

# Oxygen Bomb Calorimeter

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***Toshniwal***

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## Oxygen Bomb Calorimeter Models

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### Digital Bomb Calorimeter – Model CC01/M2



Toshniwal Bomb Calorimeter with Digital Differential Temperature/Firing Unit provides an accurate, simple and inexpensive method for determination of Heat of Combustion, Calorific Value and Sulphur contents of solids, liquids and volatile fuels.

The Bomb Jacket with top cover is provided with special highly insulated material so that temperature radiation is practically nil. Temperature correction to get the final and correct result is not required.

The readings of Toshniwal Digital Differential Temperature/Firing Unit are at par with Beckman Thermometer.

#### **Digital Bomb Calorimeter Salient Features:**

- Bomb body is machined from corrosion resistant stainless steel alloy
- Each bomb is tested at a pressure of 300 atm for 10 minutes
- Offset stirrer precludes heat from motor to Calorimeter Vessel
- Easy to handle and operate

#### **The Digital Differential Temperature/Firing Unit with Electronic Timer Features:**

- Temperature Indication: 3.5 Digit digital display
- Differential Temperature range: 0 - 10<sup>0</sup> C
- Reading Accuracy of temperature: ± 0.001<sup>0</sup> C
- Firing Voltage for Bomb: 12 VAC
- LED Indication for filament continuity
- LED Indication for Bomb Combustion

## The Bomb Calorimeter is supplied with:

- Combustion Bomb
- Calorimeter with Bomb support
- Combined Lid for Calorimeter Vessel & Jacket
- Stirrer
- Connecting Leads
- Connecting tube for Oxygen Cylinder
- Adjustable Spanner
- Ignition Wire (20 Metres), Nichrome (36 SWG)
- Valve Key
- Benzoic Acid – 10 Tablets
- Cotton Reel
- Stand for Bomb Lid
- Hook for lifting Bomb
- Crucible – 2 nos.
- Gelatine Capsules – 50 nos.
- Gas Release Valve
- O – Rings for Bomb and Stirrer
- Valve for Bomb
- Pellet Press
- Instruction Manual

## Digital Bomb Calorimeter with Software – Model CC01/M2A



The Model CC01/M2A is the advanced version of the Model CC01/M2. With all the features being the same, The M2A model has the additional feature of being connected to a computer. It is supplied with an RS 232C interface to connect it to a computer with windows operating system. The M2A model automatically calculates the Calorific value of the sample.

## Automatic Microprocessor Bomb Calorimeter – CC01/M3



The Toshniwal Microprocessor Bomb Calorimeter provides an accurate, simple & inexpensive method for determination of Heat of Combustion, Calorific Value & Sulphur contents of solids, liquids and volatile fuels, food items, bagass & other easily burning materials.

The Bomb Jacket with top cover is provided with special highly insulated material so that temperature radiation is practically nil. Temperature correction to get the final and correct result is not required.

### **Microprocessor Bomb Calorimeter Salient Features:**

- No temperature correction
- Detection of maximum rise of temperature
- Windows based software
- Temperature resolution:  $\pm 0.001^{\circ}\text{C}$
- Memory Storage: 100 data
- Battery backup for stirrer and Microprocessor unit (UPS)
- RS 232C interface to connect with computer
- Soft touch keypad
- Auto firing (With software only)
- LCD display with backlit 16 x 2 characters

**The following tasks are Automated with the Microprocessor Bomb Calorimeter with Software:**

- Automatic feeding of Calorific values of ignition wire and cotton thread
- Automatic firing in the bomb
- Automatic detection of maximum rise of temperature
- Automatic calculation of Heat Capacity of Bomb Calorimeter (Equivalent Value)
- Automatic calculation of Calorific Value of samples
- Automatic saving of Heat Capacity of Bomb Calorimeter and Calorific Values in summary format
- Automatic delivery of result data in report format

**The Microprocessor Bomb Calorimeter is supplied with:**

- SS 316 Combustion Bomb
- Calorimeter with Bomb support
- Combined Lid for Calorimeter Vessel & Jacket
- Calorimeter Jacket with insulated Material
- Stirrer
- Connecting Leads
- Connecting tube for Oxygen Cylinder
- Adjustable Spanner
- Ignition Wire (20 Metres), Nichrome (36 SWG)
- Valve Key
- Benzoic Acid – 10 Tablets
- Battery backup (UPS)
- Platinum Wire (Optional)
- Cotton Reel
- Stand for Bomb Lid
- Hook for lifting Bomb
- Crucible – 2 nos.
- Gelatine Capsules – 50 nos.
- Gas Release Valve
- O – Rings for Bomb and Stirrer
- Valve for Bomb
- Pellet Press
- Instruction Manual
- Leads to connect Microprocessor unit to PC
- Computer with Printer (Optional)
- Semi Auto Gas Filling Device (Optional)

Fuel cups (crucible) of Inconel, SS etc. as per IS:1448(P-6) for special applications are also available with us.

## Product Technical Details

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### Product Introduction:

A Bomb Calorimeter measures the amount of heat generated when matter is burnt in a sealed chamber in an atmosphere of pure Oxygen gas.

Toshniwal Bomb Calorimeter provides a simple inexpensive yet accurate method to determine the Heat of Combustion, Calorific Value and the Sulphur contents of solids & liquids and fuels. The outfit provided is complete for the analysis as per methods recommended by the Indian Standard Institute (IS: 1359 –1959), British Standard Institute (BS 1016: Part 5:1967) and the Institute of Petroleum (IP 12/63T). Each part of the outfit has been finished and tested according to the specifications laid down by these Institutions.

### Principle of Operation:

A known amount of the sample is burnt in a sealed chamber (Bomb). The air is replaced by pure Oxygen. Sample is ignited electrically. As the sample is burnt, heat is produced. The rise in temperature is measured. Since barring the loss of heat, the amount of heat produced by burning the sample must be equal to the amount of heat absorbed by the calorimeter assembly. A knowledge of the water equivalent of the calorimeter assembly and of the rise in temperature enables one to calculate the heat of combustion of the sample.

W= Water Equivalent of the calorimeter assembly in calories per degree centigrade

T= Rise in temperature in degree Celsius,

H = Heat of combustion of material in calories per gram, &

M = Mass of the sample burnt in grams,

Then  $W.T = H.M$  or  $H = W.T / M$

### Product Components:

#### 1. Bomb:

The bomb consists of three parts viz. Bomb body, lid and Union nut.

The Bomb body and the lid are machined from an ultra-strong corrosion resistant stainless steel alloy containing Cr, Ni & Mo which satisfying special ringing and bending tests for inter-crystalline corrosion.

The Bomb body is a cylindrical vessel having a capacity of 303 ml. The walls are extremely durable and can easily withstand the normal operating pressure (30 atmospheres) and also extreme pressures as high as 300 atmospheres. During burning at high pressures, the Nitrogen & Sulphur contents are oxidized to their respective acids. The corrosion resistant nature of the Bomb material protects it from the corrosive vapours.

The lid is provided with two terminals through which two metallic rods pass through. One of which is provided with a loop for placing the sample crucible and the other with a groove. The fuse wire is attached to these two rods. Each rod is also provided with a ring to press the fuse wire that is attached to it. The upper side of the lid is provided with a small hook for lifting it and with a Schrader valve for filling Oxygen in the Bomb. The Schrader valve is provided with a metallic cap.

The Gas Release Valve is used to release the pressure of gases in the bomb and also to collect gases after firing. It is screwed on the Schrader Valve provided on the lid of the bomb. The knob should be turned down clockwise to release excess of Oxygen gas out of the tube.

**Each Bomb is tested for its performance as per the requirements of the Institute of Petroleum (IP12/63T). The test is conducted at a pressure of 300 atmospheres and the pressure is maintained for a period of 10 minutes without any sign of leakage.**

## **2. Jacket:**

Jacket with top cover is provided with special highly insulated material to make temperature radiation practically nil. With this provision, temperature correction to get the final and correct results is not required. This provision also eliminates the water pipe and jacket thermometer. This jacket is essential when the system is supplied with RS232C interface and related software.

## **3. The Calorimeter Vessel:**

The Calorimeter Vessel is the vessel in which the Bomb is placed and is filled with water. It is made up of Copper and is coated with Ni-Cr to reflect the heat back into the water instead of radiating it. The Calorimeter Vessel is placed inside the Jacket

## **4. Offset Stirrer:**

The Offset Stirrer consists of a stirrer with a fan which is driven at a constant speed of 800 rpm by a motor which is connected through a heat insulator rubber belt. The motor unit is kept at a sufficient distance from the vessel to eliminate radiative heating and a heat insulator Bakelite divides the two parts of the stirrer rod. This arrangement does not raise the temperature of the water by even 0.01°C in ten minutes, thus easily meeting the specific requirements laid down by the British Standards Institution and the Institute of Petroleum and accepted by the Indian Standard Institution. The electric supply for the stirrer motor is obtained through the terminals provided on the digital firing unit.

## **5. Pellet Press:**

Powdered samples are compressed into pellets before weighing and burning. This retards the burning rate and tends to retain the particles in the capsule, thereby reducing the chances for incomplete combustion. The pellets are easier to handle than loose samples. The pellets should not be made very hard as excessive hardness leads to bursting upon ignition consequent with incomplete combustion. The Pellet Press has an 8 mm diameter punch and die.

## **6. Ignition Wire:**

Nichrome Ignition wire is supplied with the instrument.

## **7. Crucible:**

Nickel alloy crucibles are offered as standard with the instrument. Quartz, Platinum and Stainless Steel crucibles are being offered as optional.



## Important Requirements:

1. When the sample burns, the pressure of gases increases rapidly. The Bomb walls, lids & joints should be strong enough to withstand the maximum working pressures and there should be no leaks. Normal working pressure is about 30 atmospheres and overload pressures peak up to 100 atmospheres
2. The capacity of Bomb should be large enough to store enough Oxygen to ensure complete burning of the sample
3. During burning, the Nitrogen & Sulphur contents are oxidised to gases and then to nitric acid and sulphuric acid respectively. The Bomb must therefore be resistant to acidic or basic ash and should be corrosion proof
4. The stirrer unit should not generate excess amount of heat due to stirring. Furthermore, the motor heat should not reach the calorimeter, otherwise the calculations will lead to erroneous results
5. All surfaces should have high reflectance to minimize radiation losses
6. Water Equivalent of the Calorimeter Assembly should be small to ensure maximum rise in temperature of water following ignition

## Reagents, Samples & Sample Holders, Standard Samples:

### 1. Benzoic Acid:

It is commonly used as a Calorific Standard ( $6319 \pm 20$  Cal/gm). It burns easily and completely and can be pressed into pellets.

### 2. Naphthalene:

It is sometimes used as a combustion standard. It is not hygroscopic but due to its volatility it is necessary to use with care to avoid errors from sublimation.

### 3. Food Stuff and Cellulosic Material:

The high moisture content of most food stuffs will usually require that these be dried before calorific tests. The operator will have to select a method for preparing the sample that will not destroy or remove any of the combustible constituents. It may be necessary to make several preliminary tests to determine the approximate maximum allowable moisture content at which the samples can be ignited in the bomb without difficulty.

### 4. Gelatine Capsules:

Volatile liquid samples to be burnt in an Oxygen Bomb can be weighed and handled in gelatine capsules. The capsules consist of two cups, which telescope together with a fraction fit adequate to retain most liquids. Correction must be made for the heat of combustion of the gelatine when used in calorimetry.

## 5. Heavy Oils:

Oils and other liquids which are non-volatile at room temperature can be weighed directly in to crucible. The loop of the fuse should be positioned just above the surface of the sample. Non-volatile liquids can also be weighed and handled in gelatine capsules.

## 6. Explosive Oils:

Special precautions must be observed when testing materials which release large volumes of gases upon ignition or which detonate with explosive force. It is possible to test many slow burning gun powders and rocket propellants in conventional bombs, but the user must understand that these bombs are not designed to withstand the shock pressure produced by certain primers and other mixtures which detonate with explosive force. It is much safer to test these in a special high pressure oxygen bomb.

Each new explosive sample or high energy fuel introduces special problems, which can be solved only by careful experimentation. Usually it will be well to observe the burning of a small amount of a sample over an open flame to determine the explosive behaviour and then to proceed to bomb combustion using only one -tenth or one-fifth of the usual amount of sample. Further increases up to the 10,000 calories maximum permissible should be made gradually, and only after all evidences indicates the absence of violent behaviour

## Setup & Assembly:

### 1. General Precautions:

The lab in which the calorimeter is to operate should be equipped with many of the facilities commonly used for chemical analysis. These include desk space, running water, analytical balance, apparatus for making volumetric titrations and miscellaneous items of lab ware. The calorimeter should be used in a room where fluctuations in temperature should be minimum. In particular, the instrument should not be taken from one chamber to another chamber maintained at a different temperature immediately before use. Sufficient time must be allowed for equalization of temperature throughout the jacket before starting to use the calorimeter. All parts of the calorimeter should be kept clean and dry, and the inside of the jacket should be wiped clean to remove any moisture which may have condensed on the walls. Before starting to use a new calorimeter, it is advisable to assemble all parts of the apparatus without a charge in the bomb and without water in the bucket and be sure that everything is in perfect working order.

### 2. Calorimeter Parts Assembly:

Place the star support (Brass) at the bottom of the jacket and set it so that the leg having hole is fitted in the slot provided at the base of the jacket. Place the bucket on the star supporter so that the pin outside of the bucket is fitted in the hole of the leg of the star support. Place the star (metallic) in the bucket in such a way that none of the arm comes within the slot provided in the bucket. Lift the bomb on its stand by hook and place it on the star (metallic) clips inside the bucket. Attach the supply connections to the electrodes provided on the lid of the bomb. Place the combined lid of bucket and outer jacket in such a way that a pin provided on the cover plate of the jacket fits into the smaller groove provided in the lid.

Mount the stirrer assembly on the stirrer rod provided on the cover plate of the calorimeter jacket, passing the stirrer pipe through the opening provided in the combined lid of bucket and the outer jacket. The connecting leads attached with terminals provided on the bomb lid are now connected to the two terminals provided on the cover of the calorimeter jacket. The connections are then further taken to the Digital Differential Temperature/Firing Unit (DDT/FU). Similarly stirrer connections are connected to AC Mains (230 Volts, 50 Hertz).

### **3. Attaching of the Fuse:**

All manipulations prior to closing the Bomb can be performed by holding the bomb lid in the support stand. Cut a single length of 5.5 cm of fuse wire and attach it to the electrodes.

It is not necessary to submerge the wire in a powder sample. In fact, better combustion will usually be obtained if the loop of the fuse is set slightly above the surface. When using pelleted samples, bend the wire so that the loop bears against the edge of the pellet firmly enough to hold it against the side of the capsule. In case of liquid fuels, the capsule should be held as a loop of this wire. It is also a good practice to tilt the capsule slightly to one side so that the flame emerging from it will not impinge directly on the top of the straight electrode.

### **4. Filling the Bomb with Oxygen:**

While closing the Bomb, always make certain that the head gasket or the sealing ring is in good condition and care must be taken not to disturb the sample. Commercial Oxygen produced by rectification of liquid air can be used directly from the supply cylinder.

To attach the filling connection, place the bomb on its stand. Put the high pressure valve (Fine Regulating Valve) in oxygen cylinder outlet and connect the copper tube to the pressure gauge and pressure gauge to filling tube and filling tube to bomb valve and make the connections perfectly tight. Open the filling connection control valve of the cylinder slowly – slowly. Observe the gauge and allow the pressure to rise until the desired point (about 25 atmosphere), then close the collection of gas control valve. To decrease the pressure of bomb, the gas release valve can be used. By rotating the gas release valve in anti-clockwise direction, the pressure can be reduced to the desired point.

### **5. Filling the Calorimeter Vessel with Water:**

On an accurate balance determine the weight of the completely dry Calorimeter Vessel, then add 2000 grams of distilled water into the vessel. The operator must also make sure that there is no moisture on the outside surface of the vessel when it is placed in the jacket.

## Operating the Bomb Calorimeter:

It is desirable to keep the jacket temperature and the room temperature as close to the calorimeter temperature as possible. The jacket and room temperature should therefore be recorded.

### Preparing the Bomb Calorimeter:

1. Accurately weigh about one gram of the air dried material ground to pass through IS Sieve 20 (211 microns) in the crucible. If required, sample can be compressed into a pellet before weighing
2. Stretch a piece of firing fuse wire (generally Nichrome wire) across the electrodes within the bomb
3. Tie about 5.0 cm length of sewing cotton around the wire, place the crucible in position and arrange the loose ends of the thread so that they are in contact with the material. Use the same length of thread in each determination
4. Pour 2 ml of distilled water into the bomb
5. Reassemble the bomb, screw tightly with hand and avoid excessive pressure
6. Charge the bomb slowly with oxygen from a cylinder to a pressure of 30 atmospheres without displacing its original air content
7. Close the valve supplying Oxygen effectively
8. Using as little pressure as possible, detach the bomb from the oxygen supply
9. Fill the Calorimeter Vessel with enough quantity of water to sufficiently submerge the nut of the bomb to a maximum depth of 2 mm, whereby leaving the terminals projecting. Don't forget to weigh the water. Use the same weight of water for future experiments
10. Transfer the calorimeter vessel into jacket
11. Lower the bomb carefully into the calorimeter vessel after having it ascertained to be gas tight
12. Connect it to the ignition circuit through a switch for subsequent firing of the charge
13. Adjust the stirrer, place the thermometer and covers in position

### Operating the Digital Differential Temperature/Firing Unit:

1. Set the Bomb Calorimeter as mentioned above
2. Insert the RTD Probe into the Bomb Calorimeter through the combined lid
3. Connect the RTD Probe to the DDT/FU terminal marked PROBE
4. Connect the DDT/FU terminal to the Bomb Calorimeter terminal on the Jacket
5. Attach the stirrer connection to DDT /FU
6. Switch ON the instrument and stirrer
7. LED of the DDT/FU will glow which shows that the proper placement of filament is done inside the bomb
8. Timer will start giving beeps after app every one minute
9. Adjust the fine and coarse control knob to setting of ZERO or set at 1.00
10. Wait for 10 minutes for the initial temperature to stabilise. If temperature is not stabilised, extend the period for another 5 minutes for stability
11. Record the primary period (initial) temperature
12. Press the push button marked FIRE. Please note that LED will be OFF after the firing has been done. This shows that the firing has taken place
13. After firing, note down the chief period temperature after every beep given by timer
14. Record the temperature reading till there is no further rise in temperature
15. Calculate the rise in temperature
16. Switch off the instrument and stirrer

## Post Firing Process:

1. Remove the bomb from the calorimeter and after a lapse of about half an hour from the time of firing allowing the acid most to settle, release the pressure by opening the valve.
2. Verify that the combustion has been completed by noting the absence of any sooty deposit with in the bomb. The presence of any trace of sooty deposit indicates incomplete combustion and invalidate the test.
3. Wash out the contents of the bomb with hot distilled water into a hard glass beaker. Also wash the bomb cap and the crucible.
4. Add a measured excess, say 25 ml. Of 0.1 N Sodium Carbonate solution and boil it down to 16 ml to convert any metallic sulphates or nitrates to the less soluble carbonate or hydroxide. The consumption of alkali carbonate is equivalent to the sulphates or nitrates together with the free sulphuric acid and nitric acids.
5. Filter wash and make up to 100 ml. To determine the Sulphur content take 50 ml portion of this solution and follow the methods as given in section 11.
6. Determine the total acidity by titrating a 50 ml portion with 0.1 N Hydrochloric acid using methyl orange as indicator. The titre representing the excess alkali in one half of the quantity of sodium carbonate solution is added to the washings.

**Note: Disregard the above procedure if the estimate of Sulphur or Nitrogen is not required.**

## Standardising the Bomb Calorimeter:

Before a material with an unknown heat of combustion can be tested in a bomb calorimeter, the Heat Capacity of the Bomb Calorimeter also known as Energy Equivalent or Water Equivalent (W.E) must first be determined.

The W.E of the Bomb Calorimeter takes into consideration the sum of the heat capacities of the components in the calorimeter, such as the metal bomb, the bucket, the water in the bucket, the Nichrome Wire and in some cases, the Cotton Thread.

The Heat Capacity of the Calorimeter is determined empirically by burning a sample of a standard material with a known heat of combustion under controlled and reproducible operating conditions. Benzoic acid is used almost exclusively as a reference material because it burns completely in oxygen, it is not hygroscopic and it is readily available in very pure form.

The amount of heat introduced by the reference sample is determined by multiplying the heat of combustion of the standard material by the weight of the sample burned. Then, by dividing this value by the temperature rise produced in the test, we obtain a resultant Heat Capacity of the Bomb Calorimeter for this particular calorimeter.

$$W.E = \frac{(M)(H)}{\Delta T}$$

Where:

W.E = Water Equivalent | Heat Capacity of Bomb Calorimeter | Energy Equivalent

M = Mass of the Test Sample

H = Heat of Combustion of standard material (6319 in the case of Benzoic Acid)

$\Delta T$  = Rise in Temperature

Example: Consider a standardization test in which 1.651 grams of standard benzoic acid (heat of combustion 6318 cal/g) produces a temperature rise of 3.047°C.

The Water Equivalent (W.E) of the calorimeter is then calculated as follows:

$$W = \frac{(1.651)(6318)}{3.047} = 3423.37 \text{ cal/}^\circ\text{C}$$

For simplicity, the corrections usually applied for heats introduced by the Fuse Wire and by the cotton thread are omitted from the above example but should be introduced to calculate the W.E of the Calorimeter.

### W.E of Bomb Calorimeter where only Nichrome Fuse Wire is used:

In certain cases, only the Fuse Wire is used in the Bomb Calorimeter Operation. But before using the Fuse Wire, it is highly recommended that a standard Fuse Wire of standard length and standard material be used. The recommended material is Nichrome Fuse Wire. The length of the Fuse Wire can be decided by the user, but once decided, it is recommended that the same length be used for all experiments involving the Bomb Calorimeter. It is to be noted that the same gauge fuse wire be used in all experiments.

The following formula is the amended formula to calculate the W.E of the Bomb Calorimeter taking the Fuse Wire into consideration. It is to be noted that the calorie value for Nichrome Fuse Wire is known and is 333.68 Cal/gm.

$$W.E = \frac{(M)(H) + (E_w)}{\Delta T}$$

Where:

W.E = Water Equivalent | Heat Capacity of Bomb Calorimeter | Energy Equivalent

M = Mass of the Test Sample

H = Heat of Combustion of Test Sample

$\Delta T$  = Rise in Temperature

$E_w$  = Correction of Heat of Combustion for Nichrome Fuse Wire

Where:

$$E_w = (M_w)(H_w)$$

$M_w$  = Mass of Nichrome Fuse Wire

$H_w$  = Heat Capacity per Gram of Nichrome Fuse Wire | Calorie Value of Nichrome Wire

Example: Consider the previous standardization test in which 1.651 grams of standard benzoic acid (heat of combustion 6318 cal/g) produces a temperature rise of 3.047°C but a Nichrome Fuse Wire of 0.0184 gm was used and burnt.

The Water Equivalent (W.E) of the calorimeter is then calculated as follows:

$$E_w = (0.0184)(333.68) = 6.14 \text{ cal}$$

$$W.E = \frac{(1.651)(6318) + 6.14}{3.047} = 3425.39 \text{ cal/}^\circ\text{C}$$

## W.E of Bomb Calorimeter where Nichrome Fuse Wire and Cotton Thread are used:

In other cases, cotton thread is also used along with the Nichrome Fuse Wire. It is again recommended that a standard weighed sample be always used.

The following formula is the amended formula to calculate the Water Equivalent of the Bomb Calorimeter taking the Fuse Wire and Cotton Thread into consideration. It is to be noted that the Heat Capacity value for Nichrome Fuse Wire is 333.68 Cal/gm and for Cotton Thread it is 4180 Cal/gm.

$$W.E = \frac{(M)(H) + (E_w) + (E_t)}{\Delta T}$$

Where:

W.E = Water Equivalent | Heat Capacity of Bomb Calorimeter | Energy Equivalent

M = Mass of the Test Sample

$\Delta T$  = Rise in Temperature

$E_w$  = Correction of Heat of Combustion for Nichrome Fuse Wire

$E_t$  = Correction of Heat of Combustion for Cotton Thread

Where:

$$E_w = (M_w)(H_w)$$

$M_w$  = Mass of Nichrome Fuse Wire

$H_w$  = Heat Capacity per Gram of Nichrome Fuse Wire | Calorie Value of Nichrome Wire

$$E_t = (M_t)(H_t)$$

$M_t$  = Mass of Cotton Thread

$H_t$  = Heat Capacity per Gram of Cotton Thread | Calorie Value of Cotton Thread

Example: Consider the previous standardization test in which 1.651 grams of standard benzoic acid (heat of combustion 6318 cal/g) produces a temperature rise of 3.047°C but a Nichrome Fuse Wire of 0.0184 gm was used and burnt along with 0.005 gm of Cotton Thread.

The Water Equivalent (W.E) of the calorimeter is then calculated as follows:

$$E_w = (0.0184)(333.68) = 6.14 \text{ cal}$$

$$E_t = (0.005)(4180) = 20.9 \text{ cal}$$

$$W.E = \frac{(1.651)(6318) + 6.14 + 20.9}{3.047} = 3432.24 \text{ cal/}^\circ\text{C}$$

It is important to standardise the Bomb Calorimeter for Nichrome Fuse Wire only and for both Nichrome Fuse Wire and Cotton Thread. It is critical to use the same standard Nichrome Fuse Wire and Cotton Thread as used for the Standardising Process.

Please note that the energy equivalent for any calorimeter is dependent upon a set of operating conditions, and these conditions must be reproduced when the fuel sample is tested if the energy equivalent is to remain valid. It is readily apparent that a difference of one gram of water in the calorimeter will alter this value by one calorie per degree Celsius. Less obvious but equally important are the changes resulting from different bombs or buckets with unequal masses, different operating temperatures, different thermometers, or even the biases imposed by different operators

## Heat Capacity Calculation:

Heat Capacity or the Calorie Value of the test sample is calculated using the following formula:

$$H = \frac{(W.E)(\Delta T)}{M}$$

Where:

W.E = Water Equivalent | Heat Capacity of Bomb Calorimeter | Energy Equivalent

M = Mass of the Test Sample

$\Delta T$  = Rise in Temperature

In case of calculating calorific values of liquids inside gelatine capsules, place empty capsule of known weight and find out the calorific value of capsule as per above formula.

Note: Calorimetry corrections will also need to be made. See below

Once the calorie value of the gelatine capsule is calculated, take another capsule and weigh it correctly. Fill the capsule (not completely) with the liquid sample and reweigh it to know the mass of the total sample. Perform the experiment and note down the rise in temperature.

The calorific value of the liquid can be found using the following formula:

$$H = \frac{(W.E)(\Delta T) - (E_{wl} + E_{tl}) - (E_g)(M_g)}{M_t - M_g}$$

Where:

W.E = Water Equivalent | Heat Capacity of Bomb Calorimeter | Energy Equivalent

$\Delta T$  = Rise in Temperature

$E_{wl}$  = Calorific value of the leftover Nichrome Fuse Wire

$E_{tl}$  = Calorific value of the leftover Cotton Thread

$E_g$  = Calorific value of gelatine capsule

$M_g$  = Mass of the gelatine capsule

$M_t$  = Mass of the Liquid Sample + Mass of Gelatine



## Calorimetry Corrections:

Precision in bomb calorimetry is dependent upon a standard set of operating conditions. Those factors which cannot be held constant will require corrections to compensate for their effects. Factors affecting the bomb calorimeter readings are mostly the following:

1. Nichrome Fuse Wire Burning
2. Cotton Thread Burning
3. Sulphur and Nitrogen oxidation

### Nichrome Fuse Wire Burning and Cotton Thread Corrections:

The burning of the Nichrome Fuse Wire in the bomb contributes additional heat to the bomb combustion. Since the amount of fuse wire consumed in each test may vary, the energy contributed by the fuse must be determined for each test and a correction applied to compensate for this variance.

Similarly, the burning of the Cotton Thread in the bomb contributes additional heat to the bomb combustion. Since the amount of Cotton Thread consumed in each test may vary, the energy contributed by the cotton thread must be determined for each test and a correction applied to compensate for this variance.

As mentioned earlier, a constant standard length and gauge of Nichrome Fuse Wire and constant standardised weight of cotton thread must be used for all experiments. If the entire length of wire and cotton have burnt, no correction will be required since the Bomb Calorimeter was already standardised for this length and gauge of wire. But that is not always the case, there may be scenarios where not all of the wire nor all of the cotton have burnt. For such scenarios, corrections will need to be made.

To do the corrections, measure the length and the weight of the Nichrome Fuse Wire and the Cotton Thread left.

With that knowledge, the calorific value of the leftover Fuse Wire and Cotton Thread can be found using the following formula:

$$E_{wl} = (M_{wl})(H_w)$$

$E_{wl}$  = Calorific value of the leftover Nichrome Fuse Wire

$M_{wl}$  = Mass of Nichrome Fuse Wire leftover

$H_w$  = Heat Capacity per Gram of Nichrome Fuse Wire

$$E_{tl} = (M_{tl})(H_t)$$

$E_{tl}$  = Calorific value of the leftover Cotton Thread

$M_{tl}$  = Mass of Cotton Thread leftover

$H_t$  = Heat Capacity per Gram of Cotton Thread

With the knowledge of the calorific values of the leftover Fuse Wire and Cotton Thread, the corrections can be made to the Test Sample Calorific Value using the following formula:

$$H = \frac{(W.E)(\Delta T) - (E_{wl} + E_{tl})}{M}$$

Where:

W.E = Water Equivalent | Heat Capacity of Bomb Calorimeter | Energy Equivalent

$\Delta T$  = Rise in Temperature

$E_{wl}$  = Calorific value of the leftover Nichrome Fuse Wire

$E_{tl}$  = Calorific value of the leftover Cotton Thread

M = Mass of the Test Sample

### Sulphur and Nitrogen Corrections:

In normal combustion all Sulphur in a fuel is oxidized to Sulphur dioxide and discharged with the stack gases. But when the same material is burned in an oxygen bomb, the oxidation is carried further to trioxide which then reacts with moisture in the bomb to form Sulphuric acid. Likewise, in normal combustion nitrogen in the air is not affected. But when a fuel sample is burned in an oxygen bomb, some of the molecular nitrogen trapped in the bomb is oxidized and combined with water vapour to form nitric acid.

To perform the corrections, deduct 1.43 cal for each millilitre of 0.1 N nitric acid produced and 22.5 calories for each 0.01 gm of Sulphur found from the total calories developed.

### Safety Precautions:

The operator must follow the following basic points in order to operate the Oxygen Bomb Calorimeter safely:

1. Do not use too much sample. The bomb cannot be expected to withstand the effects of combustible charges which liberate more than 10,000 calories. This generally limits the total weight of combustible material (sample + gelatine, firing oil or any combustible aid) to not more than 1.10gm. Do not charge with more oxygen than is necessary and do not fire the bomb if an overcharge of oxygen should accidentally be admitted
2. Keep all parts of the bomb, especially the insulated electrode assembly in good repair at all times. Do not fire the bomb if gas bubbles are leaking from the bomb when it is submerged in water
3. Stand back from the calorimeter for at least 15 seconds after firing and above all, keep clear of the top of the calorimeter. If the bomb explodes, it is most likely that the force of explosion will be directed upwards
4. Proceed with caution and use only a fraction of the allowable maximum sample when testing new materials which burn rapidly, or which have explosive characteristics

## Causes for Poor Combustion:

An incomplete combustion in the oxygen bomb is nearly always due to one or more of the following causes:

1. Excessively, rapid admission of Oxygen gas to the bomb during charging, causing part of the sample to be blown out of the cup
2. Loose or powdery condition of the sample in the cup prior to ignition causing ejection due to violence of combustion
3. The use of a sample containing coarse particles which cannot burn readily with coal, such particles are usually too large to pass through a 60 mesh screen.
4. The use of a sample in the form of pellet which has been made too hard causing spilling and the ejection of fragments during heating.
5. Use of an ignition current too low to ignite the charge or too high causing the fuse to break before combustion is well under way.
6. Insertion of the fuse wire loop below the surface of a loose sample. Best results are obtained by barely touching the surface of the sample or even hanging the wire slightly above the surface.
7. Use of not enough oxygen to burn the charge completely or conversely, the use of a very high initial gas pressure which may retard development of the required turbulence during combustion.