Standardization of HCl solution and determination of sodium carbonate

Introduction:
In titration, it is critical to know the exact concentration of the titrant (the solution in the buret which will be added to the unknown) in order to determine the concentration of solutions being tested. Hydrochloric acid solutions can be standardized by two titrimetric methods; one is to standardize HCl solution against previously standardized sodium hydroxide and the other titrimetric method is to standardize HCl against primary-standard sodium carbonate. In this experiment, you will standardize the ~0.1 M HCl solution (the titrant) with sodium carbonate (Na_2CO_3) using methyl red as the indicator. Na_2CO_3 is a base and reacts with the strong acid HCl in the following way:

$$2HCl(aq) + Na_2CO_3\,(aq) \rightarrow NaCl\,(aq) + H_2O(l) + CO_2(g)$$

Sodium carbonate samples are commonly analyzed by titration with standard hydrochloric acid and reported as % sodium carbonate.

Experiment equipment/materials:

Bottles	Hot plates
250 mL Erlenmeyer flasks	Buret
10 mL graduated cylinder	Methyl red indicator*
Plastic pipet	0.1 M HCl solution (prepared by students)
32% HCl	Primary standard Na_2CO_3
Distilled water	Unknown samples
*Methyl red indicator: grind 0.1 g of the dye then dissolve in 3.7 mL 0.1 M NaOH, dilute to 100 mL H_2O.	

Procedure

1. Fill a 1 L bottle with about 500 mL distilled water, add 10 mL of 32% HCl solution using a graduated cylinder and add it to the bottle.
2. Fill the bottle to the mark with distilled water, mix well and keep covered.
3. **Standardize HCl as follows:**
   a. Weigh three 0.20xx g of previously dried primary-standard Na_2CO_3 (MW 106.00 g/mole) into three 250 mL Erlenmeyer flasks by difference — to avoid absorption of water from air — and dissolve it in 50 mL of distilled water (heating if necessary).
   b. Add 2-3 drops of methyl red indicator.
   c. Obtain a buret, wash it with water and soap, then rinse it 2 or 3 times with small portions of the HCl solution before filling it with it. Make certain that all air bubbles have been flushed from the tip before taking the initial volume reading. When reading the buret,

remember that you should **read** the number that is at the bottom of the meniscus and estimate each reading to the closest 0.01 mL.

d. Titrate the first Na_2CO_3 sample until the indicator has changed gradually from yellow to a definite red color. Add the HCl titrant rapidly at first, but slowly later as the endpoint is approached as indicated by the definite red color. Rinse down the sides of the flask to make sure that any splattered HCl gets a chance to react.

e. Boil the solution gently for 2 minutes to expel the CO_2 from solution that has been produced during the reaction. The color of the indicator should revert to yellow. Cover the flask with a watch glass, cool to room temperature, and continue the titration to a sharp change to red at the end point (this might take VERY little HCl so go slow!).

f. Record the volume of HCl used.

4. Repeat the above titration procedure for the other two Na_2CO_3 samples with the HCl.
5. **Calculate** the moles of Na_2CO_3, moles of HCl, molarity of HCl, average molarity of HCl, Average the molarities from the three different trials and calculate the standard deviation, G-test and 95% confidence interval.
6. <u>**Determination total carbonate of an unknown**</u>: obtain an unknown from your TA/lab instructor and record its letter.
7. <u>**Rough titration:**</u> weigh out 0.25xx – 0.35xx g sample of the impure Na_2CO_3 unknown sample into a clean 250 ml Erlenmeyer flask and add 50 ml of water and 2 or 3 drops of methyl red indicator. Titrate the unknown as rough titration as stated in step 2 above and record the HCl volume needed to reach the end point.
8. <u>**Accurate titration:**</u> repeat the rough titration procedure for other three Na_2CO_3 samples. (NOTE: *Do not discard the remaining HCl solution – you will use this for other experiment)*
9. **Calculate** the moles of HCl, moles of Na_2CO_3, grams of Na_2CO_3, % mass of Na_2CO_3, and average % mass of Na_2CO_3 of the three samples from accurate titration
10. **Calculate** the standard deviation, G-test and 95% confidence interval.

Data table 1: Standardization of HCl solution with primary-standard sodium carbonate (Na_2CO_3)

Name____ ID_________________

Partner name______________________________________ Date________________

	Trial 1	Trial 2	Trial 3
Mass of Na_2CO_3 (g)			
Initial buret reading (mL)			
Final buret reading (mL)			
Volume of HCl used (mL)			
Volume of HCl used (L)			
Moles of Na_2CO_3			
Moles of HCl			
Molarity of HCl			
Average Molarity of HCl			
Standard deviation in the HCl molarity			
95 % confidence interval			

Sample calculation: (show one sample calculation for each of the reported data—use additional paper if needed)

Data table 2: determination total CO_3^{2-} in an unknown sample

Name____ ID_________________

Partner name______________________________________ Date________________

Unknown letter: ____________	Trial 1	Trial 2	Trial 3
Mass of unknown (g)			
Initial buret reading (mL)			
Final buret reading (mL)			
Volume of HCl used (mL)			
Volume of HCl used (L)			
Molarity of HCl (M)			
Moles of HCl			
Moles of Na_2CO_3			
Moles of CO_3^{2-}			
Mass of CO_3^{2-} (g)			
% mass of CO_3^{2-} in unknown ___________			
Average % mass of CO_3^{2-} in unknown ________			
Standard deviation in the % mass of CO_3^{2-}			
95 % confidence interval			

Sample calculation: (show one sample calculation for each of the reported data—use additional paper if needed)

Example calculations:

The percentage of Na_2CO_3 in a sample can be calculated in two steps:

1- the determination of the molarity of the HCl titrant from the standardization titrations and

2- the calculation of the percentage of Na_2CO_3 from titrations of the unknown sample.

1. Molarity of HCl.

In titrations of Na_2CO_3 with HCl to the end point, 2 moles of HCl are added for each mole of Na_2CO_3:

$$2HCl + Na_2CO_3 \Longleftrightarrow H_2CO_3 + 2NaCl$$

The HCl molarity is obtained from the following relations:

Moles HCl =moles Na_2CO_3 x 2

Moles of Na_2CO_3 = grams of Na_2CO_3 used / MW of Na_2CO_3

Molarity of HCl = moles of HCl / Liters of HCl

The factor 2 is required because each mole of Na_2CO_3 reacts quantitatively with 2 moles of HCl.

2. Percentage of CO_3^{2-} in sample. The percentage of CO_3^{2-} is calculated as follows:

% mass CO_3^{2-}= (weight of CO_3^{2-}in sample)/ (g sample) x 100

Weight of CO_3^{2-} in sample = (moles CO_3^{2-}) x (mol. wt. CO_3^{2-})

Moles of CO_3^{2-} = moles of Na_2CO_3

Moles of Na_2CO_3 = moles of HCl / 2

Moles of HCl = (Liters of HCl) x (molarity HCl)

Remember: Poor results are often caused by errors in calculation rather than by faulty laboratory technique. Check all calculations and significant figures before reporting results. Ensure you have reported the correct unknown number.

Pre lab: Standardization of HCl solution and determination of sodium carbonate

Name____ ID_________________

Partner name_____________________________________ Date________________

1. Explain why primary-standard sodium carbonate should be weighed by difference.

2. What effect will boiling off carbon dioxide have on pH of the solution in the methyl red end point method?

3. 16.10 mL of 0.1114 M HCl is needed to titrate a 0.2350 g impure sodium carbonate. Calculate the % mass of carbonate in the sample.