# Calorimetry Lab

Most reactions we have considered this semester have some sort of a driving force. This driving force may be a release of heat or a net increase in disorder in the chemical system or its surroundings. The thermodynamic term for heat given off or absorbed is enthalpy. The thermodynamic term for more or less order is entropy. Together, these two quantities determine the likelihood and extent that a chemical or physical change occurs. A negative enthalpy value means a reaction is exothermic and this favors the spontaneous forward progress of a reaction.

A Coffee-Cup Calorimeter Made of Two Styrofoam Cups

As an introduction to thermodynamics this calorimetry lab will explore a way to measure the enthalpy of a reaction.
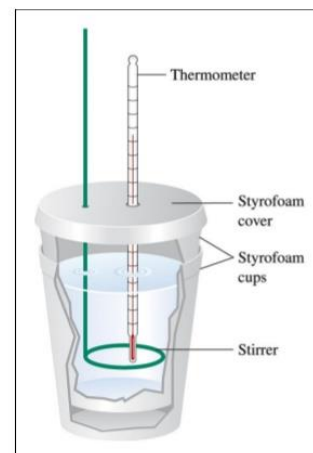
A calorimeter is an apparatus used to determine the heat that is transferred during a process and this heat transfer is denoted as *q*. Usually exothermic reactions, reactions that release heat from the system to the surroundings, are examined with a calorimeter. These reactions will then have a negative sign for q or ΔH.

The first law of thermodynamics state that energy cannot be destroyed but can be transferred from one system to another. It is stated like this:

$\Delta H_{system} + \Delta H_{surroundings} = 0$ or $q_{system} + q_{surroundings} = 0$

If we start with assuming a perfect calorimeter (one with perfectly insulating walls through which no heat can pass) all the heat given off by the system, whether it is a metal cooling down in a sample of water, a chemical reaction taking place, or even some dilutions, will release heat to the surroundings. The equations will look like these:

$$-q_{system} = q_{surroundings} \qquad -q_{metal} = q_{water} \qquad - q_{reaction} = q_{water} \qquad - q_{dilution} = q_{water}$$

However we don't have a perfect calorimeter. Therefore, the equation must account for heat loss by adding a second term on the right:

$$-q_{system} = q_{water} + q_{loss\ to\ calorimeter}$$

In the first part of this experiment you will determine the specific heats of different metals. The specific heat is related to temperature change and heat transferred by the following relationship:

$q = mC\Delta T$ q = heat, m = mass, C = specific heat capacity, and ΔT = change in temperature

The equation will look like this for a metal loosing heat to the water and calorimeter:

$$-m_{metal}C_{metal}\Delta T_{metal} = m_{water}C_{water}\Delta T_{water} + B\Delta T_{water}$$

In this equation "B" is the calorimeter constant and takes into account the heat lost to the surroundings because of an imperfect calorimeter. It has units of J/°C. Note that the ΔT for the calorimeter is the same as ΔT_{water} because the water is in the calorimeter and they are at thermal equilibrium.

This experiment consists of three parts.

In Part A, of this experiment you determine the value of your calorimeter constant, B.

In Part B, you will determine the specific heat capacity of the unknown metal, C_{metal}.   Using the Law of Delong and Petit you will identify the unknown metal.

In Part C, you will determine the heat of reaction for an acid and base reaction.   You will use Hess's Law to obtain a better value for the acid base reaction attempted in part three.

Procedure

Place your 800 mL beaker on a hot plate and start a boiling water bath. You will need it for the first half of today's lab. Obtain two coffee cups and a lid along with a thermometer that reads from 0°C to 100 °C. Set up a ring stand with an iron ring and a piece of string to hang the thermometer. You need to suspend the thermometer about 3 to 5 cm above the plate of the ring stand so that the thermometer can be suspended in the two coffee cup calorimeter without tipping it over or touching the sides or bottom. Keep in mind you will use the same thermometer for many measurements in and out of the calorimeter and you need to clean and dry it between each measurement of a different solution. Especially take care to minimize any removal of liquid from the calorimeter or any carefully measured solution, try to get the thermometer to drip all the liquid into the container from which it came. Also you will be asked to measure the temperature of strongly acidic and basic solutions, be careful with the thermometer, don't cross contaminate, and use the usual precautions for working with strong acids and bases.

Once your water bath has come to a boil take two clean large test-tubes, add 20 mL distilled water to each (25 mL if you are a slow worker) and place in the boiling water bath. Each test-tube should be 1/3 to ½ full. Take two more large clean dry test-tubes, place Al shot in the first and your unknown in the second. LOOSELY place a cork stopper on both of the two test-tubes containing the metals to prevent water from condensing in the tubes and getting the metals wet which can be a significant source of error. Label the Al test-tube "Al" and the unknown "unk" and place them in the boiling water bath. We will weigh the shot at the end of the experiment after it is in the calorimeter.

PART A Determining the "B" constant

1. Record the mass of your double cup calorimeter on the data sheet.
2. In the calorimeter place 50.0 mL of distilled water and record the mass of your double cup calorimeter + water on the data sheet.
3. Measure the temperature of the water in the calorimeter and record it on your data sheet.
4. After your two test-tubes of water have been in the boiling water bath for 15 minutes, take the temperature of the boiling water bath and record it on the data sheet.
5. Set up the calorimeter with the thermometer suspended from the ring stand through the lid of the coffee cup.
6. Using tongs one person carefully removes a test-tube of boiling water and pours it into the calorimeter while the other person secures the lid, stirs the solution and watches for the highest temperature achieved.
7. Record the highest temperature on the data sheet.
8. Carefully remove the lid and thermometer letting all the solution drip into the calorimeter.
9. Weigh the calorimeter again and record the mass on the data sheet. This last mass is to calculate the mass of hot water added in the experiment.
10. Empty the calorimeter into the sink, dry the styrofoam cups and check to make sure no liquid is on the lid.
11. Calculate your "B" constant on the data sheet.
12. Repeat this procedure with the second test-tube of hot water, calculate the "B" value and average your two determinations.

Name _____ **TA** _____ **Time** _____

PART A Determining the "B" constant

mass of two cups, _____ g, mass of two cups + water _____ g, mass after experiment_____ g

m_{WC} = Mass H_2O in calorimeter _____ g

m_{WTT} = Mass of H_2O in test tube _____ g

T_i = Initial temperature of H_2O in calorimeter _____°C

T_H = Highest T of H_2O in calorimeter after mixing _____°C

T_{TT} = T of hot H_2O in the test tube _____°C

ΔT_{WC} = Highest T of H_2O in calorimeter after mixing - initial T of H_2O in calorimeter = $T_H - T_i$ = _____ - _____ = _____°C

ΔT_{WTT} = Highest T of H_2O in calorimeter after mixing - T of hot H_2O in the test tube = $T_H - T_{TT}$ = _____ - _____ = _____°C

C_W = 4.184J/g°C

$-m_{WTT}C_W\Delta T_{WTT} = m_{WC}C_W\Delta T_{WC} + B\Delta T_{WC}$

Substitute your values in the equation above and solve for "B"

Repeat with your second sample of hot water

mass of two cups, _____ g, mass of two cups + water _____ g, mass after experiment_____ g

m_{WC} = Mass H_2O in calorimeter _____ g

m_{WTT} = Mass of H_2O in test tube _____ g

T_i = Initial temperature of H_2O in calorimeter _____°C

T_H = Highest T of H_2O in calorimeter after mixing _____°C

T_{TT} = T of hot H_2O in the test tube _____°C

ΔT_{WC} = Highest T of H_2O in calorimeter after mixing - initial T of H_2O in calorimeter = $T_H - T_i$ = _____ - _____ = _____°C

ΔT_{WTT} = Highest T of H_2O in calorimeter after mixing - T of hot H_2O in the test tube = $T_H - T_{TT}$ = _____ - _____ = _____°C

C_W = 4.184J/g°C

$-m_{WTT}C_W\Delta T_{WTT} = m_{WC}C_W\Delta T_{WC} + B\Delta T_{WC}$

Substitute your values in the equation above and solve for "B"

Average your two values for "B" _____ you will use this for "B" in the remainder of the experiments.

<u>PART B Calculation of Cp for Al</u>

1. Record the mass of your double cup calorimeter on the data sheet.
2. In the calorimeter place 50.0 mL of distilled water and record the mass of your double cup calorimeter + water on the data sheet.
3. Measure the temperature of the water in the calorimeter and record it on your data sheet.
4. After your test-tube containing Al has been in the boiling water bath for 15 minutes, take the temperature of the boiling water bath and record it on the data sheet. This is the initial temperature for Al.
5. Set up the calorimeter with the thermometer suspended from the ring stand through the lid of the coffee cup.
6. Using tongs one person carefully removes the test-tube containing Al from the water bath, removes the cork, and pours the Al into the calorimeter, WITHOUT SPLASHING ANY WATER, while the other person secures the lid, stirs the solution and watches for the highest temperature achieved. When removing the test-tube and using its contents be very careful to not let any water on the outside of these test-tubes to drip into the calorimeter.
7. Record the highest temperature on the data sheet.
8. Carefully remove the lid and thermometer letting all the solution drip into the calorimeter.
9. Weigh the calorimeter again and record the mass on the data sheet. This last mass is to calculate the mass of Al added in the experiment.
10. Decant the water out of the calorimeter and place the Al to dry in the place provided, dry the styrofoam cups and check to make sure no liquid is on the lid.

Determination of the molar mass of an unknown

Repeat the above procedure for the unknown using the last test-tube in the boiling water bath. Remember to turn off the hotplate and put your large beaker back in your drawer, you will not need them for the remainder of the lab.

PART B Calculation of Cp for Al

mass of two cups, _____g, mass of two cups + water _____g, mass after experiment_____g

m_{WC} = mass of H_2O in calorimeter _____g

m_{MTT} = mass of metal in test-tube _____g

T_i = Initial temperature of H_2O in calorimeter _____°C

T_H = Highest T of H_2O in calorimeter after mixing _____°C

T_M = T of the metal in test-tube _____°C

ΔT_{WC} = Highest T of H_2O in calorimeter after mixing - initial T of H_2O in calorimeter = $T_H - T_i$ = _____ - _____ = _____°C

ΔT_{MTT} = Highest T of H_2O in calorimeter after mixing - T of the metal in test-tube = $T_H - T_M$ = _____ - _____ = _____°C

C_W = 4.184J/g°C

$-m_{MTT}Cp\Delta T_{MTT} = m_{WC}C_W\Delta T_{WC} + B\Delta T_{WC}$

Substitute your values in the equation above and solve for Cp

Calculate your molar mass given Cp • MM = 25 J/gK _____g/mol

How does it compare to the actual value for Al? _____ %error = _____

Determination of the molar mass of an unknown

mass of two cups, _____ mass of two cups + water _____ mass after experiment_____

m_{WC} = mass of H_2O in calorimeter _____g

m_{MTT} = mass of metal in test-tube _____g

T_i = Initial temperature of H_2O in calorimeter _____°C

T_H = Highest T of H_2O in calorimeter after mixing _____°C

T_M = T of the metal in test-tube _____°C

ΔT_{WC} = Highest T of H_2O in calorimeter after mixing - initial T of H_2O in calorimeter = $T_H - T_i$ = _____ - _____ = _____°C

ΔT_{MTT} = Highest T of H_2O in calorimeter after mixing - T of the metal in test-tube = $T_H - T_M$ = _____ - _____ = _____°C

C_W = 4.184J/g°C

$-m_{MTT}Cp\Delta T_{MTT} = m_{WC}C_W\Delta T_{WC} + B\Delta T_{WC}$

Substitute your values in the equation above and solve for Cp

What is your molar mass given Cp • MM = 25 J/g°C? _____

PART C Determining Enthalpies

For the remaining sections we are going to change the calculations to work with volume instead of mass. We also want to know the amount and sign of energy that is transferred, instead of finding a specific heat or determining a molar mass. For the remainder of the experiments we will, assuming constant pressure, work with a total volume of 100.0 mL and use the density of solution along with the specific heat of water to define a constant in our equation. Also the starting temperature of water, acid and base solution needs to be the same, so all solutions need to be made the night before so they will be at the same room temperature on the day of the experiment.

$\Delta H_{reaction}$ = heat given off by reaction to the solution – heat lost to calorimeter

$\Delta H_{reaction}$ = -$m_{solution}C_{solution}\Delta T_{solution}$ + B$\Delta T_{solution}$: mC = V•d•$C_{solution}$ = 100 • 4.10 J/K

If using 100 mL of solution, the equation becomes:

$\Delta H_{reaction}$ = -410.0J/K $\Delta T_{solution}$ + B$\Delta T_{solution}$

If the temperatures of the acid and base are not the same then each volume must be calculated separately at its own starting temperature and then added together, also any volume different than 100 mL will have to be recalculated.

Heat of Solution and Neutralization of 10.0 M H_2SO_4 and 1.00 M NaOH

1. In the calorimeter place 50.0 mL of 1.00 M NaOH and 45.0 mL of distilled water.
2. Measure the temperature of the water and base in the calorimeter and record it on your data sheet.
3. In your clean dry 10 mL graduated cylinder prepare exactly 5.00 mL 10 M H_2SO_4, use a disposable pipette to get the volume exactly 5.00 mL.
4. Carefully, as described earlier, check the temperature of the 10 M H_2SO_4. If it is the same as the water and base you can continue, if not warm up graduated cylinder in your hands or cool it a little on ice.
5. Recheck the volume of the H_2SO_4 and make it exactly 5.00 mL again.
6. Set up the calorimeter with the thermometer suspended from the ring stand, through the lid of the coffee cup.
7. One person carefully pours all of the 10 M H_2SO_4 it into the calorimeter while the other person secures the lid, stirs the solution and watches for the highest temperature achieved.
8. Record the highest temperature on the data sheet.
9. Calculate the enthalpy.
10. In this experiment the solution is not completely neutralized so empty the calorimeter into your 600 mL beaker. Ask the TA to dispose of waste mixture.

Heat of Solution of 10.0 M H_2SO_4

1. In the calorimeter place 90.0 mL of distilled water.
2. Measure the temperature of the water in the calorimeter and record it on your data sheet.
3. In your clean dry 10 mL graduated cylinder prepare exactly 10.00 mL 10 M H_2SO_4, use a disposable pipette to get the volume to exactly 10.00 mL.
4. Carefully, as described earlier, check the temperature of the 10 M H_2SO_4. If it is the same as the water you can continue if not warm up graduated cylinder in your hands or cool it a little on ice.
5. Recheck the volume of the H_2SO_4 and make it exactly 10.00 mL again.
6. Set up the calorimeter with the thermometer suspended from the ring stand through the lid of the coffee cup.
7. One person carefully pours all of the 10 M H_2SO_4 it into the calorimeter while the other person secures the lid, stirs the solution and watches for the highest temperature achieved.

8. Record the highest temperature on the data sheet.
9. Calculate the enthalpy.
10. In this experiment the solution is not completely neutralized so empty the calorimeter into your 600 mL beaker. Ask the TA to dispose of waste mixture.

Heat of Neutralization of 1.00 M H$_2$SO$_4$ and 1.00 M NaOH

11. In the calorimeter place 50.0 mL of 1.00 M NaOH.
12. Measure the temperature of the base in the calorimeter and record it on your data sheet.
13. In your clean dry 100 mL plastic graduated cylinder prepare exactly 50.0 mL 1.00 M H$_2$SO$_4$, use a disposable pipette to get the volume exactly 50.0 mL.
14. Carefully, as described earlier, check the temperature of the 1.00 M H$_2$SO$_4$. If it is the same as the water and base you can continue if not warm up graduated cylinder in your hands or cool it a little on ice.
15. Set up the calorimeter with the thermometer suspended from the ring stand through the lid of the coffee cup.
16. One person carefully pours all of the 1.00 M H$_2$SO$_4$ it into the calorimeter while the other person secures the lid, stirs the solution and watches for the highest temperature achieved.
17. Record the highest temperature on the data sheet.
18. Calculate the enthalpy.
19. In this experiment the solution is not completely neutralized so empty the calorimeter into your 600 mL beaker. Ask the TA to dispose of waste mixture.

Name _____ **TA** _____ **Time** _____

Heat of Solution and Neutralization of 10.0 M H_2SO_4 and 1.00 M NaOH

T_S = Temperature of H_2SO_4 _____ T_S = Temperature of NaOH and water _____ (MUST BE THE SAME)

T_H = Highest temperature of calorimeter after mixing _____

$\Delta T_{solution}$ = Highest T of H_2O in calorimeter after mixing – initial T of H_2O solution = $T_H - T_S$ = _____ - _____ = _____°C

B = _____

$\Delta H_{reaction}$ = -410.0J/K $\Delta T_{solution}$ + B$\Delta T_{solution}$

Calculate the $\Delta H_{reaction}$ = _____

How many moles of acid were used? _____

What is the $\Delta H_{reaction}$/mol? _____

Heat of Solution of 10.0 M H_2SO_4

T_S = Temperature of H_2SO_4 _____ T_S = Temperature of water _____ (MUST BE THE SAME)

T_H = Highest temperature of calorimeter after mixing _____

$\Delta T_{solution}$ = Highest T of H_2O in calorimeter after mixing – initial T of H_2O solution = $T_H - T_S$ = _____ - _____ = _____°C

B = _____

$\Delta H_{reaction}$ = -410.0J/K $\Delta T_{solution}$ + B$\Delta T_{solution}$

Calculate the $\Delta H_{reaction}$ = _____

How many moles of acid were used? _____

What is the $\Delta H_{reaction}$/mol? _____

Heat of Neutralization of 1.00 M H_2SO_4 and 1.00 M NaOH

T_S = Temperature of H_2SO_4 _____ T_S = Temperature of NaOH _____ (MUST BE THE SAME)

T_H = Highest temperature of calorimeter after mixing _____

$\Delta T_{solution}$ = Highest T of H_2O in calorimeter after mixing – initial T of H_2O solution = $T_H - T_S$ = _____ - _____ = _____°C

$\Delta H_{reaction}$ = -410.0J/K $\Delta T_{solution}$ + B$\Delta T_{solution}$

Calculate the $\Delta H_{reaction}$ = _____

How many moles of acid were used? _____

What is the $\Delta H_{reaction}$/mol? _____

Hess's law states that enthalpies are additive, that you can calculate the enthalpy of reaction directly or by steps in a circuitous path. Does your experimental results verify Hess's law?

Enthalpy for diluting and neutralization of 10M H_2SO_4 _____J/mol

Enthalpy for diluting plus enthalpy for neutralization _____J/mol + _____J/mol = _____J/mol

Are the two enthalpies the same? _____

Are they close? _____

What are some possible sources of error?

Which method do you think gives the most accurate result, to react all at once or to calculate the heat from each step?

If you used a wet metal in the first half of the experiment, would this increase or decrease the apparent specific heat capacity, and why?