pH, *y-axis*) vs. Column C (ml NaOH, *x-axis*).
   b. use the Add Formula to calculate the first and second derivatives, then "click-drag" to Columns D and E.

5. Some instructors require that students calibrate their burets for volume delivered. The report sheet contains an entry line for such calibration data (labeled as *Buret Correction*). If you do not require calibration, simply enter 0.00 on this line.

TITRATION OF AN ACID MIXTURE

Suggested Answers to Questions/Problems

1. Calculate the pH of a solution containing 0.10 *M* HCl and 0.10 *M* acetic acid ($HC_2H_3O_2$, K_a = 1.8×10^{-5}).

   pH = 1.00, pH controlled by the HCl due to weak nature of HC_2H_3O

2. Calculate the pH of a solution containing 1.0×10^{-3} *M* HCl and 2.0 *M* formic acid (HCOOH, K_a = 1.8×10^{-4}).

   pH from HCl = $1.0 X 10^{-3}$ [H+] from HOOF ~ $1.8 X 10^{-4}$

   pH ~ -log($1.19 X 10^{-3}$) = 2.9

3. Consider the titration of an unknown mixture of HCl and H_3PO_4. A 50.0 ml sample of this mixture required 38.50 ml of 0.100 *M* NaOH to reach the first equivalence point and a total of 53.80 ml of 0.100 *M* NaOH to reach the second equivalence point. Calculate the original concentrations of HCl and H_3PO_4 in the mixture.

   38.50 ml NaOH | 0.1 mmol NaOH | 1.0 mmol HCl = 0.077 M HCl
   50.0 ml Sample | 1.0 ml NaOH | 1.0 mmol NaOH

   ml for H_3PO_4 = 53.80 - 38.50 = 15.00 ml H_3PO_4| 0.1 mmol NaOH | 1.0 mmol HCl = 0.031 M H_3PO_4
   50.0 ml Sample | 1.0 ml NaOH | 1.0 mmol NaOH

Calculations

1. Calculate the number of moles of H_3PO_4 and/or NaH_2PO_4 present in the 25 ml aliquot of unknown. Show your work.

   XX.XX ml NaOH | 0.1 mmol NaOH | 1.0 mmol H_3PO_4 = YY.YY mmol H_3PO_4
   | 1.0 ml NaOH | 1.0 mmol NaOH

   Same setup for calculating mmol of NaH_2PO_4

2. Calculate the molarity of H_3PO_4 and/or NaH_2PO_4 in the unknown. Show your work.

   XX.XX ml NaOH | 0.1 mmol NaOH | 1.0 mmol H_3PO_4 = YY.YY M H_3PO_4
   25.0 ml Sample | 1.0 ml NaOH | 1.0 mmol NaOH

   See part 2 of question 3 above for example to calculate NaH_2PO_4 for a mixture of H_3PO_4 and NaH_2PO_4

3. Calculate the number of moles of HCl present in the 25 ml aliquot of unknown. Show your work.

   XX.XX ml NaOH | 0.1 mmol NaOH | 1.0 mmol HCl = YY.YY mmol HCl
   | 1.0 ml NaOH | 1.0 mmol NaOH

4. Calculate the molarity of HCl in the unknown. Show your work.

   XX.XX ml NaOH | 0.1 mmol NaOH | 1.0 mmol HCl = YY.YY M HCl
   25.0 ml Sample | 1.0 ml NaOH | 1.0 mmol NaOH

TITRATION OF AN ACID MIXTURE

Sample Data

Molarity of Standardized NaOH: 0.1020

	First Equivalence Point	Second Equivalence Point
Final volume (ml)	14.90	23.72
Initial volume (ml)	0.76	0.76
Difference (ml)	14.14	22.96
Buret correction (ml)	0.00	0.00
Total volume NaOH delivered (ml)	14.14	22.96

ml NaOH	pH	ml NaOH	pH	ml NaOH	pH
0.76	2.07	15.20	5.22	23.56	8.50
1.96	2.12	15.30	5.42	23.64	8.80
4.38	2.14	15.52	5.68	23.72	9.15
5.00	2.17	15.78	5.83	23.80	9.54
5.40	2.19	16.16	6.03	23.94	9.83
7.60	2.31	16.72	6.23	24.04	10.00
10.20	2.51	17.44	6.41	24.30	10.23
11.96	2.71	18.38	6.62	24.60	10.44
13.00	2.93	19.30	6.80	25.08	10.63
13.80	3.13	20.24	7.00	25.78	10.81
14.28	3.35	21.14	7.21	26.86	11.00
14.52	3.52	22.00	7.41	28.70	11.20
14.80	3.90	22.52	7.63	31.54	11.41
14.90	4.24	23.02	7.88	36.12	11.60
15.02	4.82	23.30	8.12	42.78	11.80

TITRATION OF AN ACID MIXTURE

Sample *MicroLAB* Programs

Experiment file name: ***pH,temp.vs.drop.titr.0.1pH.exp***

Sensors: **drop counter**: X axis, Col. A, DD on top, units = drops; **pH**: Y1 axis, Col B, DD in middle, units = pH; **Temperature**: Y2 axis, Col C, DD on bottom, units °C. (Use of temperature at instructors discretion. If done, a Styrofoam calorimeter should be used.)

Special Program:
Read Sensors
Repeat when counter change
    If Delta pH > +/- 0.100
        Read Sensors
    Else
    End If
Until Stop Button is pressed

Comment: For best accuracy, calibrate the drop counter using one following programs. If temperature is measured with a Temp(IC) probe, it must be wrapped in Saran Wrap to prevent grounding the pH probe.

Calibration of Drop Counter by incremental volumes: The total number of drops at each of five - 2 ml increments from a buret or into a graduated cylinder is measured and graphed. The slope will give the average volume per drop.

Experiment name: ***dropcal.increm.vol.exp***.

Sensors**: drop counter**: X axis, Col. A, DD on top, units = drops; **keyboard** (**Volume**): Y1 axis, Col B, DD on bottom, units = ml.

Program: The default program works fine.

Comment: Make sure the drop counter is properly aligned to count each drop. Use the top Luer valve to shut of the titrant at each 2 ml volume.

Calibration of Drop Counter by incremental mass: The total number of drops at each of five - 2 g increments from a buret is measured and graphed. The slope will give the average grams per drop, which is then converted to volume by dividing the mass by the density of the titrant.

Experiment name: ***dropcal.increm.mass.exp***.

Sensors**: drop counter**: X axis, Col. A, DD on top, units = drops; **keyboard (mass)**: Y1 axis, Col B, DD on bottom, units = g.

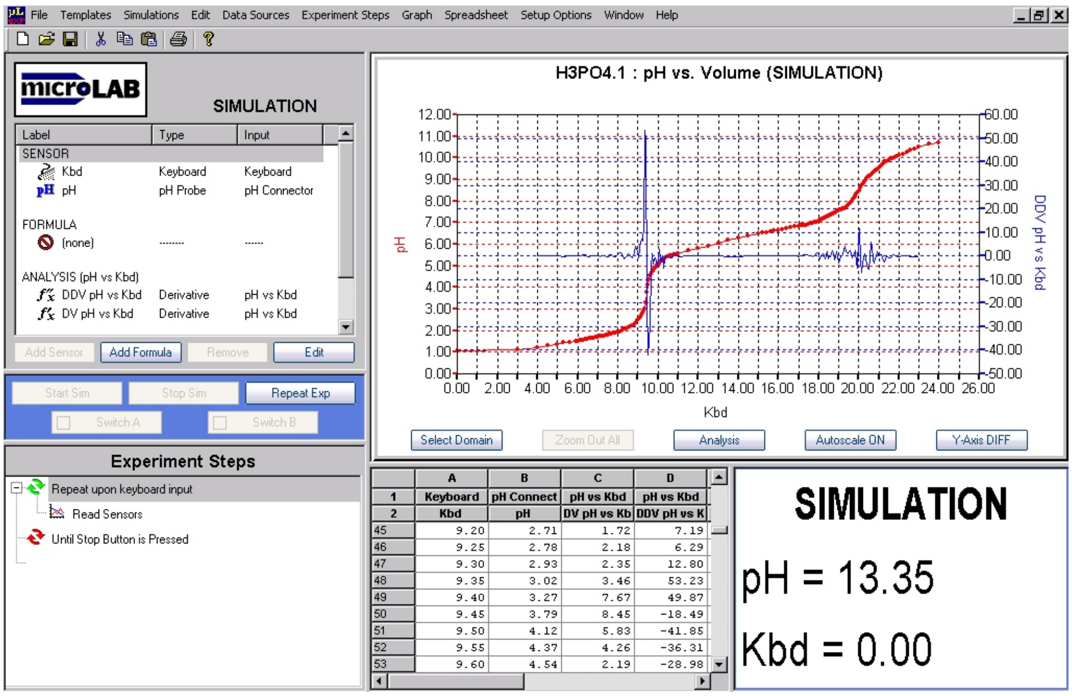
Program: Default program works fine.

Comment: Make sure the drop counter is properly aligned to count each drop. Use the top Luer valve to shut of the titrant at each 2 g mass.

TITRATION OF AN ACID MIXTURE

Sample *MicroLAB* Main Screens

MicroLAB Main Screen for a sample of pure H_3PO_4 with the two equivalence point volumes, as determined by the second derivatives, equal to each other. In a sample containing HCl and H_3PO_4, the first equivalence point volume would be greater than the second, and in a sample containing H_3PO_4 and NaH_2PO_4, the second equivalence point volume would be greater than the first.



TITRATION OF AN ACID MIXTURE

Laboratory Preparation (per student station)

Equipment

- pH electrode
- ring stand
- buret clamp
- stir bar
- magnetic stirrer
- 50 ml buret, **OR**, a ***syringe*** buret (a 60 ml plastic syringe with a Luer tip, and two 1-way Luer valves attached in series to the tip. The top valve turns the flow on and off, and the bottom valve is used to adjust the drop rate. It works really well for drop counter titrations. The syringe and Luer valves may be purchased from ***MicroLAB*** , Inc, or from Irwin Talesnick's S17 Science Supplies & Services, 57 Glen Cameron Rd. Unit 6, Thornhill, Ontario L3T 1P3, Canada)
- 25 ml pipet
- 50 ml Erlenmeyer flask
- 150 ml beaker
- 100 ml graduated cylinder

Chemicals

Actual quantities needed are given below. A 50% excess is recommended.

- buffer solution (pH 7)
- 50 ml standardized NaOH solution (~0.1 *M*)
- 40 ml unknown acid mixture (~0.025 *M* in HCl and ~0.04 *M* in H_3PO_4). The unknown can be prepared by mixing the appropriate amounts of solid NaH_2PO_4\$$H_20$, concentrated HCl, and H_2O.

Safety and Disposal

- both the acid unknown and NaOH solutions are corrosive. Make sure students wear goggles at all times.
- all resulting solutions may be neutralized and flushed down the drain with plenty of water.