

## CALORIMETRY

Purpose: You will monitor the change in heat associated with several processes and determine how they are related.

Background: Physical and chemical changes often give off heat (**exothermic**) or absorb heat (**endothermic**). As described in Burdge, we can measure such changes using a calorimeter, a device engineered to resist temperature changes itself and to minimize heat escaping to the surroundings. Coffee cups are great low-tech calorimeters, so that's what we will be using. The **law of conservation of energy** states that in normal processes, energy can be exchanged but not created or destroyed.<sup>1</sup> Thus when a reaction gives off energy, it is all absorbed by the surroundings as either heat or work. Since our coffee cups are not changing volume, no work can be done, so all the energy is transferred as heat. The heat of reaction ( $q_{\text{rxn}}$ ) can be calculated if we know the **specific heat**, mass, and change in temperature:

$$q_{\text{rxn}} = -q_{\text{calorim}} = -(\text{mass} \times \text{specific heat} \times \Delta T)$$

Since we know the specific heat of the solution in this lab, we need to use the masses of the solutions. Since the coffee cup is not air-tight, we are at constant pressure, and  $q_{\text{rxn}} = \Delta H_{\text{rxn}}$  in J. To compare different reactions, we'll need to divide by the moles of limiting reactant and convert J to kJ to get the value of  $\Delta H$  in kJ/mol. Although determining the mass is easy and specific heats can be looked up in reference books, finding a value for  $\Delta T$  is not quite as trivial. The initial temperature is obviously before the reaction starts, but what is the final temperature – right when the reaction begins, a minute later, 5 minutes later? The best way to determine  $\Delta T$  is to graph the temperature with respect to time (see Figure 1, next page) and draw two separate best fit lines: one for the data before the reaction, and one for the data after. The change in temperature is NOT the difference in the first and last temperature recorded; it is the difference in temperature at the time of mixing (arrow), which you determine by extrapolation. (In Figure 1,  $\Delta T = 33.6 - 23.4 = 10.2$  °C.) Note  $\Delta T$  can be positive (exothermic) or negative (endothermic).

Heats of reaction have been studied for many different types of reactions. Think back to the acid/base neutralization reactions you carried out during the titration labs – did you notice if these were exothermic or endothermic? If you didn't notice, don't worry. We are going to do another neutralization, between NaOH and HCl, in this lab. Heats of reaction are also associated with physical (not chemical) changes, like dissolving solids in water. **Hess's Law** (see our textbook) tells us that the heat of a reaction is constant whether it is carried out in one step or a series of steps. We will test this by carrying out a reaction in both stepwise and concerted fashions and seeing if the heats are consistent. To do this, we'll need to calculate the heat in kJ/mol because the exact number of moles used for the different steps may not be constant.

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<sup>1</sup> This law applies to normal physical and chemical changes.  $E = mc^2$  implies that pure energy and mass are interchangeable, not conserved. Physicists get around this by classifying mass as a form of energy. But since we aren't building nuclear reactors in lab, we don't have to worry about such caveats.

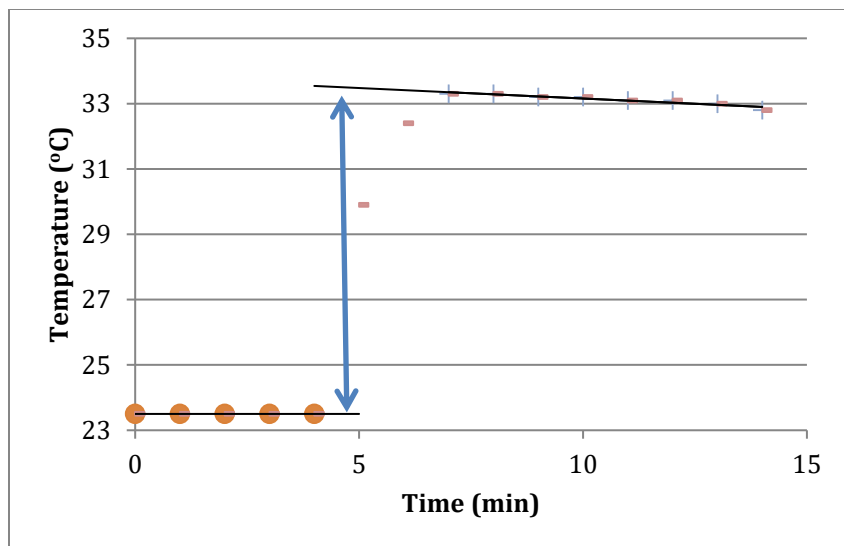


Figure 1: Finding  $\Delta T$  graphically

**Procedure:** This is the part you have to write in your notebook. Work with a partner.

From the designated point in the lab, collect two styrofoam coffee cups to use as your calorimeter, a lid, a thermometer, and an Erlenmeyer flask with a stopper. Set up the following reactions.

**Reaction 1:**  $\text{NH}_4\text{Cl (s)} \rightarrow \text{NH}_4\text{Cl (aq)}$

1. Weigh out (to 2 decimal places) about 2.5 g of solid ammonium chloride into a weigh boat. Use your graduated cylinder to pour 50.0 mL of deionized water into your nested styrofoam cup calorimeter, and place the calorimeter in an empty 250-mL beaker for stability. With the lid and thermometer in place, read the temperature for 3 minutes at 1-minute intervals to establish your initial temperature.
2. At minute 4, add the  $\text{NH}_4\text{Cl(s)}$ , and replace the lid and thermometer. Gently swirl the mixture and stir it with the thermometer to get the solid completely dissolved. Do not poke holes in your coffee cup or splash the cardboard lid. Check to make sure all the solid is dissolved. Continue the temperature readings at 1-minute intervals for 10 minutes. After 10 minutes, you can pour the solution down the drain.

**Reaction 2:**  $\text{NaOH (s)} \rightarrow \text{NaOH (aq)}$

1. Using gloves (NaOH is a strong base) weigh out (to the nearest 0.01 g) about 2 g of solid NaOH into an Erlenmeyer flask, not a weigh boat, and quickly insert a stopper. NaOH is hygroscopic, meaning it will absorb water out of the air and turn into a gooey mess if you don't stopper it. Use your graduated cylinder to pour 50.0 mL of deionized water into your coffee cup calorimeter, and place the cup in an empty 250-mL beaker for stability. With the lid and thermometer in place, read the temperature for 3 minutes at 1-minute intervals to establish your initial temperature.

- At minute 4 add the NaOH(s), and replace the lid and thermometer. Gently swirl the mixture and stir it with the thermometer to get the solid completely dissolved. Check to make sure the solid is dissolved. Continue the temperature readings at 1-minute intervals for 10 minutes. After 10 minutes, you can pour the solution down the drain.



- Measure out exactly 50.0 mL of 1.0 M HCl into the styrofoam cup calorimeter and exactly 50.0 mL of 1.0 M NaOH into a 100-mL beaker. Measure and record the temperature of the NaOH solution, and then that of the HCl solution. (These temperatures should be near room temperature and should not differ from each other by more than 2°C.) Read the temperature of the HCl in the calorimeter for 3 minutes at 1-minute intervals.
- At minute 4, quickly pour the NaOH into the HCl solution, mix thoroughly, and continue the temperature readings at 1-minute intervals for 10 minutes. After 10 minutes, you can pour the solution down the drain.



- Weigh out (to the nearest 0.01 g) about 2 g of solid NaOH in a stoppered flask. Measure about 55 mL of 1.0 M HCl, dilute this with deionized water to get a total volume of 100 mL (do not add 100 mL, get 100 mL total), and transfer this completely to your calorimeter. Read the temperature for 3 minutes at 1-minute intervals.
- At minute 4, add the NaOH (s), and replace the lid and thermometer. Gently swirl the mixture and stir it with the thermometer to get the solid completely dissolved. Check to be sure the solid is dissolved. Continue the temperature readings at 1-minute intervals for 10 minutes. After 10 minutes, you can pour the solution down the drain.

**Example Data Table:**

Data for Reaction 1		Data for Reaction 2		Data for Reaction 3		Data for Reaction 4		
Mass solute	Volume solvent	Mass solute	Volume solvent	Volume NaOH	Volume HCl	Mass NaOH	Volume HCl	Vol H <sub>2</sub> O
Time (min)	Temp °C	Time (min)	Temp °C	Temp NaOH °C	Temp HCl °C	Time (min)	Temp °C	
				Time (min)	Temp °C			

[Note: Include enough lines in the data tables in your notebook to accommodate all times and temperatures indicated in the procedure above.]

Return the clean and dry calorimeter, lid, thermometer, and Erlenmeyer flask with its stopper to the designated point in the lab.

After completing the procedure but before leaving lab, write in your notebook a brief statement (two to three sentences) on the quality and reasonableness of the data you collected. Note what you might do differently if you performed the lab again.