Analysis of a Solid Mixture

INSTRUCTOR RESOURCES

The CCLI Initiative

Learning Objectives

The objectives of this experiment are to . . .

- learn to analyze a solid unknown with volumetric techniques.
- standardize a solution of NaOH.
- determine the percentage of KHP in solid mixture of KHP and a soluble salt.

Procedure Overview

- a solution of NaOH is prepared by diluting a stock solution.
- the NaOH solution is standardized with KHP in using an indicator for experience in classic indicator titrations and also using the *MicroLAB* interface for experience in potentiometric titrations.
- the percent KHP in an unknown solid mixture is determined both by indicator and potentiometric titration.

Name _____ Section _____ Date _____

ANALYSIS OF A SOLID MIXTURE

Report Sheet

Calibration of drop size

Write the equation for conversion from drops NaOH to ml NaOH:

Standardization of NaOH – indicator titration

	Trial 1	Trial 2
mass KHP	_____ g ml	_____ g ml
initial buret reading	_____ ml	_____ ml
final buret reading	_____ ml	_____ ml
volume of NaOH used	_____ m	_____ ml
molarity NaOH	_____ M	_____ M

Standardization of NaOH – ***MicroLAB*** titration

	Trial 3	Trial 4
data file name	_____	_____
mass KHP	_____ g ml	_____ g ml
molarity NaOH	_____ M	_____ M

Average molarity for all four titrations _____

Standard deviation for all four titrations _____

Analysis of unknown solid mixture - indicator titration

Unknown #

	Trial 1	Trial 2
mass unknown	_____ g	_____ g
initial buret reading	_____ ml	_____ ml
final buret reading	_____ ml	_____ ml
volume of NaOH used	_____ ml	_____ ml
% KHP in unknown	_____ %	_____ %

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ANALYSIS OF A SOLID MIXTURE

Report Sheet

Analysis of unknown solid mixture - MicroLAB titration

	Trial 3	Trial 4
data file name	_____	_____
mass unknown	_____ g	_____ g
Equivalence Pt. vol.	_____ ml	_____ ml
% KHP in unknown	_____ %	_____ %

Average % KHP from all four titrations _____

Standard deviation for all four titrations _____

Calculations

Standardization of NaOH

1. Determine the moles of KHP.

2. Calculate the molarity of NaOH for each trial.

3. Calculate the average molarity of NaOH.

Analysis of unknown solid mixture

1. Calculate the % KHP for each trial.

2. Calculate the average % KHP.

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ANALYSIS OF A SOLID MIXTURE

Questions/Problems

1. If the unknown solid were not dried before analysis, would the calculated percent KHP be too high or too low? Explain.

2. List all of the indeterminate errors in this experiment that you can think of. Which of these is most important in determining the precision of the final result? (Use percent errors to evaluate the relative importance of the various errors.) Assume that the precision inherent in the various measuring devices are:

EQUIPMENT	PRECISION
Balance	0.2 mg
Buret	0.02 ml
Pipet	0.01 ml

3. Using this information, estimate the precision to be expected on the calculated percent KHP. To do this, use the following data:

   a. Mass of pure KHP
      i. Weighing bottle plus KHP: 21.5673 g
      ii. After KHP sample removed: 21.2106 g
      iii. Mass of KHP used: _____ g

   b. Volume of NaOH required to titrate KHP sample Buret contains NaOH solution
      i. Final buret reading: 20.10 ml
      ii. Initial buret reading: 2.45 ml
      iii. Volume of NaOH used: _____ ml

   c. Mass of unknown
      i. Mass of weighing bottle plus unknown: _____ g
      ii. Mass of bottle minus unknown: _____ g
      iii. Mass of unknown used: _____ g

   d. Volume of NaOH required to titrate the unknown solid sample Buret contains NaOH solution
      i. Final buret reading: 14.56 ml
      ii. Initial buret reading: 0.43 ml
      iii. Volume of NaOH used: ____ ml

Remember that precision accumulates as follows:
   e. when two numbers are multiplied, the **percent** precisions add, and
   f. when two numbers are added or subtracted, the **absolute** precisions add.

4. How does this accumulated precision compare with your observed standard deviation? Should they be comparable?

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ANALYSIS OF A SOLID MIXTURE

Questions/Problems (Page 2)

5. List the systematic (determinate) errors which might be present in this experiment. Do these errors affect the precision of the experiment? What do they affect?

6. How do your results from the phenolphthalein titrations compare to the results from the computer titrations? Does one method seem to be more accurate than the other? More precise? Is there a systematic offset between the two methods?

7. The most critical number in the experiment (in terms of giving an accurate end result) is the drop size. Your drop size should have been somewhere around 0.035 ml per drop (it does not need to be exactly this--drop size varies from buret to buret, but it should not be more than 10% oft). Calculate the propagated error in the final unknown determination assuming an uncertainty in this value of ± 0.0005 ml/drop. Calculate the relative uncertainty, and comment on the meaning of this.

ANALYSIS OF A SOLID MIXTURE

Tips and Traps

1. Students must use boiled, deionized water for their solutions.

2. It is helpful to show students the proper titration set-up. The tip of the buret should be 1-2 cm above the drop counter for optimum results.

3. Students should take time to align the counter properly. Most problems in the titrations result from a poorly aligned counter. No program is necessary for alignment. All students need to do is start the buret dripping and watch the counter light on the drop counter or the interface. If it blinks at each drop, alignment is correct.

4. The KHP should be dried at 110 °C for one hour.

5. Unknown mixture should be dried at l00 °C for one hour.

6. It is easiest to take all data for all trials at one sitting instead of making one solution at a time.

7. Unknown may have lumps. These should be crushed before drying.

8. Using the *MicroLAB* to get a derivative curve is discussed in the Appendix.

There is often a lot of noise in the derivatives because the collected data is not smooth, i.e., it fluctuates up and down as a function of the drop rate and the mixing rate, as seen in the Figure below under **Sample Data**. This can be avoided by using the ***pH,temp.vs.drop.titr.0.1pH.exp*** program, which collects the data only at every 0.1 pH units. The difference in the first derivative can be seen in Figure below under **Sample Data**.

ANALYSIS OF A SOLID MIXTURE

Suggested Answers to Questions/Problems

1. If the unknown solid were not dried before analysis, would the calculated percent KHP be too high or too low? Explain.

   Too low - the initial mass would include water (moisture).

2. List all of the indeterminate errors in this experiment that you can think of. Which of these is most important in determining the precision of the final result? (Use percent errors to evaluate the relative importance of the various errors.) Assume that the precisions inherent in the various measuring devices are:

EQUIPMENT	***PRECISION***	
Balance	***0.2 mg***	***Weighing: - 0.1% error***
Buret	***0.02 ml***	***Volume (Buret): -0.2% error***
Pipet	***0.0l ml***	

Weighing samples of pure KHP and the solid mixture. Measuring volumes with the buret.
The percent error in measuring volumes is higher for this experiment.

Using this information, estimate the precision to be expected on the calculated percent KHP. To do this, use the following data:

a. Mass of pure KHP
   i. Weighing bottle plus KHP: 21.5673 g
   ii. After KHP sample removed: 21.2106 g
   iii. Mass of KHP used: ***0.3567 g***

b. Volume of NaOH required to titrate KHP sample Buret contains NaOH solution
   i. Final buret reading: 20.10 ml
   ii. Initial buret reading: 2.45 ml
   iii. Volume of NaOH used: ***17.65 ml***

c. Mass of unknown
   i. Mass of weighing bottle plus unknown: _____ g
   ii. Mass of bottle minus unknown: _____ g
   iii. Mass of unknown used: _____ g

d. Volume of NaOH required to titrate the unknown solid sample Buret contains NaOH solution
   i. Final buret reading: 14.56 ml
   ii. Initial buret reading: 0.43 ml
   iii. Volume of NaOH used: ***14.13 ml***

   How does this accumulated precision compare with your observed standard deviation? Should they be comparable?

Mass of pure KHP = 0.3567 g± 0 .0004 g **(.11%)**
Volume of NaOH = 17.65 ml ± 0.04 ml **(.23%)**
M NaoH = 0.09896 ± 0.0003 ml **(.34%)**

***Mass of unknown solid* = *0.3326 g* ± *0 .0004 g* (.11%)**

ANALYSIS OF A SOLID MIXTURE

Suggested Answers to Questions/Problems

***Volume of NaOH* = 14.13 *ml* ± *0.04 ml* (.28%),**
KHP in unknown* = 1.398×10^{-3} *moles* ± 5×10^{-6} *moles
***Mass of KHP in unknown* = *0.2855 g* ± *0.0011 g.* (.39%)**

Accumulated precision should be greater than standard deviation. Indeterminate errors tend to cancel in multiple trials (that's the idea of doing several determinations).

3. List the systematic (determinate) errors which might be present in this experiment. Do these errors affect the precision of the experiment? What do they affect?

Systematic errors:
(a) Balance weighs heavy or light
(b) Buret calibration marks may be inaccurate.

Systematic errors affect the accuracy of the result, not its precision.

4. How do your results from the phenolphthalein titrations compare to the results from the computer titrations? Does one method seem to be more accurate than the other? More precise? Is there a systematic offset between the two methods?

Answers will vary depending on technique.

5. The most critical number in the experiment (in terms of giving an accurate end result) is the drop size. Your drop size should have been somewhere around 58 ml per drop (it does not need to be exactly this--drop size vary from buret to buret, but it should not be more than 10% off). Calculate the propagated error in the final unknown determination assuming an uncertainty in this value of 1.0 ml/drop. Comment on the meaning of the uncertainty.

$$\frac{(0.0005\ \text{ml})(100)}{(0.035\ \text{ml})\ \ (100)} = 1.4\ \%$$ **relative uncertainty error**

Based on the above uncertainties, this certainly is the largest. These uncertainties will add, so the accumulated error over 400 drops will be very large. This demonstrates why it is so important to minimize drop-size uncertainty.

ANALYSIS OF A SOLID MIXTURE

Sample Data

Calibration of drop size

Equation for conversion from drops NaOH to ml NaOH:

Standardization of NaOH - indicator titration

	Trial 1	Trial 2
mass KHP	0.2916 g	0.2728 g
initial buret reading	2.66 ml	1.42 ml
final buret reading	16.80 ml	14.70 ml
volume of NaOH used	14.14 m	13.28 ml
molarity NaOH	0.1010 M	0.1006 M

Standardization of NaOH - ***MicroLAB*** titration

	Trial 3	Trial 4
data file name	_____	_____
mass KHP	0.2162 g	0.2170 g
molarity NaOH	M	0.1015 M

Average molarity for all four titrations _____

Standard deviation for all four titrations _____

Analysis of unknown solid mixture - indicator titration

Unknown #

	Trial 1	Trial 2
mass unknown	0.9832 g	0.9964 g
initial buret reading	2.31 ml	3.12 ml
final buret reading	19.48 ml	20.63 ml
volume of NaOH used	17.17 ml	17.51 ml
% KHP in unknown	36.05 %	36.28 %

ANALYSIS OF A SOLID MIXTURE

Sample Data

Analysis of unknown solid mixture - MicroLAB titration

	Trial 3	Trial 4
data file name	_____	_____
mass unknown	0.9928 g	g
Equivalence Pt. vol.	_____ ml	_____ ml
% KHP in unknown	35.97%	36.62 %

Average % KHP from all four titrations _____

Standard deviation for all four titrations _____

Calculations

Standardization of NaOH

1. Determine the moles of KHP.

$$\frac{(0.2762\ g\ KHP)(1\ mol\ KHP)}{204.23\ g\ KHP} = 1.352 \times 10^{-3}\ mol\ KHP$$

2. Calculate the molarity of NaOH for each trial.

$$\frac{(1.352 \times 10^{-3}\ mol\ KHP)(1\ mol\ NaOH)(1)(0.1013\ M\ NaOH)}{1\ mol\ KHP \qquad 1\ L\ NaOH} = 0.0335\ L\ NaOH$$

3. Calculate the average molarity of NaOH.

$$\frac{0.1010\ M + 0.1013\ M + 0.1015\ M}{3} = 0.1013\ M$$

4. Calculate the % KHP for each trial.

$$\frac{(0.01717\ L\ NaOH)(0.1013\ mol\ NaOH)(1\ mol\ KHP)(204.23\ g\ KHP)}{(L\ NaOH) \qquad (1\ mol\ NaOH) \quad 1\ mol\ KHP} = 0.3552\ g\ KHP$$

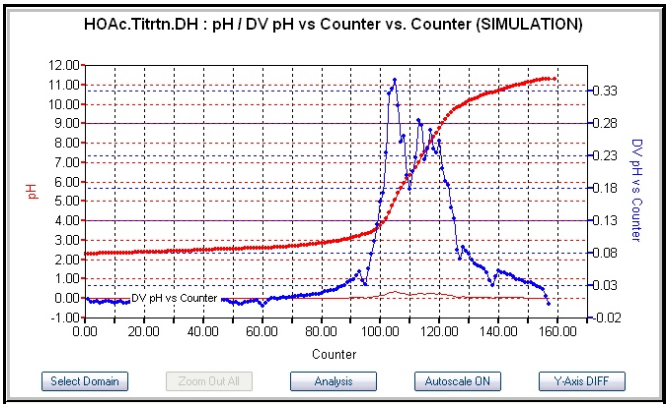
$$\frac{0.3552\ g\ KHP \times 100}{0.9832\ g\ mixture} = 36.13\%\ KHP$$

5. Calculate the average % KHP.

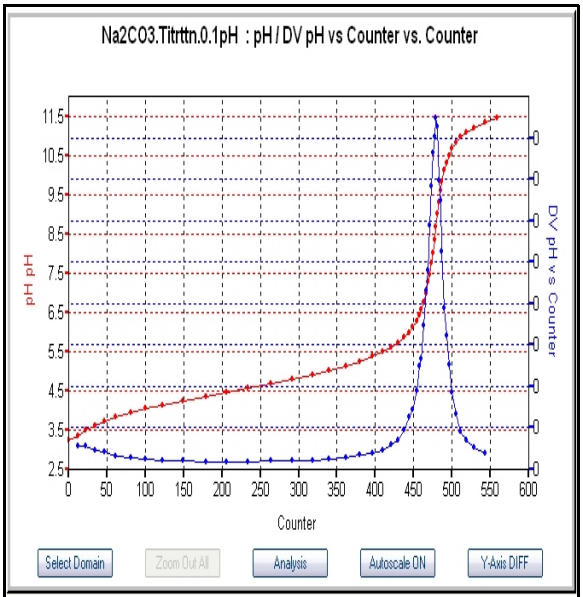
$$\frac{36.13\% + 35.97\% + 36.62\ \%}{3} = 36.24\%$$

ANALYSIS OF A SOLID MIXTURE

Sample *MicroLAB* Main Screen with Drop Counter Program



Titration curve of HOAc, with pH versus drop counter values. Note that the curve is not as smooth, and the first derivative is quite jagged.



Titration curve of HOAc with pH versus drop counter values. In this titration the pH was taken only after the pH had changed by 0.1 pH units. Note that the curve is much smoother, and the first derivative is classic in shape.

Titration Program

Acid/Base Titration, data taken every 0.1 pH units. Use for any titration of an acid with a base, or a base with an acid, recording the data each time the pH has changed by 0.1 pH units. Use of this program results in very smooth titration curves and generally very well shaped derivatives.
Experiment 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; **Temp**: Y2 axis, Col C, DD on bottom, units °C. (Use of temperature at instructors discretion.)

Special Program:
Read Sensors
*Repeat when counter change (*Sets to read only when a drop has passed through the counter.*)*
    If Delta pH > +/- 0.100 (Sets to read only when pH has changed by 0.1 pH units.)
        Read Sensors (Reads all variables selected in Data Sensors/Variables and stores in a data grid.)
    Else
    End If
Until Stop Button is pressed

Comment: Calibrate the drop counter using one of the ***dropcal.exp*** before and after the series of titrations. If temperature is measured with a Temp(IC) probe, it must be wrapped in Saran Wrap to prevent grounding the pH probe.

ANALYSIS OF A SOLID MIXTURE

Laboratory Preparation (per student station)

Equipment

- pH electrode
- ring stand
- ***MicroLAB*** drop counter
- buret clamp
- buret
- Nalgene bottle
- wash bottle (for NaOH)
- 250 ml beakers

Supplies

- paper towels

Chemicals

- KHP (3.5 g)
- unknown mixture (2.5 - 2.6 g samples), 35% - 75%% KHP, Purchase from: Thorn Smith Labs ,7755 Narrow Gauge Road, Beulah, MI.49617, Phone: (616) 882-4672 Fax: (616) 882-4804
- 6 *M* sodium hydroxide stock solution to prepare 500 ml of 0.1 M NaOH
- buffer solution (pH 4, 7 AND 10)

Safety and Disposal

- no special precautions needed Acids and bases can be flushed down the drain with lots of water.