

## EXPERIMENT: DETERMINATION OF SODA ASH (pH)

### INTRODUCTION

The measurement of soda ash in industrial samples is important. In this experiment, an HCl solution is prepared and standardized with dried sodium carbonate. The standardized HCl is then used to analyze a dried soda ash sample.

In the following procedures both indicator and pH meter methods (potentiometric titration) will be used.

### PROCEDURE:

**Indicator Method** (note that the indicators are added only for reference purposes; the important data are the pH readings)

Prepare 500 mL of a 0.1M HCl (conc. HCl stock is approx. 12.1 M) solution by diluting the appropriate quantity of conc. HCl with water. Standardize against sodium carbonate as follows.

Weigh accurately two samples of 0.2g pure, dried sodium carbonate (by difference) into a 250ml Erlenmeyer flask. Add 50mL distilled water and 1-2 drops phenolphthalein indicator. Titrate slowly (0.5mL/sec) until the pink color disappears. This is a slight excess for half the volume of acid required for total neutralization. Add two drops of methyl orange indicator, continue titrating until the color changes from straw to pale orange to pink.

Now weight accurately two 0.3 to 0.4g samples of the dried unknown soda ash and titrate with the standardized acid in duplicate with indicators as above.

At the start of the experiment on the unknown, the student should run a quick, approximate titration using the indicators to establish the approximate location of the end points and the color changes to be expected. With this information, the student should be able in the HCl required for the particular end-point, and then carefully add the remaining 10% of the HCl slowly so that an accurate endpoint is obtained.

### pH Meter Titration

Weigh accurately 0.2g pure, dried sodium carbonate (by difference) into a 400mL beaker provided with a magnetic stirring bar. Add 50mL water, dissolve, and add several drops of phenolphthalein indicator. Insert a glass and a calomel electrode into the solution, avoiding contact with the stirring bar. Add titrant and take readings until the phenolphthalein indicator changes color. In performing this titration it is best to take points at 2- 3mL intervals between the end points, and at 0.2mL increments near the end points. Add methyl orange indicator and continue the titration until the color changes again. Record the end points. Exceed the final equivalent volume with an additional 10ml of titrant. Do this titration rapidly, because the pH will tend to drift as CO<sub>2</sub> escapes from the solution.

Now weigh accurately 0.3 to 0.4 g of the dried soda ash unknown. Titrate, following the above procedure using the pH meter. For each pH meter titration, plot a curve of pH versus volume of titrant, and the Gran plot. Calculate your results based on the total volume to the second end point\* as if the soda ash unknown were sodium carbonate and contained no sodium bicarbonate. Report the results as % sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) and as % sodium oxide ( $\text{Na}_2\text{O}$ ) .

### Possible Sources of Determinate Errors.

If you compare the pH at the first end point with the pH at which you observed the phenolphthalein end point, you may find that the indicator color changes, not at the end point, but by as much as 3% to 5% of the volume of titrant after the endpoint. (For example, the equivalence point may occur at pH 8.4, whereas the red color may disappear at pH 8.0). This has some interesting consequences. One is that, with indicator, the ratio of the volumes of acid at the second to the first end point is not exactly '2' but about 1.9 (check your indicator data in the textbook to see whether the discrepancy occurs and if it agrees with this discussion). Another major difference between the two end points is that of precision. Refer to your data obtained from the pH meter titrations. The main factor influencing precision will be the ratio of changes in pH to changes in volume of added titrant at the first and second end points. To see this, examine the pH vs. V (HCl) graph. At each end point measure the V (HCl) at [pH (1)+0.5] and at [pH (1)-0.5], where pH (1) is the pH at the first end point (1), and similarly at the second end point (2). From this, calculate V (HCl) needed to change the pH by +/- 0.5 for the first and second end points.

\*Calculate your results based on a Gran Plot (see Section 12-5 of your textbook for further discussion of the theory). The Gran Plot is based on the following equation:

$$V_a \cdot 10^{+\text{pH}} = \{(1/K_a)(\gamma_B/\gamma_{\text{BH}^+})\}(V_e - V_a)$$

where  $V_a$  = is the volume of strong acid added, pH = is the pH meter reading,  $K_a$  = acid dissociation constant of the acid form of the base,  $\text{BH}^+$ , B is the weak base being titrated, and  $V_e$  = equivalence point. Note that the above equation resembles that for a straight line:  $y = mx + b$ , for regions not very near the equivalence point. The variables are ( $V_a \cdot 10^{+\text{pH}}$ ) for the ordinate (y-axis) and ( $V_a$ ) for the abscissa (x-axis). It is not necessary to know the actual values of the other parameters,  $K_a$ ,  $\gamma_B$ ,  $\gamma_{\text{BH}^+}$  and  $V_e$ . By extrapolating the straight line portion of the graph to obtain the x-intercept, one can determine the value of  $V_e$  more accurately than by a first derivative curve.

The above equation simplifies to:  $y = mx + b$  by equating

$$y = (V_a \cdot 10^{+\text{pH}}), m \text{ (the slope)} = -\{(1/K_a)(\gamma_B/\gamma_{\text{BH}^+})\}, x = (V_a) \text{ and}$$

p. 3 of 4

$b = \left( \frac{1}{K_a} \right) \left( \frac{\gamma_B}{\gamma_{BH^+}} \right) (V_e)$ . The x-intercept occurs when  $y=0$ . That occurs when  $V_a = V_e$ . It is not necessary to know the slope. \*\*\*Submit a copy of your graph with the informal results for this experiment.

**Waste Disposal:** Discard all waste in appropriate bottles as instructed. If you have any questions, please see your instructor or teaching assistant before disposing of waste.

**Informal report:**

Experiment: Determination of Soda Ash

date submitted: \_\_\_\_\_

time submitted: \_\_\_\_\_

Name: \_\_\_\_\_

Values obtained:

(1) \_\_\_\_\_ %

Locker # \_\_\_\_\_

(indicator method)

(2) \_\_\_\_\_ %

(Gran plot)

(3) \_\_\_\_\_ %

Average value = \_\_\_\_\_ %

(if applicable) Standard deviation = \_\_\_\_\_ %

	I (indicator)	II(indicator)	III(pH meter)
g dried Na <sub>2</sub> CO <sub>3</sub> used for standard solution:	_____	_____	_____ g

Standardization titration:

mLs of HCl added to reach 1 <sup>st</sup> end point	_____	_____	_____ mL
---	-------	-------	----------

mLs of HCl added to reach 2 <sup>nd</sup> end point	_____	_____	_____ mL
---	-------	-------	----------

Molarity of the HCl standard: \_\_\_\_\_ M

Sample calculation:  $M_{HCl} =$

Unknown titration:

	I (indicator)	II(indicator)	III(pH meter)
g dried unknown sample:	_____	_____	_____ g

mLs of HCl added to reach 1 <sup>st</sup> end point	_____	_____	_____ mL
---	-------	-------	----------

mLs of HCl added to reach 2 <sup>nd</sup> end point	_____	_____	_____ mL
---	-------	-------	----------

% Na <sub>2</sub> CO <sub>3</sub> in unknown: %	_____	_____	_____
---	-------	-------	-------

Sample calculation: At the 2<sup>nd</sup> equivalence point, milliequivalents, mEq of titrant, A = mEq analyte, B  
 at 2<sup>nd</sup> end point: mEq HCl = mEq Na<sub>2</sub>CO<sub>3</sub>; #mols HCl = 2 x (#mols Na<sub>2</sub>CO<sub>3</sub>);

$$M_{HCl} V_{HCl} = 2(g \text{ Na}_2\text{CO}_3) / (\text{FW of Na}_2\text{CO}_3) \Rightarrow g \text{ Na}_2\text{CO}_3 = (M_{HCl} V_{HCl}) (\text{FW of Na}_2\text{CO}_3) / 2$$

$$\% \text{ Na}_2\text{CO}_3 = (g \text{ Na}_2\text{CO}_3) (100\%) / (g \text{ of unknown sample})$$

FW of Na<sub>2</sub>CO<sub>3</sub> = 105.99 g/mol; FW of Na<sub>2</sub>O = 61.98 g/mol;