AC 2007-2839: PC-BASED MEASUREMENT OF THE HEAT OF COMBUSTION OF A SOLID FUEL USING OXYGEN BOMB CALORIMETER

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PC-Based Measurement of Heat of Combustion of a Solid Fuel Using Oxygen Bomb Calorimeter

Abstract

The paper describes an experimental system developed for measurement of the heat of combustion of a sample of solid fuel. The system is set up to use an Oxygen Bomb Calorimeter together with a temperature sensor. A data acquisition system is used to accurately record temperature versus time response before, during and after the combustion of the fuel sample. The data acquisition system includes an analog to digital converter (IOtech Personal DAQ 3005 with USB connection) a standard software package (DASYLab) to obtain the measurement under program control to determine the observed temperature rise of the system following combustion of a carefully weighed sample of solid fuel. The data processing includes several corrections to the measured temperature rise in order to determine the heating value of the fuel. The oxygen bomb calorimeter can be operated as an adiabatic system to eliminate the heat gain/loss during the experiment. However, the experiment is carried out in a non-adiabatic system to allow a greater insight in this experiment and to enhance its pedagogical value. This experimental system has been developed for an undergraduate laboratory in thermodynamics for Mechanical/Chemical Engineering students.

Introduction

The heat of combustion[1] of a fuel is the amount of heat generated by the complete combustion of a unit weight of fuel and it is expressed in Joules/kg (or other similar units such as BTU/Ib or calories/gram). It is experimentally determined by combustion of an accurately weighed sample of fuel in a calorimeter and measurement of the heat released. In this experimental system, an Oxygen Bomb Calorimeter[2] has been used. The temperature rise, ΔT, resulting from the heat released by combustion of the fuel sample is determined from the temperature ~ time response of the calorimeter. A suitable procedure is followed to determine ΔT which accounts for non-adiabatic condition of the calorimeter. Several other corrections are also applied for an accurate determination the heat of combustion of the fuel sample. This paper describes the software and hardware systems used for PC-based data acquisition/processing systems to collect temperature ~ time data and determine the heat of combustion of the fuel sample.

Theory

The gross heat of combustion, H_g, of the fuel sample is determined from the energy balance relation that the heat released by the fuel sample within the Oxygen Bomb is equal to the heat absorbed in the calorimeter. Combustion of the fuel sample results in a temperature rise of the bucket containing a measured amount of water in which the Oxygen Bomb is completely submerged. The energy balance relation is expressed by the following equation[2,3]:

$$m_f H_g = W (\Delta T) - e_1 - e_2 - e_3 \qquad (1)$$

where m_f = weight of the fuel sample, W = water equivalent of the calorimeter, e_1 = correction for heat of formation of nitric acid (HNO_3), e_2 = correction for heat of formation of sulfuric acid (H_2SO_4) and e_3 = correction for heat of combustion of fuse wire. Heat of combustion, H_g, is determined from Equation-1.

The Experimental Setup

The experimental setup (Figure 1a and 1b)[2] consists of the following components:

(i)   Oxygen Bomb (Figure 1b): Figure 1b shows the oxygen bomb with a cover which contains charging (inlet) and discharging (outlet) valves and ignition terminals. This cover can be secured onto the oxygen bomb with a screw cap after the fuel sample and fuse wire are assembled inside.

(ii)   Calorimeter Bucket (Figure 1a): It is a stainless steel bucket which contains a carefully measured amount of water in which the oxygen bomb containing the fuel sample is submerged. Combustion of the fuel sample results in a rise of temperature of water in this bucket and allows a determination of the amount of heat released from combustion of the fuel sample.

(iii)   Calorimeter Jacket: (Figure 1a): The double-wall jacket provides thermal insulation to minimize heat transfer to or from the calorimeter bucket which is placed inside the jacket during experiment. By supplying suitable mixture of hot and cold water into the jacket wall, its temperature can very closely follow the rise in bucket temperature during the experiment and maintain a nearly adiabatic condition, if desired. In this experiment, however, the outer jacket temperature remained nearly constant to provide a non-adiabatic condition to enhance its pedagogical value.

(iv)   Cover Assembly (Figure 1a): The cover assembly carries the temperature sensor (a thermocouple in this experiment) and a motor driven stirrer which can be lowered into the calorimeter bucket. The thermocouple (3.125 mm diameter, T-type) records the temperature of the bucket (referred to as bath temperature). An electric motor operates a mechanical stirrer to ensure uniform temperature within the bucket.

(v)   Ignition System: The cover of the oxygen bomb contains ignition terminals (Figure 1b) which are connected to the fuse wire in order to initiate combustion of the fuel sample inside the oxygen bomb. The lead wires from the ignition system (Figure 1a) are connected to the ignition terminals of the oxygen bomb after it is submerged in water in the calorimeter bucket. The circuit contains an ignition switch and an indicator light. In order to initiate combustion (fire the bomb), the switch is pressed and held down until the indicator light goes out.

(vi)   Other Components and Accessories: Other components and accessories of this experimental system include fuel sample, pellet press, precision weighing balance, fuse wire, titration system, 0.0725N sodium carbonate solution, methyl red, oxygen tank and

charging system. Hardware and software used for data acquisition and data analysis in this experiment are described separately under "Data Acquisition System".
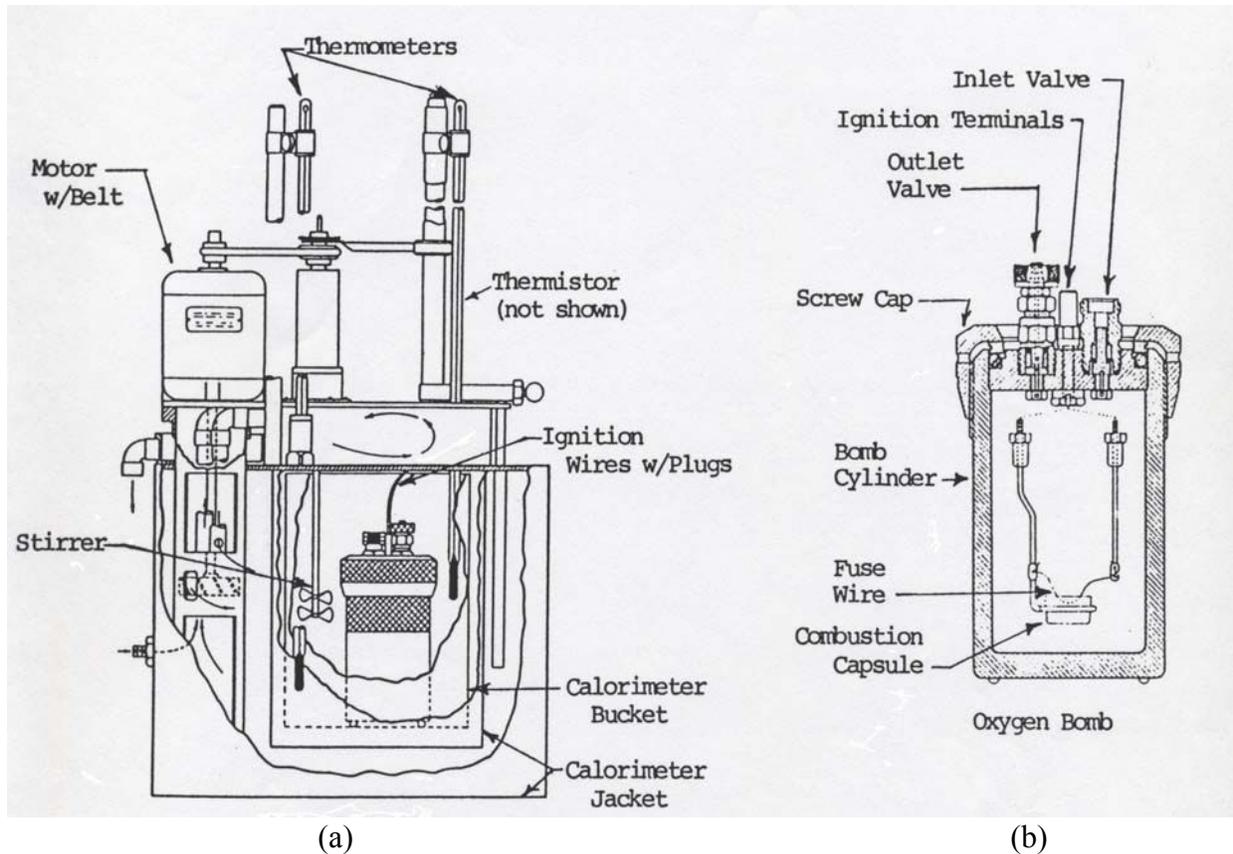

Figure 1 Schematic Diagram Showing (a) Calorimeter Cross-section[3] (b) Oxygen Bomb[2]

Experimental Procedure

The experimental process consists of the steps recommended and outlined in detail in the Instruction Manual[2] of this apparatus. The procedure is briefly described here.

1. Preparations

The preparation for this experiment consists of the following steps carried by students under proper supervision:

(i)   Fuel Sample and Fuse Wire: A sample of solid fuel in powder form is pressed to form a pellet in a pellet press. The mass of the fuel sample should be about 1 gram (maximum 1.5 gram). The fuel pellet is carefully placed in a combustion capsule in an upright position and carefully weighed in an electronic balance. The capsule is

placed in a ring attached to the cover assembly through the electrodes to which 10 cm of fuse wire is attached (Figure 1b).

(ii) Closing the Oxygen Bomb: About 1 ml of distilled water is placed inside the oxygen bomb cylinder. The cover assembly together with the fuel pellet and fuse wire is then placed on the oxygen bomb cylinder which is closed by hand-tightening the screw cap (Figure 1b).

(iii) Charging the Oxygen Bomb: The oxygen bomb is filled with oxygen to a pressure of 30 atm (but never to exceed 40 atm) to ensure complete combustion of the fuel sample. Oxygen from a high pressure cylinder is transferred, using a charging tube, very slowly through a regulator valve and the inlet valve into the oxygen bomb cylinder. The charging line is then depressurized using the bleed valve and disconnected from the oxygen bomb.

(iv) Calorimeter Bucket: The calorimeter bucket is filled with 2000 (±0.5) grams of distilled water at approximately 1.5 C below the room temperature. The bucket is then set into the calorimeter and the oxygen bomb is slowly lowered into the bucket. The ignition lead wires are inserted into the terminal sockets on the bomb head. The cover of the jacket is closed. The thermometer and stirrer assembly is lowered into the calorimeter bucket.

2. Operation

The stirrer is run for about 5 minutes and the data acquisition system is run to collect data for the next 5-minute "Pre-Period" following which the fuel is ignited by pressing the ignition switch. The data collection is continued during the "Rise-Period" during which there is rapid temperature rise and into the "Post-Period" which follows. The temperature ~ time data is processed, as outlined later (under Data Acquisition System), to obtain the temperature rise, ΔT, corrected for non-adiabatic condition of the calorimeter.

3. Further Experimental Processing for Corrections e_1, e_2 and e_3

Following the completion of the experiment, the thermometer and stirrer assembly is raised and the cover is lifted to clear the jacket opening. The oxygen bomb is carefully removed from the bucket and secured on the lab bench. The outlet valve of the oxygen bomb is slowly opened to de-pressurize it. The screw cap is then unscrewed and the cover assembly together with combustion capsule and fuse wire is removed. The residue within the bomb is washed with distilled water and collected in a beaker. To this solution, 2-3 drops of methyl red are added and the solution is prepared for titration with 0.0725N sodium carbonate solution. The volume (c_1 in ml) of the alkali solution used in acid titration is determined. Correction, e_1, for heat of formation of nitric acid is obtained from[2,3]

$$e_1 = c_1 = \text{milliliters of alkali solution used in acid titration} \qquad (2)$$

Correction, e_2, for heat of formation of sulfuric acid is obtained from the following empirical relation[2]:

$$e_2 = 14 \, c_2 \; ; \; c_2 = \text{percentage of sulfur in the fuel sample} \qquad (3)$$

All the remaining fuse wire is also collected and measured to determine the length of wire consumed (c_3) in the experiment. The correction, e_3, for heat of combustion of the fuse wire is expressed by[2]

$$e_3 \; = 2.3 \, c_3 \;\; \text{(for Parr 45C10 nickel-chromium wire used in this experiment)} \qquad (4)$$

Data Acquisition System

1. Hardware and Software

The hardware system consists of two T-type thermocouples and an IOtech Personal DAQ 3005 Series Data Acquisition System Board[4] with USB connection. One thermocouple measures the bucket temperature and the other records the outer jacket temperature. Data acquisition and storage of temperature ~ time data is carried out using DASYLab[5,6] software. Further analysis of the temperature ~ time response to obtain ΔT is done with software written in MATLAB[7]. Final determination of the heat of combustion, H_g, is obtained with MATLAB which prompts the user to input all the corrections and mass of fuel determined earlier in this experiment.

2. Data Acquisition Process

This section outlines the working of the data acquisition system. Figure 2 shows the block diagram built with DASYLab software to sample dc voltage signal (emf) from the
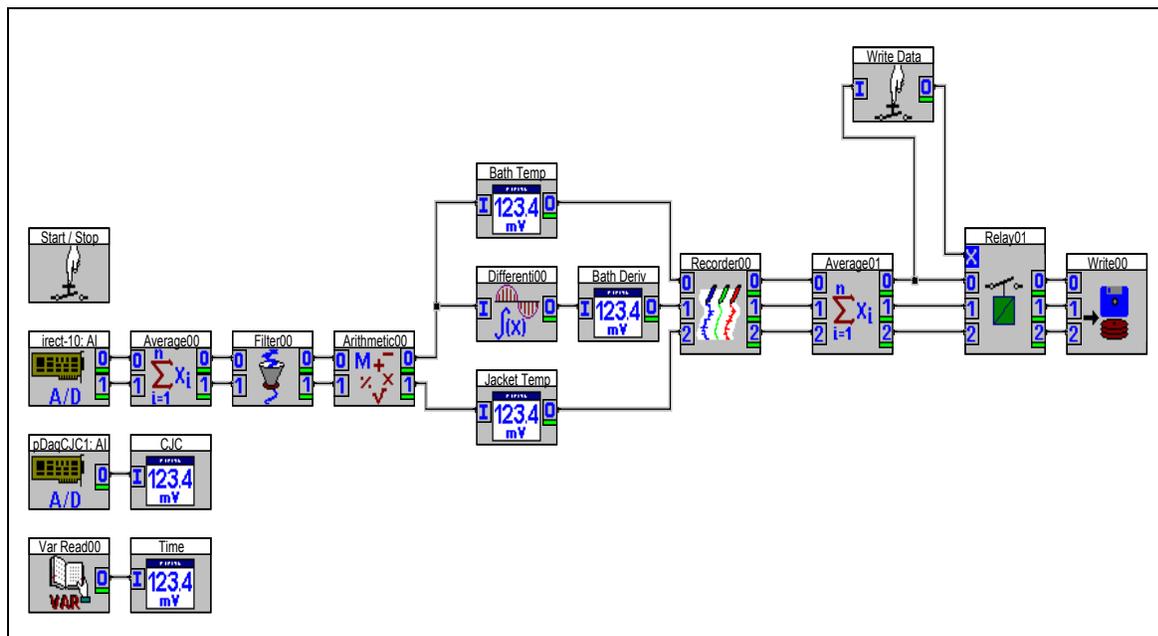

Figure 2 Data Acquisition System Block Diagram

thermocouples which are connected to IOtech Personal DAQ 3005 Series Board. The thermocouples are connected in differential mode as required setup for reading thermocouples. A sampling rate of 10 Hz is used. DASYLab software provides necessary filters for noise rejection and conversion of dc voltage readings into temperature (°C) with automatic cold junction compensation and thus avoiding the need for an ice-bath.

Temperature Measurement

The original mercury-in-glass thermometer of this apparatus has a certified accuracy and precision of ± 0.01 degree C. However, this thermometer was replaced with a T-type thermocouple for computerized measurement of temperature. The thermocouple has an error limit of ± 1 degree C but, together with the DAQ and the software DASYLab, it provides a measurement precision of 0.01 degree C. This is considered acceptable because the measurement of temperature rise (ΔT) of the bucket rather than its temperature (T) is the main objective in this experiment. Also, the temperature rise in this case is only about 1 to 2 deg C.

Once the DAQ system has obtained individual temperature readings, the data is subjected to a running average to further reduce noise. The sampled temperature is then fed to a filter set to allow frequencies below 0.1 Hz. This filter is used to eliminate noise in the derivative signal of the bath temperature.
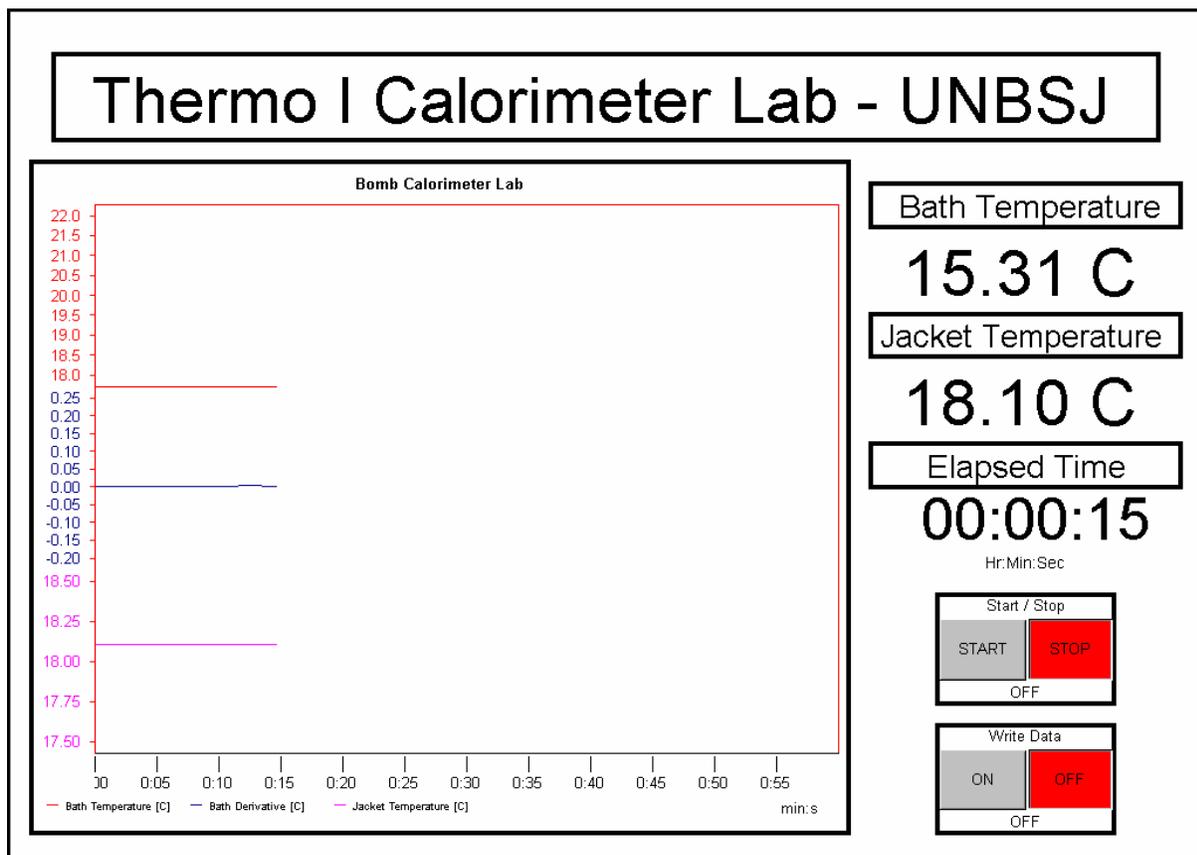

Figure 3 Data Acquisition Process on Monitor Screen

The temperatures and the derivative are then plotted on monitor screen and also recorded. Once the "write data to file" button is activated, the system averages the 10 Hz signal into a 0.5 Hz signal, allowing a smaller data file to be created for further analysis. Included in the system are other functions to allow the time and cold junction compensation to be displayed. Figure 3 shows the data acquisition process on the monitor screen. This screen allows the user to view the data acquisition process during the experiment and shows the plots of bath (bucket) temperature, its derivative and the jacket temperature as a function of time as well as digital displays of the bath (bucket) temperature, jacket temperature and elapsed time.

3. Experimental Process

The data acquisition/processing processes are designed to be user friendly and easy to complete the experimental measurement. Once the initial preparation for the experiment is complete (as outlined earlier in Experimental Procedure/Preparations), the students are introduced to computerized data acquisition system used in this experiment. The graphical programming technique used to set up the measurement system and data storage with the software DASYLab is also introduced. Students also learn how to process the experimental data (tempertaure~time, as well as other user supplied data such as m_f, c_1, c_2, and c_3) using MATLAB to determine the heating value (H_g) of the fuel sample using the heat balance equation and corrections using Equation 1. A student member of the group is then required to start the measurement program under DASYLab environment and begin the measurement. The "write to file" button is pressed and the system begins data acquisition. After the initial 5-minute pre-period the fuel is ignited by pressing the ignition switch. The data acquisition is continued for additional 14 minutes through the temperature rise-period and into the post-period. The "write to file" is turned then off first and then the data acquisition process is stopped. As indicated earlier in Experimental Procedure, next step is the determination of the corrections e_1, e_2, and e_3, following which the data analysis program determines the heat of combustion H_g.

4. Data Analysis Software

The data analysis program is written in MATLAB. This is done for several reasons. First, it is a programming language that is taught to all sophomore students enrolled in Engineering program as well as being an available analysis tool on almost all engineering computers and PCs used for a real-time data acquisition. To begin the analysis of the experimental data, the MATLAB file, Calorimeter_Lab.m, is opened and executed. The analysis software begins by reading the data file (in ASCII format) from the root directory where the software and the data-file are stored. It then asks for the parameters of the experiment, such as the mass of the fuel sample (m_f), titration volume (c_1), length of the fuse wire consumed (c_2) and the sulfur content (c_3) in the fuel sample. From here, the program converts the ASCII file into three different arrays to extract the bucket temperature, its derivative and the jacket temperature. The bucket (bath) temperature and the jacket temperature are plotted on a single graph (Figure 4).

Determination of Temperature Rise, ΔT (Figure 4)

Figure 4 shows the temperature ~ time plot obtained in this experiment. Rising bucket temperature during e-a (Figure 4) before combustion indicates heat gain due to a higher jacket

temperature. Similarly, the decreasing bucket temperature during b-h (Figure 4) is due to heat loss from the bucket which is at a higher temperature after combustion. In order to correct for the heat loss/gain during experiment, a procedure[2] is used to determine the correct temperature rise extrapolated at a time when 60% of the total temperature rise is realized. The program prompts the user to select two points (a and b in Figure 4) on bath temperature curve in Figure 4
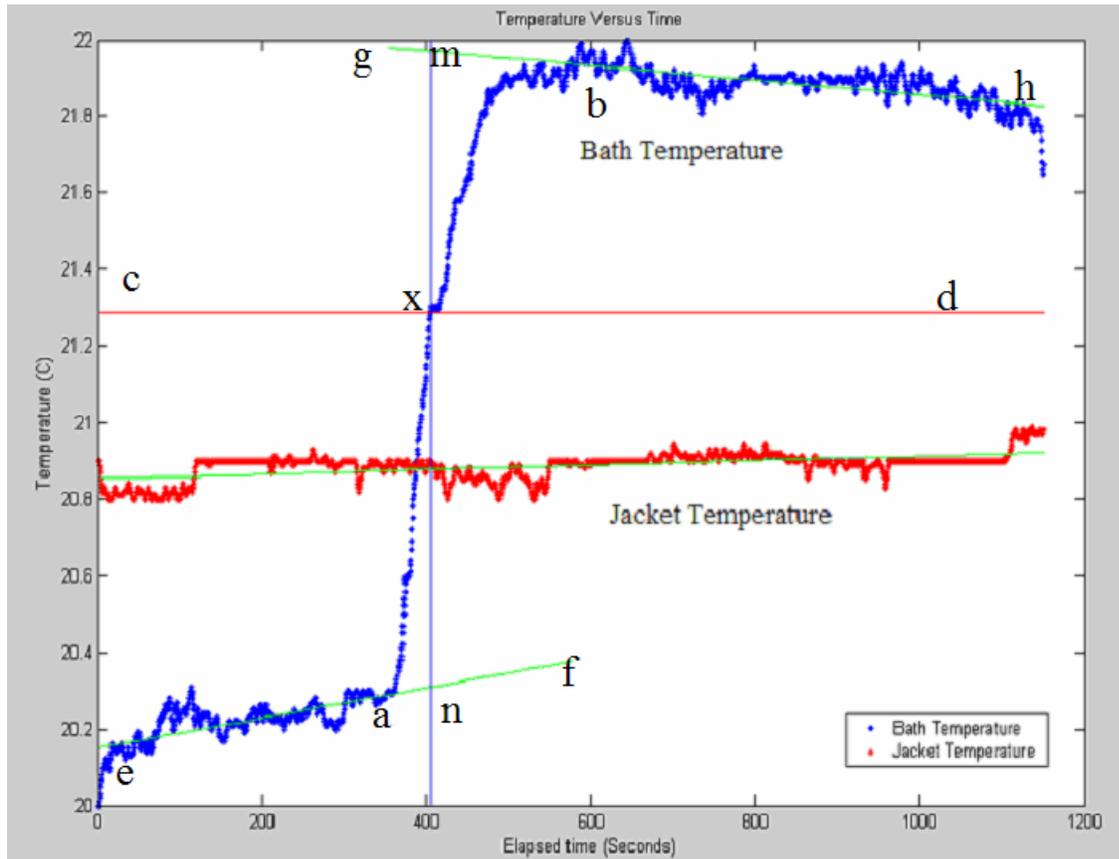

Figure 4 Bath Temperature and Jacket Temperature: Temperature ~ Time Plots

to define the nominal initial and final temperatures and determine approximate temperature difference (ΔT) in the calculation. The user selects the two points on the graph and the software creates a linear fit to the data (lines e-f and g-h in Figure 4). These two fits are used in the calculation of true and accurate temperature rise and plotted on the graph. The approximate initial and final temperatures (T_a and T_b) are used to find the reference temperature ($T_c=T_d$). It is defined as the temperature at 60% of the distance between the two temperatures selected earlier on the plot and is calculated using the following equation:

$$T_c = T_d = T_a + 0.6 \, (T_b - T_a) \tag{5}$$

A horizontal line c-d (Figure 4) representing T_c (= T_d) is drawn on the temperature ~ time plot . This reference temperature is used in an algorithm to find the time (t_x) at X where the reference temperature intersects the bath temperature data. The algorithm is a simple "for loop" with a

comparison enclosed within. The time at which the two plots intersect is passed into the equations fitted to the temperature changes shown by lines e-a and b-h before and after the ignition due to heat gain and loss respectively as shown in Figure 4. From these two extrapolated temperatures T_m and T_n, the correct temperature rise, ΔT, is calculated from the following equation:

$$\Delta T = T_m - T_n \tag{6}$$

The output of this analysis is displayed on the command line in MATLAB. Also, the jacket temperature is curve-fitted to find the average jacket temperature.

5. Determination of Heat of Combustion (H_g)

At this stage, all the parameters are known; either determined from data analysis (e.g. ΔT) or obtained by other experimental procedures completed in this experiment (e.g. m_f, c_1, c_2, and c_3). The corrections e_1, e_2, and e_3 are calculated using Equations-2, 3, and 4. Manufacturer supplied value of W, the water equivalent of the calorimeter, is used. Finally, the heat of combustion of the sample is calculated using Equation-5 which is same as Equation-1 rewritten in an alternate form.

$$H_g = \frac{W\,(\Delta T) - e_1 - e_2 - e_3}{m_f} \tag{5}$$

All the calculations are performed within MATLAB program developed for this experiment.

Results

Two trial runs were conducted with pellets made from peat moss as the solid fuel. The results obtained in these tests are presented below in Table-1. The deviation between the values of H_g determined in these trial runs is within about ±5% of each other.

Table-1 Results from the Trial Runs

Parameters	Test # 1	Test# 2
m_f = weight of fuel (gram)	1.1387	1.2389
c_1 = volume of alkali solution in acid titration (ml)	6.6	7.0
c_2 = length of wire consumed (cm)	4.0	3.5
c_3 = sulfur content in fuel sample (%)	0 (assumed)	0 (assumed)
W = water equivalent of the calorimeter (cal/°C)[*]	2418	2418
ΔT corrected for non-adiabatic condition (C)	1.83	1.89
H_g = Heat of Combustion (calculated in calories/gram)	3876	3687
H_g = Heat of Combustion (in J/gm)–by unit conversion[**]	16228	15433

[*] Data supplied by the manufacturer;　　[**] 1 cal/gm = 4.1868 J/gm

Conclusions and Recommendations

A fully computerized data acquisition and analysis system has been developed and implemented for the use with the oxygen bomb calorimeter experiment for sophomore Engineering students in Thermodynamics course. The use of these two software programs (data acquisition and data analysis programs) allow the fast and accurate analysis of the heat of combustion of a given fuel sample, allowing the student to have a greater insight into this process. The application of the data acquisition and analysis programs of this project is applicable to almost any experiment where several different temperatures are required to be recorded and analyzed in the education of undergraduate students in an introductory thermodynamics course.

Examination of the temperature ~ time plot (Figure 4) shows temperature fluctuations of about ± 0.1 deg C in temperature data recorded during e-a (before combustion) and b-h (after combustion). This is attributed to the fluctuations in the reference temperature used in the DAQ Board for cold junction compensation. Although the effect of this fluctuation on ΔT determined by the extrapolation technique used in this experiment has a second order influence, the accuracy of temperature measurement can be further improved by using a stable and constant temperature ice-point or by using a resistance temperature detector (RTD). Given the available data acquisition system's analog voltage capabilities, a RTD would be best recommended in this situation.

Acknowledgements

Bibliography

1. Steam – Its Generation and Use, 39th Edition, Publisher - Babcock and Wilcox, 1978, P6.5.
2. Parr Oxygen Bomb Calorimeter Manual No. 153, Parr Instrument Company, Moline, Il, USA
3. Instructions for the 1341 Plain Jacket Oxygen Bomb Calorimeter, Publisher – Parr Instrument Company, P8.
4. Personal Daq/3000 Series Installation Guide, IOtech, Inc, Cleveland, OH
5. DASYLab, Using IOtech Data Acquisition Products with DASYLab, p/n 472-0902 Rev 2.0, Cleveland, Ohio, USA, January 2002.
6. DASYLab (**D**ata **A**cquisition **Sy**stem **Lab**), Version 7.1, Book 1: User Guide, IOtech, Cleveland, Ohio, USA, 2002.
7. MATLAB 7.0, The MathWorks Inc., Natick, Mass., USA, 2004.