

## Physical Chemistry Lab Report Rubric -Veldman Fall 2012

### Formatting (10 Points)

- \*Used ACS template
- \*Times New Roman Font
- \*Double spaced text

### Manuscript Criteria (20 Points)

- \*1200-1500 words
- \*Maximum of 3 Labeled figures, tables, charts graphs or images with captions
- \*AT LEAST 5 Citations (ACS formatting)

### Language (10 Points)

- \*No misspelled words
- \*Correct usage
- \*Past tense for methodology and discussion
- \*Present tense when referring to data
- \*Did not use pronouns (My, me, our, we etc.)

### Content (50 Points)

\*Abstract: Is a brief summary of the report including the methods used, all pertinent results and known values, mentions any known major error that may explain major inconsistencies. Max of 150 words

\*The information section is self-contained and sufficient to explain why the experiment was performed and why the method was used and how it could be used to yield the sought after result. ~400-500 words.

\*The methodology gave a concise and complete overview of what the experiment without delving into procedural monotony. Cite the Lab manual. ~200-300 words

\* Data/Results section should systematically present the data and mention any inconsistencies and how they were addressed (such as additional analysis performed, or extra data collected). Includes a summary of the pertinent or key results and gives the literature values in an easily accessible way.

\*The discussion should be the focus of the report where the results summarized in the data section are explained and provides justification for the results or a well sported explanation for inconsistencies in the data.

\*Citations At least 5 citations in proper ACS format.

### Professionalism (10 Points)

\*Tone and explanations of the manuscript are professional and well sported with facts rather than excuses.

Levels of Achievement Criteria					
	Not Ready for Publication	Publish with Major Revisions	Publish with Minor Revisions	Ready for Publication	Comments
Formatting	<b>0 Points</b>	<b>5 Points</b>	<b>8 Points</b>	<b>10 Points</b>	
Manuscript Criteria	<b>0 Points</b>	<b>10 Points</b>	<b>15 Points</b>	<b>20 Points</b>	
Language	<b>0 Points</b>	<b>5 Points</b>	<b>8 Points</b>	<b>10 Points</b>	
Content	<b>20 Points</b>	<b>30 Points</b>	<b>40 Points</b>	<b>50 Points</b>	
Professionalism	<b>0 Points</b>	<b>5 Points</b>	<b>8 Points</b>	<b>10 Points</b>	
<b>Over all</b>	<b>/ 100 Points</b>				

# Determination of the Enthalpy of Combustion of Sucrose Using Bomb Calorimetry

## Abstract

The heat of combustion of sucrose ( $C_{12}H_{22}O_{11}$ ) was experimentally determined by adiabatic bomb calorimetry. The calorimeter constant  $C_{vcal} = 8.78 \text{ kJ/}^\circ\text{C}$  was determined via the combustion of standard benzoic acid ( $C_7H_6O_2$ ),  $\Delta U = -26.41 \text{ kJ/g}$ . Once the calorimeter had been calibrated, the change in internal energy of sucrose was determined to be  $-6.97 \pm 0.03 \text{ kJ}$ . Using the change in internal energy, the heat of combustion of sucrose  $\Delta_c H = -4599 \pm 257.9 \text{ kJ/mol}$  was estimated by substitution of the ideal gas law,  $\Delta H = \Delta U + R \cdot T_d \cdot \Delta n_{gas}$ . The calculated heat of combustion was compared to an accepted literature value  $\Delta_c H^\circ_{solid} = -5643.4 \pm 1.8 \text{ kJ/mol}$  resulting in an error of 18.50%.

## INTRODUCTION

One of the oldest known scientific methods used to measure energy transfer due to heat evolution during a chemical reaction is known as calorimetry (1). The device utilized to measure the energy transfer is known as a calorimeter. In particular, bomb calorimetry is a feasible way to determine the unknown heat of combustion value of a substance such as sucrose when a known heat of combustion value of a substance has been accurately determined and is easily accessible (2), such as that for benzoic acid.

Calorimeters are regularly enclosed by an additional water bath that is kept at equal temperature to the calorimeter in order to avoid heat loss from the system to the surroundings. Such systems are termed adiabatic because heat ( $q$ ) is not lost nor gained, thus  $q$  is equal to 0. The temperature change of the water is then measured to determine the energy output of the sample combustion. The calorimeter is treated as an adiabatic system because it was thermally insulated and was proficient in preventing heat loss.

In order to use this method to calculate heat of combustion of a substance, a substance such as benzoic acid with a known heat of com-

bustion must first be used to determine the calorimeter constant. The sample is made into a pellet, weighed, and then placed into a crucible held inside the bomb. The sample comes into contact with an ignition wire, and the bomb is pressurized with 25-30 atmospheres of oxygen gas. Once the bomb has been placed into a known volume of water, and the initial temperature has been determined and measured for a certain amount of time, the bomb is ignited and an electrical current passes through to ignite the substance. Firing of the bomb, and consequently combustion of the substance, will raise the temperature of the water quickly and significantly due to the heat evolved during the course of the reaction. This process will give initial temperature ( $T_{initial}$ ) and final temperature ( $T_{final}$ ), which will be used to determine the heat capacity ( $C_{vcal}$ ) of the calorimeter via the following equation:  $\Delta U_{benz} = -C_{vcal} \cdot (T_{final} - T_{initial})$ . Knowing the relationship between heat of combustion and internal energy, the following equation can be employed:  $\Delta U = \Delta H - \Delta(pv)$  (3). By treating the products of the combustion reaction as ideal gasses, the following equation then arises:  $\Delta H = \Delta U + RT \Delta n_{gas}$  (4), bestowing the enthalpy of combustion of a pure substance such as sucrose, which was used in this experiment.

## METHODOLOGY

The calorimeter holds a metal, thick-walled container, which is the bomb. The bomb holds the sample to be burned, which will first be benzoic acid then sucrose, in a metal crucible. A small length of wire, about 10 cm, feeds through and is in direct contact with the sample. An electric current is passed through the wire, heating it rapidly, thus initiating combustion. The bomb must then be sealed tightly and filled with pure oxygen at 25-30 atm. It is important to fill and vent the oxygen a couple of times first to flush out any atmospheric nitrogen, then fill a third time without venting to pressurize the bomb. The bomb is then ready to be placed in the calorimeter. Water is poured into the calorimeter, completely submerging the bomb, and must constantly be stirred to help ensure a uniform temperature throughout the bath, which itself is isolated from the surroundings.

Equipped with a thermometer, the temperature is measured before combustion for 10 minutes, once the bomb has been fired for approximately 10 minutes, and after the sample has been combusted. For speedy and complete combustion of the sample, the system is connected electrically and a current is passed through the wire. The temperature then rises rather quickly and the sample is ignited, during which, the wire may also be mostly or completely burned. The walls of the bomb are made of thick alloy metal to make certain that combustion takes place at constant volume. We are therefore primarily concerned with change in internal energy,  $U$ . When this internal energy is released from the sample being combusted, it will be maintained in the water bath and the walls of the bomb due to the fact that the system is adiabatic as the apparatus is thermally insulated. Finally, by measuring the rise in temperature and the heat capacity of the calorimeter via the calibration run, it is possible to measure the internal energy released by the reaction.

## DATA AND DISCUSSION

The energy ( $\Delta U$ ) of benzoic acid is known to be  $-26.41 \text{ kJ/g}$  (4). This value made possible the determination of the calorimeter constant ( $C_{vcal}$ ), which was calculated to be  $8.78 \text{ kJ/}^\circ\text{C}$ . Figure 1 shows temperature vs. time for the benzoic acid combustion. After the ignition of the sucrose sample with an unknown heat of combustion, the experimental heat of combustion for sucrose can be determined. The literature value for the heat of combustion ( $\Delta H$ ) of sucrose is  $-5643 \text{ kJ/mol}$  (5). The literature value was also determined via bomb calorimetry so the experimental differences should be negligible. Table 1 summarizes the results of the bomb calorimetry experiment. Trial 1 of this experiment yielded a value of  $-4782 \text{ kJ/mol}$  and trial 2 produced a value of  $-4417 \text{ kJ/mol}$ , thus the average  $\Delta H$  was  $-4599 \pm 257.9 \text{ kJ/mol}$ . Figure 2 shows temperature vs. time for the two sucrose combustion trials. However, this average value does not quite compare to the literature value, and the percent error equates to 18.50%. Figure 2, however, display the data as expected; initial temperature is stable, there is a steep and steady increase once the bomb is ignited, and then the temperature begins to slowly decrease after the maximum temperature has been reached. This behavior is typical of a combustion reaction in which bonds are breaking and releasing energy in the form of heat.

**Table 1:** Bomb Calorimetry Results.

$C_{v(cal)}$ (kJ/ $^\circ\text{C}$ ):	8.78
$\Delta H_{\text{sucrose 1}}$ (kJ/mol):	-4782
$\Delta H_{\text{sucrose 2}}$ (kJ/mol):	-4417
Avg. $\Delta H_{\text{sucrose}}$ (kJ/mol):	-4599
Std. Dev. (kJ/mol):	257.9
Literature Value (kJ/mol):	-5643.4
Percent Error (%):	18.50

The 18.50% difference could be the result of several factors. The pressure of the oxygen in the bomb varied with each trial. There was 26 atm in the calibration trial, 23 atm in sucrose trial 1 run, and 22 atm in sucrose trial

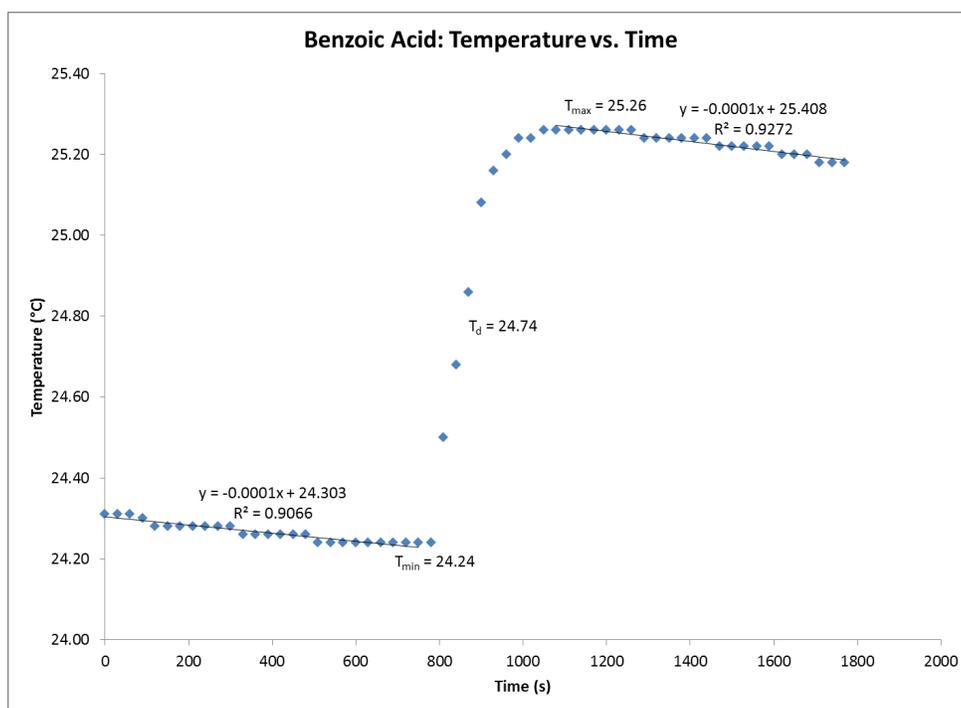


Figure 1: Temperature vs. time graph for the combustion of benzoic acid. The mass of the benzoic acid pellet was 0.4200 g and the pressure inside the bomb was 26 atm.

2 run. Also, the initial temperature of the water should have been above 25 °C but was close to 22 °C for each trial. These numbers, though small, can still lead to significant error when calculating values pertaining to energy. It is also true that the process was not completely adiabatic. Heat is still capable of escaping through the walls of the calorimeter, thus adding uncertainty to the recorded temperature values. The nichrome wire is also a potential source of uncertainty in the experiment. For example, completely neglecting the change in internal energy caused by the combustion of the nichrome wire would decrease the experimentally determined calorimeter constant which result in a smaller  $\Delta H$  of sucrose which would increase error of the experiment to 18.96%. The amount of water surrounding the bomb is also a source of error. Even a small amount of uncertainty in the amount of water measured could lead to error in the experiment. For example, if 2001 mL was measured rather than the expected 2000 mL, the water

would require 4.186 more Joules of energy to increase the temperature by 1 °C. Still, the values properly indicate that this process was highly exothermic, and the error is not large enough to invalidate the results.

## CONCLUSION

Benzoic acid was used as a standard to determine the calorimeter constant of the bomb calorimeter used in this experiment. The calorimeter constant was found to be 8.73 kJ/°C. The heat of combustion of sucrose was determined to be  $-4599 \pm 257.9$  kJ/mol. Comparison of our result to the literature value of  $-5643.4 \pm 1.8$  kJ/mol results in an error of 18.50%. This difference is most likely due to the multiple sources of error described in the discussion section. We can conclude that the use of bomb calorimetry to determine the heat of combustion of sucrose was successful despite moderate experimental error.

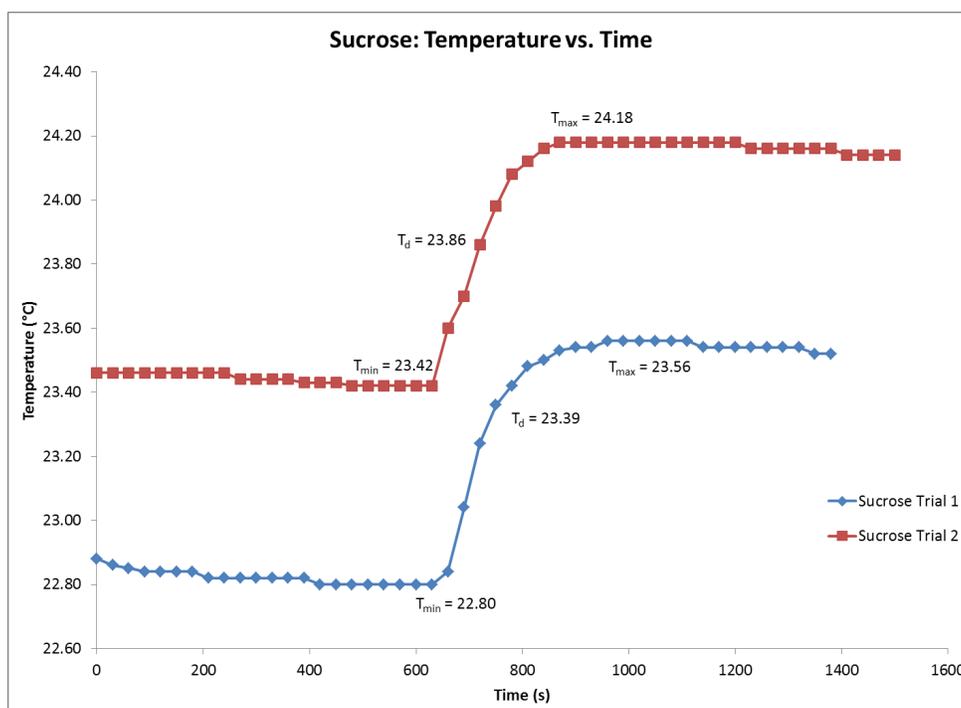


Figure 2: Temperature vs. time graph for the combustion of sucrose. Trial one used sucrose mass 0.4977 g at 23 atm, and trial two used 0.5353 g at 22 atm.

## REFERENCES

- [1] Silbey, R.; Alberty, R.; Bawendi, M. *Physical Chemistry*. Wiley: Hoboken, NJ, 4th Edition, 2004.
- [2] Salzberg, H.; Morrow, J.; Green, M. *Physical Chemistry Laboratory: Principles and Experiments*. pp 65-68, Macmillian: New York, NY, 1978.
- [3] Atkins, P.; De Paula, J. *Physical Chemistry*. pp 54-74, Oxford University Press: Oxford, UK, 9th Edition, 2010.
- [4] CSU Channel Islands. *Physical Chemistry Laboratory Manual*. pp 19-28, Fall Edition, 2012.
- [5] Ponomarev, V.V.; Migarskaya, L.B., *Heats of combustion of some amino-acids*. Russ. J. Phys. Chem. (Engl. Transl.), 34, pp 1182-1183, 1960.

Levels of Achievement Criteria		Comments
Formatting	<b>10 Points</b>	Full journal formatting was not required, but very nice.
Manuscript Criteria	<b>20 Points</b>	
Language	<b>10 Points</b>	
Content	<b>50 Points</b>	
Professionalism	<b>10 Points</b>	
<b>Over all</b>	<b>100 / 100 Points</b>	

# Determination of the Enthalpy of Combustion of Sucrose using Bomb Calorimetry

## Abstract

Calorimetry is the science of measuring the heat of chemical reactions and the subsequent change in internal energy. An experiment was conducted to determine the enthalpy of combustion of sucrose using Bomb Calorimetry. A benzoic acid calibration was performed to determine the specific heat capacity of the bomb calorimeter, which was found to be  $-9.146 \text{ kJ/K}$ . The specific heat capacity was in turn used to calculate the enthalpy of combustion of sucrose:  $-5199.8 \text{ kJ/mol}$ . The final change in enthalpy was  $5199.77 \text{ kJ/mol}$ , deviating  $9.26\%$  from the literature value of  $-5643.4 \text{ kJ/mol}$ .<sup>2</sup>

## Introduction

A Bomb calorimeter is a constant-volume calorimeter utilized to measure the heat of combustion of the enclosed chemical reaction. The heat of reaction is related to the change in internal energy and is shown by:

$$\Delta U = q + w \quad (1)$$

where  $q$  is the heat absorbed or produced and  $w$  is the work done on or by the system. The system is at constant volume and does not undergo expansion work. Therefore, the change of internal energy of the system is equal to the heat of combustion.

$$\Delta U_{v_{\text{rxn}}} = q_{v_{\text{sys}}} \quad (2)$$

All the energy released by the combustion reaction is transferred as heat into the surroundings. This allows for the determination of the heat of combustion of the system at constant volume and is related to the specific heat capacity,  $C_v$ , of the system and the temperature change. The heat of combustion can be obtained from the following equation,

$$-q_{\text{rxn}} = q_{\text{sys}} = C_{v_{\text{sys}}} \Delta T \quad (3)$$

If the system does not perform any additional work during the reaction, the change in enthalpy,  $\Delta H$ , is equal to the heat transferred to the system at constant pressure and moles. Any additional work from increased pressure is negligible:

$$\Delta H = \Delta U + \Delta nPV \quad (4)$$

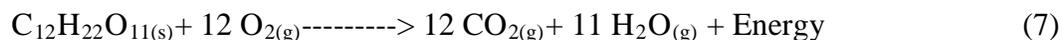
The following equation is then utilized to calibrate the calorimeter:

$$\Delta H \approx C_{v,p} \Delta T \quad (5)$$

Benzoic acid will be used as a means of obtaining the heat capacity of the calorimeter. Benzoic acid was chosen because it burns completely in oxygen, is not hygroscopic, and is readily available in pure form, and the literature values (enthalpy of combustion, etc) are well established.<sup>2</sup> The following reaction occurs during combustion of benzoic acid:<sup>4</sup>



Our study will be on the enthalpy of combustion of sucrose:



However, as seen in equation 4, not all of the energy released goes to heating the water. Due to logistics of the experiment, there is contribution of the fuse wire to internal energy and its burning used to start the reaction:

$$\Delta U_{\text{comb}} + \Delta U_{\text{wire}} = -C_v \Delta T \quad (8)$$

The heat capacity for the bomb calorimeter can be calculated where  $\Delta U_{\text{comb}}$  is the internal energy of the benzoic acid standard and  $\Delta U_{\text{wire}}$  is the energy released due to combustion of the nichrome wire. If we assume ideal behavior for all gases due to the gaseous products of the reaction, the combustion reaction can be manipulated to,

$$\Delta H = \Delta U + R^* T_d^* \Delta n_{\text{gas}} \quad (9)$$

where R is the ideal gas constant,  $T_d$  is the midpoint temperature, and  $\Delta n$  is the stoichiometric relationship between gaseous products to gaseous reactants.

### Methodology

A Parr-type constant-volume bomb calorimeter was utilized for determination of the enthalpy of combustion for sucrose. The calorimeter consisted of the bomb, an adiabatic container, and a firing unit. The bomb was filled with oxygen at 27atm, sealed, and surrounded with water. The bomb and cap were precisely made for exact fit. A one-way needle valve allowed for venting the bomb after experiment completion. The bomb was loaded with ~1g of sample.<sup>1</sup>

The complete combustion of the sample was initiated by passing a current through the wire in contact with the sample (benzoic acid or sucrose). Upon reaction, a temperature change was observed and recorded in the bomb and surrounding water. The stirrer ensured even heat distribution throughout the surrounding water where the temperature change was recorded via high precision thermometer. A calibration run was performed using benzoic acid to calculate the experimental heat capacity of the bomb. The combustion enthalpy for benzoic acid in the solid state is -26.41kJ /g.<sup>5</sup>

The calorimeter was plugged into the ignition unit and the stirrer was turned on and temperature was recorded every 30 seconds for 10 minutes. The bomb was then fired and temperature taken every 30 seconds and 10 minutes after the maximum temperature was achieved. The bomb was vented and the wire weighed to determine the mass combusted.<sup>4</sup> The temperature was measured

for 10 minutes prior to ignition to account for any addition of thermal energy contributed by the rotating stirrer, which would affect the heat capacity; a constant temperature is desired.

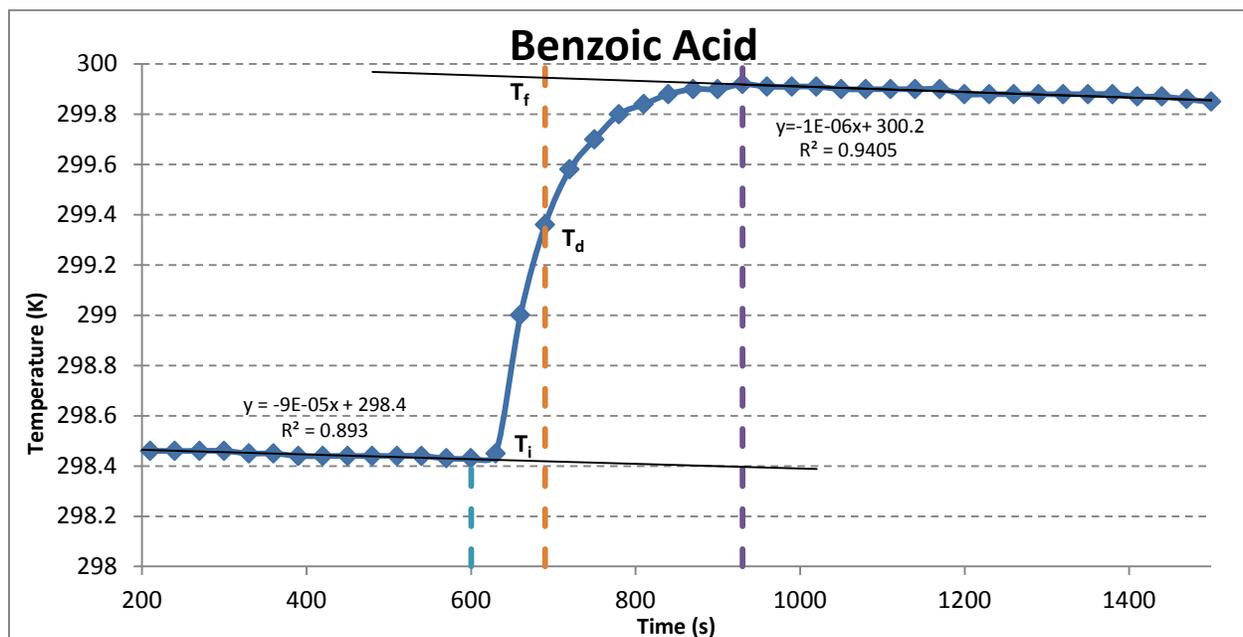
### Results

The change in temperature for the combustion of benzoic acid was plotted versus time to determine the initial (pre-fire) and final (post-max) temperatures, and to extrapolate the midpoint temperature with a linear trendline at  $t_{\text{fire}}$  and  $t_{\text{max}}$ . These temperatures were used to find the change in temperature during combustion and to further calculate the specific heat for benzoic acid.

Sample	$\Delta$ mass of wire (g)	Temp water bath (°C)	Bomb Pressure (atm)	Bomb volume (mL)
Benzoic Acid	0.0088	25.6	27	341
Sucrose	0.0082	25.8	24	341

**Table 1:** Raw preliminary data for benzoic acid and sucrose.

Preliminary data shown in Table 1 were used in calculations for the determination of benzoic acid from the run diagram shown in Figure 1.

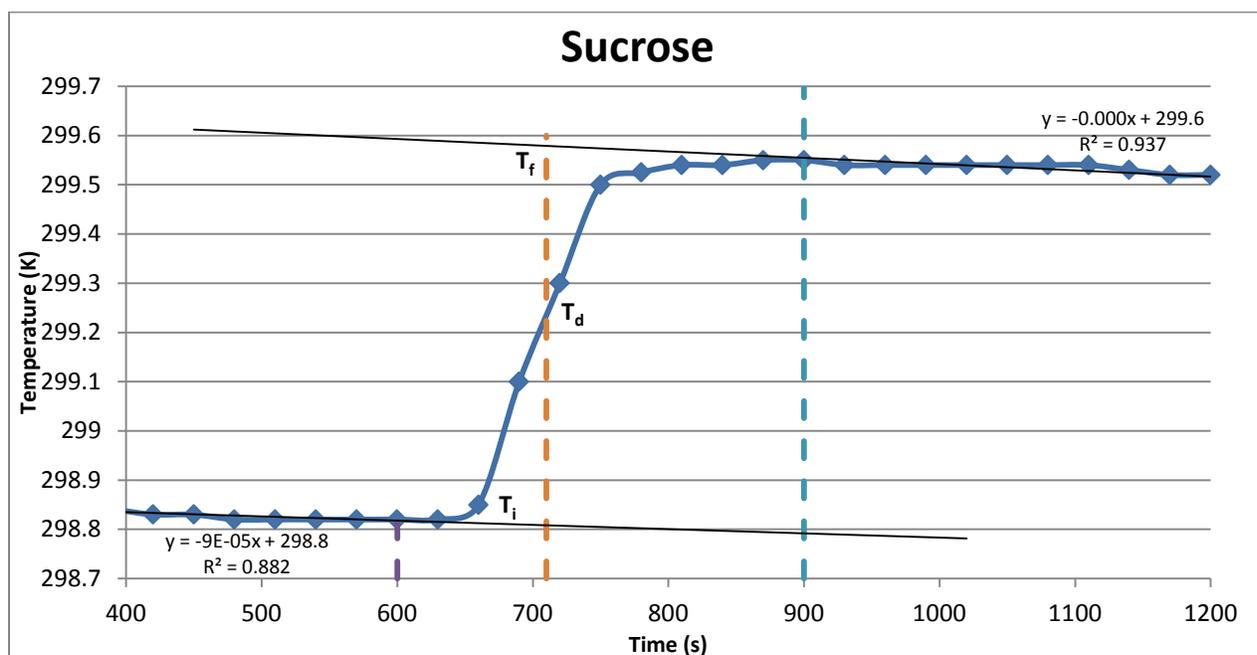


**Figure 1.** Temperature profile for a real calorimeter for the combustion of benzoic acid. Line at time=600s is the time the filament was fired,  $t_{\text{fire}}$ , and line at time=920s is the maximum temperature. The midpoint temperature,  $t_d$ , is determined with line at  $t=690$ s.

The heat capacity of the bomb and calorimeter,  $C_{v,\text{cal}}$ , was found by determining the near-isolated temperature change obtained from the specific enthalpy of combustion of benzoic acid and fuse wire using equation (8), yielding a value of 21.66kJ/gK. The change in internal energy of the fuse wire is incorporated into the equation with a value of -5.86kJ/g. These values all contribute to determining the enthalpy of combustion and change in internal energy for sucrose.

Sample	Mass of pellet (g)	$\Delta T$	$\Delta U_{\text{comb}}$ (kJ/g)	$C_v$ (kJ/gK)
Benzoic Acid	0.5868	1.49	-26.41	21.66
Sucrose	0.572	0.73	-15.1	-----

**Table 2:** Combustion of benzoic acid with calculated values.



**Figure 2:** Temperature profile for the combustion of sucrose in a realistic calorimeter.

The midpoint temperature,  $T_d$ , is necessary to find for a bomb calorimeter because a realistic combustion does not follow the ideal situation in which the slopes from  $t=0$  to  $t_{\text{fire}}$  and  $t_{\text{max}}$  to the end of the run are not zero. From the trendlines constructed from these temperatures, the midpoint temperature is determined and used to calculate the enthalpy of combustion.<sup>4</sup>

The values above were used to calculate the change in enthalpy of sucrose, which was found to be  $-8.689\text{kJ/g}$ . The values utilized in this calculation included  $t_{\text{fire}}$ ,  $t_{\text{max}}$ , and  $t_d$ . The midpoint temperature,  $t_d$ , is necessary to find for a bomb calorimeter because a realistic combustion does not follow the ideal situation in which the slopes from  $t=0$  to  $t_{\text{fire}}$  and  $t_{\text{max}}$  to the end of the run are not zero. Using the trendlines constructed from these temperatures, the midpoint temperature was determined and used to calculate the enthalpy of combustion. The first change in enthalpy of sucrose was used to find the corrected enthalpy with equation (9), resulting in an enthalpy change of  $\Delta H = -5199.8\text{kJ/mol}$ . Compared with the literature value of  $-5643.4\text{kJ/mol}$ , experimental values deviated by 9.26%. Error was calculated by determining the quotient of the literature value for the enthalpy of sucrose over the experimental value, subtracted from one, shown in the equation below:

$$\% \text{ Error} = \left| 1 - \frac{\Delta H_{\text{suc(lit)}}}{\Delta H_{\text{suc(exp)}}} \right| * 100 \quad (10)$$

## Discussion

The difference between the enthalpy of combustion and change in internal energy for sucrose was  $34.46\text{kJ/mol}$ . According to equation (5), the enthalpy of combustion and change in internal energy are both equal to the heat capacity of the bomb multiplied by the change in temperature. This made the values nearly equal to each other.

The enthalpy of combustion for the nichrome wire was very important to include in the calculations for  $\Delta U$  of sucrose. If the enthalpy for the nichrome wire were not included in calculations for the enthalpy for sucrose, values would have resulted in a 28.7% error.

Sources of error include the percent error in water measurement and incomplete vaporization of the nichrome wire. The internal volume of the Parr-bomb was measured to be 341mL. Inaccurate measurements could lead to water vaporization within the bomb after ignition, therefore changing the values of internal energy and enthalpy of vaporization because the system would no longer be at constant pressure.<sup>3</sup> If vaporization of water occurred during the run, the enthalpy would have increased significantly; a value of -77476kJ/mol was determined for increased water vapor in the bomb- only 3 more moles of water vaporized. If there had been incomplete burning of the nichrome wire, the change would have been smaller. The enthalpy of vaporization would change to -4756.7kJ/mol. In the actual calculation for sucrose, the change in moles is negligible if all the products are in the condensed phase.<sup>4</sup>

Ways of minimizing vaporization of water are by carefully monitoring temperature and not firing the bomb unless the temperature change is no more than 0.01°C per minute. Methods of reducing error include accurate temperature measurements more accurate volume determination.

## References

<sup>1</sup> Atkins, P; de Paula, J. Eds. *Atkins' Physical Chemistry*. 8<sup>th</sup> Edition; Oxford University Press: Oxford, 2010; 44-73.

<sup>2</sup> Mira R. Bissengaliyeva, Daniil B. Gogol, Shynar T. Taymasova, and Nuraly S. Bekturganov *Journal of Chemical & Engineering Data* **2011** 56 (2), 195-204

<sup>3</sup> N.K Smith, R.C Stewart, A.G Osborn, D.W Scott, *The Journal of Chemical Thermodynamics*, 1980; 919-926

<sup>4</sup> *Physical Chemistry Lab Manual*; California State Channel Islands: Camarillo, 2012

<sup>5</sup> [Ponomarev, V.V.](#); [Migarskaya, L.B.](#), *Heats of combustion of some amino-acids*, **Russ. J. Phys. Chem. (Engl. Transl.)**, 1960, 34, 1182-1183

Levels of Achievement Criteria					
	Not Ready for Publication	Publish with Major Revisions	Publish with Minor Revisions	Ready for Publication	Comments
Formatting	<b>0 Points</b>	<b>5 Points</b>	<b>8 Points</b>	<b>10 Points</b>	
Manuscript Criteria	<b>0 Points</b>	<b>10 Points</b>	<b>15 Points</b>	<b>20 Points</b>	*Only one graph for finding $T_{max}$ and $T_{min}$ is needed. *A summary table of pertinent data would make the analysis easier to follow
Language	<b>0 Points</b>	<b>5 Points</b>	<b>8 Points</b>	<b>10 Points</b>	Tense issues
Content	<b>20 Points</b>	<b>30 Points</b>	<b>40 Points</b>	<b>50 Points</b>	*U is related to H through the change in moles not volume. ( $H=U+nRT$ ) *Additional analysis needed
Professionalism	<b>0 Points</b>	<b>5 Points</b>	<b>8 Points</b>	<b>10 Points</b>	
<b>Over all</b>	<b>83/ 100 Points</b>				

## Bomb Calorimetry

### Abstract:

The purpose of this experiment was to determine the change in internal energy of a sucrose combustion system by calibrating a bomb calorimeter in order to find the heat capacitance of the calorimeter. Bomb calorimetry can measure the change in enthalpy because there is no change in volume so there is no work done by expansion and any internal energy is measured as a heat transfer. The specific heat capacitance of the bomb was found to be 6.098 by using equation 2. The average change in internal energy ( $\Delta U$ ) was found to be 46.65J, and the average change in enthalpy ( $\Delta H$ ) was found to be 113.195J. Although the results weren't similar to the ones given as literature values, they were similar enough to those of our peers, resulting in a remotely successful experimental determination of organic compounds using bomb calorimetry.

This is how it was submitted.

### Introduction:

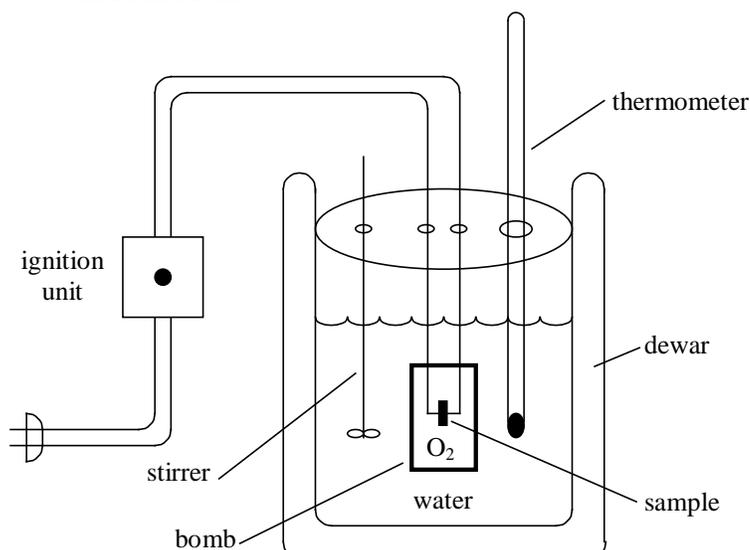
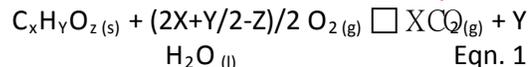


Figure 1 Schematic drawing of the bomb calorimeter.

Bomb calorimetry is used to measure the change in internal energy of combustion reactions with the general formula given in Equation 1 (Eqn. 1):



Calorimetry is a method that is used in order to measure the change in internal energy of a system by accounting for the transfer of heat in a system and the set-up is similar to that of Figure 1. The change in internal energy is equivalent to the sum of the work done on or by the system, and the heat lost or gained by the system as shown in Equation 2.

$$\Delta U_{\text{calorimeter}} = q_{\text{calorimeter}} + w_{\text{calorimeter}} \quad \text{Eqn. 2}$$

In order to only measure the heat of the system, there must be no expansion work done on the system, which is accomplished by using a container that keeps a constant volume so that all energy is measured in heat as demonstrated in Equation 3.

$$W_{\text{cal}} = 0, \text{ and } \Delta U_{\text{cal}} = q_{\text{cal}}. \quad \text{Eqn. 3}$$

The heat lost by the reaction is equivalent to the heat gained by the system because the system is at constant volume, which is interconnected to the heat specific heat capacity ( $C_v$ ) as well as the change in temperature as shown in Equation 4:

$$-q_{v_{\text{rxn}}} = q_{v_{\text{sys}}} = C_{v_{\text{sys}}} \cdot \Delta T \quad \text{Eqn. 4}$$

Calorimetry can be a very accurate method of calculating enthalpies of reactions if it is performed with care. Each calorimeter system must be calibrated in order to account for any error that may occur due to the set up of the system. One of the two ways to calibrate the system is to run a sample that is replaced by an electric heater, and the electrical work ( $w_{\text{electric}}$ ) done from the heater is transmitted into the system as heat. The change in the temperature of the system that occurs can be utilized to figure out the heat capacity of the calorimeter illustrated in Equation 5.

$$C_{v_{\text{sys}}} = \frac{w_{\text{electric}}}{T_{\text{final}} - T_{\text{initial}}} \quad \text{Eqn. 5}$$

Electrical work can be calculated by calculating the amount of power (P) used by the heater during the time the experiment was operational. The current (I) is introduced into the system at a certain voltage (V), which is equivalent to the power used to operate the heater as shown in Equation 6.

$$w_{electric} = P \cdot \Delta t = V \cdot I \cdot \Delta t \quad \text{Eqn. 6}$$

The other way to calibrate the heat capacity of the calorimeter is to perform a reaction with a sample with known thermodynamic properties like the change in internal energy and illustrated in Equation 7.

$$\Delta U_{known} = -C_{v_{calorimeter}} \cdot (T_{final} - T_{initial}) \quad \text{Eqn. 7}$$

The calorimeter heat capacity is then used in further experiments of substances with unknown thermodynamic properties by plugging in the heat capacity in Equation 3.

The change in enthalpy ( $\Delta H$ ) is used more often the internal energy because it is equal to the bond energy as in Equation 8 where  $p$  is the pressure, and  $v$  is the volume:

$$\Delta U = \Delta H - \Delta(p \cdot v) \quad \text{Eqn. 8}$$

Although it is hard to measure reactions adiabatically, calorimetry quantifies enthalpy results due to the association shown in Equation 8. The ideal gas law can be plugged into Equation 8 if it is presumed that the gas will act ideally, and would convert into Equation 9:

$$\Delta H = \Delta U + R \cdot T_d \cdot \Delta n_{gas} \quad \text{Eqn. 9}$$

The change in enthalpy is associated with the change in moles,  $n(\text{products}) - n(\text{reactants})$ , due to  $R$  and the midpoint temperature being fairly constant. The last variable set becomes insignificant when the products are in the condensed phase and the change in enthalpy becomes Equation 10:

$$\Delta H = \Delta U \quad \text{Eqn. 10}$$

### Methodology:

The purpose of this experiment was to determine the change in internal energy by using a bomb calorimeter to measure the combustion of hydrocarbons. Benzoic acid's known enthalpy of combustion was used in order to obtain the heat capacitance of the calorimeter. The heat capacitance was then used in order to find the change in internal energy from the combustion of sucrose. All experiments were carried out using deionized (DI) water, and using sucrose and Benzoic acid provided by the CSUCI Chemistry stock room. The bomb calorimeter used in this experiment was made by Parr Instrument Co. In the first week each partner was assigned to either prepare the Parr bomb, the calorimeter, or the sample of Benzoic acid.

The sample was prepared by crushing some Benzoic acid in a mortar, weighing roughly 5.0 grams (g) of crushed Benzoic acid, and the crushed powder was turned into a pellet using a pellet press. The pellet was weighed to be 0.393g. The sucrose pellets were prepared the same way, and were weighed to be 0.510g, and 0.548g.

Roughly 10cm of nichrome wire was cut and attached to the two electrodes on the lid of the Parr bomb. The pellet prepared previously was then placed in the crucible, and the nichrome wire was bent in order to touch and hold down the pellet. The volume of the bomb was measured at 3.41L by pouring in a known amount of water so that the volume of the container would be known for later calculations. The lid was then carefully placed on the Parr bomb, and the bomb was filled with oxygen until the pressure was 30atm, and then gas was released twice; the third time the oxygen was left inside the bomb and placed into a stainless steel pail.

The pail was filled with 2.0L of DI water at a temperature between  $298.15 \pm 0.5K$ , and care was taken to remove bubbles around the bomb, and the bomb was watched for 5 minutes to ensure no new bubbles formed so air didn't interfere with the system. The lid of the bomb enclosure was placed on top of the apparatus, a band was placed on the gears of the stirring rod located on the outside of the bomb lid, and care was given in making sure the stirring rod attached to the lid on the inside was not hitting the apparatus's wall. The thermometer was gently inserted in the lid through a hole.



## Discussion:

After calculating the results for the sucrose sample, it was observed that the values for internal energy and enthalpy were extremely low compared to the literature value, as shown in Table 1. Benzoic acid was used as the standard to calibrate the bomb calorimeter values and give rise to the heat capacity to be used in the calculations of sucrose. The value is seen in Table 1.

Change in internal energy for the sucrose run was found to be -13.682 kJ, and our change in enthalpy was found to be -13.705 kJ. These negative values indicate that energy was given off during the combustion of the sucrose, which makes sense because when the sample burns, carbon dioxide and water are the products. The change in internal energy and the change in enthalpy are relevant at the midpoint temperature because as in equation 9 the second term has a constant temperature, moles, and R so then the equation transforms into equation 10 and the change in enthalpy and internal energy are equivalent. The final pressure was calculated to be 26.3 atmospheres which did not exceed the design's safe pressure capacitance of 45 atmospheres. The error in having a 1.0 mL difference in the pail would produce an error of 0.0590J. Electrical energy method and the known sample method for calibrating the calorimeter both have their advantages, but for the purpose of the experiment the known sample was used due to finding out the closest value to what would be done to our system. The change in enthalpy of the NIST standard was reported to be  $-5664 \pm 0.69$  kJ/mol, with our sucrose run resulting in a -13.697 kJ/mol heat of combustion of sucrose, and having a percent error of 99%.

Although the difference between the two values is dramatic, the values obtained by the other groups during the lab calculations are similar to our own. The calculated values are in Table 1. Our group also used two different bomb calorimeters during the two-week laboratory sessions, which might have caused an error in our calculations because each calorimeter has a different insulating capacity and that needs to be constant as well. With so much not being kept constant with our data, it is not surprising that we calculated such different values between the changes in enthalpy of sucrose.

All of our attempts at igniting the benzoic acid were unsuccessful due to a procedural error from leaving the pressure release valve open to the atmosphere allowing atmospheric nitrogen to re-enter the bomb reaction chamber during the oxygen flushing process. Due to this, we weren't able to ignite the wire and, in effect, neither the benzoic acid nor the sucrose in our early attempts. The benzoic acid data was collaboratively analyzed with another group along with one of the sucrose runs. This collaboration contributed to the large difference between the literature and the calculated values. The different masses of pellets, as well in the change in the mass of the wire need to be consistent between groups due to the calculation of the specific heat capacity of the benzoic acid and the sucrose.

Overall, and thanks to the comparison between the other groups' values, we were actually able to successfully perform this bomb calorimetry lab. Unfortunately, we weren't able to execute it well enough to compare the values calculated to the literature values on the NIST website.

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	Not Ready for Publication	Publish with Major Revisions	Publish with Minor Revisions	Ready for Publication	Comments
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