As always, start with a fresh right-hand page, and write the title and purpose of lab 5. Include also your name, your lab partner’s name and the date.

You may cut and paste the “Purpose” through “Waste Disposal” section. Following this, begin a “Data” section (see hints in the Data section as to how to set it up).

Follow that with the “Analysis” section and then the “Conclusions” section. After that, please write an abstract (see hints in that section).

Photocopy the appropriate parts of your lab notebook and attach the abstract to the front. Make sure the title and name are legible. This lab is due Monday, February 13 in class.

Chemistry 160

Lab 5: The thermodynamics of the solubility of borax

Purpose: To determine the thermodynamic quantities $\Delta H^\circ$ and $\Delta S^\circ$ for the solvation reaction of borax in water by measuring the solubility product constant over a temperature range.

Introduction: Much of the derivations that follow may be found in the Thermodynamics chapter in the text.

The standard free energy change of a reaction $\Delta G^\circ$ is related to the equilibrium constant for the reaction by the equation $\Delta G^\circ = -RT \ln K$. The reaction is thermodynamically favored if $\Delta G^\circ < 0$; this means that $K > 1$. If $K$ is significantly larger than 1, the reaction should proceed primarily to products.

The free energy expresses the net effects of two thermodynamic quantities: the enthalpy $H$ and the entropy $S$. Under constant temperature conditions, the quantities are related by the equation $\Delta G^\circ = \Delta H^\circ - T \Delta S^\circ$. $\Delta H^\circ$ indicates that amount of heat absorbed or released during in the reaction and $\Delta S^\circ$ represents the amount of molecular disorder created or removed during the reaction.

Equating the two equations in the previous paragraphs, you get $\Delta H^\circ - T \Delta S^\circ = -RT \ln K$, which is rearranged to:

$$\ln K = -\frac{\Delta H^\circ}{R} \left(\frac{1}{T}\right) + \frac{\Delta S^\circ}{R}$$

Let $y = \ln K$ and $x = (1/T)$ and the equation has the form of the linear relationship $y = mx + b$, where $m$ is the slope of the line, given by $-\left(\Delta H^\circ / R\right)$, and $b$ is the $y$-intercept, given by $\left(\Delta S^\circ / R\right)$. Thus, if the quantities $\ln K$ and $1/T$ could be plotted, you could measure the slope and intercept of a best-fit line you could draw through those data points and determine $\Delta H^\circ$ and $\Delta S^\circ$ for this reaction.
The equation on the previous page is valid only if $\Delta H^\circ$ and $\Delta S^\circ$ are not temperature-dependent quantities. Over the small temperature range that you are investigating, the assumption is reasonably valid for borax.

So what is $K_c$ for this reaction? Note that the borax solvation reaction equilibrium constant is the solubility product $K_{sp}$ for borax or $K_{sp} = [\text{Na}^+]^2 [\text{B}_4\text{O}_5\text{(OH)}_4^{2-}]$. By the stoichiometry of the reaction $[\text{Na}^+] = 2 [\text{B}_4\text{O}_5\text{(OH)}_4^{2-}]$, so $K_{sp} = 4 [\text{B}_4\text{O}_5\text{(OH)}_4^{2-}]^3$.

The amount of $\text{B}_4\text{O}_5\text{(OH)}_4^{2-}$ in solution is determined by titration with HCl solution using an indicator to mark the equivalence point.

**Skills:**
- Developing a temperature-series set of solubility measurements
- Use of an indicator for qualitative determination of the acidity/alkalinity of a solution
  - Use of a graph to determine a best-fit line
  - Use of a best-fit line equation to determine thermodynamic quantities

Chemical equation: $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O} (s) \rightleftharpoons 2 \text{Na}^+ (aq) + \text{B}_4\text{O}_5\text{(OH)}_4^{2-} (aq) + 8 \text{H}_2\text{O} (l)$

Chemicals needed:
- Borax (make sure it is not “cut” with other cleaning ingredients)
- A precisely-known concentration hydrochloric acid solution (about 0.2 M)
- A concentrated solution of sodium hydroxide (over 1 M)
- Indicator solutions (phenolphthalein)

Equipment needed:
- five clean 250 mL beakers
- one clean 150 mL beaker
- one clean 100 mL beaker
- thermometer
- buret

**Procedure**

1. Pick an appropriate indicator: thymol blue ($pK_{in} = 1.5$), methyl red ($pK_{in} = 5.1$) or phenolphthalein ($pK_{in} = 9.4$). The $pI$ of borax is about 4.9. After choosing the indicator, obtain about 10 mL of the HCl solution and add two drops of the indicator to that; stir. Then add drops of concentrated NaOH solution, stirring all the while, until the color change occurs. Note the color change (before and after) in your notebook for future reference.

2. Obtain the five 250 mL beakers and label them #1 through #5.

3. In a 100 mL beaker, prepare a saturated solution of borax by adding roughly 25 g of borax to 50 mL of distilled water. Obtain a hot plate and set the heat setting at about midpoint; place the borax solution on the hot plate and stir. You may use the thermometer as a stirring rod, but be gentle. Alongside that beaker, heat another beaker with about 100 mL of distilled water.
4. Take temperature readings constantly while stirring; make sure the bulb of the thermometer is not touching the sides or bottom of the beaker. When the temperature of the solution reaches 57 or 58°C, remove the beaker from the hot plate. **Heating over 60°C will decompose** (not dissolve) the borax. Keep monitoring the temperature of the solution.

5. With the beaker sitting on the countertop, stir the solution until it has cooled to about 55°C. Let the beaker stand with the thermometer in it until the solid has settled out of solution. While the solution is settling, rinse a 10 mL graduated cylinder with the hot water from the hot plate. (If you pour the hot borax solution into a cold graduated cylinder, the borax will instantly precipitate against the cold glass. When the solid has settled out, read and record the temperature to the nearest 0.1°C, then immediately decant exactly 5.0 mL into the warmed 10 mL graduated cylinder. Then transfer the contents of the cylinder into beaker #1. Rinse the cylinder with hot water several times and pour these rinses into the beaker as well.

6. Now allow the solution to cool to about 45°C; make sure you are stirring the solution frequently. When the solution is about 45°C, let the solution stand (as above) until the solid settles out and follow the sample collection process in step 5 for transferring 5.0 mL of the solution to beaker #2. Remember to record the temperature to the nearest 0.1°C. Do not forget to rinse the graduated cylinder and add the rinses to the beaker.

7. Repeat this procedure near 35°C, 25°C and 15°C (the last couple of temperatures may require an ice water bath — be careful not to let the beaker tip over in the bath), transferring 5.0 mL aliquots to beakers #3, #4 and #5, respectively.

8. Obtain about 100 mL of hydrochloric acid (note its precise concentration) in a 150 mL beaker. Rinse and fill the buret with the acid.

9. Add enough distilled water to beakers #1 through #5 so they each have about 100 mL total (the marking on the beaker is sufficient precision). Add five drops of your chosen indicator to each beaker. Stir the solutions in the beakers to make sure all of the borax is redissolved; you may need to warm beakers #1 and #2 to redissolve the borax.

10. Titrate each beaker with hydrochloric acid from the buret. For beakers #1 and #2, make sure the buret contains a full 50 mL of hydrochloric acid. Clearly, the amount of acid needed for titration should decrease from beaker #1 to beaker #5.

**Waste disposal:** Place all waste in the “aqueous waste” container in the hood.

**Data section:**

Remarks about the choice of indicator, given what titration was to be done later

Remarks about the indicator’s color change — both endpoint colors as well as any transitional color
Mass of borax initially added

Table of beaker number, volume of borax solution collected and temperature of collection

Observations of how cloudy the collect solution was or how much material crystallized on the inside of the graduated cylinder before the rinses

Concentration of hydrochloric acid solution (and units)

Table of beaker number and endpoint (equivalence point) volume

**Analysis**

1. Determine the concentration of the dissolved $B_4O_5(OH)_4^{2-}$ in each sample titrated; show a complete calculation for beaker #1.

2. Calculate $K_{sp}$ for each borax solution.

3. Prepare a graph of your results, plotting the two quantities $x$ and $y$ (as defined in the introduction). Using either your calculator’s functions or the graphing program’s functions, determine the **equation of the best-fit line** through your data points.

4. From the parameters of the best-fit line equation, calculate $\Delta H^\circ$ and $\Delta S^\circ$ for this reaction. Make sure to keep track of units, especially for $R$, so that the units of $\Delta H^\circ$ and $\Delta S^\circ$ work out sensibly.

5. Obtain the rest of the class’s $\Delta H^\circ$ and $\Delta S^\circ$ values and calculate the mean and standard deviation of each for the class. Determine if there are any outliers, then recalculate the mean and standard deviation, if necessary. Pay attention to the units and sig figs.

**Conclusion**

The literature values for enthalpy and entropy of the dissolution of borax in water are 110 kJ/mol and 380 J/mol K, respectively. Determine the percent error in your experimentally determined values and the class’s mean values. Comment on the confidence you have about this procedure to determine $\Delta H^\circ$ and $\Delta S^\circ$ for this reaction accurately.

**Abstract**

First sentence: What are your results? (The reaction is called borax solvation)
Second sentence: Enlarge to include class results (report mean and std. dev. in proper format with units); comment about your results compared to the class results
Third sentence: In comparison to the literature values, how well did the procedure work? Appropriate to add comments about possible error sources, if significant