I. **PREPARATION**

Read "Principles of Calorimetry" in Ch. VI of “Experiments in physical chemistry”, Sixth Edition by Shoemaker, Garland, and Nibler (SGN6, pp. 145-151), as well as Experiment 6 (pp. 152-158). This experiment is quite similar to SGN's Experiment 6, except that we have a Parr two-valve bomb. Besides reading this writeup and the experiment in SGN, you may want to review your thermochemistry in your Freshman text. See also the section in SGN6 on temperature measurement, Ch. XVIII, especially pp. 589-604

Work out the exercise in the Data Analysis section *before* you come to lab and submit it to the TA in charge.

II. **PROCEDURE**

The procedure is similar in spirit to that described in SGN's Experiment 6, however, since we have the Parr bomb there are a number of minor differences. We will use fuse wire made from Chromel C instead of iron.

A. **Preparation of the Pellet**

1. When making pellets, use the following amounts:
   
   Benzoic Acid ................................ 0.9 - 1.1 g  
   Unknowns A, B, C, D, E ................. 0.5 - 0.75 g.

2. Prepare the pellet in the press and weigh it to 0.0002 g. Make sure you are using the vernier properly. Handle the pellet with tweezers. Don't touch it with your hands.

B. **Loading the Bomb**

1. Cut about 10 cm of ignition wire from the spool and record its weight.

2. Attach the wire to the posts in the bomb. Make sure that the wire presses against the pellet but does not touch the cup.

3. Add 1.0 ml of water to the bomb.

4. Flush the bomb twice with oxygen at about 10 atm and then fill it to about 30 atm. Always fill or bleed the oxygen slowly.

5. Check the circuit with an ohmmeter.
C. The Calorimeter

1. Place the bomb in the bucket, then fill the bucket with 2 l of water. The amount of water must be reproducible (so that the heat capacity of the calorimeter will be constant), but it is not necessary that it be exactly 2 l. We will use an automatic pipet manufactured by the Parr company which reproducibly delivers about 2 l of temperature-controlled water. You might want to measure the temperature to see if it is constant from run to run. Avoid dipping your hands in the water.

2. Place the bucket in the calorimeter and connect the electrodes.

3. Close the calorimeter, CAREFULLY lower the thermometer and the stirrer and start the motor. Be sure that the stirrer is not fouled by the wires.

D. Adiabatic Jacket

1. Bring the jacket temperature to the bucket temperature and maintain for about 4-5 minutes, taking readings every 30 seconds until the temperature is constant or the temperature rise is linear. Read the thermometer as precisely as you can (to ca. 0.002 °C).

2. Vibrate the thermometer, record the bucket temperature, and fire the bomb.

3. Match the bucket and jacket temperatures as closely as possible. During the rapid temperature rise following the firing of the bomb, it may be difficult to make the jacket temperature track exactly with the bucket temperature. They should be kept as close as possible. During the leveling off period, you should be able to match the two temperatures to within the precision of your measurements.

4. Once the temperature rise has slowed, begin taking readings from the bucket thermometer every 30 seconds. Continue to take readings until four successive readings yield the same result or the slope is linear. Record this final temperature.

E. Unloading

1. Stop the motor, CAREFULLY raise the thermometer and the stirrer and remove the bucket and bomb.

2. Pour the water back into the reservoir and dry the bucket for the next run.

3. Release the pressurized gas slowly from the bomb and then open the bomb.

4. Carefully remove any unburned fuse wire, straighten it, measure its weight and record that figure.
5. Rinse the bomb with water and dry it.

**IMPORTANT NOTES**

1. NEVER USE A WRENCH OF ANY SORT ON THE PARR BOMB!

2. NEVER ALLOW THE MERCURY IN THE THERMOMETERS TO GET BELOW THE EXPANSION BULB, i.e., DO NOT LEAVE THE COLD WATER ON!

3. IF THE BOMB LEAKS (emitting bubbles at a rate greater than 10 per minute), **DO NOT FIRE!!**..FIND OUT WHY IT LEAKS AND GET IT FIXED!

4. The amount of water added to the bucket depends on temperature. Either use the same temperature (in the volumetric flask) or apply a correction in your calculations.

5. If you are the last group to perform the experiment before an extended break (mid-semester, Spring, Summer, etc.) turn off the temperature controller/heater for the hot water bath and carefully remove the heater from the bath. This is necessary to deter corrosion of the apparatus by the water.

6. Do at least three runs with benzoic acid and at least three runs with one unknown using the adiabatic calorimeter.

**III. DATA ANALYSIS**

The general equation of interest here is 

$$\Delta E = C_v \Delta T.$$

The benzoic acid runs will determine $C_v$ for the calorimeter and, this being known, the heats of combustion of the unknown can be calculated. The heat of combustion of the ignition wire will have to be taken into account also:

$$C_v = \frac{(\Delta E_{BA} \infty m) + e}{\Delta T}$$

where:

\[m\] = weight of benzoic acid sample

\[e\] = error due to wire burning = 1400 cal/g \times mass of wire burned in g
\( \Delta T \) = temperature change, i.e., the difference between the corrected final and initial temperature readings

\( \Delta E_{BA} \) = heat of combustion of benzoic acid = 6314.6 cal/g

The same equation can be rewritten to get \( \Delta E \) for the unknown:

\[
\Delta E = \frac{C_V \Delta T - e}{m}
\]

**Exercise.** Write a Matlab routine to analyze the calorimetric data in the Chem381 Web site. Calculate the heat capacity of the bomb and \( \Delta E \) for the unknown. Turn in your Matlab program, plots of Temperature vs time, and values of \( C_V \) and \( \Delta E \). For this exercise it is not necessary to consider an analysis of the errors.

**IV. Written Report and Error Analysis**

You may find the discussion in SGN6, pp 10-24 useful in preparing your report.

**Introduction, Experimental, and Data:** Except for complicated mathematical expressions your report must be typewritten. Your report should include a brief (~half-page) introduction, summarizing the theoretical background, and the objective and approach of the experiment. If the experimental method described in this writeup and in SGN was simply followed, so state, but be sure to mention any procedural changes. Include your raw data, preferably in tabular form.

**Results:** Turn in your results together with sample calculations. They should be plainly marked and neatly arranged. The mean values you obtained for the heat of combustion of your unknown must be explicitly given together with your estimate of their uncertainty (below). Specify units for all numbers! Be sure to indicate which unknown was used. When you have completed your calculations, identify your unknown by comparing the heat of combustion you have obtained with those given in the table below.

**Discussion and Error Analysis:**

For this experiment a simplified error analysis will suffice. Estimate the uncertainty in \( C_V \) and \( \Delta E \) by calculating the standard deviation of the mean for each (SGN pp. 43-44). Since the value of \( \Delta E \) for your unknown depends on your value of \( C_V \) as well as on the calorimetric measurements with the unknown, the uncertainty in \( C_V \) adds additional uncertainty to \( \Delta E \) beyond that estimated from the standard deviation of the mean of \( \Delta E \). How much?

It is useful (but maybe somewhat discouraging!) to consider the accuracy you should have gotten. This determination relies only on temperature, weight, and volume measurements, and you can estimate how much the uncertainty in each affects the final answer. For example, you should be able to weigh to ±0.5 mg (and accuracy of ±0.1 is attainable). How much does this
affect the final result? What is the biggest source of error? Discuss your results, identifying as best you can the most important sources of random and systematic errors, human, mechanical or otherwise.
# LIST OF POSSIBLE UNKNOWNS

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<tr>
<th>Compound</th>
<th>$\Delta E$, cal/g</th>
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<tr>
<td>Menthol</td>
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