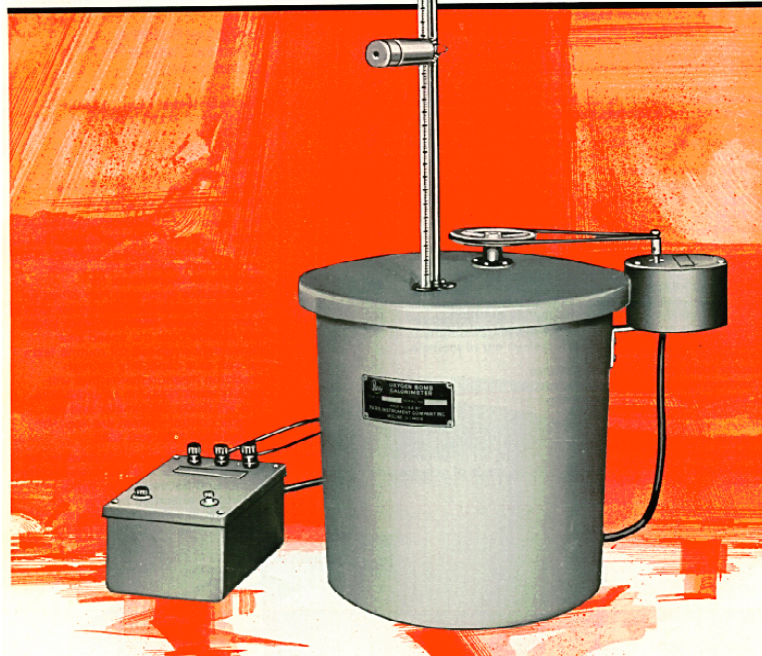


Plain Oxygen Bomb Calorimeter



INSTRUCTIONS FOR THE
1341 PLAIN JACKET
OXYGEN BOMB CALORIMETER

SCOPE	
THE 1341 CALORIMETER	3
SETTING UP THE CALORIMETER	3
Installing the Thermometer	4
The Ignition System	4
The 1108 Oxygen Bomb	6
CHARGING THE BOMB	6
Allowable Sample Size	6
Attaching the Fuse	6
Water in the Bomb	7
Closing the Bomb	7
Oxygen Filling Equipment	7
Filling the Bomb	7
1101 and 1102 Oxygen Bombs	7
SAFETY REQUIREMENTS	8
OPERATING THE CALORIMETER	8
Filling the Bucket	8
Assembling the Calorimeter	8
The Test Run	9
Opening the Calorimeter	9
The Acid Titration	10
The Fuse Correction	10
The Sulfur Correction	10
CALCULATING THE HEAT OF	
COMBUSTION	10
Assembly of Data	10
Temperature Rise	10
Thermochemical Corrections	10
Gross Heat of Combustion	10
Example	10
DISCUSSION OF CALCULATIONS	11
Thermometer Corrections	11
Acid Correction	11
Sulfur Correction	11
Fuse Wire Correction	11

Radiation Correction	12
Conversion to Other Bases	12
STANDARDIZING THE CALORIMETER	13
Standard Samples	13
The Energy Equivalent Factor	13
Standardization Procedure	13
Example	13
SAMPLES AND SAMPLE HOLDERS	14
Selection and Preparation	
of Solid Samples	14
Anthracites and Coke	14
Foodstuffs and Cellulosic Materials	14
Sample Pellets	15
Combustion Capsules	15
Gelatin Capsules	15
Glass Ampoules	15
Volatile Sample Holders	15
Heavy Oils	16
Explosives and High Energy Fuels	16
OPERATING SUGGESTIONS	16
Poor Combustions	16
DETERMINATION OF SULFUR	17
Sulfur in Combustible Solids	17
Sulfur in Combustible Liquids	17
Other Bomb Combustion Methods	18
MAINTENANCE INSTRUCTIONS	18
Bomb Maintenance	18
Hydrostatic and Proof Tests	18
Calorimeter Maintenance	18
Pellet Making with Parr Pellet Press	19

SCOPE

This Manual describes the operation of the Parr 1341 oxygen bomb calorimeter for measuring heats of combustion of samples which can be burned in oxygen. It gives step-by-step instructions for installing, operating, standardizing and maintaining the calorimeter as well as calculating heats of combustion from test data. Since this is primarily an operating manual, the user may want to consult other writings for additional information on calorimetric theory and high precision temperature measurements. Suggested references include:

ASTM Method D240 64, "Method of Test for Heat of Combustion of Liquid Hydrocarbon Fuels by bomb Calorimeter."

ASTM Method D271-70, "Laboratory Sampling and Analysis of Coal and Coke."

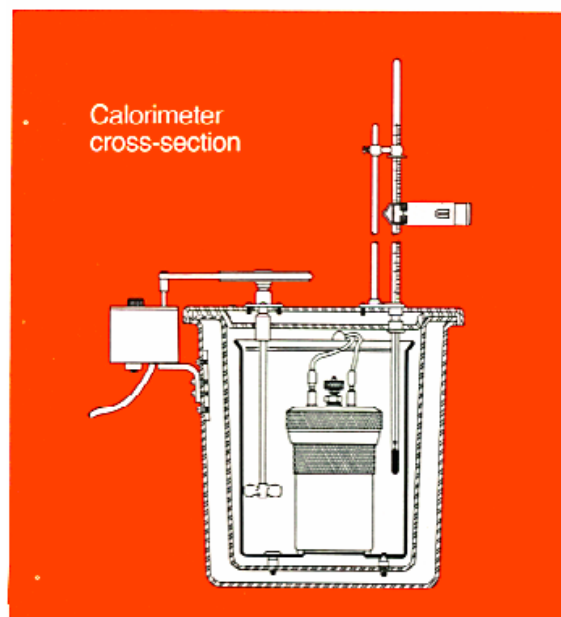
Experimental Thermochemistry-Measurement of Heats of Reaction, Rossini, Frederick D., Interscience Publishers, New York (1956) .

National Bureau of Standards Monograph No. 7, "Precise Measurement of Heat of Combustion with a Bomb Calorimeter", by R. S. Jessup.

Reprints of the above ASTM Methods are furnished by Parr as a supplement to this Manual. Copies of these individual methods can also be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA. 19103. The NBS Monograph can be purchased from the Supt. of Documents, Government Printing Office, Washington, D.C.

THE 1341 CALORIMETER

The 1341 calorimeter is an improved version of a bomb calorimeter which has been made by Parr for more than sixty years. Although commonly called a "plain" calorimeter because of its simple design, technically this is a static jacket instrument which operates at or near room temperature with no provision for temperature adjustment or control. Compensation for any heat loss (or gain) during a test is made by applying a correction computed from heat leak measurements taken immediately before and after each test. When operated with reasonable care, the precision attainable with this calorimeter will approach the capability of most general purpose adiabatic calorimeters, but the speed with which a test can be performed will be slower due to the additional time needed for measuring and computing a heat loss correction for each test.



Calorimeter cross-section

The operating principle for this calorimeter is the same as in all bomb calorimeters. A weighed sample is burned in an oxygen-filled metal bomb while the bomb is held in a measured quantity of water within a thermal insulating jacket. By observing the temperature rise of the water and knowing the energy equivalent of the calorimeter, the amount of heat released from the sample can be calculated. Test results are commonly expressed in calories per gram (cal/g), British thermal units per pound (Btu/lb) or in the large Calorie per gram units commonly used for foods. They may also be expressed in joules per kilogram (J/kg) as used in the SI system of international units. In this manual the International Table calorie is used. This is defined as equal to 4.1868 joules. The following conversion factors may be used:

$$\begin{aligned} 1 \text{ cal/g} &= 1.8 \text{ Btu/lb} \\ 1 \text{ cal/g} &= .001 \text{ Cal/g} \\ 1 \text{ cal/g} &= 4186.8 \text{ J/kg} \end{aligned}$$

SETTING UP THE CALORIMETER

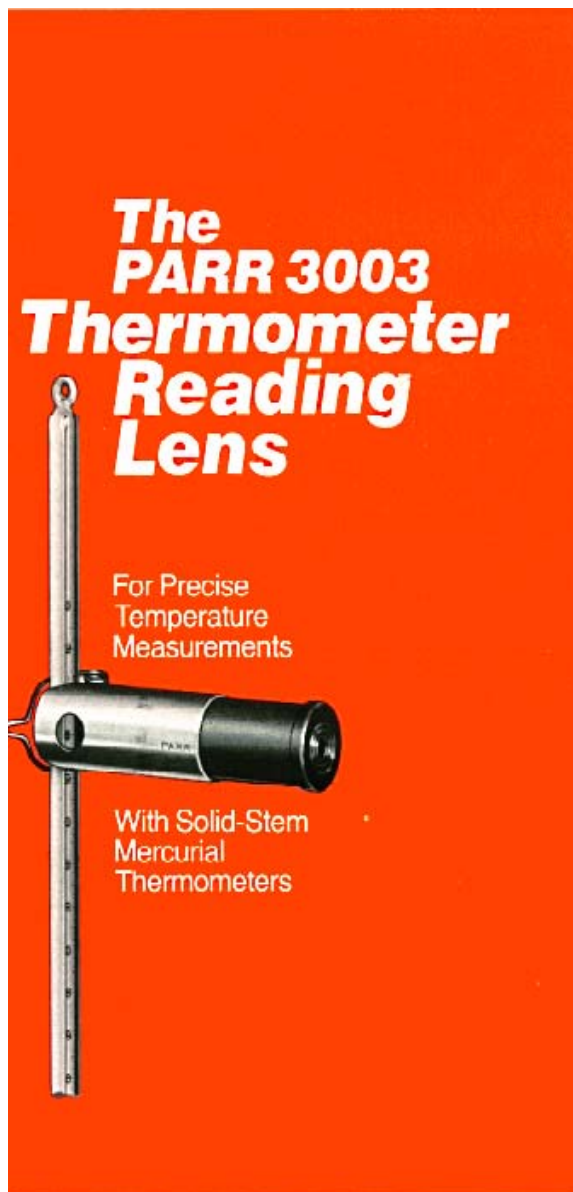
Unpack the calorimeter carefully and check the packing list to be sure that all parts are present and in good condition. Assemble the calorimeter on a sturdy bench or table in a location free from drafts and protected from sources of radiant heat, preferably in an air conditioned room. Temperature changes in the room should be minimal. There should be convenient access to cold running water, to a drain and to a 115

or 120 volt electric outlet. About 8 sq. ft. of work space will be required. Other necessary accessories include: a tank of oxygen (99.9S% minimum purity), a chemical balance sensitive to 0.1 mg and a solution, trip or torsion balance capable of weighing; up to 2.0 kg with 0.1 g sensitivity.

Screw the thermometer support rod into the support plate on the jacket cover and attach the stirrer drive motor to the jacket. Remove the middle two screws in the vertical row of four on the side of the jacket and attach the motor at this location. Never remove more than two screws at once from the row of four. Assemble the A37A calorimeter cover support stand and the A38A bomb head support stand.

Installing the Thermometer. Unwrap the thermometer carefully and inspect it for mercury separations as described in Instruction Sheet 1600 which is included with the thermometer test certificate and scale correction chart. Temperature extremes and rough handling in shipment will frequently cause mercury separations which must be removed before the thermometer can be used. The steps to be followed in this inspection and removal process are fully explained in the instruction sheet. After all gas bubbles and mercury-separations have been removed, moisten the two 52C thermometer support washers and slide them onto the thermometer stem. Position one washer at the bottom graduation on the thermometer and the other 2 1/2 inches from the top of the stem. Slide the bulb through the hole in the calorimeter cover; then with the lower washer resting against the cover bushing, slide the A39C bracket onto the top of the rod and lock it in position with the bracket bearing against the top washer. Clip the 3003 reading lens onto the thermometer stem and focus the magnifier by sliding the eyepiece.

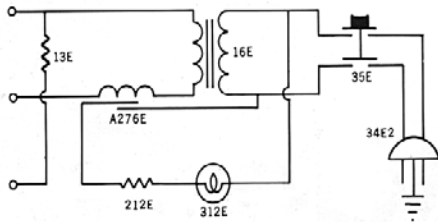
Once installed, the thermometer can remain attached to the cover during all operations. Always hold the thermometer upright when the cover is removed from the calorimeter jacket. Do not lay the cover on the table top. Always set it in the supporting ring on the A37A stand to keep the thermometer upright and to protect the thermometer bulb and the stirring shaft.



The Ignition System. While any electrical system capable of furnishing approximately 23 volts can be used to ignite the fuse wire in the oxygen bomb, most users will prefer to use a Parr 2901 ignition unit for this purpose. This unit operates from any standard electrical outlet to provide the proper low voltage firing current as well as a convenient push switch, an indicating lamp and connecting terminals.



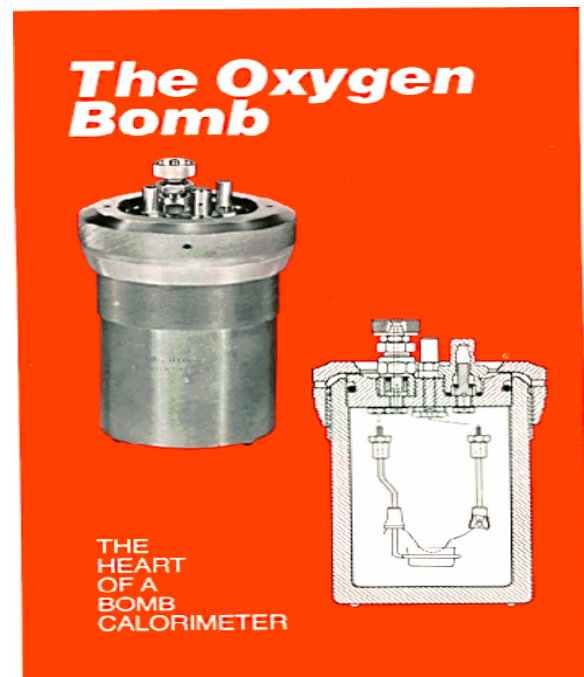
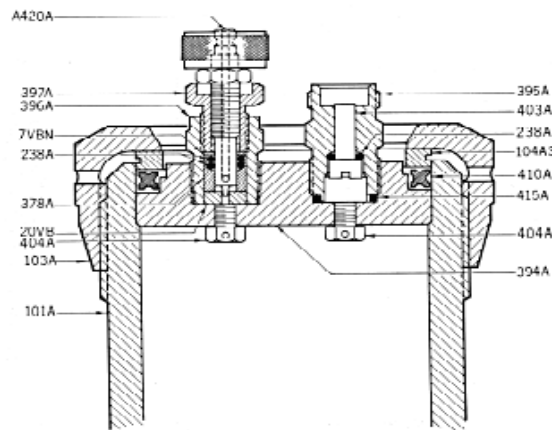
Connect the "10 CM FUSE" terminals on the ignition unit to the binding posts on the calorimeter jacket using the A38E lead wire which is provided; then plug the power cord into any 115 or 120 volt, 50 or 60 Hz grounded outlet. After the unit has been plugged into an outlet, do not press the firing button unless the lead wires in the calorimeter are connected to a bomb. If the bare terminals on the wire happen to be in contact with each other when the circuit is closed, the short circuit can damage the ignition

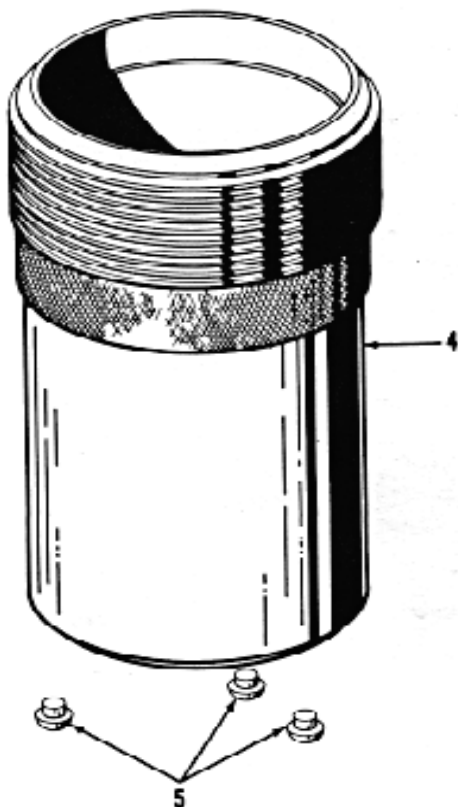


When the firing button is depressed the indicator light will come on while the fuse is heating, and the light will go out when the fuse burns off and breaks the circuit. Normally this takes place within about 1/2 second, but it is good practice to keep the push switch closed for about 5 seconds regardless 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. On the other hand, if the light does not come on when the button is pressed, there is either an open circuit in the system or the indicator light has burned out. An open circuit can usually be located with an ohmmeter. To check the firing voltage, attach an a.c. voltmeter across the ignition terminals. The output across these terminals should read 23 volts when

operating from a 115 v line. The red indicator light will not come on during this test since it glows only when a measurable current is flowing through the circuit to the bomb.

The 1108 Oxygen Bomb. Unless otherwise specified, each 1341 calorimeter is equipped with a Parr 1108 oxygen bomb. This is a 342 ml combustion bomb which can be sealed by simply turning a knurled cap until it is hand tight. Sealing forces develop internally when the bomb is pressurized, but after the pressure has been released the cap can be unscrewed easily and the head can be removed from the cylinder.



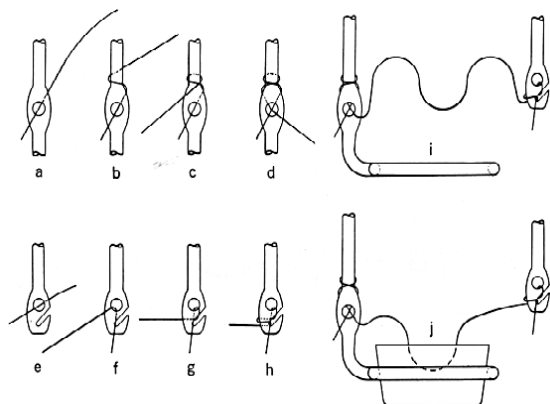


The 1108 bomb has two valves with replaceable stainless steel bodies. On the inlet side there is a check valve which opens when pressure is applied and closes automatically whenever the supply is shut off. On the outlet side gases are released through an adjustable needle valve with gases passing through a longitudinal hole in the valve stem and discharging from a short hose nipple at the top. Gas flow through the outlet valve is controlled by turning a knurled adjusting knob. A deflector nut on the inlet passage diverts the incoming gas so that it will not disturb the sample. A similar nut on the outlet side reduces liquid entrainment when gases are released.

CHARGING THE BOMB

Allowable sample Size. Care must be taken to avoid overcharging the bomb for it must be realized that the peak pressure developed during a combustion is proportional to the size of the sample and to the initial oxygen pressure. 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. Stand the initial oxygen pressure should never exceed 35 atmospheres (515 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 A3BA support stand and attach a 10 cm. length of fuse wire as shown on page 6. Parr 45C10 nickel alloy wire will be used for most tests, but the same procedure applies when using 36 ga. or finer platinum wire. In either case a pair of forceps or tweezers will be helpful for binding the wire to the electrodes. First, fasten one end of the wire to the loop electrode (steps a thru d); then attach the other end to the straight electrode (steps e thru h). Pull the loop downward to tighten the connections; then bend the wire upward as in detail i; Place the capsule in the loop holder and bend the wire down to touch the surface of the charge as shown in detail j.



Steps in Binding Fuse Wire to 4A and 5A Electrodes

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.

Water in the Bomb. Place 1.0 ml. of distilled water in the bomb from a piped

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. Be sure that the 104A2 contact ring is in place above the sealing ring and that the sealing ring is in good condition; then slide the head into the cylinder and push it down as far as it will go. It will be convenient (but not essential) to hold the bomb in an A124A2 bench clamp during the closing operation and while filling the bomb with oxygen. Set the screw cap on the cylinder and turn it down firmly by hand. Do not use a wrench or spanner on the cap. Hand tightening should be sufficient to secure a tight seal.

Oxygen Filling Equipment. Commercial oxygen as supplied in a standard 1A cylinder with CGA No. 540 outlet is usually of sufficient purity for calorimetric work. Connections to the oxygen cylinder are made with a Parr 1825 filling connection which is supplied with the calorimeter. This connection has a needle valve which controls the flow into the bomb and a 0-55 atm. gage which shows the pressure to which the bomb has been charged. It also has an automatic relief valve to prevent overcharging. To attach the filling connection, unscrew the protecting cap from the

oxygen 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 cylinder outlet and draw up the union nut tightly with a wrench, keeping the 0-55 atm gage in an upright position.

Filling the Bomb. Press the fitting on the end of the oxygen hose into the inlet valve socket and turn the knurled union nut finger tight. Close the control 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 not more than 30 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 connecting 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 release valve is opened, the check



A38A Bomb Head support Stand

valve in the bomb head is not operating properly. This trouble will have to be corrected before the bomb can be used.

1101 and 1102 Oxygen Bombs. Any existing 1101, 1102 or other Parr oxygen bomb (except 1107) can be used in the 1341 calorimeter by changing the terminals on the bomb head to accept banana plugs and by adding a grounding terminal if one is not already available. Bombs which have a grounding connection can be converted to fit the new

calorimeter by simply installing 411A terminal nuts on the insulated electrode and in the grounding socket. If the bomb head does not have a grounding connections this can be added by returning the head to Parr with instructions to install 411A terminal nuts.

If an existing 1101 or other older bomb is to be used interchangeably with a new 1108 bomb, an A427A adapter should be added to the oxygen inlet valve on the older bomb so that both can be filled from the same 1825 oxygen connection. Conversion parts are available for changing existing 1823 and other older oxygen connections to the 1825 style.

SAFETY REQUIREMENTS

The high pressures and nearly explosive reactions which occur within an oxygen bomb need not be considered unusually hazardous, provided that the bomb is in good operating condition and that the operator follows the recommended test procedure. All persons concerned with safe operation of the calorimeter should insist upon compliance with the directions given herein. Particular emphasis should be given to the following basic safety requirements:



1825 Oxygen Filling Connection

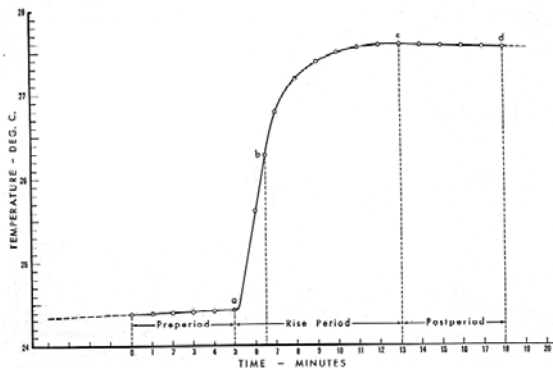
- Do not use too much sample. The total charge placed in the Parr 1108 bomb must not liberate more than 8000 calories when burned in oxygen at an initial pressure of 35 atmospheres. This usually limits the Samples weight to not more than 1.1 grams.

- Use a small fraction of the maximum allowable sample weight when testing unfamiliar materials which may burn either rapidly or explosively.
- Do not fill the bomb with more oxygen than is necessary to obtain complete combustion. Do not fire the bomb if it is pressurized to more than 35 atmospheres.
- Keep all parts of the bomb, especially the valves and insulated electrode, in good repair at all times. If gas bubbles escape from the bomb when it is submerged in water, do not fire the charge.
- Keep away from the top of the calorimeter for 30 seconds after firing. If the bomb should rupture, it is most likely that the resultant forces will be directed along the vertical axis.
- Never use lubricants on valves or fittings in contact with high pressure oxygen.

OPERATING THE CALORIMETER

Filling the Bucket. Weigh the dry calorimeter bucket on a Solution or trip balance, then add 2000 (plus or minus 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. The bucket may be filled from an automatic pipet or from any other volumetric device if the water is measured with comparable precision.

Assembling the Calorimeter. Set the filled bucket in the calorimeter with the bomb locating boss in the bottom of the bucket facing the front of the calorimeter. Attach the lifting handle to the two holes in the side of the screw cap and lower the bomb into the water. Before the head is submerged, push the ignition wires into the two terminal sockets on the bomb head; then push any excess wire back through the hole in the jacket liner. Lower the bomb into the water with its feet spanning the boss in the bottom of the bucket. Handle the bomb carefully during this process so that the sample will not be disturbed.



Typical Temperature Rise Curve for 1341 Plain Calorimeter

Remove the lifting handle and shake any drops of water back into the bucket; then set the cover on the calorimeter with the thermometer facing toward the front and with the cover locating pin inserted into the hole in the jacket rim. Turn the stirrer by hand to be sure that it runs freely; then attach the drive belt and start the motor. The calorimeter is now ready for the test run.

The Test Run. Let the stirrer run for 5 minutes to reach thermal equilibrium before starting a measured run. At the end of this period, record the time or start a timer and read the temperature to the nearest 0.002°C (or to 0.005°F if a Fahrenheit thermometer is used). Continue to read and record the temperature at one-minute intervals for exactly 5 minutes; then press the button on the ignition unit to fire the charge at the start of the sixth minute. 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. The vibrator sets on top of the thermometer stem and exerts a series of sharp impulses to level the mercury meniscus when activated by a control button on an A84C power supply.

At approximately 20 seconds after ignition the temperature will begin to rise. The rate of rise will be large at first and then decrease as the bomb, water and bucket approach a new equilibrium temperature. The nature of the temperature rise is indicated by the typical temperature rise curve shown on page 8. It is not necessary to plot a similar curve for each test, but accurate time and temperature observations must be recorded to identify the critical points needed to calculate the calorific value of the sample or the energy equivalent for the calorimeter.

If the net temperature rise can be estimated from previous tests with similar samples, add 60 per cent

of the expected total rise to the observed temperature at ignition and locate this point on the thermometer scale. Record the time in minutes and decimal fractions of a minute when the mercury column reaches this temperature. This observation can be taken without the thermometer magnifier since the temperature will be rising rapidly at this point and it will be difficult to keep the magnifier in focus.

If the net temperature rise at the 60% point cannot be estimated before ignition, the time required to reach the 60% point can be found by linear interpolation from readings taken during the rise period. This requires temperature observations at 45, 60, 75, 90 and 105 seconds after firing. These can be taken without a magnifier since readings; estimated to the nearest 0.02° are sufficient at this point.

After the rapid rise period (about 4 or 5 minutes after ignition) adjust the reading lens and record temperatures to the nearest 0.002°C (0.005°F) 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 upward 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.

Opening the Calorimeter. After the last temperature observation has been made, stop the motor, remove the belt and lift the cover from the calorimeter. Wipe the thermometer bulb and the stirrer with a clean cloth to remove any drops of water 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.

Open the knurled valve knob on the bomb head to release the residual 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.

The Acid Titration. Wash all interior surfaces of the bomb with a jet of distilled water and collect the

washings in a beaker. Titrate the washings with a standard sodium carbonate solution using methyl orange or methyl red indicator. A 0.0725N sodium carbonate solution is recommended for this titration to simplify the calculation. This is prepared by dissolving 3.84 grams of Na₂CO₃ in water and diluting to one liter. NaOH or KOH solutions of the same normality may be used.

The Fuse Correction. Carefully 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. (This correction may be omitted when using 36 ga. of finer platinum fuse wire.)

The Sulfur Correction. The sulfur content of the sample should be determined if it exceeds 0.1 percent. This analysis can be made by any standard gravimetric, volumetric or nephelometric method for sulfur using the solution remaining from the acid titration. A suggested procedure is given on page 15 in this Manual, also in ASTM Method D271, Sections 22 and 23.

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

r₁ = rate (temperature units per minute) at which temperature was rising during the 5-min. period before firing

r₂ = 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, r₂ is negative and the quantity -r₂(c—b) becomes positive and must be added when computing the corrected temperature rise.

c₁ = milliliters of standard alkali solution used in the acid titration

c₂ = percentage of sulfur in the sample

c_s = 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:

e₁ = correction in calories for heat of formation of nitric acid (HNO₃)

= c₁ if 0.0725N alkali was used for the titration

e₂ = correction in calories for heat of formation of sulfuric acid (H₂SO₄)

= (14) (c₂) (m)

e₃ = correction in calories for heat of combustion of fuse wire

= (2.3) (c₃) when using Parr 45C10 nickel-chromium fuse wire, or

= (2.7) (c₃) 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:

$$H_g = (tW - e_1 - e_2 - e_3)/m$$

Example.

a = 1:44:00 = 1:44.0

b = 1:45:24 = 1:45.4

e = 1:52:00 = 1:52.0

t_a = 24.428 + .004 = 24.432 °C

t_c = 27.654 + .008 = 27.662 °C

r₁ = + .010 °C/5 min. = + .002 DC/min.

r₂ = -0.004 °C/5 min. = -0.001 °C/min.

c₁ = 23.9 ml.

c₂ = 1.02% Sulfur

c₃ = 7.6 cm. Parr 45C10 wire

W = 2426 calories/°C

m = 0.9936 grams

t = 27.662 - 24.432 - (0.002) (1.4) - (-0.001) (6.6)

= 3.234 °C

e₁ = 23.9 calories

e₂ = (14) (1.02) (.9936) = 14.2 calories

e₃ = (2.3) (7.6) = 17.5 calories

H_g = ((3.234) (2426) — 23.9 - 14.2 - 17.5)/0.9936

= 7840 calories/gram

= (1.8) (7840) = 14,110 Btu/lb

$$= (4186.8) (7840) = 3.282 \times 10^7 \text{ joules/kg}$$

DISCUSSION OF CALCULATIONS

Thermometer corrections. Thermometers furnished with Parr 1341 calorimeters have been tested for accuracy at intervals of not less than 1.5° C or 2.5° F 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 or 2.5° F. 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 = 0.00016 for Centigrade thermometers, or 0.00009 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 13.8 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. This is adjusted by a separate computation based upon the sulfur content of the sample.

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 Kcal per mol. But, a correction of 2×13.8 Kcal per mol of sulfur is included in the nitric acid correction. Therefore the additional correction which must be applied for sulfur will be $72 - (2 \times 13.8) = 44.4$ Kcal per mol. or 1.4 Kcal per gram of sulfur. For convenience, this is expressed as 14 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 D271. 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.).

Conversion to Other Bases. The calculations described here 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 basis by determining the moisture content of the air-dry sample and using conversion formulae published in ASTM Method D271 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:

$$H_n = 1.8H_g - 91.23H$$

Errors. In addition to incomplete combustions, errors in calorimetric tests may be introduced in several other ways. Errors in thermometry can be extremely troublesome. It is essential that temperatures be measured with a reliable calorimetric thermometer and that scale corrections be applied where applicable. Even the best mercurial thermometer may develop separations or have gas bubbles introduced into the mercury during shipment or by improper handling. Usually this condition can be corrected by the user. Instructions for handling calorimetric thermometers and for removing gas bubbles from the mercury are furnished with each Parr thermometer.

The thermometer which was used with the calorimeter when its energy equivalent was determined should always be used in subsequent tests. If it is necessary to substitute a new thermometer, the energy equivalent should be rechecked.

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 .004° C. in measuring the temperature rise will change the thermal value 9.6 cal.

If all of these errors were in the same direction, the total error would be 22.4 cal.

STANDARDIZING THE CALORIMETER

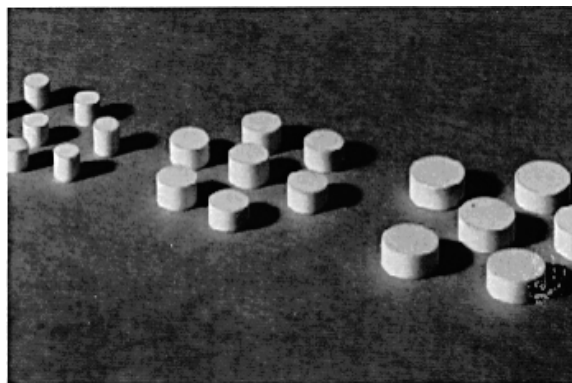
Standard Samples. A vial of ten one-gram benzoic acid pellets is furnished with each calorimeter for

standardization purposes. Additional benzoic acid pellets or powder can be obtained from Parr. For high precision measurements, a primary standard benzoic acid powder can be purchased from the National Bureau of Standards, Washington, D. C. The NBS also offers standard 2,2,4-trimethylpentane (Isooctane) for checking calorimeters which are to be used for testing volatile fuels. Benzoic acid is usually compressed into tablet or pellet form for standardizing bomb calorimeters because the pellets are easy to handle and they burn more slowly than loose samples, thereby minimizing incomplete combustions.

The Energy Equivalent Factor. The term “standardization” as used here denotes the operation of the calorimeter on a standard sample from which the energy equivalent or effective heat capacity of the system can be determined. The energy equivalent, W , of the calorimeter is the energy required to raise the temperature one degree, expressed as calories per degree Celsius or calories per degree Fahrenheit.

Parr furnishes a certificate with each complete calorimeter showing the energy equivalent for that calorimeter as determined in the Parr laboratory prior to shipment. No certificate is issued for an incomplete calorimeter, that is, one purchased without a thermometer or without a bomb because an exchange of these parts might change the factor. It is recommended that the user standardize his calorimeter and use the factor which he has determined rather than to assume that the value furnished by Parr will apply in his laboratory. If there is a large difference between his value and the factory certificate, the factors affecting the energy equivalent should be investigated. Standardization tests should be repeated after changing any parts of the calorimeter, and occasionally as a check on both the calorimeter and operating technique.

Standardization Procedure. The procedure for a standardization test is exactly the same as for testing a fuel sample. Use a pellet of calorific grade benzoic acid weighing not less than 0.9 nor more than 1.25 grams. Determine the corrected temperature rise, t , from the observed test data, also titrate the bomb washings to determine the nitric acid correction and measure the unburned fuse wire. Compute the energy



Calorific Standard
Benzoic Acid Powder and Pellets

equivalent by substituting in the following equation:

$$W = \frac{H_m + e_1 + e_2}{t}$$

W = energy equivalent of the calorimeter in calories per degree Celsius (Centigrade), or calories per degree Fahrenheit

H = heat of combustion of the standard benzoic acid sample in calories per gram

m = mass of the standard benzoic acid sample in grams

t = net corrected temperature rise in degrees C or F

e_1 = correction for heat of formation of nitric acid in calories

e_3 = correction for heat of combustion of the firing wire in calories

Example. Standardization with a 1.1651 gram benzoic acid sample (6318 cal/gm) produced a net corrected temperature rise of 3.047° C. The acid titration required 11.9 ml of standard alkali and 8 cm of fuse wire were consumed in the firing. Substituting in the standardization equation:

$H = 6318$ cal/gram

$m = 1.1651$ gram

$e_1 = (11.9 \text{ ml}) (1 \text{ cal/ml}) = 11.9 \text{ cal}$

$e_3 = (8 \text{ cm}) (2.3 \text{ cal/cm}) = 18.4 \text{ cal}$

$t = 3.047 \text{ C}$

$W = ((6318)(1.1651) + 11.9 + 18.4)/3.047$
 $= 2426 \text{ cal per deg C}$

SAMPLES AND SAMPLE HOLDERS

Selection and Preparation of Solid Samples. Solid

samples should be air-dry and ground until all particles will pass a 60-mesh screen. The particle size is important because the combustion reaction proceeds to completion within a few seconds and if any of the individual particles are too large they will not burn completely. A sample that is too finely divided may also be difficult to burn because extremely small particles can be swept out of the combustion capsule by the turbulent gases. If they fall to the bottom of the bomb without being ignited, the test will be unsatisfactory. The Parr pellet press offers a possible solution to the problem of incomplete combustion when working with powdered or finely divided samples.

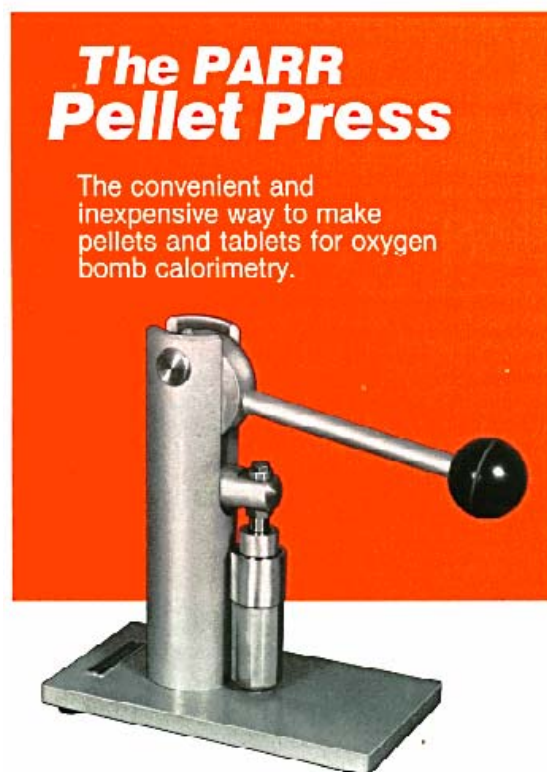
Some operators prefer to work with moisture-free or bone-dry samples instead of using air-dried samples as suggested above. There is no objection to this procedure if care is taken to prevent overheating and loss of volatile constituents when preparing the bone dry sample. Care must also be taken to avoid moisture absorption from the atmosphere when the sample is weighed.

Care in sampling is of the outmost importance. The best practice for obtaining a truly representative sample for a calorific test is a broad subject that requires more extensive treatment than can be given here. The user is urged to consult standard references such as ASTM Methods D2013 and D2234 if he is not familiar with the essential details.

Anthracites and Coke. When testing anthracite coals, coke or other slow-burning materials it may be difficult to secure ignition and complete combustion of the entire sample. In these cases it will be desirable to have the sample ground fine enough to pass a 100-mesh screen. These materials may be difficult to test, either because they are not easily ignited, or because they burn so slowly that the particles become chilled below the ignition point before complete combustion is obtained. For materials which are extremely difficult to ignite, a small weighed amount of a standard combustible, such as powdered benzoic acid, can be mixed with the sample. The heat of combustion of the additive is then subtracted from the total observed amount to obtain the net value for the sample. For samples which do not ignite easily it is sometimes helpful to make four or six small loops in the fuse wire and position these loops directly over the sample to concentrate the heat in one area.

Foodstuffs and Cellulosic Materials. The high moisture content of foodstuffs may require that they

be dried before making a calorific test, but this is not true in all cases. Samples containing up to 40% moisture can usually be burned in an oxygen bomb without preliminary drying, but samples with higher moisture contents may have to be dried. If possible, use a portion of the sample as received for the calorimetric test and run the moisture determination on a separate portion. When dealing with new materials it may be necessary to make several preliminary tests to determine the approximate maximum allowable moisture content at which the sample will ignite and burn completely. A certain amount of moisture is desirable in calorimetric samples since a very dry sample may burn so rapidly



that the particles will be carried out of the capsule to strike the cold wall of the bomb, leaving a smudge of unburned carbon as an indication that complete combustion was not obtained. Rapid burning rates can be slowed by simply adding a small amount of water to the weighed sample and allowing time for the moisture to be absorbed uniformly throughout the sample before firing the bomb.

Sample Pellets. It is helpful to compress coal or other powdered samples into a pellet or tablet before

the sample is weighed. Pellets are easier to handle than loose samples and they burn slower in an oxygen bomb, thereby reducing the chances for incomplete combustion. They are usually made in a Parr pellet press with one-half inch diameter punch and die. Instructions are supplied with each press. It will be convenient to make several pellets from the same sample and hold them in a stoppered vial or weighing bottle until they are to be weighed into a fuel capsule. Pellets should be handled with forceps or a small pair of tongs and not touched with the fingers.

Combustion Capsules. Non-volatile samples to be tested in Parr oxygen bombs are weighed and burned in shallow capsules measuring approximately 1" dia. and 7/16" deep. These are available in stainless steel, fused silica and platinum alloyed with 31/2% rhodium. Six stainless steel capsules (43AS) are furnished with each calorimeter. These are suitable for all tests except those in which a non-metallic (fused silica) holder is desired, or where the superior corrosion resistance of a Pt-Rh cup is required. The stainless capsules will become dull after repeated use and any loose oxide scale should be removed by polishing with fine emery cloth. Any capsule which has been used to a point where the bottom or the rim has worn thin should be discarded because of the danger that it might ignite and burn with an intensity that will damage the bomb.

Gelatin capsules. Volatile liquid samples to be burned in an oxygen bomb can be handled conveniently in Parr gelatin capsules. These are offered in several sizes, each consisting of two cups which telescope together with a friction fit adequate to retain most liquids. Corrections must be made for the heat of combustion of the gelatin and for its sulfur content. The sulfur content is usually about 0.35% by weight and the heat of combustion is approximately 4600 calories per gram. Blank tests must be made to determine the exact corrections. The capsules should be stored in sealed bottles and handled with due regard for their hygroscopic nature. The blank tests should be repeated at frequent intervals since values determined on a weight basis will change if there are variations in the moisture content of the gelatin.

Glass Ampoules. Thin-walled glass bulbs or ampoules are excellent for handling volatile samples, but glass-blowing skill is required to prepare them. They are blown from soft glass tubing with a 1 to 1.5 cm. thin wall bulb and a long capillary tip. The

weight of an empty bulb is usually about 0.2 gram. The bulb is filled by alternately warming and cooling, with the capillary immersed in the liquid sample during the cooling cycle. When it is about half filled, seal the capillary tip in a flame and let the bulb cool to room temperature before weighing. Place the sealed bulb in an open fuel capsule and wrap the fuse wire around the stem near the bulb. The glass will break and release the sample when the fuse is heated.

Volatile Sample Holders. Volatile samples can be handled conveniently in a Parr A185A volatile sample holder using a "Scotch" tape disc to seal the metal cup. The tape disc can be cut with a cork borer or by holding the end of the cup against a strip of tape and trimming with a sharp blade. The weight of the tape disc must be determined separately so that a correction can be applied for its heat of combustion. A blank test using about one-half gram of tape will indicate the correction to be applied. The size and weight of the tape disc will usually be sufficiently uniform so that after a single determination successive discs can be cut from the same roll without weighing and computing the correction for each piece.

After placing the liquid sample in the weighed cup, apply the tape disc and use the top ring to press the tape against the brim. Reweigh to determine the mass of the sample; then place the cup in a 5A2 loop electrode attached to the bomb head. Attach a 10 cm. fuse and bend the wire so that it touches only the center of the tape. Assemble the bomb in the usual manner but just before closing, puncture the tape with a sharp needle. Fill slowly with oxygen and fire the bomb in the usual manner. Some operators prefer to remove the top ring before closing the bomb to avoid the trace of unburned disc which sometimes remains in the joint between the ring and the rim of the cup.

A similar procedure for handling volatile samples is described in ASTM Method D2382. This uses a low form platinum cup with a spun rim (Parr 43A6) which can be covered and sealed with 11/2" "Scotch" tape. A Parr 43AS alloy capsule with a smooth, flat rim may be used in the same manner.

Heavy Oils. Oils and other liquids which are not volatile at room temperature can be weighed directly into open combustion capsules. The loop of the fuse should be positioned just slightly above the surface of the sample. Some operators place one end of a short piece of fine cotton thread over the fuse loop,

with the other end touching the liquid. In any case, the fuse wire itself should not be submerged in the liquid.

Several precautions must be observed when testing heavy oils because of the intense heat which they develop. If the wall of the metal combustion capsule is thin, or if some of the sample happens to have been spread on the thin rim of the capsule, it is possible that the metal may become heated to the point where it will ignite. This condition is serious because of the excessive heat liberated when metal burns in oxygen. Also, the molten metal oxides may damage the interior of the bomb. In extreme cases the bomb electrodes may also ignite and burn with similar results. For these reasons, be sure that any capsule holding a heavy oil is in good condition and not worn thin from prior usage. Also, bend the straight electrode so that it does not project over the cup where it will receive the full flame from the sample. It is always desirable to tilt the capsule slightly in the loop holder so as to direct the flame away from both electrodes. Some operators prefer to use a 10 to 13 ml. platinum crucible for holding heavy oil samples because the added depth in a crucible promotes slower combustion and a milder flame.

Explosives and High Energy Fuels. Special precautions must be observed when testing materials which release large volumes of gas upon ignition, or which detonate with explosive force. Although most slow-burning gun powders and rocket propellants can be tested in the conventional 1108 bomb, the user must understand that this bomb is not designed to withstand the shock pressures produced by primers and high explosives. It is much safer to test these materials in a Parr 1104 high pressure bomb which can be substituted for the 1108 bomb in the 1341 calorimeter.

OPERATING SUGGESTIONS

Poor Combustions. The difference in combustion characteristics of the wide variety of materials which may be burned in an oxygen bomb make it difficult to give specific directions which will assure complete combustions for all samples. However, two fundamental conditions may be stated. First, some part of the sample must be heated to its ignition temperature to start the combustion and, in burning, it must liberate sufficient heat to support its own combustion regardless of the chilling effect of the adjacent metal parts. Second, the combustion must produce sufficient turbulence within the bomb to

bring oxygen into the fuel cup for burning the last traces of the sample.

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

1. Excessively rapid admission of 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 which will permit unburned particles to be ejected during a violent combustion.
3. The use of a sample containing coarse particles which will not burn readily. Coal particles which are too large to pass a 60-mesh screen may not burn completely.
4. The use of a sample pellet which has been made too hard or too soft. Either condition sometimes causes spilling and the ejection of unburned fragments.
5. The use of an ignition current too Low to ignite the charge, or too high, causing the fuse to break before combustion is 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 or by having the wire slightly above the sample.
7. The use of insufficient oxygen to burn the charge, or conversely, the use of a very high initial gas pressure which may retard the development of sufficient gas turbulence within the bomb.

Operators sometimes disagree as to the most desirable oxygen charging pressure. As a rule, it is best to use the lowest gas pressure that will give complete combustion. Lower pressures permit higher gas temperatures and greater turbulence, both of which help to secure better combustion. The range of charging pressures for Parr oxygen bombs is between 20 and 35 atmospheres. Most methods permit a variation in the charging pressure but specify that the same pressure must be used in all tests, including standardization.

DETERMINATION OF SULFUR

The ease with which samples may be completely burned in an oxygen bomb without loss of

combustion products has lead to the acceptance of the bomb method as a basic procedure for determining sulfur, halogens and other elements in a broad range of combustible materials. The 1108 bomb furnished with the 1341 calorimeter is used for many of these analytical procedures, particularly for determining sulfur. Since the combustion operations for a sulfur determination and for a calorific test are the same, both tests can be performed with the same sample by simply recovering the bomb washings from the calorific test and proceeding with the sulfur determination. The gravimetric procedure described below is recommended as a reliable procedure for this determination, but any other standard gravimetric, volumetric or nephelometric method for sulfur may be used.

Sulfur in Combustible Solids. Collect the bomb washings following the combustion of a sample weighing not more than 1.0 gram. If this sample has not been used for a calorific test, let the bomb stand in water bath for at least 10 minutes after firing; then remove the bomb from the water and release the residual gases slowly and at an even rate so that the pressure is reduced to atmospheric in not less than one minute. Open the bomb and wash all parts of its interior, including the combustion capsule, valve passages and electrodes, with a fine jet of distilled water containing 1 ml of a saturated solution of methyl orange indicator per liter. Wash until no acid reaction is observed, collecting the washings in a beaker. If necessary, use a rubber policeman to transfer any precipitate from the bomb or capsule to the beaker.

If a calorimetric test has been made, titrate the washings with standard sodium carbonate solution to determine the acid correction.

After neutralizing the solution, add 1 ml of ammonium hydroxide, heat the solution to boiling, and filter through a rapid qualitative paper. Wash the residue and filter paper with hot distilled water and add sufficient water to bring the total volume of solution to approximately 250 ml. Neutralize with concentrated hydrochloric acid and add 2 ml in excess. Add 10 ml of saturated bromine water and evaporate to approximately 200 ml on a hot plate or other source of heat. Adjust to a slow boil and stir constantly while adding 10 ml of a 10% barium chloride solution from a pipette. Continue stirring for two minutes, cover with a fluted watch glass and keep just below boiling on a steam bath or hot plate until the volume is reduced to 75 ml, then allow the precipitate to settle for another hour while cooling.

Filter through an ashless filter paper and wash with warm water until free from chlorides.

Transfer the paper and precipitate to a weighed crucible, dry at low heat, char the paper without flaming, then raise the temperature to a good red heat (approximately 925° C) and heat to constant weight. If the crucible is placed in a cold electric muffle furnace and the current turned on, drying, charring and ignition will usually occur at the desired rate. After ignition is complete, allow the crucible to cool to room temperature and weigh. Determine the exact weight of the barium sulfate precipitate and calculate the percentage of sulfur in the sample as follows:

$$\text{Sulfur, \%} = \frac{\text{Wt. BaSO}_4 \times 13.734}{\text{Wt. Sample}}$$

Sulfur in Combustible Liquids. For oils or other liquids containing 5% sulfur or less, use a sample weighing from 0.6 to 0.8 gram. If the sample contains 5% sulfur, use a sample weighing from 0.3 to 0.4 gram and add an equal amount of sulfur-free U.S.P. white oil. If the sample is not readily miscible with white oil, some other low sulfur combustible diluent may be used. However, the combined weight of sample and white oil or other combustion aid must not exceed 1.0 gram. If the sample is volatile it must be weighed in a sealed holder.

The procedure for filling the bomb, firing, recovering the washings and determining sulfates is the same for liquid samples as described in the preceding method for solids.

Other Bomb Combustion Methods. The following standard test methods may also be of interest to users of the 1341 calorimeter for developing the full capability of the calorimeter as an analytical instrument in addition to its uses for heat measurements.

ASTM Method D271-70, "Sulfur in Coal and Coke by the Bomb Washing Method".

ASTM Method D129 64, "Sulfur in Petroleum Products by the Bomb Method".

ASTM Method D2361-66, "Chlorine in Coal".

ASTM Method D808 63, "Chlorine in New and Used Petroleum Products (Bomb Method)".

MAINTENANCE INSTRUCTIONS

Bomb Maintenance. Under normal usage a Parr oxygen bomb will give long service if it is handled with reasonable care. However, the user should remember that the bomb is continually subjected to high temperatures and pressures which apply heavy stresses to the sealing mechanism. The mechanical condition of the bomb must therefore be watched carefully and any parts that show signs of weakness or deterioration should be replaced before they fail. Otherwise a serious accident may occur. The parts which probably will require most frequent replacement are the sealing rings. An extra supply of these is furnished with each calorimeter.

Check the 410A sealing ring frequently and replace it if there is any uncertainty as to its age or condition. Also check and replace the sealing rings in the valves and insulated electrode if there is any evidence of gas leakage at these points. DO NOT fire a bomb if gas bubbles indicate a possible leak at any point. Disassemble the bomb and install new seals immediately. Also, do not use extreme force when closing the outlet valve. A moderate but firm turn on the valve knob should be sufficient to stop all gas flow. Excessive pressure will deform the valve seat and possibly close the gas passage. If this happens, unscrew the valve body and replace the 20VB valve seat. Always keep the 397A packing nut tightened firmly in order to maintain a tight seal between the 20VB valve seat and the valve body.

Never under any circumstances use oil on valves or fittings which handle compressed oxygen. This precaution applies to all of the oxygen bomb parts as well as to the oxygen filling connections.

The threads on the screw cap may become worn after long and continued use, making it necessary to replace the cap as a safety precaution before this condition goes too far. A new cap made to a standard gage probably will not fit an old bomb cylinder because of the unpredictable extent to which the cylinder threads will have worn. Therefore when ordering a new screw cap it is best to return the bomb cylinder to the factory. The replacement cap can then be fitted individually to the old cylinder, producing an assembly having the strength and safety of a new unit.

Although Parr oxygen bombs are made from alloys which will withstand most corrosive gases, these bombs will not resist chlorine, fluorine or bromine in the presence of moisture. If samples yielding

appreciable amounts of these elements are burned in a Parr bomb, the interior surfaces may become etched or corroded. In such cases the bomb should be emptied and washed as quickly as possible after each combustion.

If the interior of the bomb cylinder should become etched as mentioned above, the resistance of the metal to further attack can be improved by restoring the surface to its original highly polished condition. Bombs needing repolishing or other repair work can be returned to the factory. A periodic overhaul and test at the factory will help to keep any Parr oxygen bomb in first-class condition. When ordering replacement parts, always give part numbers if the correct number is known. It will also be helpful if the order shows the catalog number and the serial number of the bomb or calorimeter for which the parts are intended.

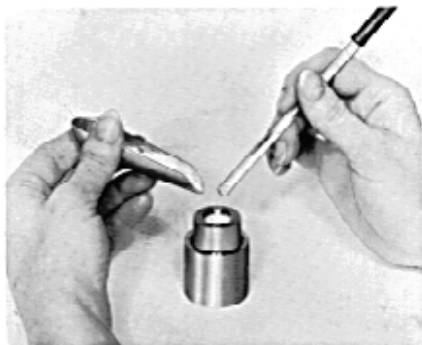
Hydrostatic and Proof tests. In addition to mechanical repairs, the Parr Instrument Co. will retest any Parr bomb and issue a new test certificate covering hydrostatic and proof tests as specified in ASTM E144-64, "Recommended Practice for Safe Use of Oxygen Combustion Bombs." It is recommended that each bomb be retested after each 500 firings, or at more frequent intervals if the bomb has been subjected to extreme usage, or if it shows evidence of damage from corrosion, overheating or from any other cause. Bombs returned to the factory for this purpose should be shipped prepaid to:

Parr Instrument Company
Attn: Repair Department
211- 53rd Street
Moline, Illinois 61265

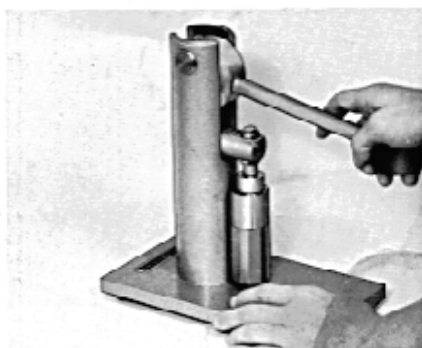
An order or a letter of instructions covering the repair work should be mailed to the same address, as no repairs will be started without specific instruction. Prompt individual attention will be given to all repair orders.

Calorimeter Maintenance. The use of non-rusting materials in Parr calorimeters reduces the maintenance of these instruments to a minimum. The inner calorimeter chamber should be kept clean and dry, and the high polish on the calorimeter bucket should be maintained. This polished chromium finish minimizes any heat transfer to or from the bucket. If the finish becomes dulled, the bucket should be replated or replaced.

PELLET MAKING WITH A PARR PELLET PRESS



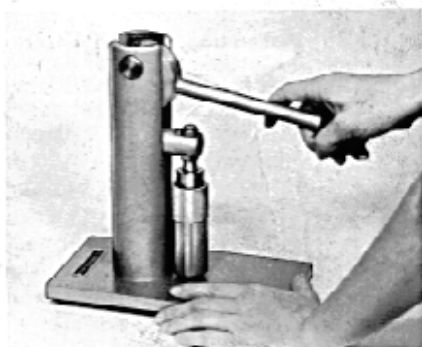
1. Fill the die



2. Compress the charge



3. Reverse the holder



4. Eject the pellet

The steps required to produce pellets or tablets with a Parr Pellet Press have been speeded and simplified by eliminating the die plug and receiving cup used in earlier models. There are only two loose parts in this improved design: a polished stainless steel die and a die holder with working surfaces on both ends. This new die arrangement is an extension of a design previously used only for 1/8" and decimal size pellets. All dies for the Parr press are now made to this design which will produce pellets in four simple steps as described below.

Fill the die. set the die and its holder on the base of the press with the beveled edge of the die cavity facing upward and with the bottom of the die resting on the flat surface in the reversible holder. Pour the charge into the die cavity and tamp with a stirring rod, if necessary.

Compress the charge. Transfer the die and its holder to the press and push the lever down to compress the charge. To obtain maximum compression, the lever should require a firm push as it moves through its full stroke. If a full stroke is not obtained, turn the anvil to lower the die until the full mechanical advantage of the press can be utilized. Conversely, if the lever moves through its full stroke without encountering sufficient resistance, raise the die until firm compression can be applied.

Reverse the die holder. Raise the lever and slide the die and its holder out of the press. Reverse the holder to bring the deep cavity under the die and return the parts to their original position. The clearance under the punch will be limited when making thin pellets. In such cases it will be more convenient to grasp the die with one hand and slide it upward on the punch, holding it in that position while reversing the die holder with the other hand.

Eject the pellet. Bring the lever down gently to eject the pellet into the cavity in the holder. If a thick pellet not ejected by this stroke, turn the anvil to raise the die. The pellet will then drop out freely. Remove the pellet with tweezers or forceps; reverse the holder and repeat the cycle if additional pellets are required.