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
Prepare 500 mL of a 0.1 M HCl (conc. HCl stock is approx. 12.1 M) solution by diluting the appropriate quantity of conc. HCl with distilled water. Fill your burette with the HCl solution and standardize it against sodium carbonate as follows.

Weigh accurately two samples of 0.1g pure, dried sodium carbonate (by difference) into two 250ml Erlenmeyer flasks. Add 50mL distilled water and 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 2 drops of methyl orange indicator and continue titrating until the color changes from straw to pale orange to pink. Calculate the molarity of the HCl solution, which is standardized.

Now weigh accurately two 0.2 to 0.3g samples of the dried unknown soda ash and titrate with the standardized acid in duplicate with indicators as above (note: 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 to estimate the required HCl for the particular end-point, and then carefully add the remaining 10% of the HCl slowly so that and accurate endpoint is obtained.)

pH Meter Titration (note: the important data are the pH readings; the indicators are added only for reference purposes.)
Weigh accurately 0.1-0.2g pure, dried sodium carbonate (by difference) into a 400mL beaker provided with a magnetic stirring bar. Add 50mL water, dissolve, and add 2 drops of phenolphthalein indicator. Insert a glass and calomel electrode into the solution (note: avoid contact by the elctrodes with the stirring bar). Add titrant and record pH readings in approximately equal steps. To determine the volume step size to be added for each reading, take the total volume of titrant needed to reach the second endpoint in your indicator titrations and divide this value by 25. Proceed with the pH meter titration by adding the step size volume and recording the pH from the meter. The pH value will change rapidly as the titration passes through the first and second endpoints. Once you have passed the second endpoint (the pH value should be less than 3.0) add five more volumess of titrant, recording the pH value for each additions. Do this titration rapidly, because the pH will tend to drift as CO_2 escapes from the solution.

Now weigh accurately 0.2 to 0.3 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 (Na_2CO_3) and as % sodium oxide (Na_2O) *.

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. Vol. of HCl graph. At each end point, measure the Vol. of HCl at (pH+0.5) and at (pH-0.5). From these, calculate Vol. of 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:

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

➔ y = $m \cdot x + b$

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, 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 $V_a \cdot 10^{+pH}$ 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 , γ_B , γ_{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
$y = V_a \cdot 10^{+pH}$, m (the slope) = $-\{(1/K_a)(\gamma_B/\gamma_{BH+})\}$, $x = V_a$ and b = $(\{(1/K_a)(\gamma_B/\gamma_{BH+})\}(V_e))$. 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 <u>appropriate bottles</u> as instructed. If you have any questions, please see   your   instructor   or   teaching   assistant   before   disposing   of   waste.

Informal report: date submitted: _____
Experiment: Determination of Soda Ash   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_2CO_3 used for standard solution:	_____	_____	_____ g

Standardization titration:

	I (indicator)	II(indicator)	III(pH meter)
mLs of HCl added to reach 1st end point	_____	_____	_____ mL
mLs of HCl added to reach 2nd 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 1st end point	_____	_____	_____ mL
mLs of HCl added to reach 2nd end point	_____	_____	_____ mL
% Na_2CO_3 in unknown: %	_____	_____	_____

Sample calculation: At the 2nd equivalence point, milliequivalents, mEq of titrant,A =mEq analyte, B
        at 2nd end point: mEq HCl = mEq Na_2CO_3; #mols HCl = 2 x(#mols Na_2CO_3) ;
         $M_{HCl}V_{HCl}$=2(g Na_2CO_3)/(FWof Na_2CO_3) => g Na_2CO_3 =($M_{HCl}V_{HCl}$)(FWof Na_2CO_3)/2
        % Na_2CO_3 = (g Na_2CO_3)(100%)/(g of unknown sample)

        FW of Na_2CO_3 = 105.99 g/mol; FW of Na_2O = 61.98 g/mol;