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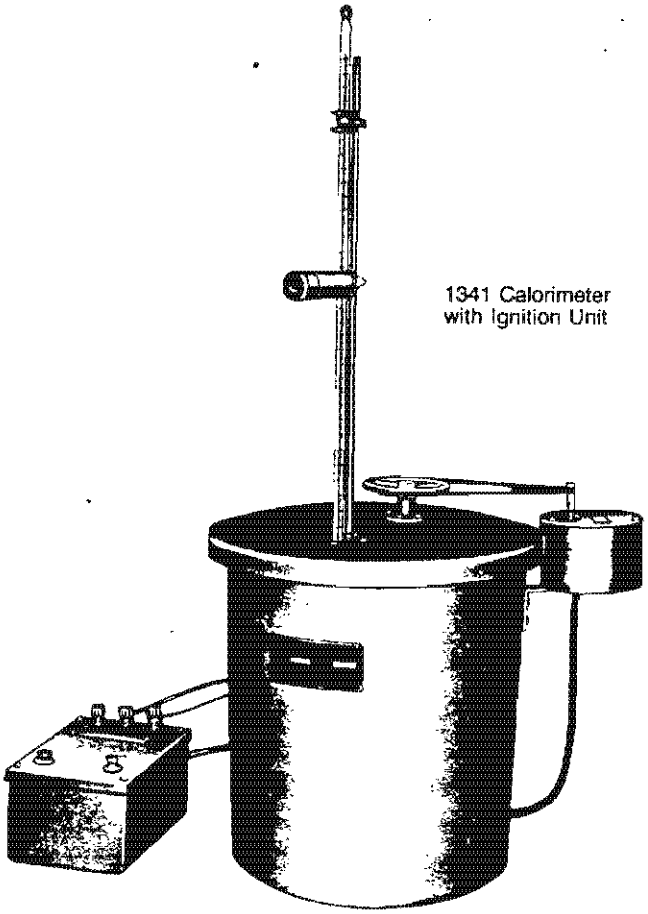
No. 204M

OPERATING INSTRUCTIONS

for the 1341 Oxygen Bomb Calorimeter



1341 Calorimeter
with Ignition Unit

SCOPE

These instructions cover the steps to be taken in setting up and operating a Parr 1341 Plain Oxygen Bomb Calorimeter. The user should study these instructions carefully before starting to use the calorimeter so that he will fully understand the capabilities of the equipment, and so that he will be well aware of the safety precautions to be observed in its operation. Instructions covering the operation of the 1108 Oxygen Bomb and the use of other related apparatus are provided in separate instruction sheets listed below. The separate sheets which apply to a particular calorimeter installation should be added to and made a part of these instructions.

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RELATED INSTRUCTIONS

SHEET No.	
205M	Instr. for the 1108 Oxygen Combustion Bomb
211M	Instr. for Mercurial Calorimetric Thermometers
212M	Instr. for the 1661 Recording Calorimetric Thermometer
214M	Instr. for the 1841 Autocharger
201M	Limited Warranty

OPERATING THE 1108 OXYGEN BOMB

Detailed instructions for preparing the sample and charging the 1108 oxygen bomb are given in Instruction Sheet No. 205M. Follow these instructions carefully, giving particular attention to the precautions to be observed in charging and handling the bomb.

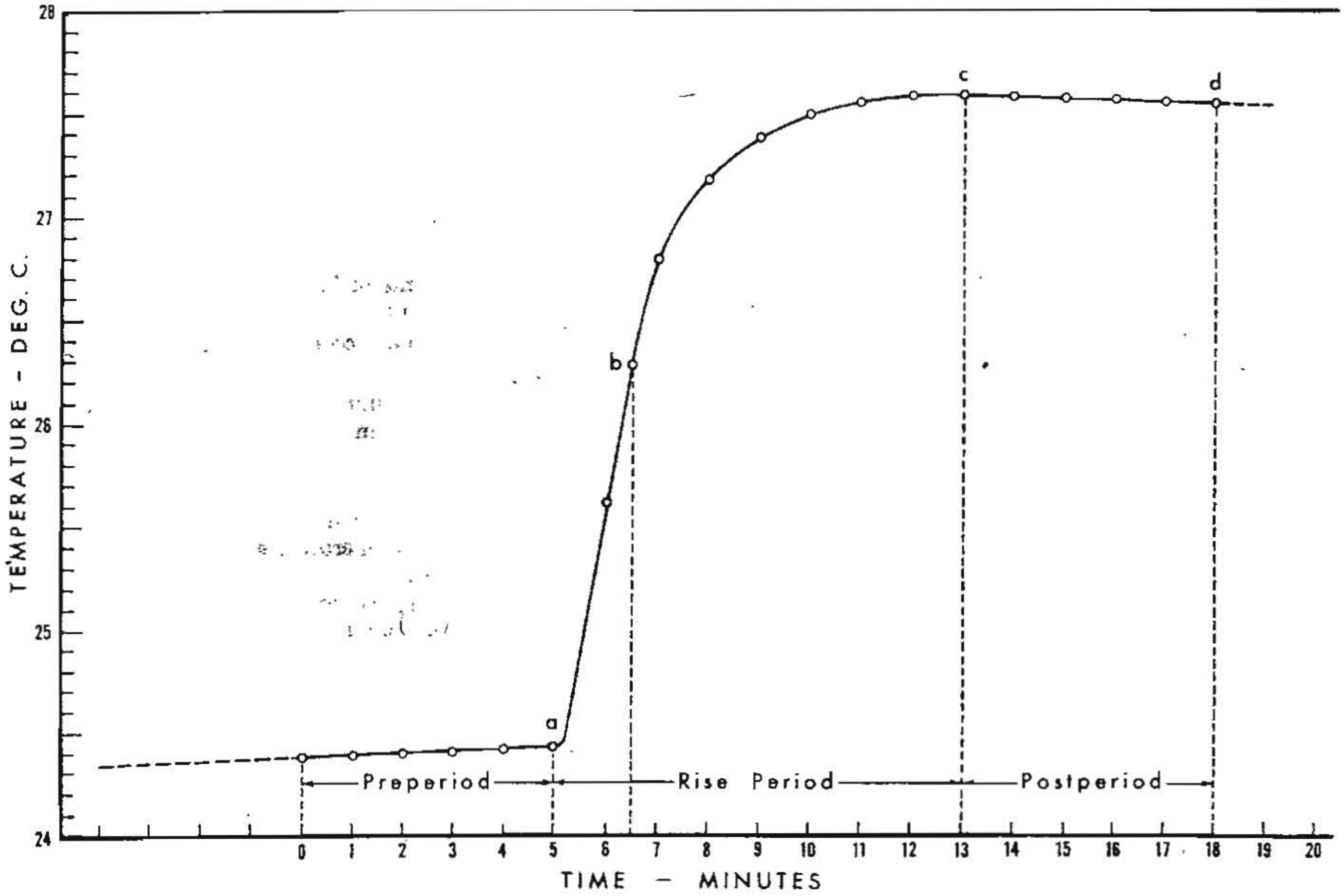
OPERATING THE CALORIMETER

All operations required to test an unknown sample or to standardize the 1341 plain calorimeter should proceed step-wise in the following manner:

1. **Prepare the sample and charge the oxygen bomb** as described in Instruction Sheet No. 205M.

2. **Fill the calorimeter bucket** by first taring the dry bucket on a solution or trip balance; then add 2000 (+/−0.5) grams of water. Distilled water is preferred, but demineralized or tap water containing less than 250 ppm of dissolved solids is satisfactory. The water temperature should be approximately 1.5° C below room temperature, but this can be varied to suit the operator's preference. It is not necessary to use exactly 2000 grams, but the amount selected must be duplicated within +/−0.5 gram for each run. Instead of weighing the bucket it can be filled from an automatic pipet or from any other [illegible] device if the repeatability of the filling system is within +/−0.5 ml. and the water temperature is held within a 1° C range.

3. **Set the bucket in the calorimeter;** attach the lifting handle to the two holes in the side of the screw cap and lower the bomb into the water with its feet spanning the circular boss in the bottom of the bucket. Handle the bomb carefully during this operation so that the sample will not be disturbed. Remove the handle and shake any drops of water back into the bucket; then push the two ignition lead wires into the terminal sockets on the bomb head, being careful not to remove any water from the bucket with the fingers.



Typical Temperature Rise Curve for 1341 Plain Calorimeter

4. Set the cover on the jacket with the thermometer facing toward the front. Turn the stirrer by hand to be sure that it runs freely; then slip the drive belt onto the pulleys and start the motor.

5. Let the stirrer run for 5 minutes to reach equilibrium before starting a measured run. At the end of this period record the time or start a timer and read the temperature to one-tenth of the smallest scale division. Always tap the thermometer with a pencil or rod to vibrate the mercury before taking a reading. Or, use a Parr 3010 Thermometer Vibrator for this purpose.

6. Read and record temperatures at one-minute intervals for 5 minutes. Then, at the start of the 6th minute...

7. Stand back from the calorimeter and fire the bomb by pressing the ignition button and holding it down until the indicator light goes out. Normally the light will glow for only about ½ second but release the button within 5 seconds regardless of the light. **Caution: Do not have the head, hands or any parts of the body over the calorimeter when firing the bomb; and continue to stand clear for 30 seconds after firing.**

8. The bucket temperature will start to rise within 20 seconds after firing. This rise will be rapid during the first few minutes; then it will become slower as the temperature approaches a stable maximum as shown by the typical temperature rise curve on page 3. It is not necessary to plot a similar curve for each test, but accurate time and temperature observations must be recorded to identify certain points needed to calculate the calorific value of the sample.

9. Measure the time required to reach 60 per cent of the total rise by estimating the temperature at the 60% point and observing the time when the rising mercury thread reaches that level. If the 60% point cannot be estimated before ignition, take temperature readings at 45, 60, 75, 90 and 105 seconds after firing and interpolate between these readings to identify the 60% point after the total rise has been measured. These readings can be taken without a magnifier since estimates to the nearest 0.02° C are sufficient at this point.

10. After the rapid rise period (about 4 or 5 minutes after ignition) adjust the reading lens and record temperatures to one-tenth of the smallest scale division at one-minute intervals until the **difference** between successive readings has been constant for five minutes. Usually the temperature will reach a maximum; then drop very slowly. But this is not always true since a low starting temperature may result in a slow continuous rise without reaching a maximum. As stated above, the **difference** between successive readings must be noted and the readings continued at one-minute intervals until the **rate** of the temperature change becomes constant over a period of 5 minutes.

11. After the last temperature reading, stop the motor, remove the belt and lift the cover from the calorimeter. Wipe the thermometer bulb and stirrer with a clean cloth and set the cover on the A37A support stand. Lift the bomb out of the bucket; remove the ignition leads and wipe the bomb with a clean towel.

12. Open the knurled knob on the bomb head to release the gas pressure before attempting to remove the cap. This release should proceed slowly over a period of not less than one minute to avoid entrainment losses. After all pressure has been released, unscrew the cap; lift the head out of the cylinder and place it on the support stand. Examine the interior of the bomb for soot or other evidence of incomplete combustion. If such evidence is found, the test will have to be discarded.

13. Wash all interior surfaces of the bomb with a jet of distilled water and collect the washings in a beaker.

14. Remove all unburned pieces of fuse wire from the bomb electrodes; straighten them and measure their combined length in centimeters. Subtract this length from the initial length of 10 centimeters and enter this quantity on the data sheet as the net amount of wire burned.

15. Titrate the bomb washings with a standard sodium carbonate solution using methyl orange or methyl red indicator. A 0.0709N sodium carbonate solution is recommended for this titration to simplify the calculation. This is prepared by dissolving 3.76 grams of Na_2CO_3 in water and diluting to one liter. NaOH or KOH solutions of the same normality may be used.

16. Analyze the bomb washings to determine the sulfur content of the sample if it exceeds 0.1 per cent. Methods for determining sulfur are discussed in Instruction Sheet No. 207M.

CALCULATING THE HEAT OF COMBUSTION

Assembly of Data. The following data should be available at the completion of a test in a 1341 calorimeter:

a = time of firing
b = time (to nearest 0.1 min.) when the temperature reaches 60 per cent of the total rise
c = time at beginning of period (after the temperature rise) in which the rate of temperature change has become constant
t_a = temperature at time of firing, corrected for thermometer scale error
t_c = temperature at time c, corrected for thermometer scale error
r_1 = rate (temperature units per minute) at which temperature was rising during the 5-min. period before firing
r_2 = rate (temperature units per minute) at which the temperature was rising during the 5-min. period after time c. If the temperature was falling instead of rising after time c, r_2 is negative and the quantity $-r_2(c-b)$ becomes positive and must be added when computing the corrected temperature rise
c_1 = milliliters of standard alkali solution used in the acid titration
c_2 = percentage of sulfur in the sample
c_3 = centimeters of fuse wire consumed in firing
W = energy equivalent of the calorimeter, determined under STANDARDIZATION
m = mass of sample in grams

Temperature Rise. Compute the net corrected temperature rise, t, by substituting in the following equation:

$$t = t_c - t_a - r_1(b-a) - r_2(c-b)$$

Thermochemical Corrections: Compute the following for each test:

e_1 = correction in calories for heat of formation of nitric acid (HNO_3)
= c_1 if 0.0709N alkali was used for the titration
e_2 = correction in calories for heat of formation of sulfuric acid (H_2SO_4)
= $(13.7)(c_2)(m)$
e_3 = correction in calories for heat of combustion of fuse wire
= $(2.3)(c_3)$ when using Parr 45C10 nickel chromium fuse wire, or
= $(2.7)(c_3)$ when using No. 34 B. & S. gage iron fuse wire

Gross Heat of Combustion. Compute the gross heat of combustion, H_g, in calories per gram by substituting in the following equation:

$$H_g = \frac{tW - e_1 - e_2 - e_3}{m}$$

Example.

$a = 1{:}44{:}00 = 1{:}44.0$
$b = 1{:}45{:}24 = 1{:}45.2$
$c = 1{:}52{:}00 = 1{:}52.0$
$t_a = 24.428 + .004 = 24.432$ C
$t_c = 27.654 + .008 = 27.662$ C
$r_1 = + .010$ °C/5 min. $= + .002$ °C/min.
$r_2 = - .004$ °C/5 min. $= - .001$ °C/min.
$c_1 = 23.9$ ml.
$c_2 = 1.02\%$ Sulfur
$c_3 = 7.6$ cm. Parr 45C10 wire
$W = 2426$ calories/°C
$m = .9936$ grams
$t = 27.662 - 24.432 - (.002)(1.4) - (-.001)(6.6)$
$= 3.234$ °C
$e_1 = 23.9$ calories
$e_2 = (13.7)(1.02)(.9936) = 13.9$ calories
$e_3 = (2.3)(7.6) = 17.5$ calories

$$H_g = \frac{(3.234)(2426) - 23.9 - 13.9 - 17.5}{.9936}$$

$= 7841$ calories/gram
$= (1.8)(7841) = 14{,}114$ Btu/lb

Conversion to Other Bases. The calculations described above give the calorific value of the sample with moisture as it existed when the sample was weighed. For example, if an air-dried coal sample was tested, the results will be in terms of heat units per weight of air-dry sample. This can be converted to a moisture free or other dry basis by determining the moisture content of the air-dry sample and using conversion formulae published in ASTM Method D3180 and in other references on fuel technology.

The calorific value obtained in a bomb calorimeter test represents the gross heat of combustion for the sample. This is the heat produced when the sample burns, plus the heat given up when the newly formed water vapor condenses and cools to the temperature of the bomb. In nearly all industrial operations this water vapor escapes as steam in the flue gases and the latent heat of vaporization which it contains is not available for useful work. The net heat of combustion obtained by subtracting the latent heat from the gross calorific value is therefore an important figure in power plant calculations. If the percentage of hydrogen, H, in the sample is known, the net heat of combustion, H_n in Btu per pound can be calculated as follows:

$$H_n = 1.8H_g - 91.23H$$

No factory standardization is currently recorded for this calorimeter. Your W values should be approximately 2420 ±10 cal/° C.

DISCUSSION OF CALCULATIONS

Thermometer Corrections. Thermometers furnished with Parr 1341 calorimeters have been tested for accuracy at intervals of not less then 1.5° C over the entire graduated scale. The corrections to be applied at each of these test points are reported on a certificate and plotted on a correction chart. The correction for any observed temperature may be read from the chart, and it must be added or subtracted as indicated.

During the operation of a plain jacket calorimeter the difference between the calorimeter temperature inside the jacket and the ambient room temperature will usually be less than 1.5° C. Since this difference is within one thermometer test interval, an accuracy adequate for most calorimetric tests can be obtained without applying a differential stem correction. However, if the greatest accuracy obtainable from the equipment is desired, or if there is a regular difference of more than 1.5° C between the jacket and its surroundings, then a differential stem correction should be made and applied to the corrected temperature rise, t, in all tests, including standardization. This correction is computed as follows:

$$\text{Stem Corr.} = K\,(t_c - t_a)\,(t_c + t_a - L - T)$$

where:

K = differential expansion coefficient for mercury in glass = .00016 for Centigrade thermometers, or .00009 for Fahrenheit thermometers

L = scale reading to which thermometer was immersed

T = mean temperature of emergent stem

t_a = initial temperature reading

t_c = final temperature reading

When using Beckmann thermometers, both a differential emergent stem correction and a "setting" correction must be applied to initial and final calorimeter temperature readings.

Acid Correction. Since combustion in the bomb takes place in an atmosphere of nearly pure oxygen at high temperature and pressure, several reactions take place which would not occur in burning the same material under normal atmospheric conditions. These side reactions are important because they generate an appreciable amount of heat which cannot be credited to the sample, and for which a correction must be made.

For example, in the normal combustion of coal, all sulfur is oxidized and liberated as SO_2 but nitrogen in the material usually is not affected. Likewise, no change occurs in the nitrogen of the air required for normal combustion. But, when the same coal is burned in the oxygen bomb, oxidation of the sulfur is carried further to form SO_3 which combines with water vapor to form H_2SO_4; and some of the nitrogen in the bomb is also oxidized and combined with water vapor to form HNO_3. These two side reactions result in acids within the bomb, and require a correction to account for the heat liberated in their formation.

In computing the correction for acid formation it is assumed that all of the acid titrated is nitric acid (HNO_3), and that the heat of formation of 0.1N HNO_3 under bomb conditions is −14.1 Kcal per mol. Obviously, if sulfuric acid is also present, part of the correction for H_2SO_4 is included in the nitric acid correction. The sulfur correction described below takes care of the difference between the heats of formation of nitric and sulfuric acids.

Sulfur Correction. A correction of 1.4 Kcal must be applied for each gram of sulfur converted to sulfuric acid. This is based upon the heat of formation of 0.17N H_2SO_4 which is −72.2 Kcal per mol. But a correction of 2 × 14.1 Kcal per mol of sulfur is included in the nitric acid correction. Therefore the additional correction which must be applied for sulfur will be 72.2 − (2 × 14.1) or 44.0 Kcal per mol, or 1.37 Kcal per gram of sulfur. For convenience, this is expressed as 13.7 calories for each percentage point of sulfur per gram of sample.

Fuse Wire Correction. The wire used as a fuse for igniting the sample is partly consumed in the combustion. Thus the fuse generates heat both by the resistance it offers to the electric firing current, and by the heat of combustion of that portion of the wire which is burned. It can be assumed that the heat input from the electric firing current will be the same when standardizing the calorimeter as when testing an unknown sample, and this small amount of energy therefore requires no correction. However, it will be found that the amount of wire consumed will vary from test to test, therefore a correction must be made to account for the heat of combustion of the metal.

The amount of wire taking part in the combustion is determined by subtracting the length of the recovered unburned portion from the original length of 10 cm. The correction is then computed for the burned portion by assuming a heat of combustion of 2.3 calories per cm. for Parr 45C10 (No. 34 B & S gage "Chromel C") wire, or 2.7 calories per cm for No. 34 B & S gage iron wire

Radiation Correction. The method recommended for calculating the correction for heat gain or loss from a plain calorimeter is that specified by the American Society for Testing and Materials as published under ASTM Designations D240 and D3286. It is based upon the work of Dr. H. C. Dickinson at the National Bureau of Standards who showed that the amount of heat leak during a test could be approximated by assuming that the calorimeter is heated by its surroundings during the first 63 per cent of the temperature rise at a rate equal to that measured during the 5-minute preperiod. The method then assumes that the cooling (or heating) rate during the remaining 37 per cent of the rise is the same as the rate observed during the 5-minute postperiod. For most experimental work the dividing point between these two periods is taken as that point in time, b, when the temperature has reached six-tenths (instead of 63%) of the total rise. Note that these two time intervals must be expressed in minutes and decimal fractions (Example: 1.4 min. and 6.6 min.).

OPERATING SUGGESTIONS

Thermometer Reliability. It is essential that temperatures be measured with a reliable calorimetric thermometer and that scale corrections be applied where applicable. Rough handling in shipment or by the user may cause mercury separations which must be eliminated before a thermometer will operate properly. Detailed instructions for checking and correcting this condition are furnished with each Parr thermometer. The same thermometer which was used when the energy equivalent was determined should remain in place for all subsequent tests with unknown samples. If it becomes necessary to replace a thermometer, the energy equivalent should be rechecked.

Recording Calorimetric Thermometer. To simplify the procedure for measuring and computing the net temperature rise in a 1341 calorimeter, Parr offers a thermistor thermometer (No. 1661) developed specifically for measuring the temperature change in the calorimeter with better resolution than can be obtained with the usual mercurial thermometer. The 1661 thermometer consists of a special thermistor probe and bridge which can be used with a strip chart recorder to produce a permanent graphic record of the temperature rise during a calorimetric test. A graphic method can then be used to determine the net rise directly from the recorded trace without having to compute and apply specific heat leak rates. This is an easily operated system which is well matched to the 1341 calorimeter, offering an attractive means for simplifying temperature measurements and upgrading the calorimeter at a modest price. Complete operating details are provided in Instruction Sheet No. 212M.

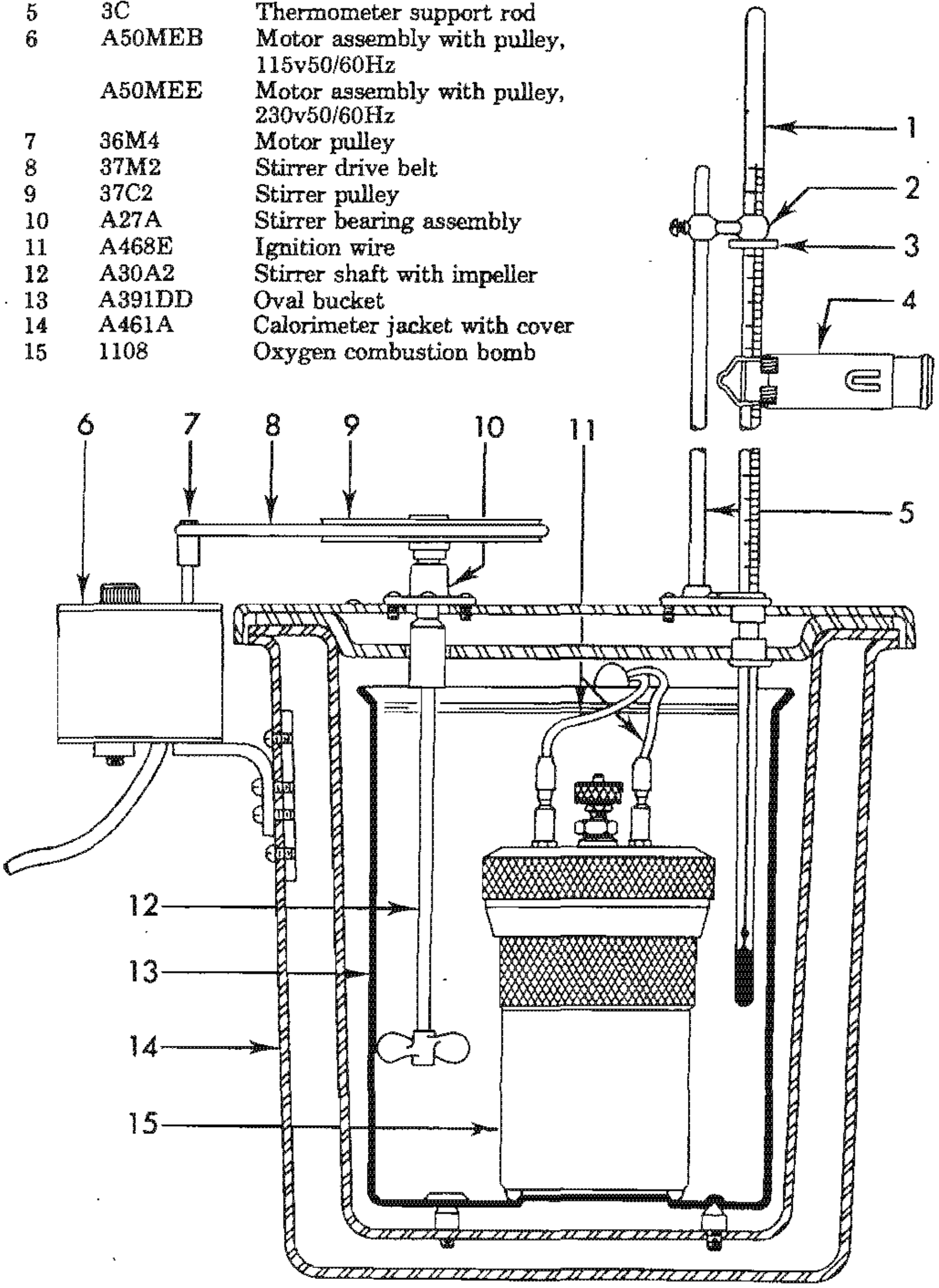
Magnitude of Errors. The following examples illustrate the magnitude of errors which may result from faulty calorimeter operations. They are based upon an assumed test in which a 1.0000 gram sample produced a 2.800° C temperature rise in a calorimeter having an energy equivalent of 2400 calories per deg. C.

> An error of 1 milliliter in making the acid titration will change the thermal value 1.0 cal.
>
> An error of 1 centimeter in measuring the amount of fuse wire burned will change the thermal value 2.3 cal.
>
> An error of 1 gram in measuring the 2 kilograms of water will change the thermal value 2.8 cal.
>
> An error of 1 milligram in weighing the sample will change the thermal value 6.7 cal.
>
> An error of .002° C. in measuring the temperature rise will change the thermal value 4.8 cal.

If all of these errors were in the same direction, the total error would be 17.6 cal.

PARTS FOR THE 1341 CALORIMETER

Key No.	Part No.	Description
1	1603	Thermometer, 19-35° C.
2	A39C	Thermometer bracket
3	52C	Thermometer support washer
4	3003	Thermometer reading lens
5	3C	Thermometer support rod
6	A50MEB	Motor assembly with pulley, 115v50/60Hz
	A50MEE	Motor assembly with pulley, 230v50/60Hz
7	36M4	Motor pulley
8	37M2	Stirrer drive belt
9	37C2	Stirrer pulley
10	A27A	Stirrer bearing assembly
11	A468E	Ignition wire
12	A30A2	Stirrer shaft with impeller
13	A391DD	Oval bucket
14	A461A	Calorimeter jacket with cover
15	1108	Oxygen combustion bomb



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OPERATING THE 1108 OXYGEN BOMB

Precautions. Combustion with oxygen in a sealed bomb is a very effective and reliable method for releasing all heat energy obtainable from a sample and for preparing hydrocarbon compounds and carbonaceous materials for analysis, but there are certain precautions which must always be observed when using this equipment. In particular:

- Do not overcharge the bomb with too much sample or with a sample which might react with explosive violence.
- Do not overcharge the bomb with too much oxygen. The initial charging pressure should not exceed 40 atm (590 psig).
- Do not fire the bomb alone on an open bench without providing a protective cooling medium. Usually the bomb should be completely submerged in water during firing.
- Do not fire the bomb if gas bubbles are released from any point on the bomb when it is submerged in water.
- Stand away from the bomb during firing and do not handle the bomb for at least 20 seconds after firing.
- Keep the bomb in good condition at all times. Any parts that show signs of weakness or deterioration must be replaced promptly.
- Read the maintenance and safety instructions on p. 8 before starting to use the bomb, and urge all operating personnel to re-read these instructions often.

Allowable Sample Size. To stay within safe limits, the bomb should never be charged with a sample which will release more than 8000 calories when burned in oxygen, and the initial oxygen pressure should never exceed 40 atmospheres (590 psig.). This generally limits the mass of the combustible charge (sample plus benzoic acid, gelatin, firing oil or any combustion aid) to not more than 1.1 grams. When starting tests with new or unfamiliar materials it is always best to use samples of less than one gram, with the possibility of increasing the amount if preliminary tests indicate no abnormal behavior. To avoid damage to the bomb and possible injury to the operator, it should be a standing rule in each laboratory that the bomb must **never** be charged with more than 1.5 grams of combustible material.

Attaching the Fuse. Set the bomb head on the A38A support stand and fasten a 10 cm length of fuse wire between the two electrodes. Parr 45C10 nickel alloy wire is used for most tests, with platinum wire offered as an alternate for certain special procedures. The 45C10 wire is furnished on cards from which uniform 10 cm lengths can be cut without further measurement.

Quick-grip electrodes now installed in all new 1108 oxygen bombs eliminate most of the threading and twisting formerly required when binding the wire to plain electrodes. To attach the fuse to quick-grip electrodes, insert the ends of the wire into the eyelet at the end of each stem and push the cap downward to pinch the wire into place. No further threading or twisting is required. The procedure for binding the fuse to the 4A and 5A plain electrodes in older Parr bombs is illustrated in instruction manuals furnished with the original equipment. For convenience, it is recommended that the user purchase and install new 4A10 and 5A10 quick-grip electrodes as replacements for the 4A and 5A style in older equipment.

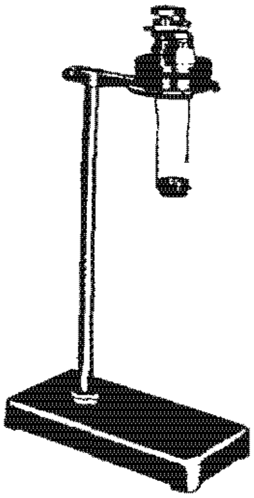
Place the fuel capsule with its weighed sample in the electrode loop and bend the wire downward toward the surface of the charge. It is not necessary to submerge the wire in a powdered sample. In fact, better combustions will usually be obtained if the loop of the fuse is set slightly above the surface. When using pelleted samples, bend the wire so that the loop bears against the top of the pellet firmly enough to keep it from sliding against the side of the capsule. It is also good practice to tilt the capsule slightly to one side so that the flame emerging from it will not impinge directly on the tip of the straight electrode.

Liquids in the Bomb. Most bomb combustion procedures call for a small amount of liquid to be placed in the bottom of the bomb as a sequestering agent and absorbent. If the amount and type of liquid are not otherwise specified, add 1.0 ml of distilled water from a pipet.

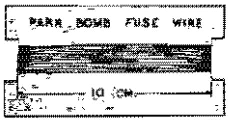
Closing the Bomb. Care must be taken not to disturb the sample when moving the bomb head from the support stand to the bomb cylinder. Check the sealing ring to be sure that it is in good condition and moisten it with a bit of water so that it will slide freely into the cylinder; then slide the head into the cylinder and push it down as far as it will go. For easy insertion, push the head straight down without twisting and leave the gas release valve open during this operation. When working with older bombs which have a removable compression ring, be sure that the 104A2 ring is in place above the gasket before attaching the screw cap. Current model A416A3 bomb heads do not require a separate compression ring. Set the screw cap on the cylinder and turn it down firmly by hand as far as it will go. It is important that the cap be turned down to a solid stop but it is not necessary to use a wrench or spanner on the cap. Hand tightening should be sufficient to secure a tight seal. It will be convenient—but not essential—to hold the bomb in the A124A2 bench clamp during the closing operation and while filling the bomb with oxygen.

Filling the Bomb. Parr offers either an automatic or a manual system for filling the bomb with oxygen. Instructions for the 1841 Auto Charger automatic system are given in a separate instruction sheet, No. 214M. The instructions below describe the manual system using the 1825 Oxygen Filling Connection furnished with all Parr calorimeters and oxygen bomb apparatus.

Oxygen for the bomb can be drawn from a standard commercial oxygen tank. Unscrew the protective cap from the tank and inspect the threads on the valve outlet to be sure they are clean and in good condition. Place the ball end of the connection into the outlet socket and draw up the union nut tightly with a wrench, keeping the 0-55 atm gage in an upright position.



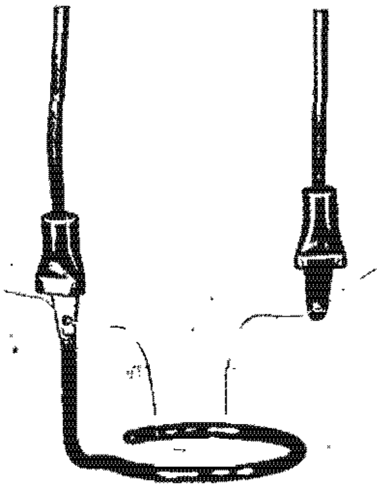
A38A Bomb Head Support Stand



45C10 Fuse Wire

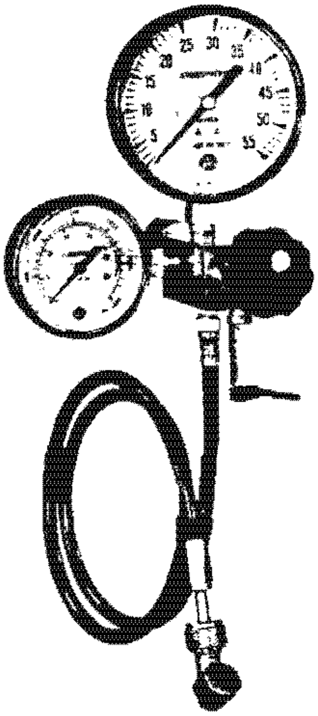
Set the bomb head on the A38A support stand when attaching the fuse and arranging the sample.

To attach the fuse: raise the cap; insert the wire through the eyelet; then pull the cap downward to complete the assembly.



The pressure connection to the bomb is made with a slip connector on the oxygen hose which slides over the gas inlet fitting on the bomb head. Older bombs use a threaded connector with a knurled coupling which must be turned finger tight. Slide the connector onto the inlet valve body and push it down as far as it will go. If it does not slide easily, a drop of water spread around the inlet valve will lubricate the sealing rings.

Close the valve on the filling connection; then open or "crack" the oxygen tank valve not more than one-quarter turn. Open the filling connection control valve slowly and watch the gage as the bomb pressure rises to the desired filling pressure (usually 30 atm., but never more than 40 atm.); then close the control valve. The bomb inlet check valve will close automatically when the oxygen supply is shut off, leaving the bomb filled to the highest pressure indicated on the 0-55 atm. gage. Release the residual pressure in the filling hose by pushing downward on the lever attached to the relief valve. The gage should now return to zero. If the pressure drops slowly and a large amount of gas escapes when the pressure relief valve is opened, the check valve in the bomb head is not operating properly. This trouble will have to be corrected before the bomb can be used. If too much oxygen should accidently be introduced into the bomb, **do not** proceed with the combustion. Detach the filling connection; exhaust the bomb; remove the head and reweigh the sample before repeating the filling operation.

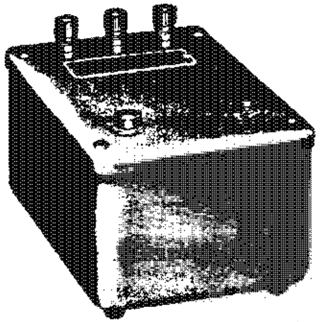


1825 Oxygen
Filling Connection

Firing the Bomb. If the bomb is to be used in a calorimeter, attach the 421A lifting handle to the two holes in the side of the screw cap and lower the bomb into the calorimeter water bucket with its feet spanning the circular boss in the bottom of the bucket. Remove the handle and shake any drops of water back into the bucket; then push the two ignition wires into the terminal sockets on the bomb head, being careful not to remove any water from the bucket with the fingers.

If the bomb is to be used alone for analytical purposes it should be used with a Parr 2901 ignition unit and held in an A378A or similar water bath during firing. Set the bomb in the water bath and attach ignition wires from the 10 cm binding posts on the ignition unit to the two terminals on the bomb head, pressing the banana plugs firmly into the terminal sockets.

In all operations, check the bomb for leaks before firing. If any gas leakage is indicated, no matter how slight, **do not fire the bomb.** Instead, remove it from the water bath; release the pressure and eliminate the leak before proceeding with a combustion test. If no leakage is indicated, adjust the water flow rate so that the bomb will be covered by a continuous flow of cold water during the firing period, then stand back and press the firing button on the ignition unit to fire the charge. **Caution: Do not have the head, hands or any parts of the body directly over the bomb during the firing period and do not go near the bomb for at least 20 seconds after firing.**



2901 Ignition Unit

Fire the charge by pressing the firing button on the ignition unit or on the calorimeter control panel, keeping the circuit closed for about 5 seconds. The indicator light will come on when the button is depressed and will remain on while current flows through the fuse. When the fuse burns off and breaks the circuit, the light will go out. Normally this takes about ½ second, but it is good practice to keep the push switch closed for about 5 seconds regardless of the light. If the light continues to glow while the button is depressed, there is either a short circuit in the firing system or the fuse was not properly arranged. If a 26 ga. platinum wire is used to fire the charge, hold the firing button down for only one or two seconds which should be sufficient to ignite the auxiliary fuse. A longer period may melt the wire. If the wire melts, use the 7 cm terminals on the ignition unit to obtain a lower firing voltage; or add a heavy, one-ohm resistor to the 10 cm firing circuit to lower the voltage.

If the indicator light does not come on when the firing button is pressed there is either an open circuit in the system or the A276E magnetic switch has burned out. An open circuit can usually be located with an ohmmeter. Flex the lead wires during any continuity check as the wires may be broken and making only intermittent contact. If the red indicator light glows during ignition but the bomb fuse does not burn, check the system for a voltage leak to ground, most likely in the insulated electrode on the bomb head. Check the electrode using the high impedance scale on an ohmmeter and replace the electrode insulator and seal if leakage is indicated.

Recovering the Combustion Products. Let the bomb stand in the calorimeter or water bath for at least 3 minutes, then lift it out of the water and wipe with a clean towel. Open the valve knob slightly to release all residual gas pressure before attempting to remove the screw cap. Gas release should proceed slowly over a period of not less than one minute to avoid entrainment losses. After all pressure has been released, unscrew the cap; lift the head out of the cylinder and place it on the support stand. Do not twist the head during removal. Pull it straight out to avoid sticking. Examine the interior of the bomb for soot or other evidence of incomplete combustion. If such is found the test will have to be discarded. Wash all interior surfaces of the bomb and the combustion capsule with a jet of distilled water and collect the washings. If any precipitate or residue is present, remove it with a rubber policeman. Do not filter the washings as this might remove valuable constituents. Titrate the washings and measure the unburned fuse wire as required for calorific tests, then analyze the washings for sulfur and other elements, if required.