PRE-LABORATORY ASSIGNMENT EXPERIMENT 6

1. Is the sign of $\Delta_r H$ for an exothermic reaction positive or negative? Why?

___________________________________________________________________________________________

2. When 4.21 grams of potassium hydroxide are added to 250. mL of water in a coffee cup calorimeter, the temperature rises by 4.14 °C. Assume that the density and specific heat of the dilute aqueous solution are the same as those of H$_2$O and calculate the molar heat of solution of potassium hydroxide in kJ/mol. $C = 4.184$ J/g·K

3. Using Hess' Law, Calculate the standard heat of formation of copper(I) oxide given the following data:

   $\text{CuO(s)} \rightarrow \text{Cu(s)} + \frac{1}{2} \text{O}_2(g) \quad \Delta_r H^\circ = 157.3$ kJ/mol

   $4\text{CuO(s)} \rightarrow 2\text{Cu}_2\text{O(s)} + \text{O}_2(g) \quad \Delta_r H^\circ = 292.0$ kJ/mol
EXPERIMENT 6 THERMOCHEMISTRY

Introduction:
Thermochemistry is the study of the relationship between chemical reactions and energy changes. Thermochemistry has many practical applications. For example, using thermochemistry:

(1) mining engineers can calculate how much fuel will be needed to prepare metals from their ores,
(2) structural engineers can determine how much heat will be evolved by cement as it sets, and
(3) the number of Calories in an edible compound can be determined.

The experimental measurement of the heat change that results from a chemical or physical process is known as calorimetry. Calorimetry is of interest to chemists because one of the fundamental characteristics of a chemical reaction is the evolution or absorption of heat. This heat change is the result of the breakage or formation of chemical bonds during the chemical reaction. Breaking a chemical bond requires energy; thus, it is an endothermic process. Forming a chemical bond releases energy; thus, it is an exothermic process. For example, coal is burned in oxygen (combusted) to produce carbon dioxide and heat in the following exothermic reaction:

\[
\text{coal (s)} + \text{O}_2 \text{(g)} \rightarrow \text{CO}_2 \text{(g)} + \text{Energy (heat)}
\]

In this example, heat is released because the products are lower in energy than the reactants. The energy released in the formation of new chemical bonds is greater than the energy required to break the original reactant bonds thus the overall process releases energy in the form of heat.

A calorimeter is an apparatus that is used to measure heat flow. It consists of an insulated container (you will use a Styrofoam cup in this experiment) fitted with a thermometer. The aqueous solution to be studied is placed inside the insulated container and the temperature change of the solution is monitored as the reaction proceeds. The calorimeter and its contents can be thought of as an isolated system, insulated from its surroundings, from which or into which no heat can be transferred.

Refer to the sections on calorimetry and enthalpy in your textbook for additional information and sample problems.

Heat of Solution

When an ionic solid dissolve in a water, the cations and anions that result are surrounded by water molecules due to the electrostatic attractive forces between the formal charge of the ion the dipole of water. Each individual solute ion is completely surrounded by water molecules thereby isolating the oppositely charged species form one another. This process is called solvation, and, in the case where water is the solvent, hydration. The hydration process for a salt can be either exothermic or endothermic. The enthalpic quantity associated with solvation/hydration is called the molar heat of solution. This is defined as the quantity of heat evolved or absorbed when per mole of a solid solute is solvated/hydrated.

A commercial application of an endothermic heat of solution is the instant cold packs used in athletics to treat sprains. In these cold packs, water and salt are initially separated and then mixed by squeezing the pack to break the separation device. Heat is absorbed in the endothermic hydration process, so the pack quickly becomes extremely cold. Hot packs, which take advantage of the exothermic heat of solution, work in an analogous manner, releasing heat when the contents are mixed.

In the experimental measurement of the heat of solution of a substance, a known mass of solute is added to a known volume of water in a calorimeter. The temperature of the water is measured before mixing and as a function of time after mixing. These data are plotted to find the change in temperature, \( \Delta T \), and then the heat absorbed or generated in the reaction \( q_{\text{reaction}} \) and the molar heat of solution \( \Delta H \) are calculated. In part A-(1) of this experiment, you will determine the molar heats of solution of an ionic solutes, potassium nitrate (KNO₃).
**Heat of Neutralization**

An acid (an H⁺ donor) reacts with a base (an OH⁻ donor) to form water in a neutralization reaction represented by the following net ionic equation:

\[ \text{H}^+ (aq) + \text{OH}^- (aq) \rightarrow \text{H}_2\text{O}(l) \]

The energy of a reaction associated with acid/base neutralization is referred to as the *heat of neutralization*. In part A-(2) of this experiment, you will react solutions of hydrochloric acid (HCl) and sodium hydroxide (NaOH) to measure the heat of neutralization. The heat of neutralization is dependent on the number of moles of H⁺ and OH⁻ present, not the particular acid or base used. The nature of the spectator ions, sodium and chloride in this case, is not important.

**Hess’s Law – Part B**

In part B of this experiment, we will be finding the enthalpy of formation for MgO(s) using an indirect method. According to Hess’s Law, if two or more reactions can be added to give a net reaction, \( \Delta H^\circ \) for the net reaction is simply the sum of the \( \Delta H^\circ \)'s for the reactions which are added. Consider the following three reactions:

1. \( \text{Mg}(s) + 2 \text{H}^+(aq) \rightarrow \text{Mg}^{2+}(aq) + \text{H}_2(g) \quad \Delta H_1^\circ \)
2. \( \text{Mg}^{2+}(aq) + \text{H}_2\text{O}(l) \rightarrow \text{MgO}(s) + 2 \text{H}^+(aq) \quad \Delta H_2^\circ \)
3. \( \text{H}_2(g) + \frac{1}{2} \text{O}_2 \rightarrow \text{H}_2\text{O}(l) \quad \Delta H_3^\circ \)

\[
(4) \quad \text{Mg}(s) + \frac{1}{2} \text{O}_2(g) \rightarrow \text{MgO}(s) \quad \Delta H_4^\circ = \Delta H_1^\circ + \Delta H_2^\circ + \Delta H_3^\circ
\]

Notice that when the three reactions are added, they form the formation reaction for MgO. Analogously, when we add the enthalpies for the three reactions, they give the enthalpy change associated with the formation reaction.

**Objectives**

In this experiment, you will measure the heat of solution of potassium nitrate and measure the heat of neutralization for an acid-base reaction then compare your experimental results with calculated values. You will also utilize Hess’s Law to determine the enthalpy of reaction for a reaction you will not actually perform, the formation of MgO(s).

**Experimental Procedure**

**Part A.**

Measurements will be made using a calorimeter consisting of a Styrofoam cup and cover, thermometer, and magnetic stir bar. For stability, the Styrofoam cup should be placed in a beaker. The solutions should be stirred with a magnetic stir bar and a magnetic stirrer. You will work with a partner for this experiment. Check out a digital thermometer from your instructor.

The balanced net ionic equations for the two reactions in part A are:

1. \( \text{KNO}_3(s) \rightarrow \text{K}^+(aq) + \text{NO}_3^-(aq) \)
2. \( \text{OH}^- (aq) + \text{H}^+(aq) \rightarrow \text{H}_2\text{O}(l) \)

1. **Heat of Solution of KNO₃.** Weigh approximately 5 g of potassium nitrate (KNO₃) on an analytical balance and record the mass to the nearest 0.0001 g. Place 50.0 mL of distilled water in your calorimeter using a graduated cylinder. Record the volume of water to the nearest 0.5 mL. Using your thermometer, record the initial temperature of the water at 30-second intervals for three minutes. Add the solid KNO₃ to the
water and stir rapidly with a magnetic stirrer as your partner takes temperature readings every 10 seconds. Continue taking temperature readings for at least five readings after the direction of the temperature change reverses or remains constant. When done, rinse and dry the calorimeter.

2. Heat of Neutralization. Place 50.0 mL (record the volume to the nearest 0.5 mL) of 1.0 M HCl in your calorimeter and record the initial temperature at 30-second intervals for three minutes. Pour 50.0 mL (record the volume to the nearest 0.5 mL) of 1.0 M NaOH into the HCl solution in the calorimeter and stir rapidly with a magnetic stirrer as the temperature is recorded at 10-second intervals. Remember to take five readings after the direction of the temperature change reverses or remains constant. Rinse and dry the calorimeter when you are finished.

Experimental Procedure

Part B. Enthalpy of Reaction and Verification of Hess’s Law

(Again work in partners.)

The calorimeter and the thermometer setup will be the same as that used in Part A. Rinse the inner cup of your calorimeter thoroughly with deionized water and dry. Assemble your calorimeter, being certain that there is no water in the space between the two cups. Check out a thermometer from your instructor.

(a) Weigh a sample containing 0.45 - 0.55 g of 20-mesh granulated magnesium using a weigh boat or weighing paper. Load the calorimeter with 100 mL of 1.0 M HCl (record to ±0.5 mL) and your stir bar. Measure the initial temperature at 30 second intervals for three minutes. Add the Mg(s) quickly. As the metal reacts record the temperature every 30 seconds. Continue taking temperature readings for at least five readings after the direction of the temperature change reverses or remains constant.

(b) When the reaction has subsided, pour the solution into the waste container in the fume hood labeled "metals waste". DO NOT POUR IN THE STIR BAR! When done, rinse and dry the calorimeter.

(c) Calculate the mass of MgO that would contain the same number of moles of Mg as you used in part (a). Weigh enough MgO to be within 5% of this value. Load once again with 100 mL of 1.0 M HCl (record to ±0.5 mL). Proceed as in part (a), but note that it may take longer for the MgO(s) to react than it did for the Mg(s). If you observe a temperature change of less than 5°C, repeat the reaction of MgO. Dispose of the reaction waste as before, DO NOT POUR IN THE STIR BAR! Rinse and dry the calorimeter and please the cups and lids back in the drawers.

\[ \text{g Mg} \rightarrow \text{mols Mg} \rightarrow \text{mols MgO} \rightarrow \text{g MgO} \]

Example: If your mass was 0.5000g Mg, then a range of 0.87g to 0.79g ± 0.0001g of MgO would be acceptable.
Graphical Treatment of Data

In this experiment, it is not valid to make the assumption that no heat is exchanged between the contents of the calorimeter and the room. This heat transfer may be corrected for using graphical techniques if data are collected over a period of time. For this reason, at least five measurements were made after the direction of the temperature change reversed or remained constant. You will plot graphs of the four data sets in part A and two in part B for a total of six graphs in order to correct for the heat exchanged between the calorimeter and the surroundings. Figure 1 shows a set of data taken for the determination of a heat of solution. The time of mixing was at 190 seconds and data were recorded for 330 seconds.

In order to determine the temperature change, $\Delta T$, which would have occurred if the mixing was instantaneous, it is assumed that the loss of heat to the room is constant and linear. This is a reasonably valid assumption if the temperature of the calorimeter contents is not vastly different from room temperature. This assumption then allows the extrapolation back to the instant the reagents were mixed, 190 seconds in this example.

To determine $\Delta T$ as in Figure 2, use the following extrapolation procedure:

1. Draw a vertical line using a ruler, which passes through the time corresponding to the time of mixing. In this example, the line was drawn at 190 seconds.
2. Draw best-fit straight lines through the approximately linear before mixing and after mixing data points which intersect the vertical line drawn in step one.
3. Read the initial temperature ($T_i$) and final temperature ($T_f$) from the points of intersection of the vertical line and the two best-fit straight lines. In this example, $T_i$ is 23.2 °C and $T_f$ is 29.4 °C. The difference between these two temperatures ($T_f - T_i$) is the temperature change, $\Delta T$. In this case, $\Delta T$ is 6.2 °C (29.4 °C - 23.2 °C). See Figures 1 and 2 below.

![Graph](image)

Figure 1. Plotted temperature-time data for an exothermic process.

Note that the plot for an endothermic process will show a drop in temperature between $T_i$ and $T_f$. 
Figure 2. Extrapolation of temperature-time data in Figure 1 to find the temperature change, ΔT.

**Calculations**

All calculations that use a temperature change must have that temperature change (ΔT) determined from a plot like the one above. Points will be deducted if this is not the case!

First, plot, using Excel, a graph of temperature versus time for each of your four data sets. For each data set, determine Ti, Tf, and ΔT from the temperature versus time graph using the extrapolation procedure (by hand) given above. Calculate $q_{\text{solution}}$ and then reverse the sign on $q_{\text{solution}}$ to obtain $q_{\text{reaction}}$. Lastly, convert $q_{\text{reaction}}$ to the molar change in enthalpy, ΔH. A sample calculation is provided below.

**Sample Calculation for Heat of Solution**

**Given:** 5.20 g of sodium carbonate (Na$_2$CO$_3$) is dissolved in 75.0 mL of distilled water in a calorimeter. The temperature of the water before the addition of the Na$_2$CO$_3$ was 26.0 °C. The temperature after mixing was 29.9 °C.

1. **Mass of solution**
   
   Calculate the mass of the water, $m_w$, in the calorimeter using the density of water, 1.00 g/mL.
   
   $m_w = \text{volume of water} \times \text{density of water}$
   
   $= 75.0 \text{ mL} \times 1.00 \text{ g/mL}$
   
   $= 75.0 \text{ g}$

   The total mass of solution equals the mass of the water ($m_w$) plus the mass of solute ($m_s$).
   
   $m_T = m_w + m_s = 75.0 \text{ g} + 5.20 \text{ g} = 80.2\text{ g}$

2. **$q_{\text{solution}} \ (J)$**

   Calculate the heat gained by the solution, $q_{\text{solution}}$. Use the specific heat of water, 4.184 J/g.°C, for $C_w$.

   Note, you must find the ΔT by graphing your data and determining the initial and final temperatures as described in the section on the graphical treatment of the data.
\[ q_{\text{solution}} = m_{\text{C}_w} \Delta T \]
\[ = 80.2 \text{ g} \times 4.184 \text{ J/g} \cdot ^\circ \text{C} \times (29.0 \ ^\circ \text{C} - 26.0 \ ^\circ \text{C}) \]
\[ = (80.2 \text{ g} \times 4.184 \text{ J/g} \cdot ^\circ \text{C} \times 3.9 \ ^\circ \text{C} \]
\[ = 1300 \text{ J} \]

3. \( q_{\text{reaction}} \) (J)

Reverse the sign on \( q_{\text{solution}} \) to obtain the heat given off in the solvation process, \( q_{\text{reaction}} \).

\[ q_{\text{reaction}} = -q_{\text{solution}} \]
\[ q_{\text{reaction}} = -1300 \text{ J} \]

4. \( \Delta H \) Experimental

Remember that the enthalpy change associated with this process, \( \Delta H \), is a molar quantity expressed in kJ/mol. Therefore, the mass of \( \text{Na}_2\text{CO}_3 \) solvated must be converted to moles using the molecular weight of \( \text{Na}_2\text{CO}_3 \).

\[ \text{moles of \( \text{Na}_2\text{CO}_3 \)} = \frac{\text{grams of \( \text{Na}_2\text{CO}_3 \)} \text{ used}}{\text{molecular weight of \( \text{Na}_2\text{CO}_3 \)}} \]
\[ = \frac{5.20 \text{ g} \ \text{Na}_2\text{CO}_3}{106 \text{ g} \ \text{Na}_2\text{CO}_3/\text{mol} \ \text{Na}_2\text{CO}_3} \]
\[ = 0.0491 \text{ mol} \ \text{Na}_2\text{CO}_3 \]

Note that if the solute is a liquid, as in step A-(2) of the experiment, the moles of solute can be found by multiplying the volume in liters by the molarity:

\[ \text{moles of solute} = (\text{volume of solute [L]} \times \text{molarity of solute [mol/L]}) \]

Then, divide \( q_{\text{reaction}} \) by the moles of \( \text{Na}_2\text{CO}_3 \) to give \( \Delta H \) in J/mol.

\[ \Delta H = \frac{q_{\text{reaction}}}{\text{mol} \ \text{Na}_2\text{CO}_3} \]
\[ = \frac{(-1300 \text{ J})}{(0.0491 \text{ mol} \ \text{Na}_2\text{CO}_3)} \]
\[ = -26000 \text{ J/mol} \]

Finally, convert joules to kilojoules to give \( \Delta H \) in the proper units.

\[ \Delta H = -26000 \text{ J/mol} \times 1 \text{ kJ} / 10^3 \text{ J} \]
\[ = -26 \text{ kJ/mol} \]

The negative sign on \( \Delta H \) indicates that the solvation of \( \text{Na}_2\text{CO}_3 \) is exothermic.
5. Comparison of Experimental Results with Literature Values

You will also compare your experimental results with literature values. To do this:

a. Write a balanced net ionic equation for the reaction.

b. Use the heats of formation in Table 1 to calculate the actual value of $\Delta H$.

$$\Delta H_{\text{reaction}} = (\text{sum of } \Delta H_f \text{ of products}) - (\text{sum of } \Delta H_f \text{ of reactants})$$

Table 1. Heats of Formation*\(^1\)

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<thead>
<tr>
<th>Compound</th>
<th>$\Delta H_f$ (kJ/mol)</th>
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<tbody>
<tr>
<td>KNO$_3$ (s)</td>
<td>-492.7</td>
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<td>H$_2$O (l)</td>
<td>-285.9</td>
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<tr>
<td>H$^+$ (aq)</td>
<td>0.0</td>
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<tr>
<td>OH$^-$ (aq)</td>
<td>-229.9</td>
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<tr>
<td>KNO$_3$ (aq)</td>
<td>-457.8</td>
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6. Percent Error

Calculate a percent error by comparing your experimental value with the calculated result. Remember that errors of 10% or more are not unreasonable.

$$\% \text{ error} = \frac{|\text{experimental value} - \text{calculated value}|}{\text{calculated value}} \times 100$$

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Part A Data  
(\textit{DO NOT TURN IN THIS PAGE})

\textbf{Solution 1: Heat of Solution of KNO}_3

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Solution 2: *Heat of Neutralization.*

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Data and Results:  
Name: ______________________________

Part A  (Turn this and the following pages in)  
Section: __________

Reaction 1: Heat of Solution KNO₃

Mass KNO₃ _________  Moles KNO₃ _________  H₂O volume _________ (± 0.5 mL)  
(show all calculations)

Mass of solution (g) _________  ΔT (from plot) _________ (careful of your sign)  
(show all calculations)

q solution (J) _________  q reaction (J) _________  
(show all calculations)

ΔH experimental _________  ΔH literature _________ (see page 9)  
(show all calculations)

Percent Error _________  
(show all calculations)
Data and Results: Name: ____________________________
Part A Section: __________

Reaction 2: Heat of Neutralization \[ M(HCl) \quad \text{and} \quad M(NaOH) \]

\[ \text{mL HCl(aq)} \quad \text{(± 0.5 mL)} \quad \text{Moles } H^+ \quad \text{__________} \]
\( (\text{show all calculations}) \)

\[ \text{mL NaOH(aq)} \quad \text{(± 0.5 mL)} \quad \text{Moles } OH^- \quad \text{__________} \]
\( (\text{show all calculations}) \)

\[ \text{Mass of Solution (g)} \quad \text{__________} \quad \text{ΔT (from plot)} \quad \text{__________} \quad \text{(careful of your sign)} \]
\( (\text{show all calculations}) \)

\[ q \text{ solution (J)} \quad \text{__________} \quad q \text{ reaction (J)} \quad \text{__________} \]
\( (\text{show all calculations}) \)

\[ \Delta H \text{ experimental} \quad \text{__________} \quad \Delta H \text{ literature} \quad \text{__________} \quad (\text{see page 9}) \]
\( (\text{show all calculations}) \)

\[ \text{Percent Error} \quad \text{__________} \]
\( (\text{show all calculations}) \)
Summary of Results Part A:  
(Staple this page to the top of your data sheets and plots)  

| Name: ______________________________ | Lab: Section: ________ |

- Turn in this Page with pages 12, 13, 14 & 15 along with your plots of temperature vs. time by the next lab meeting.
- Each student must produce their own graphs. The determination of $\Delta T$ from each plot must be clearly labeled on each plot. Each plot must be titled labeled appropriately.
- Points will be deducted if this is not so!

### Part A Reaction 1: Heat of Solution KNO$_3$

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<th>$\Delta H$ (Literature)</th>
<th>% error</th>
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### Part A Reaction 2: Heat of Solution Neutralization

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Instructor Comments: ________________________________________________________________
Part B Data (DO NOT TURN IN THIS PAGE)

Mg Reaction Data

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MgO Reaction Data

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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
**Data and Results:**

<table>
<thead>
<tr>
<th></th>
<th>Mg</th>
<th>MgO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass of weighing paper</td>
<td>_______ g</td>
<td>_______ g</td>
</tr>
<tr>
<td>Mass of sample and weighing paper</td>
<td>_______ g</td>
<td>_______ g</td>
</tr>
<tr>
<td>Mass of sample</td>
<td>_______ g</td>
<td>_______ g</td>
</tr>
<tr>
<td>Moles of sample</td>
<td>_______mol</td>
<td>_______mol</td>
</tr>
<tr>
<td>Volume of solution</td>
<td>_______mL</td>
<td>_______mL</td>
</tr>
<tr>
<td>Initial temperature (T_i)</td>
<td>_______ °C</td>
<td>_______ °C</td>
</tr>
<tr>
<td>Final temperature (T_f)</td>
<td>_______ °C</td>
<td>_______ °C</td>
</tr>
<tr>
<td>ΔT reaction (watch sign)</td>
<td>_______ °C</td>
<td>_______ °C</td>
</tr>
<tr>
<td>Mass of solution</td>
<td>_______ g</td>
<td>_______ g</td>
</tr>
<tr>
<td>q solution</td>
<td>_______ J</td>
<td>_______ J</td>
</tr>
<tr>
<td>q reaction</td>
<td>_______ J</td>
<td>_______ J</td>
</tr>
<tr>
<td>ΔH reaction</td>
<td>_______ kJ/mol</td>
<td>_______ kJ/mol</td>
</tr>
</tbody>
</table>

*(show all calculations)*

**Hess’s Law Calculation:** Refer to reactions in introduction.

\[ ΔH^°_1 \quad \text{(Mg(s) + HCl(aq))} \]

\[ ΔH^°_2 \quad \text{(MgO(s) + HCl(aq))} \]

\[ ΔH^°_3 = -285.9 \text{ kJ/mol} \quad \text{(heat of formation of water)} \]

Combine the appropriate heats of reaction and use Hess’s law to find \( ΔH^° \) (MgO).

SHOW YOUR WORK HERE:

Don’t forget to add the enthalpies of rxn’s (1), (2) & (3) so that they yield Mg(s) + \( \frac{1}{2} \) O_2(g) → MgO(s)

**Literature value for \( ΔH^° \) MgO (Text or CRC handbook)**

\[ \text{___________ kJ/mol} \]

**Percent Error Experimental vs. Literature**

\[ \text{___________} \]

*(show all calculations)*