Experiment 9. Thermodynamics of the Dissolution of Borax

Experimental Procedure

Lab 406

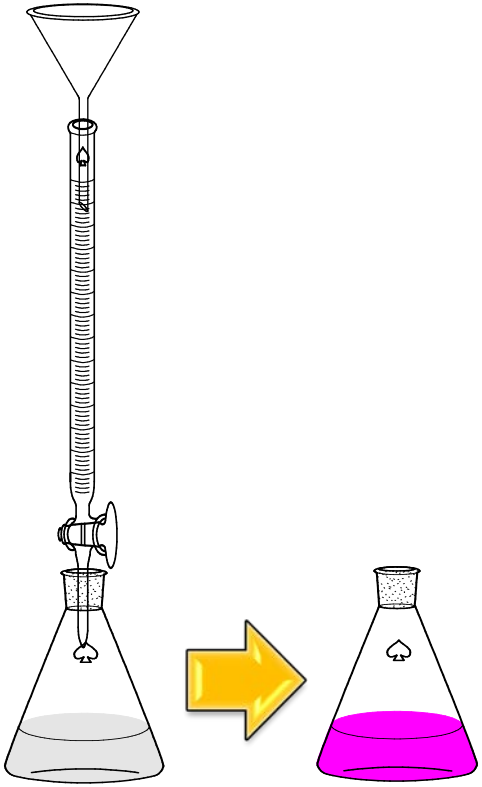
Overview

This experiment is to be complete in cooperation with other chemists/chemist groups in the laboratory. In PART A, a standardized solution of hydrochloric acid is to be prepared. In PART B, four warm water baths are to be set up at the beginning of the laboratory, each at a different temperature, but at a maximum 60^{o}C. The water baths then are to be shared. In conjunction with four warm water baths for PART B, about 150 mL of warm (~55^{o}C) deionized water is to be prepared for PART C.1. Begin those preparations.

A. Standardization of HCl Solution

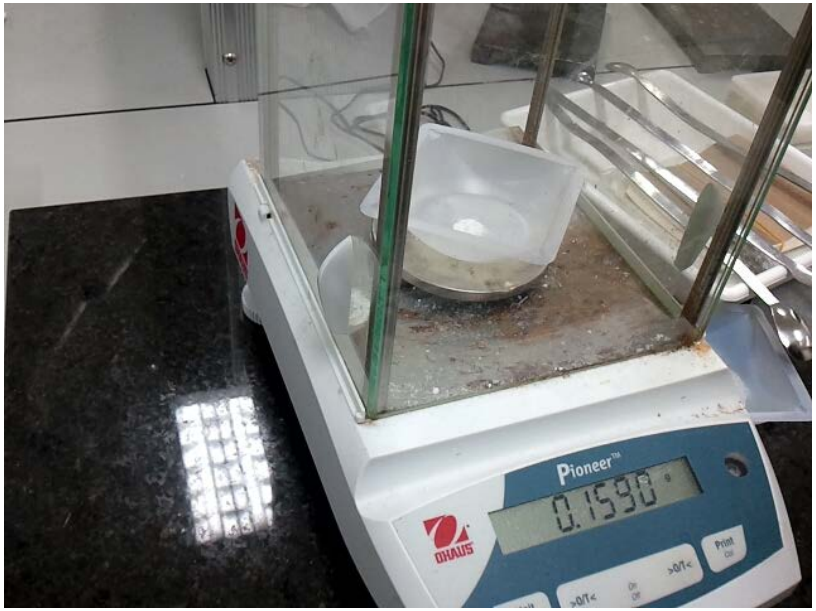
1. Prepare the HCl solution.

Prepare 0.20 M HCl solution in a 250 mL volumetric flask from 6 M HCl solution per experimental group)



2. Prepare the primary standard

Calculate the mass of sodium carbonate that ***neutralizes 15 mL of 0.20M HCl*** *at the stoichiometric point. Measure this mass on a tared piece of weighing paper or dish and transfer to a 125mL Erlenmeyer flask. Prepare at least three samples of sodium carbonate for the analysis of the HCl solution.*



3. Prepare the buret

Clean a buret and rinse with several 3-portions of the ~0.20M HCl solution. Use a clean funnel to fill the buret with the ~0.20M HCl solution and record the volume of HCl solution in the buret.

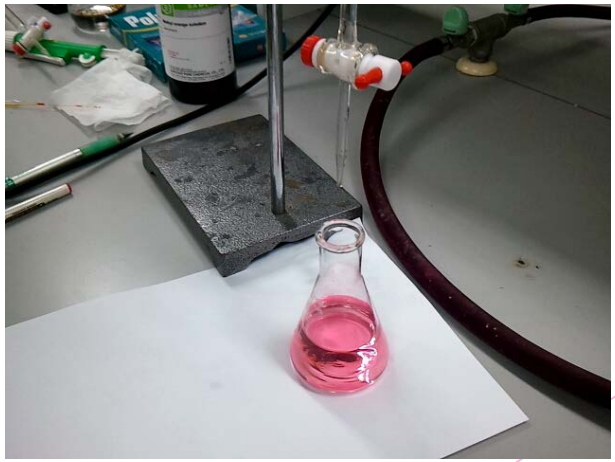
4. Titrate the primary standard

To each solid sodium carbonate sample, add ~50 mL of deionized water and several drops of methyl orange indicator. Place a sheet of white paper beneath the Erlenmeyer flask.

Dispense the HCl solution from the buret, swirling the Erlenmeyer flask during the addition. Carefully add additional HCl titrant until the endpoint is reached and the color persists for 30 seconds. (a color change caused by the addition of one additional drop of the HCl solution from the buret) Stop the HCl titrant. After 10-15 seconds, record the volume of HCl in the buret.

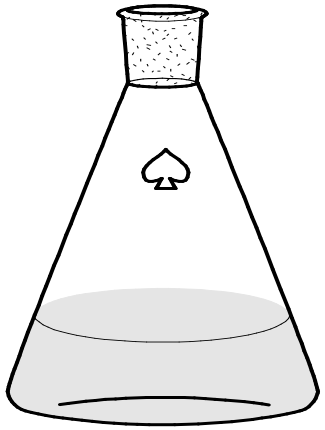
5. Repeat the analysis and do the calculations

Repeat two more analyses and calculate the molar concentration of the prepared HCl solution.



B. Preparation of Borax Solutions

[We will share water baths to keep a more accurate water temperature. Label the test tubes (tube number and your experimental group)].



~~1. Calibrate test tubes~~

~~*Pipet 5 mL of deionized water into six clean medium-sized test tubes. Mark the bottom of the meniscus. Discard the water and allow the test tubes to air- or oven-dry. Label the test tubes.*~~

2. Prepare stock solution of borax

Using a 250-mL Erlenmeyer flask, add ***35 g of borax to 100 mL of deionized water.*** ***A****gitate carefully the mixture for several minutes to prepare the saturated solution.*

3. Prepare the test solutions of borax

Label a set of six clean, medium-sized test tubes. (Test tube # and experimental group) Again, thoroughly agitate the borax stock solution and then half-fill this set of medium-sized test tubes with the stock solution. Place the test tubes in the respective baths ***(Bath 1:≈56°C, Bath2: ≈48°C, Bath3: ≈40°C, Bath4: ≈32°C, Your beaker: Ambient, yellow box: ≈5°C).***

4. Prepare saturated solutions of borax

a. Saturate the solutions. *Occasionally agitate the test tubes in the baths to make solution in each tubes saturated. Solid borax should always be present and add more if necessary.*

b. Allow sample to settle (not change temperature). *Allow the borax to settle until the solution is clear (this will require several minutes, be patient !) and has reached thermal equilibrium. Allow 10-15 minutes for thermal equilibrium to be established.*

≈ 56 ºC

≈ 48 ºC

≈ 40 ºC

≈ 32 ºC

Ambient

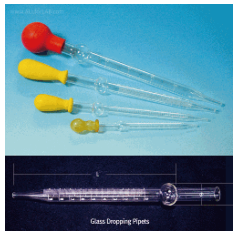
≈ 5 ºC (Ice in the yellow box)


56°C

C. Analysis of Borax Test Solutions

1. Transfer the samples

a. Prepare for sample transfer. *Set up and label a set of six clean labeled 250-mL Erlenmeyer flasks.*



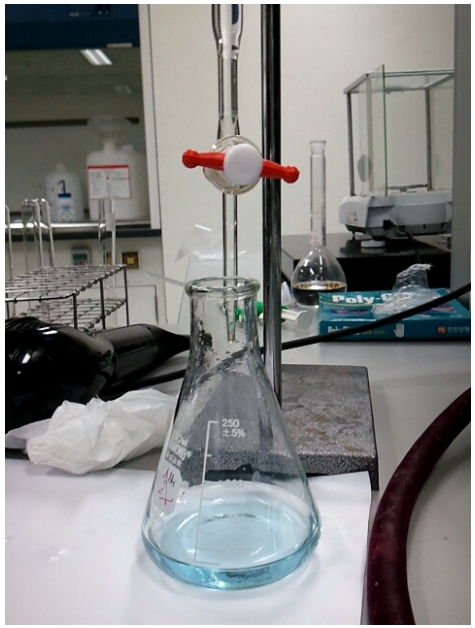
b. Transfer the samples with a 5-mL pipet. *After the sample is clear in the test tube, transfer the solution with a 5-mL pipet to the correspondingly labeled Erlenmeyer flask(Do not transfer any of the solid borax), rinse the pipet with two or three ~5 mL portions of warm, deionized water (~ 55 °C) and combine the washings with the sample.*

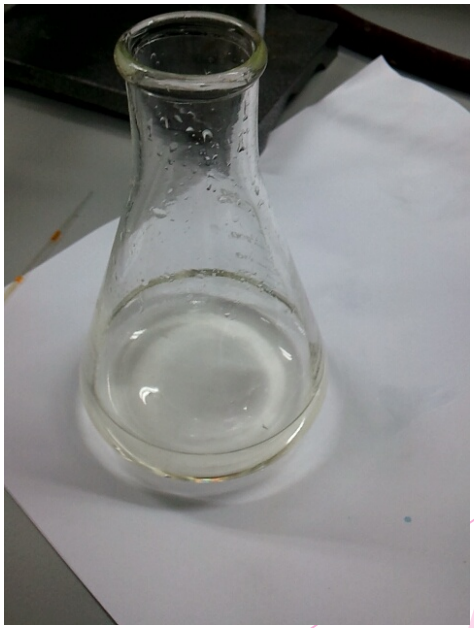
"The warm distilled water (50 ~ 55 °C)will be prepared in the assigned water bath. Also share it."

(Caution! Borax is stable in aqueous solutions at temperatures less than 60 °C; at higher temperatures, some dehydration of the $B_4O_5(OH)_4^{2-}$ anion occurs)

2. Titrate the samples.

Dilute each sample to about 25 mL with warm, deionized water. Add 2-3 drops of bromocresol green. Titrate each of the six samples to a yellow endpoint with the standardized HCl solution prepared in Part A. Remember to record the buret readings before and after each analysis of a sample.





D. Data Analysis(after experiment)

Six repeated calculations are required to establish the data plot for the determination of ΔH^o and ΔS^o for the dissolution of borax. The lengthy task of completing the calculations and for minimizing errors in the calculations can be reduced with the use of an Excel spreadsheet. The data from the calculations can then be plotted using the embedded graphing capabilities of Excel.

1. *Calculate the molar solubility of borax at each of the measured temperatures.*
2. *Calculate the solubility product of borax at each of the measured temperatures.*
3. *Plot the natural logarithm of the solubility product versus the reciprocal temperature $1/T(K^{-1})$ for each sample and draw the best straight line.*
4. *Determine the slope of the linear plot equal to $-\Delta H^o/R$ and calculate the standard enthalpy of solution for borax.*
5. *Determine the y-intercept(at $x=0$) of the linear plot equal to $\Delta S^o/R$ and calculate the standard entropy of solution for borax.*

Cleaning of the test tube and chemical disposal

After completing the experiment, clean your test tubes in a hot stainless water bath to remove the remaining borax in the tube effectively.

Dispose of the analyte and titrant in the waste container in the chemical hood.

Cleaning the Buret

In order for your buret to perform optimally, it must be properly cleaned. To clean the buret, use the following procedure:

1. Rinse with distilled water:

With the stopcock closed, add some distilled water to the buret. Tip and roll the buret, allowing the water to have contact with all of the inside surfaces. Open the stopcock and allow the water to drain. If the water drains without leaving any droplets on the side, repeat the rinse twice more then move to step two. If droplets remain on the inside surface, wash the buret with detergent solution, rinse several times with tap water, then rinse three times with distilled water.

2. Rinse with solution:

After draining the final distilled water rinse, close the stopcock and add about 5 mL of the solution to be dispensed from the buret. Again, roll and tip the buret so the solution has contact with all the inside surfaces. Open the stopcock and allow the solution to drain. Repeat this twice more. Discard the solution used in the rinses.

After you are finished with the buret in your experiment, rinse it by filling it with distilled water and allowing it to drain.

Once the buret is clean, clamp it to a stand using a buret clamp. Always make sure the burette is clamped in a perfectly vertical position before taking any readings.

When adding solutions to the buret, make sure the stopcock is closed (horizontal position). Unclamp the buret and tilt it slightly while pouring the solution slowly down the inside surface. This will prevent the formation of air bubbles.

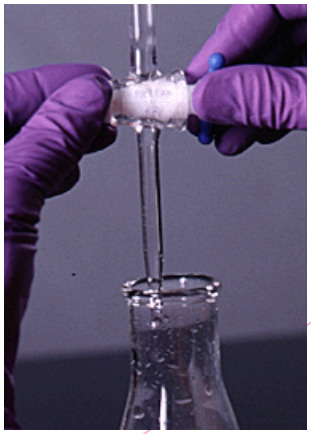
1. Get the appropriate amount of solution in a clean, dry beaker.

2. Pour a few milliliters of solution into the buret. Open the stopcock all the way in order to force all the air out of the stopcock and tip. Close the stopcock before the solution drains below the stopcock. If the tip still contains air, add a few more milliliters of solution and repeat the process. Repeat this until you are convinced no more air is left in the stopcock or tip. Discard the solution that you have run through the buret.

3. Using the procedure described above for adding solutions to the buret, fill it to a level just above 0.00 mL. Drain the buret to just under 0.00 mL. This will properly form the meniscus. DO NOT ATTEMPT TO ADJUST THE MENISCUS TO EXACTLY 0.00 mL. THIS IS AN INCREDIBLE WASTE OF TIME.

4. Touch the tip of the buret to the inside wall of a beaker in order to remove any drops on the tip. Do not wipe the tip. Wait a few seconds for the solution to drain to the top of the fluid level, then record the initial buret reading in your notebook.

5. Loosely cover the top of the buret with a cocked, small beaker or a loosely fitting piece of aluminum foil. This will keep dust out of the buret.



In order to make the meniscus easier to see, place a white card with a black mark on it behind the buret. Align the black mark so that it is just under the meniscus.

1. Get your eye level with the bottom of the meniscus. Looking up or down on the meniscus will cause a parallax error.

2. Read the buret to the nearest 0.01 mL. The marks occur every 0.1 mL, so the last number will have to be an estimate. With practice, you should be able to do this quite accurately.