Analysis of Soda Ash

INSTRUCTOR RESOURCES

A CCLI Initiative

Learning Objectives

The objectives of this experiment are to . . .

- understand the titration curve for a diprotic weak base.
- use the titration curve to calculate the percent Na_2CO_3 and $NaHCO_3$ in an unknown sample of soda ash.

Procedure Overview

- after calibration of the drop size and the pH electrode, a hydrochloric acid solution is standardized against pure Na_2CO_3
- an unknown sample of soda ash is analyzed for its Na_2CO_3 content using the standardized HCl.

Name _____ Section _____ Date _____

ANALYSIS OF SODA ASH

Report Sheet

Calibration of drop size

Equation for conversion from drops of HCl to ml of HCl:

Standardization of HCl

	Trial 1	Trial 2	Trial 3
mass of Na_2CO_3	_____ g	_____ g	_____ g
volume of HCl	_____ ml	_____ ml	_____ ml
data file name	_____	_____	_____
molarity of HCl	_____	_____	_____

Analysis of unknown soda ash

Unknown # _____

	Trial 1	Trial 2	Trial 3
mass of soda ash	_____ g	_____ g	_____ g
volume of HCl	_____ ml	_____ ml	_____ ml
data file name	_____	_____	_____
mass of Na_2CO_3	_____	_____	_____
% Na_2CO_3	_____	_____	_____

Name _____ Section _____ Date _____

ANALYSIS OF SODA ASH

Report Sheet (page 2)

Calculations

Standardization of HCl

1. Determine the moles of Na_2CO_3.

2. Calculate the molarity of HCl for each trial.

3. Calculate the average molarity for HCl.

Titration of soda ash unknown

1. Calculate the average percent Na_2CO_3 in the soda ash unknown.

Name _____ Section _____ Date _____

ANALYSIS OF SODA ASH

Questions/Problems

1. The assay of a soda ash sample was reported as 65.21% Na_2CO_3. Express the carbonate content in terms of percent carbon dioxide and percent carbonate ion.

2. A 0.166 g sample of an unknown pure carbonate salt was dissolved in 50.0 ml of 0.215 *M* HCl. The solution was boiled to remove dissolved carbon dioxide and backtitrated with 39.9 ml of 0.213 *M* NaOH. What is the cation in the carbonate salt?

3. A 0.322 g sample of soda ash required 15.7 ml of 0.130 *M* HCl to reach the first equivalence point, and an additional 22.1 ml volume of the acid to reach the second equivalence point. What is the percent Na_2CO_3 and percent $NaHCO_3$ in the sample?

4. In a soda ash analysis, indicate how each of the following would affect the reported values for Na_2CO_3 and $NaHCO_3$?

   (a) the hydrochloric acid was actually 0.1053 *M* though believed to be 0.1104 *M*.

   (b) the endpoint was overrun by 0.25 ml.

   ©) the actual mass of sample was 0.006 g higher than believed.

   (d) there is an error of +0.05% in the volume delivered.

   (e) the sample was dissolved in 100 ml of boiled deionized water instead of 80 ml.

ANALYSIS OF SODA ASH

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 0.5 cm above the drop counter for optimum results and aligned with the cross hairs such that the red light flashes with each drop. The drop counter is then properly aligned.

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 interface. If it blinks at each drop, alignment is correct. Be sure to clamp everything tight at that point so accidental movement does not cause loss of drops.

4. The Na_2CO_3 and soda ash unknown should be dried at 110 C for two hours.

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

6. Be sure to include the instructions on setting up and calibrating the drop counter, and performing drop counter titrations from the **Useful Titration Operations** file, obtained from the **Useful Tools** folder on the CD.

ANALYSIS OF SODA ASH

Sample Data (page 2)

Calibration of drop size

Sample equation for conversion from drops of HCl to ml of HCl.

$$\frac{\text{\# titration drops} \mid \text{Cal. vol.} \mid}{\text{Cal. drops}} = \text{ml titrant}$$

Standardization of HCl

	Trial 1	Trial 2	Trial 3
mass of Na_2CO_3	0.2488 g	0.3722 g	0.2873 g
volume of HCl	49.00 ml	75.14 ml	57.94 ml
data file name	_____	_____	_____
molarity of HCl	0.0958 *M*	0.0935 *M*	0.0936 *M*

Analysis of unknown soda ash

Unknown # _____

	Trial 1	Trial 2	Trial 3
mass of soda ash	0.5001 g	0.4965 g	0.4478 g
volume of HCl	42.50 ml	42.57 ml	38.74 ml
data file name	_____	_____	_____
mass of Na_2CO_3	_____	_____	_____
% Na_2CO_3	42.45	42.83	43.22

ANALYSIS OF SODA ASH
Sample Data (page 2)

Calculations

Standardization of HCl

1. Determine the moles of Na_2CO_3.

$$\frac{\#\ ml\ HCL \mid \#\ mmol\ HCL \mid 1\ mmol\ Na_2CO_3 \mid}{1\ ml\ HCL \mid 2\ mmol\ HCL \mid} = mmol\ Na_2CO_3$$

2. Calculate the molarity of HCl for each trial.

$$\frac{X\ g\ Na_2CO_3 \mid 1 mol\ Na_2CO_3 \mid 2\ mol\ HCL \mid}{\mid 222\ g\ Na_2CO_3 \mid 1\ mol\ Na_2CO_3 \mid Y\ L\ HCL} = Z\ M\ HCL$$

3. Calculate the average molarity for HCl.

$$0.0958\ M + 0.0935\ M + 0.0936\ M = 0.0943\ M$$

Titration of soda ash unknown

1. Calculate the average percent Na_2CO_3 in the soda ash unknown.

$$42.45 + 42.83 + 43.22 = 42.83\ \%\ Na_2CO_3$$

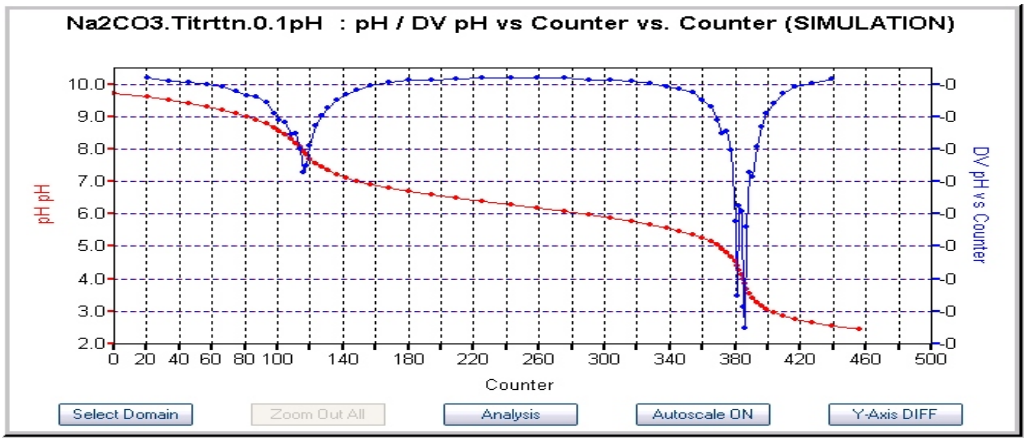
ANALYSIS OF SODA ASH

Sample Data (page 3)

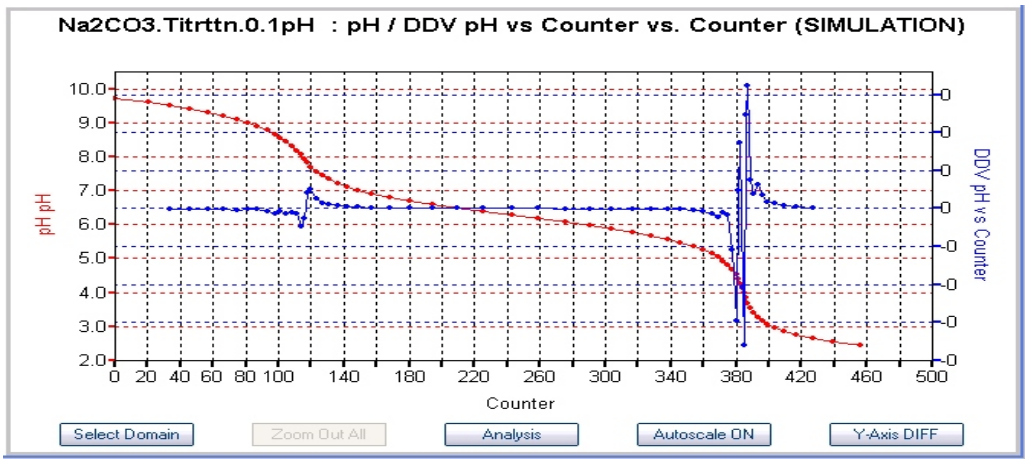
Using *MicroLAB* to get a derivative curve

See the discussion in **The Measurement Manual** for all details for operation of the ***MicroLAB*** interface.

Sample titration curve for Na_2CO_3, with first derivative curves, data at 0.1 pH intervals.



Sample titration curve for Na_2CO_3, with second derivative curves, data at 0.1 pH intervals.



If there are not smooth transitions along the titration curve, the derivatives may be noisy, with a lot of hash up and down. The large peak should be roughly symmetrical, and there should be a definite highest point. Ideally, there should be one large peak at the equivalence point. This can be overcome by programming the MicroLAB to take points every 0.1 pH units, as was done in the sample titration shown here. This smooths the curve and gives more ideal derivative curves. The second derivative will give the exact volume of titrant required to reach the equivalence point by interpolation. See the Measurement Manual for this information.

ANALYSIS OF SODA ASH

Suggested Answers to Questions/Problems

1. The assay of a soda ash sample was reported as 65.21% Na_2CO_3. Express the carbonate content in terms of percent carbon dioxide and percent carbonate ion.

   27.09% CO_2 ***36.93% CO_3^{2-}***

2. A 0.166 g sample of an unknown pure carbonate salt was dissolved in 50.0 ml of 0.215 *M* HCl. The solution was boiled to remove dissolved carbon dioxide and back titrated with 39.9 ml of 0.213 *M* NaOH. What is the cation in the carbonate salt?

   $SrCO_3$

3. A 0.322 g sample of soda ash required 15.7 ml of 0.130 *M* HCl to reach the first equivalence point, and an additional 22.1 ml volume of the acid to reach the second equivalence point. What is the percent Na_2CO_3 and percent $NaHCO_3$ in the sample?

   ***67.14%* Na_2CO_3** ***21.71%* $NaHCO_3$**

   plus impurities

4. In a soda ash analysis, indicate how each of the following would affect the reported values for Na_2CO_3 and $NaHCO_3$?

   (a) the hydrochloric acid was actually 0.1053 *M* though believed to be 0.1104 *M*.

   Calculated percents are too high.

   (b) the endpoint was overrun by 0.25 ml.

   Calculated percents are too high.

   ©) the actual mass of sample was 0.006 g higher than believed.

   Calculated percents are too high.

   (d) there is an error of +0.05% in the volume delivered.

   Calculated percents are too high.

   (e) the sample was dissolved in 100 ml of boiled deionized water instead of 80 ml.

   no effect

ANALYSIS OF SODA ASH

Laboratory Preparation (per student station)

Equipment

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

Supplies

- paper towels

Chemicals

- pure Na2CO3 (0.75 g)
- unknown soda ash samples (1.35 ± 0.1 g)
- Obtain from: Thorn Smith Labs, 7755 Narrow Gauge Road, Beulah, MI. 49607, Phone: (618) 882-4672, Fax: (618) 882-4804
- buffer solution (pH 7.0)
- 0.1 *M* HCl solution (1 liter), can be prepared by students from 12 *M* HCl (8.4 ml to 1 L solution) or prepared in bulk by stockroom for student use
- 50% (w/w) NaOH solution

Safety and Disposal

- no special precautions necessary. Neutralize the solutions and flush down the drain with lots of water.