Operating Instruction Manual

534M

6400

Calorimeter

TABLE OF CONTENTS

PREFACE

Scope
This manual contains instructions for installing and operating the Parr 6400 Calorimeter. For ease of use, the manual is divided into 13 chapters.

Installation
Quick Start
Operation
Menu Descriptions
Calculations
Reports
Memory Management
Maintenance
Troubleshooting
Technical Service
Parts Lists
Drawings
Tables

Subsections of these chapters are identified in the Table of Contents.

To assure successful installation and operation, the user must study all instructions carefully before starting to use the calorimeter to obtain an understanding of the capabilities of the equipment and the safety precautions to be observed in the operation.

Additional instructions concerning the installation and operation of various component parts and peripheral items used with the 6400 calorimeter should be made a part of these instructions. Additional instructions for the optional printer are found in the respective printer package and should be made a part of this book.

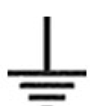
No.	Description
201M	Limited Warranty
207M	Analytical Methods for Oxygen Bombs
230M	Safety in the Operation of Laboratory and Pressure Vessels
483M	Introduction to Bomb Calorimetry

Note:
The unit of heat used in this manual is the International Table (IT) calorie, which is equal to 4.1868 absolute joules.

Purpose

Heats of combustion, as determined in an oxygen bomb calorimeter such as the 6400 Isoperibol Calorimeter, are measured by a substitution procedure in which the heat obtained from the sample is compared with the heat obtained from a standardizing material. In this test, a representative sample is burned in a high-pressure oxygen atmosphere within a metal pressure vessel or “bomb”. The energy released by the combustion is absorbed within the calorimeter and the resulting temperature change is recorded.

Explanation of Symbols

I	On position
O	Off position
~	Alternating Current (AC)
	This **CAUTION** symbol may be present on the Product Instrumentation and literature. If present on the product, the user must consult the appropriate part of the accompanying product literature for more information.
	**ATTENTION**, Electrostatic Discharge (ESD) hazards. Observe precautions for handling electrostatic sensitive devices.
	**Protective Earth (PE) terminal**. Provided for connection of the protective earth (green or green/yellow) supply system conductor.
	**Chassis Ground**. Identifies a connection to the chassis or frame of the equipment shall be bonded to Protective Earth at the source of supply in accordance with national and local electrical code requirements.
	**Earth Ground**. Functional earth connection. NOTE: This connection shall be bonded to Protective earth at the source of supply in accordance with national and local electrical code requirements.

Safety Information

To avoid electrical shock, always:

1. Use a properly grounded electrical outlet of correct voltage and current handling capability.
2. Ensure that the equipment is connected to electrical service according to local national electrical codes. Failure to properly connect may create a fire or shock hazard.
3. For continued protection against possible hazard, replace fuses with same type and rating of fuse.
4. Disconnect from the power supply before maintenance or servicing.

To avoid personal injury:

1. Do not use in the presence of flammable or combustible materials; fire or explosion may result. This device contains components which may ignite such material.
2. Refer servicing to qualified personnel.

Intended Usage

If the instrument is used in a manner not specified by Parr Instrument Company, the protection provided by the equipment may be impaired.

General Specifications

Electrical ratings

115VAC, 5.0 Amps. 50/60 Hz
230VAC, 3.0 Amps, 50/60 Hz

Before connecting the calorimeter to an electrical outlet, the user must be certain that the electrical outlet has an earth ground connection and that the line, load and other characteristics of the installation do not exceed the following limits:

Voltage: Fluctuations in the line voltage should not exceed 10% of the rated nominal voltage shown on the data plate.

Frequency: Calorimeters can be operated from either a 50 or 60 Hertz power supply without affecting their operation or calibration.

Current: The total current drawn should not exceed the rating shown on the data plate on the calorimeter by more than 10 percent.

Environmental Conditions

Operating:	15ºC to 30ºC; maximum relative humidity of 80% non-condensing. Installation Category II (overvoltage) in accordance with IEC 664. Pollution degree 2 in accordance with IEC 664. Altitude Limit: 2,000 meters.
Storage:	-25ºC and 65ºC; 10% to 85% relative humidity.

INSTALLATION

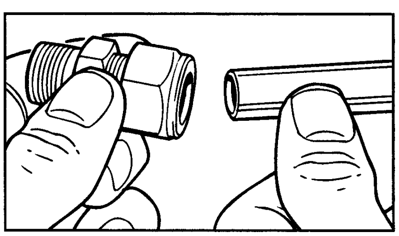
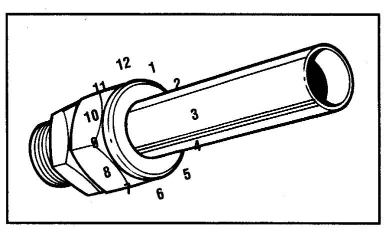
Note:
Some of the following manual sections contain information in the form of warnings, cautions and notes that require special attention. Read and follow these instructions carefully to avoid personal injury and damage to the instrument. Only personnel qualified to do so, should conduct the installation tasks described in this portion of the manual.

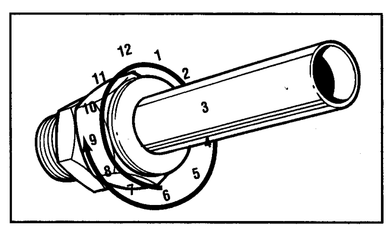
Each Parr 6400 Calorimeter was completely assembled and thoroughly tested prior to shipment. Unpack the calorimeter and carefully check the individual parts against the packing list. If shipping damage is discovered, save the packing cartons and report it immediately to the delivering carrier.

This apparatus is to be used indoors. It requires at least 4 square feet of workspace on a sturdy bench or table in a well-ventilated area with convenient access to an electric outlet, running water and a drain. The supply voltage must be within ± 10% of marked nominal voltage on the apparatus. The supply voltage receptacle must have an earth ground connection.

When Swagelok Tube Fittings are used, the instructions for installation are:

1. Simply insert the tubing into the Swagelok Tube Fitting. Make sure that the tubing rests firmly on the shoulder of the fitting and that the nut is finger-tight.
2. Before tightening the Swagelok nut, scribe the nut at the 6 o'clock position.
3. While holding the fitting body steady with a back-up wrench, tighten the nut 1-1/4 turns. Watch the scribe mark, make one complete revolution and continue to the 9 o'clock position.
4. For 3/16" and 4mm or smaller tube fittings, tighten the Swagelok nut 3/4 turns from finger-tight.



Swagelok tubing connections can be disconnected and retightened many times. The same reliable leak-proof seal can be obtained every time the connection is remade using the simple two-step procedure.

1. Insert the tubing with pre-swaged ferrules into the fitting body until the front ferrule seats.
2. Tighten the nut by hand. Rotate the nut to the original position with a wrench. An increase in resistance will be encountered at the original position. Then

tighten slightly with a wrench. Smaller tube sizes (up to 3/16" or 4mm) take less tightening to reach the original position than larger tube sizes.

The type of tubing and the wall thickness also has an effect on the amount of tightening required. Plastic tubing requires a minimal amount of additional tightening while heavy wall metal tubing may require somewhat more tightening. In general, the nut only needs to be tightened about 1/8 turn beyond finger tight where the ferrule seats in order to obtain a tight seal.

Over tightening the nut causes distortion (flaring) of the lip of the tube fitting where the ferrule seats. This in turn causes the threaded portion of the body to deform. It becomes difficult to tighten the nut by hand during a subsequent re-tightening when the fitting body becomes distorted in this manner.

Consumables, Utilities and Power Requirements

The 6400 Calorimeter requires availability of oxygen, 99.5% purity, with CGA 540 connection, 2500 psig, maximum.

The 6400 Calorimeter requires availability of nitrogen or air, oil and water free, with CGA 580 connection, 2500 psig, maximum.

Approximately 16L of distilled water are required to fill the external pressurized rinse tank.

Approximately 2L of tap water, with a total hardness of 85 ppm or less, are required for filling the internal cooling reservoir.

The power requirements for the sub-assemblies of the 6400 Calorimeter are:

Calorimeter
5A @ 120 VAC
3A @ 230 VAC

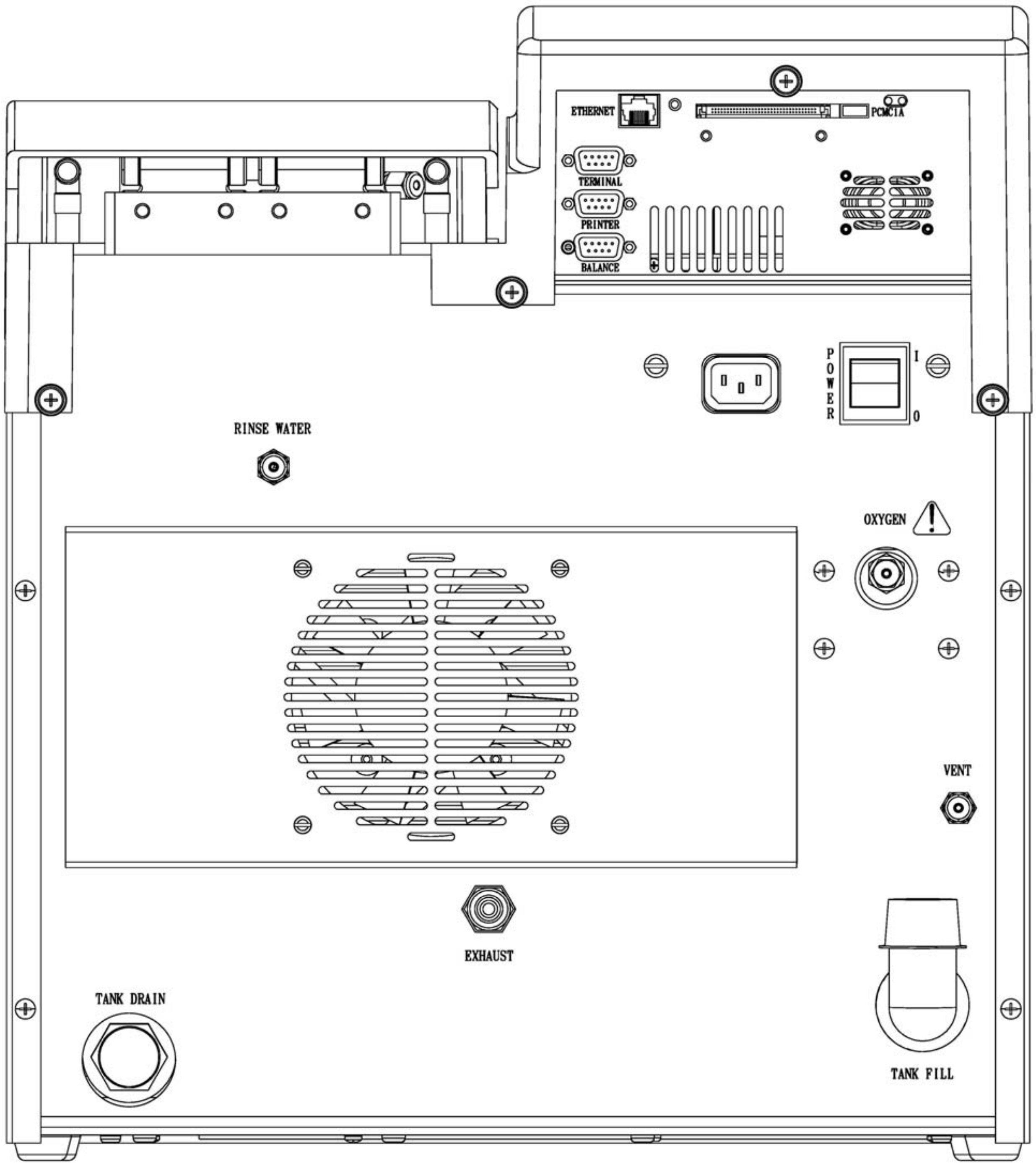
Printer
(100 to 240 VAC, 50/60 Hz) 0.35 A

Electrical Connection

Plug the power line into any grounded outlet providing proper voltage that matches the specification on the nameplate of the calorimeter. Grounding is very important not only as a safety measure, but also to ensure satisfactory controller performance. If there is any question about the reliability of the ground connection through the power cord, run a separate earth ground wire to the controller chassis.

Turn the power switch to the ON position. After a short time, the Parr logo will appear on the LCD display followed by a running description of the instrument boot sequence. When the boot sequence is complete, the Main Menu is displayed.

Figure 1 - 6400 Calorimeter Back Panel - Label



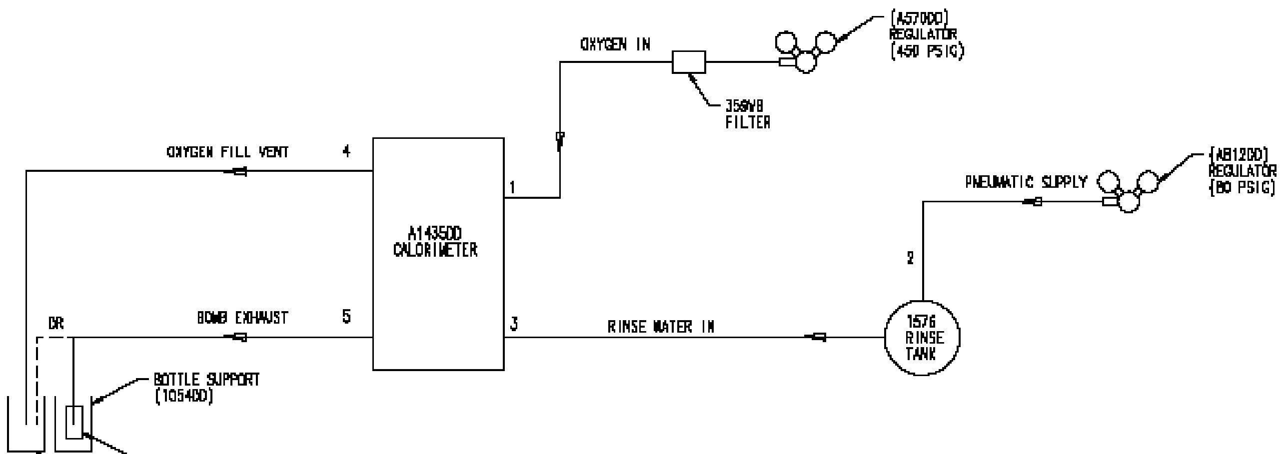
Water Connection

Remove the cap plug on the water filling elbow and fill the internal reservoir tank with water having a total hardness of 85 ppm or less, until the water level is at the bottom of the filling elbow. The calorimeter water tank will initially accept about 2 liters.

Fill the external rinse tank with about 16 liters of distilled water through the large opening at the top of the tank. The cover for this opening is removed by lifting up on the handle, pushing down on the lid, tilting and removing. Replace and close the cover after filling.

The two connections between the calorimeter and the 1576 Rinse Tank should be made with two pieces of 1/8" nylon pressure hose (HX0012TB024).

Figure 2 - 6400 External Plumbing



TUBE #	DESCRIPTION & (PART NUMBER)	MAX LENGTH
1	1/8" OD NYLON (HX0012TB024)	5 FT
2	1/4" OD NYLON (HJ0025TB035)	5 FT
3	1/8" OD NYLON (HX0012TB024)	5 FT
4	1/8" OD NYLON (HX0012TB024)	10 FT
5	3/8" OD BEV-A-LINE (HX0038TB062)	12 FT

Gas Connection

Make the connections to the oxygen supply at this time. Refer to Figure 2. 1/8" O.D. nylon pressure hose (HX0012TB024) is used to connect the oxygen supply. The inlet connection incorporates a flow restrictor just behind the inlet connection. When making the oxygen connection, a back-up wrench should be placed on the restrictor to insure a secure connection and to prevent over tightening the flow restrictor. The delivery pressure for oxygen should be set to 450 psig. To install the regulator, unscrew the protecting cap from the tank and inspect the threads on the tank outlet to be sure they are clean and in good condition. Place the ball end of the regulator in the tank outlet and draw up the union nut tightly, keeping the gages tilted slightly back from an upright position. Open the tank valve and check for leaks. The bomb must never be filled to more than 600 psig (40 atm).

Make the connections to the nitrogen supply at this time. Refer to Figure 2. 1/4" O.D. nylon pressure hose (HJ0025TB035) is used to connect the nitrogen supply. When making the nitrogen connection, a back-up wrench should be placed on the restrictor to insure a secure connection and to prevent over tightening the flow restrictor. The delivery pressure for nitrogen should be set at 80 psig. To install the regulator, unscrew the protecting cap from the tank and inspect the threads on the tank outlet to be sure they are clean and in good condition. Place the ball end of the regulator in the tank outlet and draw up the union nut tightly, keeping the gages tilted slightly back from an upright position. Open the tank valve and check for leaks.

Note:
A hissing sound will occur while the rinse tank is being pressurized. This is normal. Adjust the pneumatic supply regulator to 80 psig as needed.

During extended periods of inactivity, close the tank valve to prevent depleting the tank in the event of a leak. Close the tank valve prior to removing the regulator when changing tanks. Do not use oil or combustible lubricants in connection with any part of the oxygen filling system. Keep all threads, fittings and gaskets clean and in good condition.

Bomb Exhaust Connections

The exhaust and vent connections at the rear of the calorimeter, are made with the dual tube A1006DD assembly. The end of the assembly with the bomb exhaust diffuser should be placed into the 10 liter carboy (231C2). The carboy should be placed at or below the level of the calorimeter to facilitate complete draining of these lines.

Alternatively:
The A1050DD Bomb Rinse Container Assembly is provided as an accessory to the 6400 Calorimeter. See Figure 28. This device allows for complete and systematic recovery of the bomb combustion products. These combustion products include the initial line exhaust after the fill cycle and the portion expelled during the bomb rinse cycle. The Bomb Rinse Container Assembly is connected to the rear of the calorimeter, in place of the portion of the waste tube assembly that is connected to the bomb exhaust fitting. Combustion products are discharged from the bomb in two steps. The first step occurs during the initial rapid release of the residual bomb gases. The 1053DD bottle has sufficient strength and volume to deal effectively with this sudden pressure release. Gas is expelled from the four holes on the perimeter of the 1052DD bottle cap, leaving any discharged liquid in the bottle. As an additional safety measure, the bottle is supported in a 1054DD acrylic cylinder which serves to keep the bottle upright and contained in the unlikely event the bottle ruptures. At the end of the bomb exhaust step the aqueous combustion products reside in the bomb, associated tubing as well as the 1053DD bottle. The bomb rinse step flushes these combustion products from the bomb and the tubing into the 1053DD bottle. The bottle can then be unscrewed from the assembly and capped, until the sample is to be analyzed. Some users find it useful to add the contents of the rinsed combustion capsule to the washings collected in the bottle. Three 1053DD bottles are provided with the assembly. Additional bottles may be ordered separately from Parr.

Communication Connections

There are three RS-232 serial ports at the rear of the calorimeter. These ports are designated Terminal, Printer and Balance. The pin-out of these three ports are identical and can be found in Table 1.

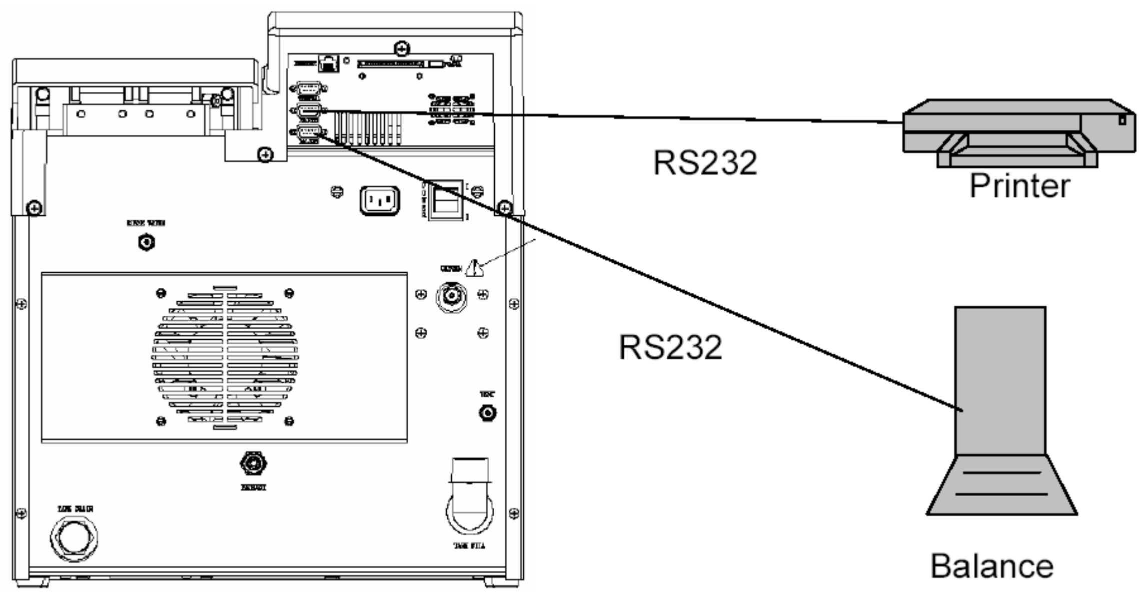
Table 1 - 6400 Calorimeter Serial Ports Pin-Out

9 pin D Connector Pin #	Description	Direction (6400 – External Device)
2	Received Data	←
3	Transmitted Data	→
4		
5	Signal Ground	← →
6		
7	Ready to Send (RTS)	→
8	Clear to Send (CTS)	←

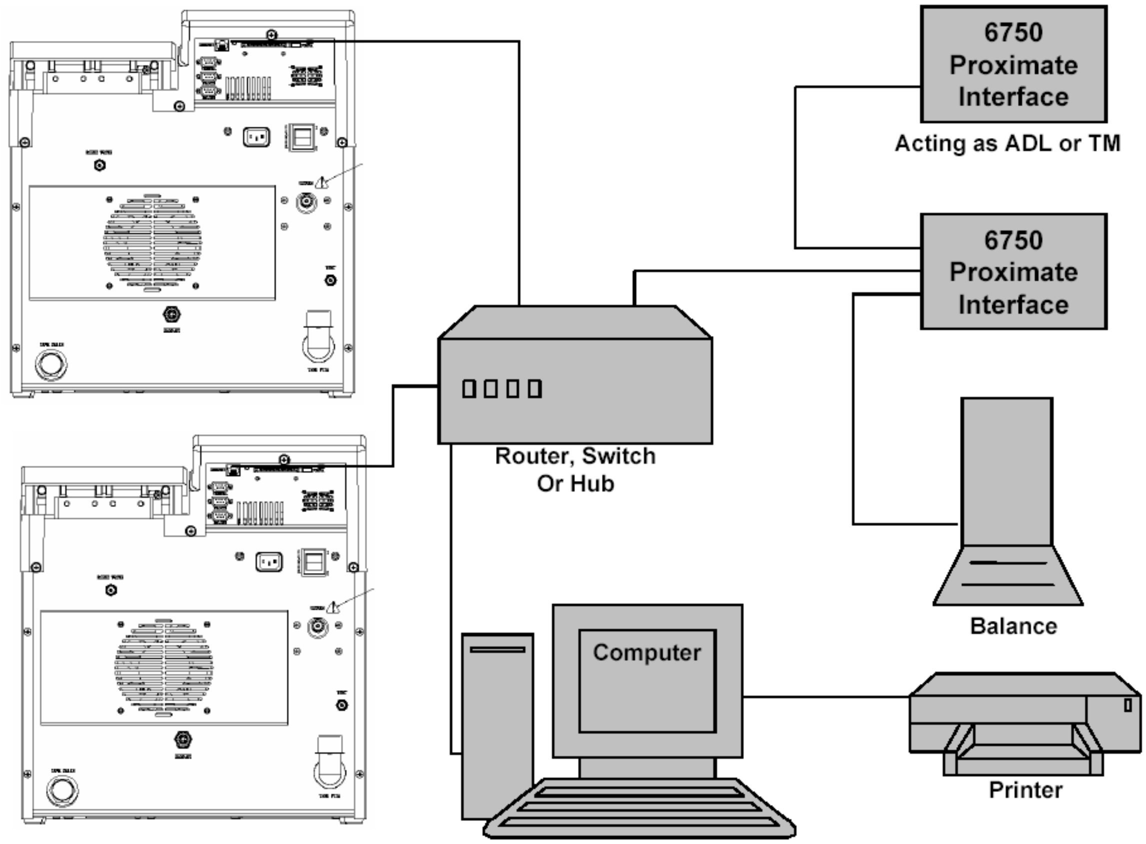
The RS232 balance port is a female port whereas the RS232 printer port is a male port. The 6400 Calorimeter is also equipped with an RJ45 Ethernet port for connection to a computer. Before making any of these connections, the data transmission rate of the Calorimeter and the printer, balance or computer must be matched. Generally the baud rates on either device can be changed to achieve this match.

The 6400 will also allow the user to specify the IP addresses of one or more Balance Interface devices on the network by selecting the Network Data Device menu in the Communications Controls menu. Balance Interface devices are polled from device 1 to 15 for sample and / or spike weights when the weight entry mode is set to Network.

Figure 3 - 6400 Calorimeter Peripherals



Multiple Alternate Configurations



Printer Connections

The printer port settings are on the Communication Controls Menu: Printer Port Communications Menu. The default parameters for the 6400 are set up for use with the Parr 1757 Printer. Table 1 identifies and describes the pin-out for the RS-232 port.

Balance Connections

The 6400 Calorimeter supports input from the multiple balance types. Additionally, a generic input driver is provided for communications with balances that do not conform to the eight supported protocols. A new feature supported by all balance input drivers is the ability to change the expected number of characters in the data field. The number of data characters indicated for each of the drivers, below, are default values. This feature virtually eliminates the need for balance input drivers to be re-written in the event the balance manufacturer elects to alter the output string of a balance when new models are introduced.

The format of an unknown balance can be determined by logging the balance output to the printer attached to the calorimeter. Those protocols which send a command string to the balance will do so while logging is active. In order for the logging to produce meaningful results, the cable connecting the balance to the balance input port of the calorimeter must be correctly wired or configured. In addition, the specifics of the data frame, such as the baud rate, # of data bits, parity, # of stop bits and handshaking (if used) must be the same for both the balance and the calorimeter.

Mettler 011/012 Interface
The ID field must contain “S_” to indicate a stable mass. The data field contains the current mass, right justified, with a decimal point. The balance should be configured to send continuously.

Field	Length
ID	2
space	1
data	9
space	1
g	1
CR	1
LF	1

Sartorious Interface
The polarity field must contain either a “+” or a space. Leading zeros in the data field are blanked, except for the one to the left of the decimal point. The stability field must contain “g_” for the calorimeter to accept a mass. The balance should be configured to transmit data upon receipt of the following command string:
[ESC] P [CR] [LF]

Field	Length
polarity	1
space	1
data	8
space	1
stability	2
CR	1
LF	1

Note:
The automatic data output option should not be used.

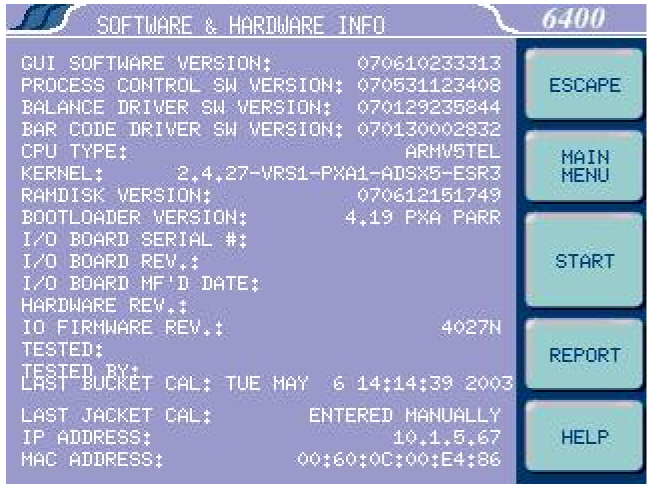
Generic Interface
The data field should consist of 9 numeric characters (0 through 9, +, - and space) terminated with a carriage return (CR). Leading zeros may be blanked as spaces and are counted. Non-numeric characters are ignored and will reset the input buffer if the data field has not been filled. Any characters received after filling the data field and before the carriage return are ignored.

Field	Length
data	8
CR	1

Bar Code Port

The use of barcodes in the laboratory has become a highly accurate, rapid and inexpensive way to identify samples. When purchasing this feature, the user must supply Parr with the MAC address of the calorimeter (found in the Software & Hardware Info menu screen). This allows Parr to activate the feature key. In order to enable the calorimeter to use the bar code feature, the feature key needs to be entered into the instrument. Select the “Program Information and Control” key from the Main Menu. Next, select “Feature Key” and enter the feature key purchased from Parr Instrument Company into the instrument by using the touchpad. Pressing the key labeled “ABC” allows the user to switch from upper case letters, to lower case letters, to numerals, and finally to symbols. A CD containing all the necessary documentation and setup information for using both the scanner and the printer is provided at the time of purchase. A PC based program used for printing bar coded labels is also provided on this CD.

Computer Connections

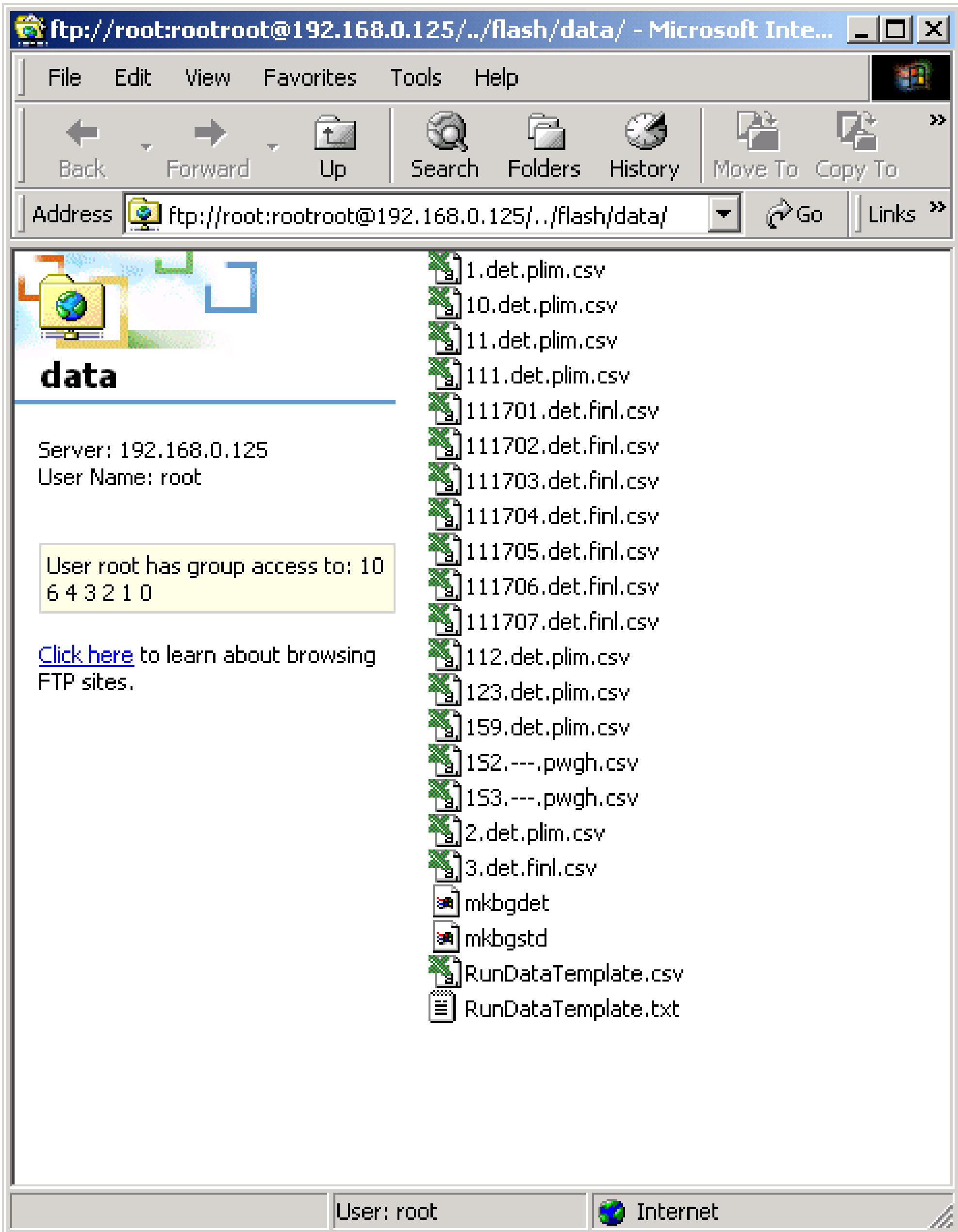


If the 6400 Calorimeter is to be connected to a computer, the Ethernet connection should be used. Test data can be transferred to an Ethernet network connected computer using the FTP File Transfer Protocol. First, you must know the IP address of the network-connected calorimeter. The network DHCP (Dynamic Host Configuration Protocol) server provides this address shortly after the calorimeter is turned on. The address can be seen on the Software & Hardware Info" screen, under Program Info and Control Menu (see the example screenshot). Users who don't have a network infrastructure can create a simple network by connecting a router with DHCP server capability to the calorimeter using an ordinary CAT 5 network cable. The calorimeter should be connected to LAN side of the router. The PC in turn is also connected to the LAN side of the router using a similar CAT 5 cable. A D-Link 614+ router is recommended for this purpose. For this router, operated without a WAN connection, the primary DNS address of the router (WAN setup) must be set to the IP address of the router found on the LAN setup page. Other routers behave differently in the absence of a WAN connection. Providing an active upstream connection to the WAN port of most routers generally minimizes the use of any obscure setup configurations. An FTP enabled web browser can be used to access stored test data. The URL is of the following form:

ftp://root:rootroot@192.168.0.125/../flash/data/

In this case, 192.168.0.125 is the IP address of the calorimeter.

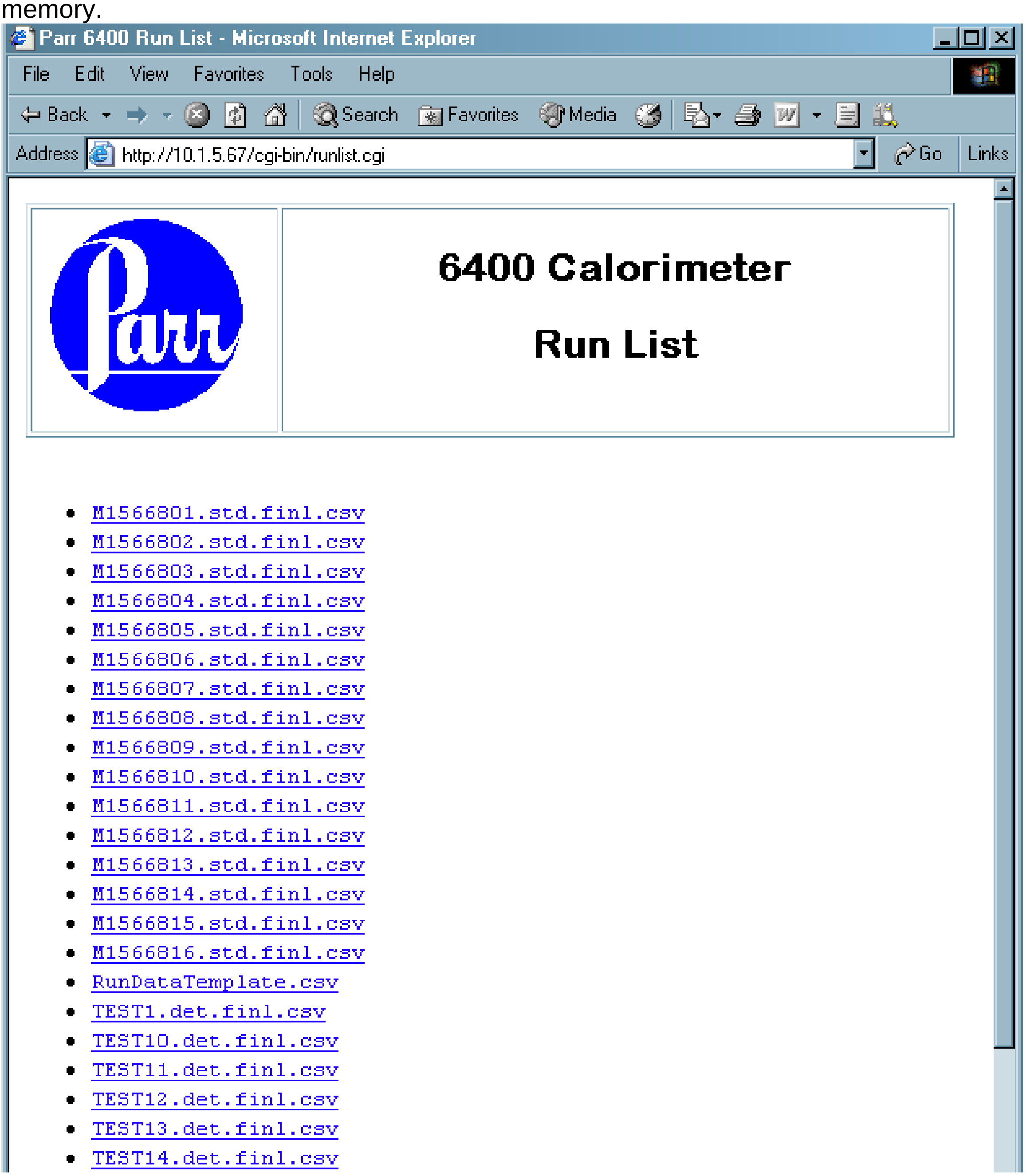
The following screenshot illustrates the contents of the calorimeter data directory as presented by a web browser.



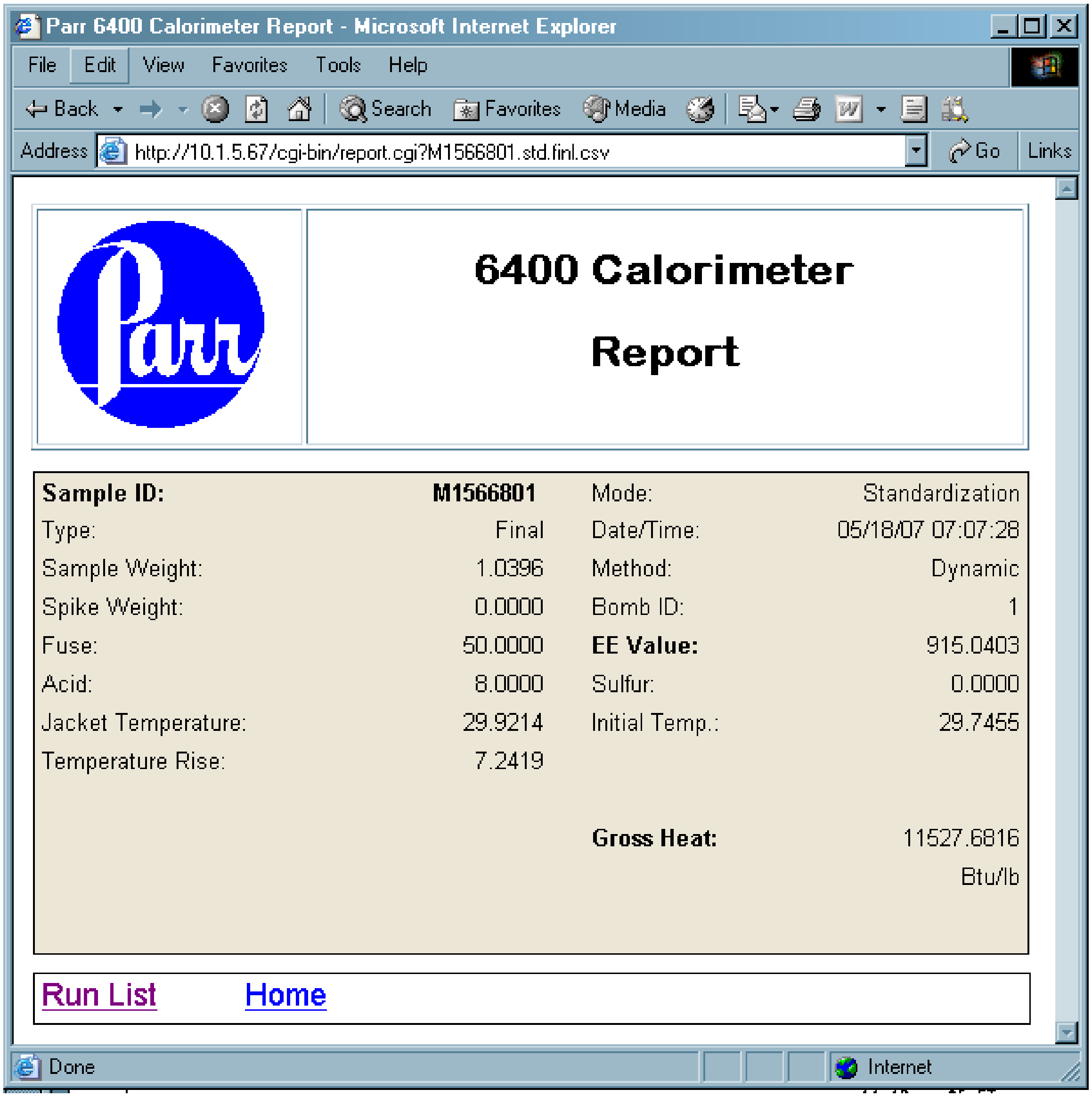
You can drag and drop or copy and paste test data files (with the csv suffix) from the web browser window to any convenient folder or directory on the PC.

The calorimeter offers a web server service. Test reports can be viewed with a web browser using a URL of the following form: http://192.168.0.125, where 192.168.0.125 is the IP address of the calorimeter.

Clicking on the Sample Data tab displays a list of reports currently in the instrument memory.



Clicking on any given report will provide a display similar to the following:



QUICK START

1. Turn on the heater and pump in the Calorimeter Operation menu. Allow at least 20 minutes for the calorimeter to warm up.

2. Initiate a pretest to run the calorimeter through the fill and cool/rinse cycles. This function is used to pre-condition the calorimeter if it has been sitting idle for an extended period of time (greater than 15 minutes).

3. Prepare and weigh the sample to 0.0001g.

4. Gently tap capsules that contain powdered samples to compact the material. (Pellets are easier to handle than loose samples and they burn slower in the bomb, thereby reducing the chances for incomplete combustion).

5. Carefully place the capsule into the capsule holder, attach 10 cm of ignition thread and install the bomb head in the calorimeter.

6. Close the calorimeter cover making sure that the latch is engaged.

7. Select determination or standardization as appropriate on the Calorimeter Operation page, by toggling the operating mode key. Press the Start Key. The calorimeter will now prompt the operator for Bomb ID number, sample ID number, sample weight and spike weight in accordance with the instructions set into the operating controls page.

8. The calorimeter will now take over and conduct the test. During the time it is establishing the initial equilibrium, it will display PREPERIOD on the status bar. Just before it fires the bomb, it will sound a series of short beeps to warn the user to move away from the calorimeter. Once the bomb has been fired, the status bar will display POSTPERIOD. The calorimeter will check to make certain that a temperature rise occurs and will then look for the final equilibrium conditions to be met. If it fails to meet either the initial or final equilibrium conditions, or if it fails to detect a temperature rise within the allotted time, the test will terminate and advise the user of the error.

9. At the conclusion of the test, the calorimeter will signal the user.

10. Open the cover and remove the head. 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.

11. Titrate the bomb washings with a standard sodium carbonate solution using methyl orange, red or purple 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 the water and diluting to one liter. NaOH or KOH solutions of the same normality may be used.

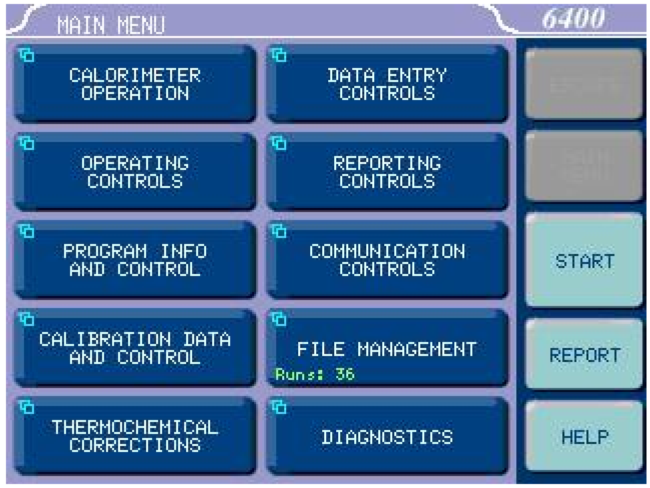
12. Analyze the bomb washings to determine the sulfur content of the sample if it exceeds 0.1%. Methods for determining sulfur are discussed in Analytical Methods for Oxygen Bombs, No. 207M.

OPERATION

Menu System

All configurations and operations are handled by a menu-driven system operated from the bright touch screen display. The settings and controls are organized into ten main sections as displayed on the MAIN MENU.

> ***Note:***
> *Keys with a "double box" in the upper left hand corner lead to sub-menus.*



Menu Keys

The controls that change the data field information in the menus will be one of the following:

1. **Toggles.** These data fields contain ON/OFF or YES/NO choices. Simply touching the key on the screen toggles the choice to the other option. The current setting is displayed in the lower right corner of the key.
2. **Option Selection.** These data fields contain a list of options. Touching the key on the screen steps the user through the available choices. The current setting is displayed in the lower right corner of the key.
3. **Value Entry Fields.** These data fields are used to enter data into the Calorimeter. Touching the key on the screen brings up a sub-menu with a key pad or similar screen for entering the required value. Some keys lead to multiple choices. Always clear the current value before entering a new value. Once entered the screen will return to the previous menu and the new value will be displayed in the lower right corner of the key.
4. **Data Displays.** Most of these keys display values that have been calculated by the calorimeter and are informational only. Certain ones can be overridden by the user entering a desired value through a sub-menu. The value is displayed in the lower right corner of the key.

> ***Note:***
> *Some keys will respond with an opportunity for the user to confirm the specified action to minimize accidental disruptions to the program and/or stored data.*

Control Keys

There are five control keys which always appear in the right column of the primary displays. These keys are unavailable when they are gray instead of white.

1. **Escape.** This key is used to go up one level in the menu structure.
2. **Main Menu.** This key is used to return to the main menu touch screen from anywhere in the menu structure.
3. **Start.** This key is used to start a test.
4. **Report.** This key is used to access the test results stored in the calorimeter, to enter thermochemical corrections, and to initiate a report on the display, printer or attached computer.
5. **Help.** This key is used to access help screens related to the menu currently displayed on the touch screen.

Programming

The program in the 6400 Calorimeter can be extensively modified to tailor the unit to a wide variety of operating conditions, reporting units, laboratory techniques, available accessories and communication modes. In addition, the calculations, thermochemical corrections and reporting modes can be modified to conform to a number of standard test methods and procedures. Numerous provisions are included to permit the use of other reagent concentrations, techniques, combustion aids and short cuts appropriate for the user's work.

> ***Note:***
> *Changes to the program are made by use of the menu structure. Any of these items can be individually entered at any time to revise the operating program.*

Default Settings

Units are preprogrammed with default settings. See Table 2 for a listing of the factory default settings. A more in-depth explanation of these parameters is found on the corresponding parameter group help pages. These default settings remain in effect until changed by the user. Should the user ever wish to return to the factory default settings, go to the Program Info and Control Menu, User/Factory Settings, touch Reload Factory Default Settings and YES. Non-volatile memory is provided to retain any and all operator initiated program changes; even if power is interrupted or the unit is turned off. If the unit experiences an intentional or unintentional "Cold Restart", the controller will return to the last known settings.

The default parameters of the 6400 calorimeter can be changed to guarantee that the calorimeter, when cold restarted, will always be in the desired configuration before beginning a series of tests. Users who wish to permanently revise their default settings may do so using the following procedure:

- Establish the operating parameters to be stored as the user default settings.
- Go to the Program Info and Control Menu, User/ Factory Settings, User Setup ID, and enter the desired User Setup ID.
- Select Save User Default Settings

To re-load the user default setting, go to the Program Info and Control Page, User/Factory Settings, Re-load User Default Settings, and YES.

Sample Preparation

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. The initial oxygen pressure is set at 30 atmospheres (450 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. To avoid damage to the bomb and calorimeter, 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.

When starting tests with new or unfamiliar materials, it is always best to use samples of less than 0.7 grams with the possibility of increasing the amount if preliminary tests indicate no abnormal behavior and sample will not exceed the 8000 calorie limit.

Samples containing sulfur should contain no more than 50 mg of sulfur and have a calorific value of at least 9000 BTU/lb.

Samples containing chlorine should be spiked to insure that sample contains no more than 100 mg of chlorine and liberates at least 5000 calories

Particle Size and Moisture Content

Solid samples burn best in an oxygen bomb when reduced to 60 mesh, or smaller, and compressed into a pellet with a *2811 Parr Pellet Press*. Large particles may not burn completely and small particles are easily swept out of the capsule by turbulent gases during rapid combustion.

> ***Note:***
> *Particle size is important because it influences the reaction rate. Compression into a pellet is recommended because the pressure developed during combustion can be reduced as much as 40% when compared to the combustion of the material in the powder form. In addition to giving controlled burn rates, the formation of pellets from sample material keeps the sample in the fuel capsule during combustion.*

Materials, such as coal, burn well in the as-received or air-dry condition, but do not burn completely dry samples. A certain amount of moisture is desirable in order to control the burning rate. Moisture content up to 20% can be tolerated in many cases, but the optimum moisture is best determined by trial combustions. If moisture is to be added to retard the combustion rate, drop the water directly onto the loose sample or onto a pellet after the sample has been weighed. Then let the sample stand to obtain uniform distribution. Low volatile samples with high water content, such as urine or blood, can be burned in an open capsule by absorbing the liquid on filter paper pulp or by adding a combustion aid, such as ethylene glycol.

Sample Types

Because of the difference in combustion characteristics of the many different materials which may be burned in an oxygen bomb, it is difficult to give specific directions which will assure complete combustions for all samples.

The following fundamental conditions should be considered when burning samples:

- 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.
- The combustion must produce sufficient turbulence within the bomb to bring oxygen into the fuel cup for burning the last traces of the sample.
- A loose or powdery condition of the sample which will permit unburned particles to be ejected during a violent combustion.
- The use of a sample which contains coarse particles will not burn readily. Coal particles which are too large to pass a 60 mesh screen may not burn completely.
- The use of a sample pellet which has been made too hard or too soft can cause spalling and the ejection of unburned fragments.
- The bottom of the cup should always be at least one-half inch above the bottom of the bomb or above the liquid level in the bomb to prevent thermal quenching.
- If the moisture, ash and other non combustible material in the sample totals approximately 20% or more of the charge, it may be difficult to obtain complete combustion. This condition can be remedied by adding a small amount of benzoic acid or other combustion aid.

Foodstuffs and Cellulosic Materials

Fibrous and fluffy materials generally require one of three modes for controlling the burn rate. Fibrous materials do not pelletize readily and generally require either moisture content or a combustion aid such as mineral oil to retard the burn rate and avoid development of high pressures. Partial drying may be necessary if the moisture content is too high to obtain ignition, but if the sample is heat sensitive and cannot be dried, a water soluble combustion aid such as ethylene glycol can be added to promote ignition. Material such as Napthalene should not be burned in loose powder form but should be formed into a pellet.

Coarse Samples

In most cases it may be necessary to burn coarse samples without size reduction since grinding or drying may introduce unwanted changes. There is no objection to this if the coarse sample will ignite and burn completely. Whole wheat grains and coarse charcoal chunks are typical of materials which will burn satisfactorily without grinding and without additives or a special procedure.

Corrosive Samples

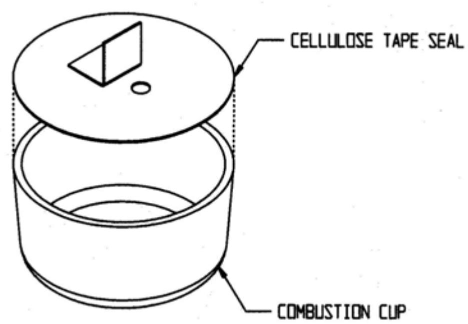
The 1138 bomb is made from alloy 20; a special niobium stabilized stainless steel selected for its resistance to the mixed nitric and sulfuric acids produced during the combustion process. The 1138CL is made from the halogen resistant Hastelloy G30™. Hastelloy 30™ is an alloy rich in cobalt and molybdenum and is able to resist the corrosive effects of free chlorine and halogen acids produced when burning samples with significant chlorine content. While no alloy will completely resist the corrosive atmospheres produced when burning samples containing halogen compounds; users who intend to test these materials are urged to select the 1138CL Bomb. These bombs are 250 mL in volume and are rated to a maximum working pressure of 2000 psi. The bombs are hydrostatically tested to 3000 psi and the sample range is ~1g or 5000 – 8000 calories.

Explosives and High Energy Fuels
Materials which release large volumes of gas which detonate with explosive force or burn with unusually high energy levels, should not be tested in this calorimeter. Rather, they should be tested in a model 6100 or 6200 Calorimeter which can be equipped with an 1104 High Strength Oxygen Bomb designed specifically for these types of samples.

Volatile Sample Holders
Volatile samples are defined as one with an initial boiling point below 180ºC per ASTM D-2. Volatile samples can be handled in a Parr 43AS Alloy Capsule which has a sturdy wall with a flat top rim. These holders can be sealed with a disc of plastic adhesive tape prepared by stretching tape across the top of the cup and trimming the excess with a sharp knife. The seal obtained after pressing this disc firmly against the rim of the cup with a flat blade will be adequate for most volatile samples. The tape used for this purpose should be free of chlorine and as low in sulfur as possible. Borden Mystic Tape, No. M-169-C or 3M Transparent Tape, No. 610, are recommended for this purpose. The 3M Transparent Tape can be ordered through Parr, Part No. 517A.

The weight of the tape disc must be determined separately and a correction applied for any elements in the tape which might interfere with the determination. The approximate Heat of Combustion of the tape is 6300 cal/g. An actual amount should be determined by running a blank test with tape alone using a sample weighing 1.0 gram. The compensation for heat of tape may be done through the spike option; see Spike Controls, Heat of Combustion of Spike.

Figure 4 - Volatile Sample Technique



Note:
Tape should always be stored in a sealed container to minimize changes in its moisture and solvent content.

Use the following procedure when filling and handling any of these tape-sealed sample holders:

1. Weigh the empty cup or capsule; then cover the top with tape, trim with a knife and press the trimmed edge firmly against the metal rim. Also cut and attach a small flag to the disc (see Figure 4).
2. Puncture the tape at a point below the flag, then re-weigh the empty cup with its tape cover.
3. Add the sample with a hypodermic syringe; close the opening with the flag and re-weigh the filled cup.

4. Set the cup in the capsule holder and arrange the auxiliary fuse so that it touches the center of the tape disc.
5. Just before starting the test, prick the disc with a sharp needle to make a small opening which is needed to prevent collapse of the disc when pressure is applied.
6. Fill the bomb with the usual oxygen charging pressure.
7. The calorimeter will fire the bomb and complete the test in the usual manner.

Combustion Aids

Some samples may be difficult to ignite or they may burn so slowly that the particles become chilled below the ignition point before complete combustion is obtained. In such cases white oil or other suitable material of known purity can be mixed with the sample. Ethylene glycol, butyl alcohol or decalin may be used for this purpose.

> ***Note:***
> *It must be remembered, that a combustion aid adds to the total energy released in the bomb and the amount of sample may have to be reduced to compensate for the added charge.*

When benzoic acid is combusted for standardization runs, it should be in the form of a pellet to avoid possible damage to the bomb which might result from rapid combustion of the loose powder.

Combustion Capsules

Non-volatile samples to be tested in Parr oxygen bombs are weighed and burned in shallow capsules measuring approximately 1" diameter and 7/16" deep. These are available in stainless steel, fused silica and platinum alloyed with 3-1/2% rhodium.

Stainless steel capsules (43AS) are furnished with each calorimeter. The stainless steel capsules will acquire a dull gray finish after repeated use in an oxygen bomb due to the formation of a hard, protective oxide film. This dull finish not only protects the capsule, but it also promotes combustion and makes it easier to burn the last traces of the sample. New capsules are heated in a muffle furnace at 500ºC for 24 hours to develop this protective coating uniformly on all surfaces. This treatment should be repeated after a capsule has been polished with an abrasive to remove any ash or other surface deposits. Heating in a muffle is also a good way to destroy any traces of carbon or combustible matter which might remain in the capsule from a previous test. Capsules should be monitored for wear. Do not use the capsule if the wall or base thickness is less than 0.025”.

> ***Note:***
> *After heating, place the capsules in a clean container and handle them only with forceps when they are removed to be weighed on an analytical balance.*

When combusting samples that contain metal particles such as aluminum or magnesium, the non-metallic (fused silica) 43A3 Capsule is required.

When superior corrosion resistance is needed, the Platinum Rhodium 43A5 Capsule is required.

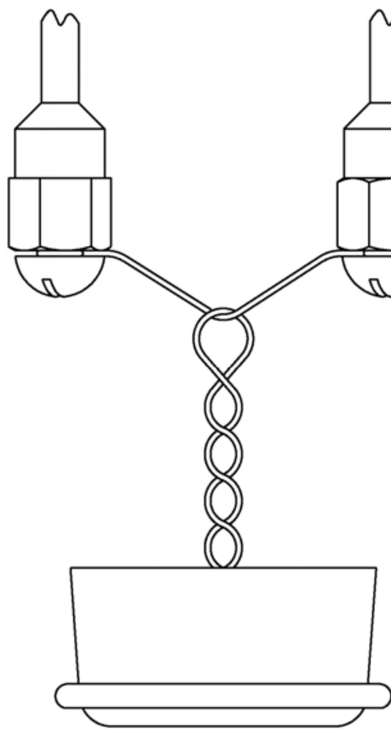
Test Process

Loading the sample

Prepare and weigh the sample to 0.0001g. Gently tap capsules that contain powdered samples to compact the material. (Pellets are easier to handle than loose samples and they burn slower in the bomb, thereby reducing the chances for incomplete combustion).

Carefully place the capsule into the capsule holder. A cotton thread (845DD2) is used as an auxiliary fuse to ignite the sample. Remove any moisture from the heating wire prior to attaching the cotton thread.

Figure 5 - Cotton Thread Assembly



Four inches of thread is recommended for this auxiliary thread which is looped over the heating wire, doubled on itself, twisted to form a single strand and fed into the sample cup to lay on the sample. When contact is made through the heating wire, the thread will ignite, drop into the sample cup and ignite the sample. One spool of thread, part number 845DD, is 563 yards. Part number 845DD2 contains approximately 1000 pieces of thread pre-cut to 4 inches.

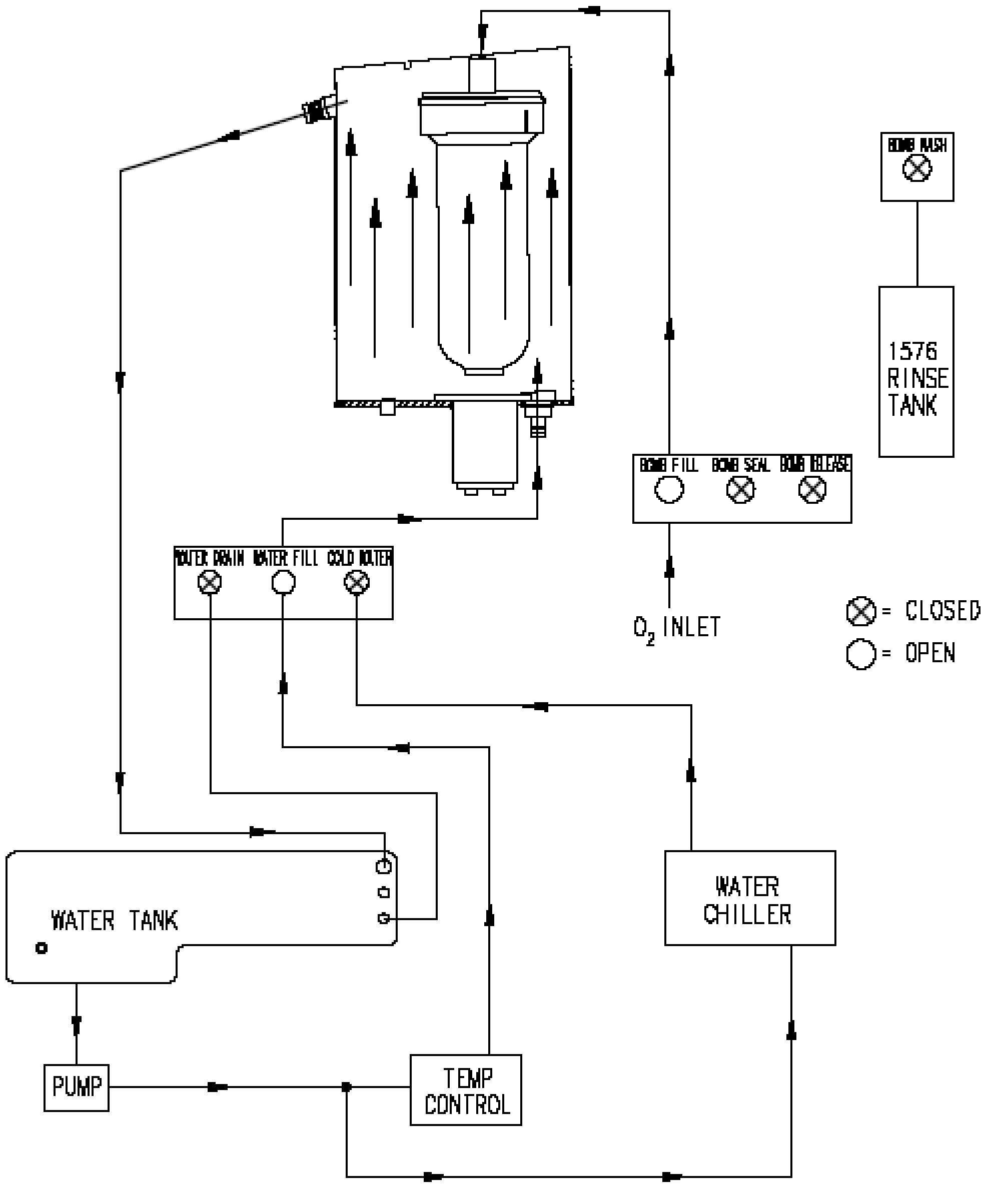
Closing the bomb

Care must be taken not to disturb the sample when moving the bomb head from the support stand to the bomb cylinder in the calorimeter. 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.

Notice that the bomb head grounding lug extends beyond the outside diameter of the bomb head. A slot for this lug is cut into the top of the calorimeter bucket which holds the bomb cylinder. Position this lug approximately 20 degrees to the operators right and slide the head into the cylinder and push it down as far as it will go. Now rotate the bomb head 20 degrees to the left until the lug contacts the left edge of the cut out and is pointed to the front of the calorimeter.

Fill Cycle
Once the calorimeter is started and the cover is closed, the fill sequence begins.

Figure 6 - Bucket Fill Flow Diagram



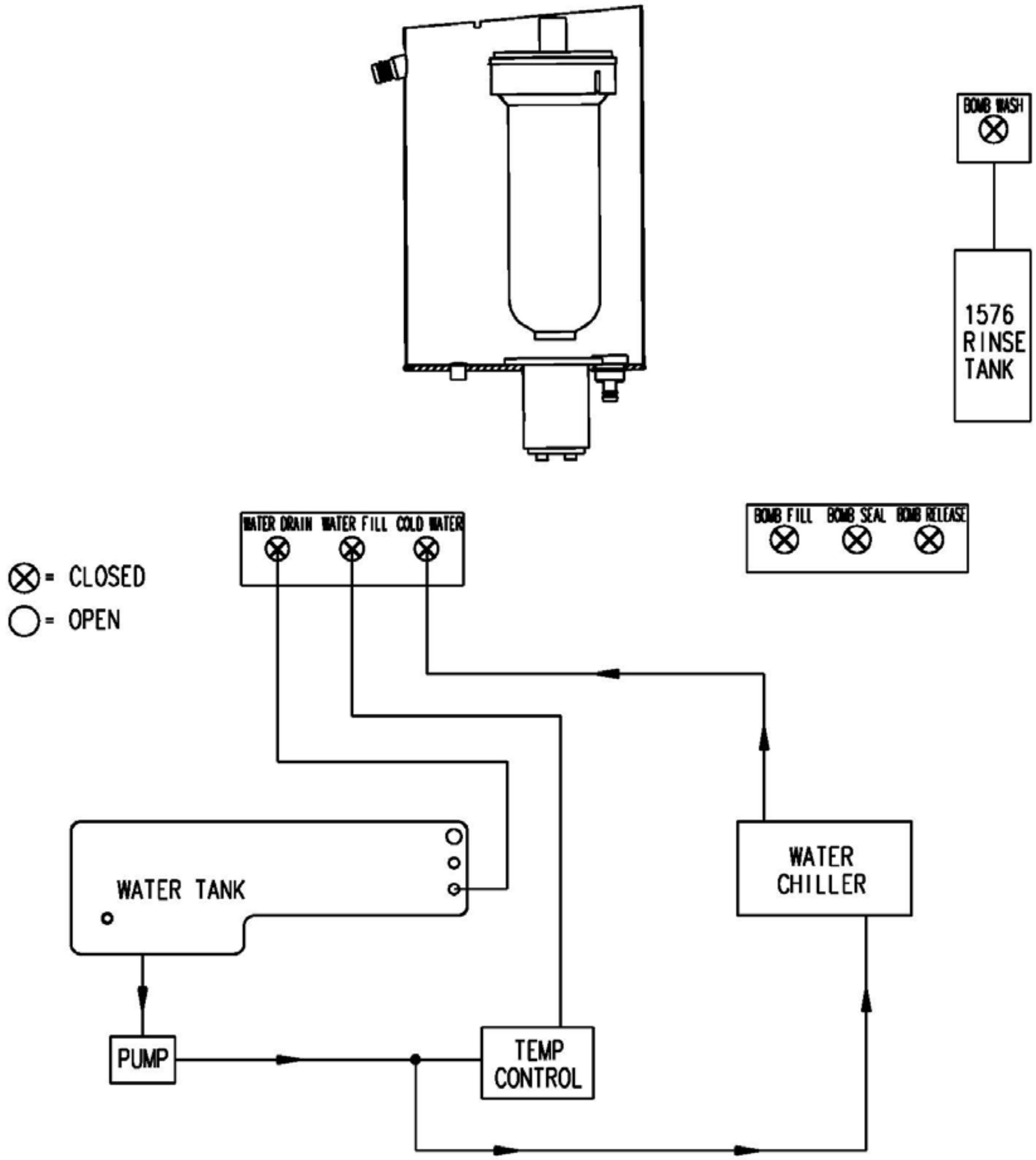
1. The calorimeter checks the bomb ignition circuitry for continuity.

2. The water fill solenoid opens and water is pumped from the internal tank into and through the bucket that surrounds the bomb. Overflow from the bucket is returned to the closed water tank. Because the jacket and bucket are both filled with water from the closed water tank, initial equilibrium will be reached quickly.
3. The oxygen fill solenoid is opened and oxygen is added slowly to the bomb to bring its pressure to approximately 30 atm.

Pre-Period

At the completion of the fill sequence, the pre-period begins.

Figure 7 - Pre-Period / Post-Period Flow Diagram



1. The water fill solenoid valve closes and isolates the water in the bucket from the rest of the system. Water within this bucket is circulated by the stirrer. Water

continues to circulate from the closed water system through the jacket surrounding the bucket.

2. The oxygen filling valve closes and the pressure in the filling line is vented. The automatic check valve at the top of the bomb closes and isolates the bomb from the oxygen filling line.
3. The controller monitors the operating temperature until it confirms that the initial equilibrium has been established.

Bomb Firing

Once the initial equilibrium is confirmed, the controller initiates the firing sequence. There are no changes to the circulation pattern, as shown in Figure 7, from the pre-period through the bomb firing and post-period. A warning of short beeps is sounded indicating the bomb is about to be fired.

Post-Period

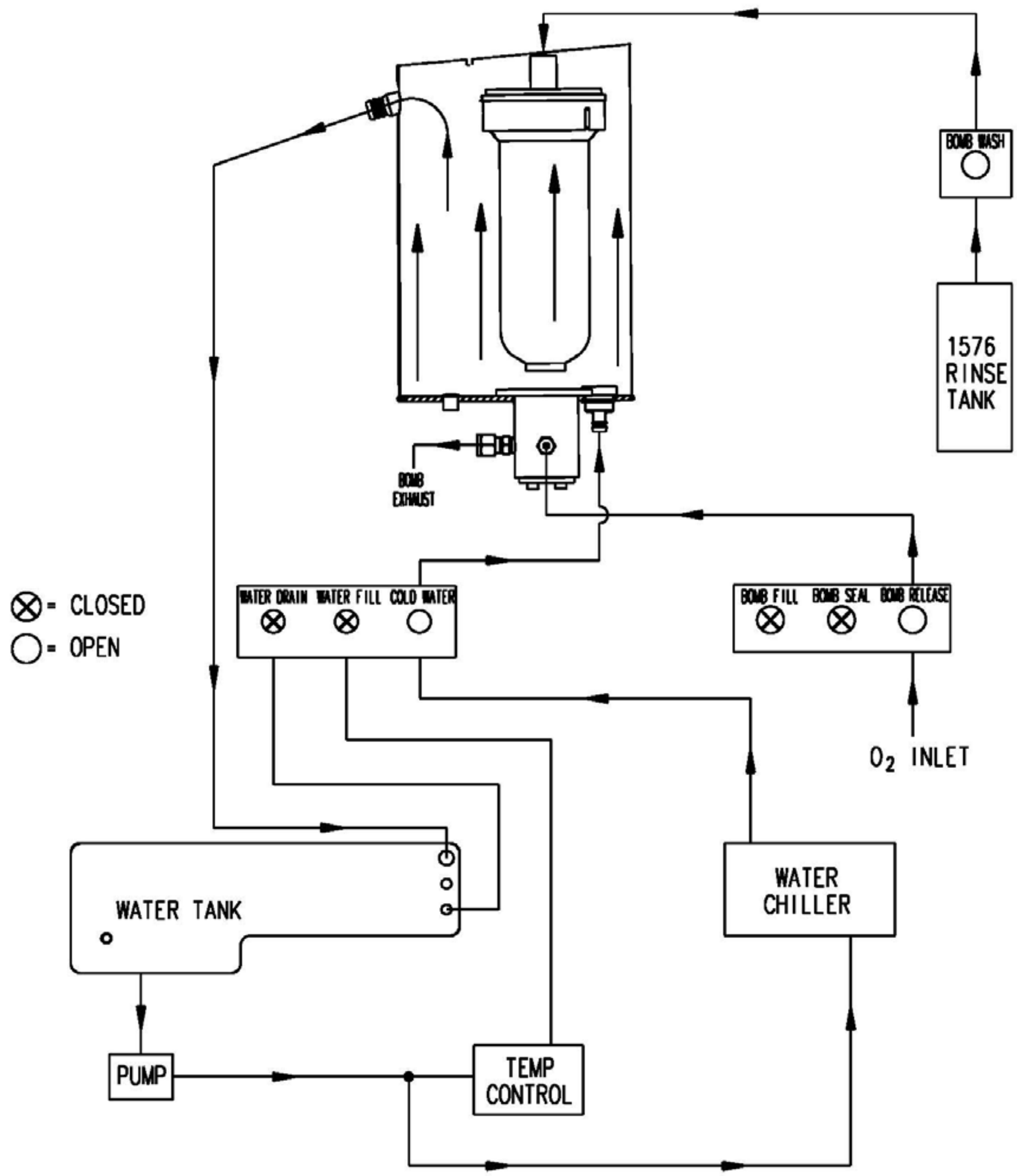
A minimal temperature rise will confirm that the sample has ignited. After this verification, the post-period begins. See Figure 7.

1. The controller monitors the temperature rise and determines the final temperature rise by either the dynamic or equilibrium criteria as established by the user.
2. Once the final temperature rise is determined, it is recorded with the test results.

Cool / Rinse

At the completion of the post-period, the rinse and cool sequence begins.

Figure 8 - Rinse & Cool Flow Diagram



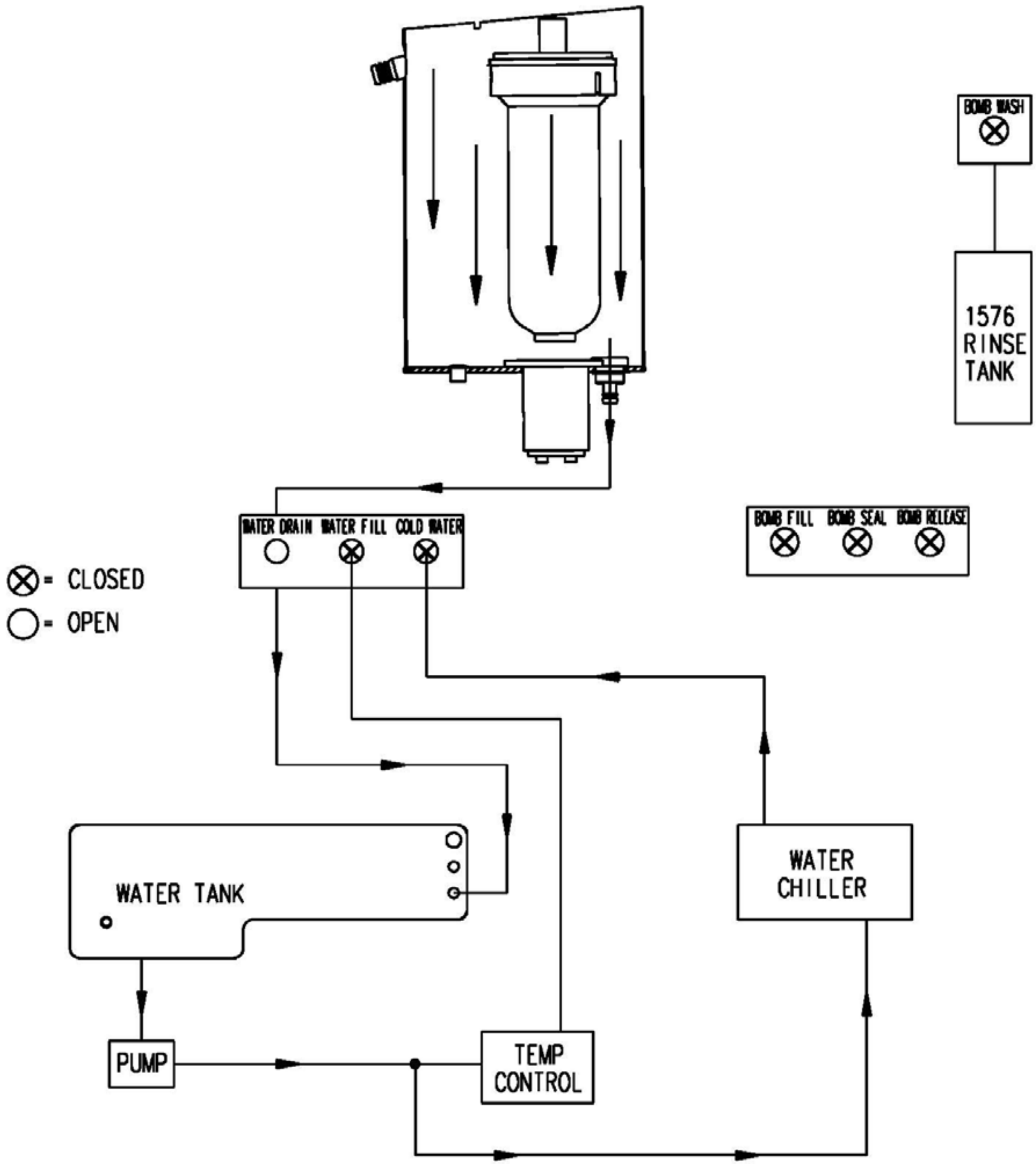
1. Chilled water is circulated through the bucket to cool the bomb to the starting temperature.
2. The release valve in the bottom of the bomb is opened and the residual pressure is released through the bomb exhaust line.
3. Once the excess oxygen is vented, the bomb rinse water from the rinse water tank is admitted through the bomb rinse solenoid valve and the check valve at the top of the bomb. The bomb rinse water is released to the wash bottle.

Note:
Several rinse patterns may be configured by the user to meet various operational and analytical requirements.

4. The bomb is filled one more time with oxygen to help flush the water residue from the interior of the bomb.

Drain
At the completion of the bomb rinse sequence, the drain sequence begins.

Figure 9 - Drain Flow Diagram

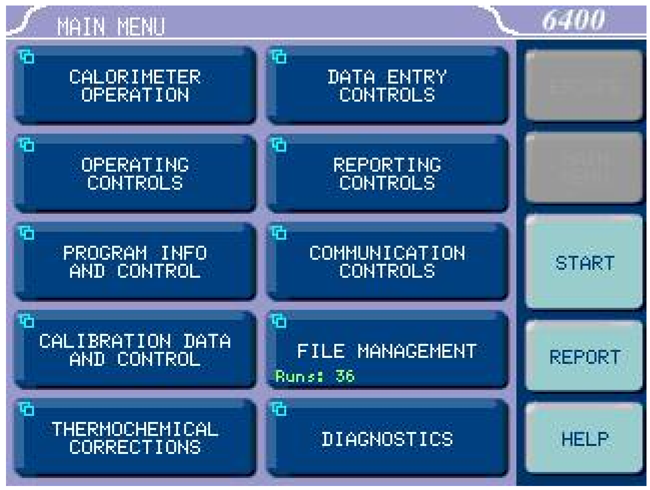


The water in the bucket is drained out of the bucket and routed to the drain connection. Once the bucket is drained, the calorimeter may be opened to remove the bomb head and load the next sample. The test result will then print or be displayed.

MENU DESCRIPTIONS

Note:
Keys which make global changes to the setup of the calorimeter contain a YES or NO response to make certain that the user wishes to proceed. This two step entry is intended to prevent inadvertent global program changes.

Main Menu



Escape Key:
Selecting the Escape key on any menu will return you to the menu one level up.

Main Menu Key:
Selecting the Main Menu key on any menu will return you to the screen pictured on the right of this page.

Start Key:
Press the Start key to begin any Determination or Standardization run.

Report:
Press the Report key to begin the reporting process.

Help:
Press the Help key on any screen to display the explanation text for that screen.

Calorimeter Operation Menu

The Calorimeter will normally be operated from the Calorimeter Operation Menu, although tests can always be started from any menu screen.

Operating Mode:
Sets the operating mode by toggling between Standardization (for instrument calibration) and Determination (for test runs).

Temperature Graph:
Press this key to display a real-time plot of the bucket and / or jacket temperature on the Temperature vs. Time Plot screen.

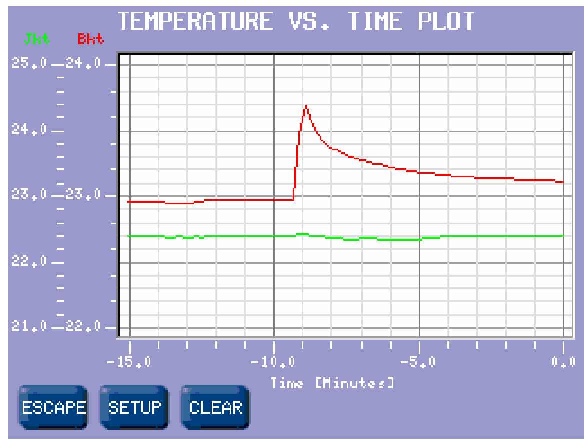
Bomb / EE:
Used to identify the bomb presently installed in the Calorimeter and its EE value.

Start Preweigh:
This key is used to start the sample pre-weigh process. The user is presented with or prompted for a sample ID. Next, the user is asked to key in the associated sample mass or alternatively the mass is retrieved from a connected balance.

Heater and Pump:
The heater and pump must only be turned on after the calorimeter water tank is filled with water.

> ***Note:***
> *The heater and pump must be turned ON to bring the jacket to the correct starting temperature before testing can commence.*

Start Pretest:
This key is used to initiate a pretest cycle. A pretest will cycle the calorimeter through the fill and cool/rinse process. This function is used to pre-condition the calorimeter.



Temperature vs. Time Plot

Press the Setup key to access the Temperature Plot Setup Menu, which has many keys that permit the user to fully customize both the x (time) axis and the scaling of the y axis.

Temperature Plot Setup Menu

Enable Bucket: Toggles ON/OFF.

Bucket Autoscale: Toggles ON/OFF.

Enable Jacket: Toggles ON/OFF.

Jacket Autoscale: Toggles ON/OFF.

Time Mode: Toggles between Autoscale, Window, and Range.

Bucket Plot Symbol: Toggles between:

- No Point
- Small Dot
- Round
- Square
- Up Triangle
- Down Triangle
- Diamond

Press this key to access its numeric dialog box to set a minimum bucket value.

Bucket Min Value: Press this key to access its numeric dialog box to set a minimum bucket value.

Jacket Plot Symbol: (same as Bucket Plot Symbol, above).

Jacket Min Value: Press this key to access its numeric dialog box to set a minimum jacket value.

Time Window: Sets the time scale for the X-axis

Time Units: Toggles between minutes and seconds.

Bucket Plot Color: Toggles between:

- Red
- Green
- Yellow
- Blue
- Magenta
- Cyan
- White
- Black

Bucket Max Value: Press this key to access its numeric dialog box to set a maximum bucket value.

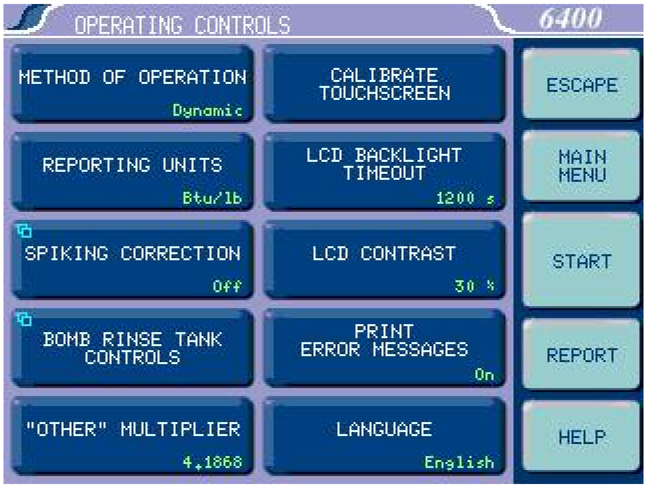
Jacket Plot Color: Toggles between:(same as Bucket Plot Color, above).

Jacket Max Value: Press this key to access its numeric dialog box to set a maximum jacket value.

Time Minimum: Press this key to access its numeric dialog box to set the least amount of time for the display.

Time Maximum: Press this key to access its numeric dialog box to set the greatest amount of time for the display.

Operating Controls Menu



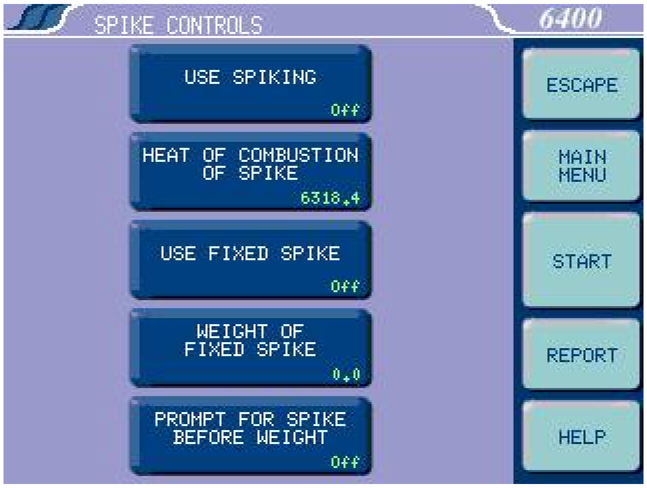
Method of Operation:
Offers an operating mode of either dynamic or equilibrium. In most cases, the dynamic mode with its curve matching capability will save approximately 3-4 minutes per test and will produce the same operating precision as the slower equilibrium mode.

Reporting Units:
Offers a choice of Btu/lb, cal/g, J/kg, or MJ/kg for the reporting units. A user selected set of reporting units may be chosen by selecting "other".

Spiking Correction:

Accesses the Spike Controls sub-menu:

"Spiking" is the addition of material, such as benzoic acid or mineral oil, to samples which are difficult to burn in order to drive the combustion to completion.



- *Use Spiking*. When set to ON, the calorimeter will prompt for the weight of the spike added and will compensate for the heat of combustion in the calculations.
- *Heat of Combustion of Spike*. The heat of combustion of spike is entered on sub-menu keyboard in cal/g.
- *Use Fixed Spike*. When set to ON, a constant amount of spike is to be added to each test.
- *Weight of Fixed Spike.* The weight of the fixed spike is entered on sub-menu keyboard.

Note:

The precision of tests with fixed spikes can be no better than the accuracy of the spike weight.

- *Prompt for Spike before Weight.* When set to ON, the calorimeter will prompt the user for the weight of the spike and the weight of the sample. Normally the calorimeter will prompt the user for the weight of the sample and then the weight of the spike.

Bomb Rinse Tank Controls:

Accesses the Rinse Tank Controls submenu:

Wash water for the bomb is drawn from the Bomb Rinse Tank.

- *Report Rinse Tank Empty.* When turned on the calorimeter will notify the user when it believes the rinse tank is empty based upon capacity of tank and number of tests.
- *Rinse Tank Capacity.* Sets the number of tests available from a container refill. If the rinse timing controls have been changed, then the value must be changed proportionally.

- *Reset Rinse Tank Counter.* Resets the counter when the rinse tank has been refilled. This counter must be reset after the rinse tank is refilled.

- *Rinse Time.* This value establishes the time that the rinse water solenoid is turned ON for each rinse cycle. When the rinse water solenoid is ON, distilled water from the rinse tank is pumped, under pressure, into the bomb cylinder. This rinses the cylinder walls and the bomb head. These rinsings are then pooled and collected at the exhaust port of the calorimeter. The factory default value is 2.5s. This value, along with the # of rinse cycles, determines the total volume of recovered rinse. These default values will yield a total of 50 ml (approx.) of bomb washings.

- *# Rinses Left.* This value provides an estimate of how many rinses are left in the tank. This number is simply a counter not an actual measurement of the volume in the tank.

- *Rinse Flush Time.* This value is used to establish a time between rinse cycles. During this time the rinse solenoid is turned OFF. This off time permits the rinse water to drain out before the next rinse cycle begins. The factory default value is 2s.

- *Clear Time.* This time value is used to establish a post-rinse oxygen filling time for the bomb. This step is used to clear the lines and valves of any residual rinse water prior to the next test. The factory default value is 10s.

- *# of Rinse Cycles.* This value establishes the number of distinct rinse cycles used to rinse the bomb. The factory default value is 3 rinse cycles

"Other" Multiplier:
Press this key to display the Other Multiplier dialog box, where the user can enter a final multiplier to be used when the reporting units are set to "Other".

Calibrate Touchscreen:
This key prompts the user to touch the screen at predefined points in order to facilitate touch screen calibration. It is important that a touch screen stylus, rather than a finger, be used in order to realize an accurate calibration.

LCD Backlight Timeout:
The unit is equipped with an automatic circuit to shut off the backlight when it is not being used. The back light will shut off if there is no keyboard activity for the number of seconds entered. Pressing any key will automatically turn the back lighting ON. A setting of 0 will keep the backlight ON at all times.

LCD Contrast:
This key accesses a sub-menu with a slide control which adjusts the contrast on the LCD display for optimum viewing.

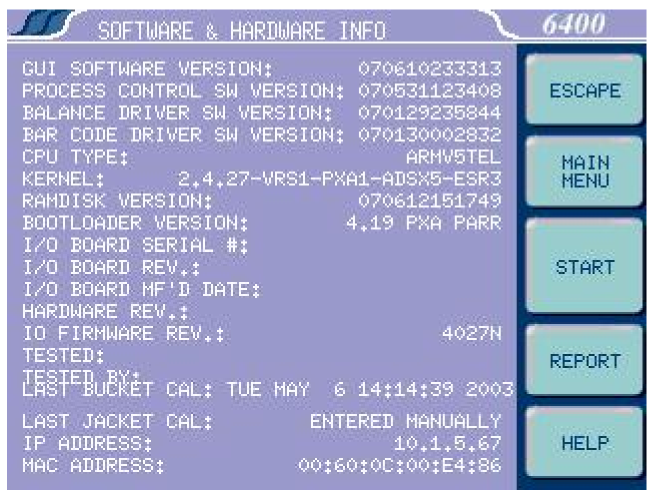
Print Error Messages:
When turned ON, all error messages will be printed on the printer as well as displayed on the screen. When turned OFF, messages will only display on the screen.

Language:
Steps the Calorimeter through the installed operating languages.

Program Information and Control Menu

Date:
Displays current date and accesses sub-menu on which date is set in (YY/MM/DD) format.

Time:
Displays current time and accesses sub-menu on which time is set in (HH:MM) format.



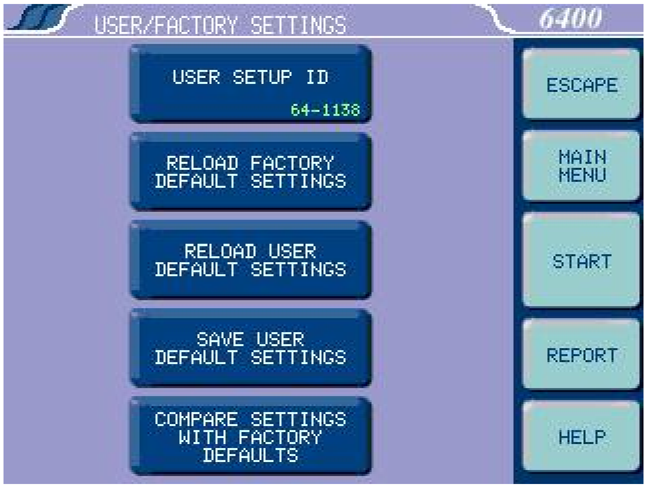
Software and Hardware Info:
This screen displays important information such as the main software version, I/O board information, CPU information, and Controller IP address assigned by the network DHCP server.

Settings Protect:
Provides protection for the program options and settings on the menus. If this is turned ON, the user will be warned that enumeration keys are locked when a key is pressed. Enumeration Keys either toggle a value (ON / OFF) or select from a predefined list. This feature is used primarily to protect the instrument settings from accidental changes if one were to inadvertently touch or bump up against the touchscreen.

User/Factory Settings:
This key leads to a sub-menu that allows the user to save or recall user defined instrument settings. Additionally, factory preinstalled settings supporting different bombs or special operating modes can also be recalled.

- *User Setup ID*
  Used to enter a unique identifier for recalling user settings. Parr offers a unique program within the 6400 identified as "63-FAST". The program will "overlay" the factory settings and shorten the run time by approximately 2 minutes however the user should be aware that a loss of precision will occur.

- *Reload Factory Default Settings:*
  Used to erase all of the settings and restore the factory default settings.



- *Reload User Default Settings:*
  Used to restore the user's setup should the program in the instrument be corrupted for any reason.

- *Save User Default Settings:*
  Used to record the setup to the memory once the user has configured the instrument to their operating requirements.

Feature Key:
This key displays a screen which allows the user to input a code to access special calorimeter features such as the bar code capabilities or remote calorimeter operation.

Bomb Type Select:
This key toggles through the different bomb models available for the calorimeter. When the user chooses a bomb, the instrument must be re-booted to load the correct version of the software. (Note that the calorimeter will not let you exit this function without re-booting the system).

User Function Setup:
This key leads to sub menus that support the configuration of five factory / user definable function keys. The function keys are accessible from the Diagnostics page.

Cold Restart:
This is essentially the same as cycling power on the unit. All valid test data will be retained during this cold restart procedure.

Calibration Data and Controls Menu

Calibration Run Limit:
Displays the maximum number of runs that will be included in determining the EE value of a bomb and bucket combination and accesses the sub-menu on which this limit is set. Most test methods suggest 10 tests. Tests in excess of the most recent ones used are still available but are not used in the calculation of the EE value. For example if 11 standardization tests have been run, the calorimeter will only use the most recent 10. The 11th is still stored in the memory and is available for viewing or printing. Only runs that are at final status will be used in this calculation.

EE Max Std Deviation:
Displays the maximum relative standard deviation in percent that will be permitted for any EE value calculated by the Calorimeter and accesses the sub-menu on which this limit is set. If this value is exceeded, the user will be warned to take corrective action before proceeding with testing. A setting of zero disables this check.

Heat of Combustion of Standard:
Displays the heat of combustion in calories per gram for the material used to standardize the calorimeter and accesses the sub-menu on which this value is set. For benzoic acid, this value is 6318.4 calories per gram.

Bomb Service Interval:
Displays the maximum number of times a bomb may be fired before it is flagged as due for service and accesses the sub-menu on which this limit is set. Parr recommends 500 firings for this service interval. (Parts may need to be replaced on a more frequent basis depending upon the nature of the sample).

Control Chart Parameters:
A control chart is a graphical way to interpret test data. In its simplest form, a selected reference sample is measured periodically and the results are plotted sequentially on a graph. This key will define the charted value; either the heat of combustion of the standard or the Energy Equivalent. It also defines the process sigma.

Use Bomb:
Displays the ID of the bomb currently being used in the Calorimeter and toggles through the four possible bomb numbers.

Bomb 1:

Leads to sub-menu, Bomb 1. Displays standardization information for bomb and bucket combinations. While only one bomb and bucket is installed in the calorimeter at a time, a spare may be used for servicing and for more rapid turn-around. The respective EE values for each bomb can be stored in memory.

> ***Note:***
> *For rapid turn around between tests, the user may wish to use an extra head. Each head should be assigned a bomb ID. On the Data Entry Controls Menu, set the* ***Prompt for Bomb ID*** *to "ON".*

The following four values are displayed for the Bomb # shown in the title on top of the screen.

- Bomb EE Value. Displays the calculated EE value.
- # Runs, EE Val. Displays how many runs have been used to determine the EE value.
- Rel. Std. Dev. Displays the relative standard deviation for the series of tests used to determine the current EE value in percent of the EE value.
- Bomb Fire Count. Displays the current bomb firing count or the number of times the bomb has been fired since it was last serviced. When this count matches the limit set by Bomb Service Interval (on the Calibration Data and Controls screen), the user will be informed that the bomb is ready for service.

Protect EE Value:

Toggles between OFF and ON. When set to OFF, the 6400 automatically updates the EE value as new tests are run. When set to ON, it keeps the EE value protected, whether it has been revised manually via the Manual EE Entry key or calculated by the instrument.

Update Statistics:

If the Protect EE Value is set to OFF, pressing this key will cause the EE Value for this Calorimeter to be updated using all standardization runs currently in memory to the limit established in the Calibration Data and Controls menu. If the Protect EE value is set to ON, this key is not functional.

Manual EE Entry:

This key allows the user to manually enter an EE or calibration factor for a given calorimeter ID or bomb head. If an EE value is manually entered, it is necessary to turn the Protect EE Value ON in order to prevent this value from being overwritten by an automatic update.

Print Standardization Runs:
This key will print all of the tests that have been incorporated into the calculated EE value. This will be helpful in evaluating a series of tests which fail to produce a satisfactory EE value and relative standard deviation.

Reset Bomb Fire Count:
After bomb service, press this key to reset the fire count to zero.

Control Chart Plot:
Each data point represents a Standardization run used in the calculation of the EE Value for the Bomb ID being displayed. The left side of the graph contains the oldest runs and the right represents the most recent. To view the run data used for a particular data point, click on the data point with a stylus and the run data for that point is displayed.

Thermochemical Corrections Menu

The Thermochemical Corrections Menu permits three types of fixed corrections for standardization (instrument calibration) runs, and the same three types for determination (test) runs. Pressing the LEFT side of each key toggles the correction ON or OFF. Press the RIGHT side of each key to access the specific numeric dialog box where that fixed value can be set. Each value entered for these fixed corrections is used in all preliminary reports.

When any fixed correction is set to ON, the specified value will be used in the final reports, and the 6400 will not prompt for actual corrections to be entered. (If all corrections are fixed, only a final report will be generated).

When any fixed correction is set to OFF, during the data entry reporting steps the user will be prompted to enter an appropriate desired value which will be used in the final report.

Standardization Corrections

Fixed Fuse Correction:

Press this key on the LEFT side to toggle ON or OFF the fixed fuse correction for standardization runs. Press it on the RIGHT side to access the Fixed Fuse numeric dialog box on which the value can be set. An appropriate fixed fuse value is 50 calories.

Fixed Acid Correction:

Press this key on the LEFT side to toggle ON or OFF the fixed acid correction for standardization runs. Press it on the RIGHT side to access the Fixed Acid numeric dialog box on which the value can be set. An appropriate fixed acid (nitric acid) value is 8 calories when one-gram benzoic acid pellets are used to calibrate the instrument.

Fixed Sulfur Correction:

Press this key on the LEFT side to toggle ON or OFF the fixed sulfur correction for standardization runs. Press it on the RIGHT side to access the Fixed Sulfur numeric dialog box on which the value can be set. When benzoic acid is used as the calibrant, a fixed sulfur value of 0 should be used.

Determination Corrections

Fixed Fuse Correction:

Press this key on the LEFT side to toggle ON or OFF the fixed fuse correction for determination runs. Press it on the RIGHT side to access the Fixed Fuse numeric dialog box on which the value can be set. An appropriate fixed fuse value is 50 calories.

Fixed Acid Correction:
Press this key on the LEFT side to toggle ON or OFF the fixed acid correction for determination runs. Press it on the RIGHT side to access the Fixed Acid numeric dialog box on which the value can be set.

Fixed Sulfur Correction:
Press this key on the LEFT side to toggle ON or OFF the fixed sulfur correction for determination runs. Press it on the RIGHT side to access the Fixed Sulfur numeric dialog box on which the value can be set.

> ***Note:***
> *When fixed corrections are turned ON, the value in the specified field will be used in both the preliminary and final reports. The calorimeter will not prompt for actual corrections. If all corrections are fixed, only final reports will be generated. If any correction value is entered and the toggle is set to OFF, then the preliminary report will use the displayed fixed value, but the final report will use the value entered when prompted during the reporting process.*

Calculate Net Heat of Combustion:
Toggles ON or OFF the calculations for the net heat of combustion for materials with significant hydrogen content. When set to ON, the 6400 will prompt for entry of the hydrogen content during the reporting data entry step.

Calculation Factors:
Accesses the Calculation Factors sub-menu, which provides for setting a number of options for the way the thermochemical corrections are applied.

Calculation Factors Menu

Acid Value is Nitric Acid Only:
When set to ON, the acid value is nitric acid only. When set to OFF, it represents both nitric and sulfuric acid.

Acid Multiplier:
This multiplier is the normality of the sodium carbonate used during the acid correction titration. The default value of 0.0709 allows for direct entry of the acid correction in calories. If the bomb rinses are titrated in order to determine the acid correction, press this key to display the Acid Multiplier numeric dialog box, where you can change the multiplier to represent the concentration of the base (equivalents/L) or normality used for titration. If this is the case, the acid correction is entered as milliliters of base used to titrate the bomb rinses.

Sulfur Value is Percent:
When set to ON, the sulfur value is being entered as weight percent sulfur. If another system is to be used, this must be turned OFF and the sulfur multiplier set accordingly.

Sulfur Multiplier:
Values entered by the user to be used for the sulfur correction are multiplied by this value to get the product into units of milliequivalents. The default number (0.6238) requires that the sulfur value be entered in weight percent.

Fuse Multiplier:
The fuse corrections represent the number of calories liberated by the burning fuse wire used to ignite the sample. If another measurement is used, the correction factor must be entered here. Press this key to access the Fuse Multiplier numeric dialog box and enter this multiplier value.

Use Offset Correction (ISO):
The thermochemical calculations used for treatment of nitric acid and sulfuric acid corrections in the ISO and B. S. methods require an offset correction to compensate for the back titration that is made. To use these calculations, toggle this to ON and enter the appropriate value as the offset value.

Offset Value:
The value used when Offset Correction is turned ON. Press this key to access the Offset Value numeric dialog box and change its value.

Fixed Hydrogen:
Press the LEFT side to toggle this setting On / Off. Press the RIGHT side to display the Fixed Hydrogen numeric dialog box and change its value.

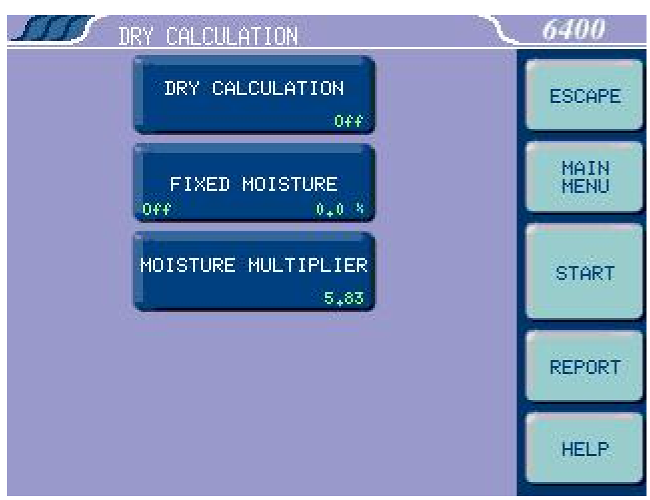
Hydrogen Multiplier:
This value is associated with the net heat of calculation. It is related to the heat of formation of water. Press this key to display the Hydrogen Multiplier numeric dialog box and change its value.

Dry Calculation:
Displays whether set to ON or OFF. Press this key to display the Dry Calculations sub-menu to toggle this setting and update Fixed Moisture and Moisture Multiplier data.

Dry Calculation Menu

Dry Calculation:
Toggles the dry calculation ON or OFF.

Fixed Moisture:
Press the LEFT side to toggle ON or OFF whether to use the entered moisture correction. Press the RIGHT side to access the Fixed Moisture numeric dialog box and set the value. Units are weight %.



Moisture Multiplier:
Press to access the Moisture Multiplier numeric dialog box and set the value. This value is associated with the net heat calculation. It is related to the heat of vaporization of water.

Data Entry Controls Menu

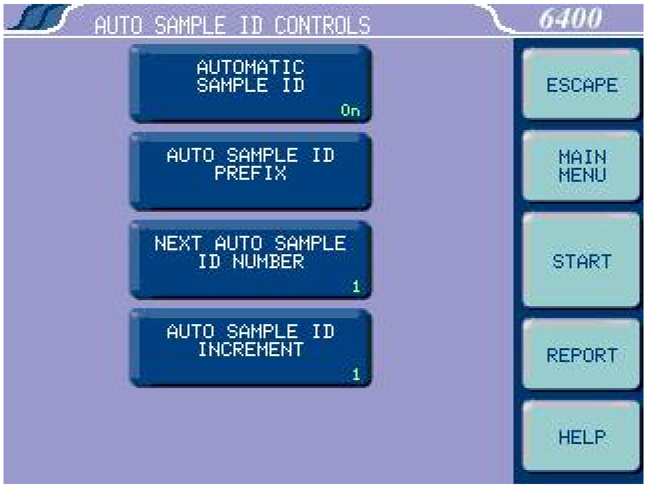
Prompt for Bomb ID:
Toggles ON or OFF. In the ON position the controller will prompt for a Bomb ID (1-4) when a test is started.

Weight Entry Mode:
This key steps through the options for entering sample weights either manually through the touch screen, balance port or through the network.

Acid Entry Mode:
This key steps through the options for entering acid correction value either manually through the touch screen or automatically through the balance port.

Hydrogen Entry Mode:
This key steps through the options for entering hydrogen content for calculating the net heat of combustion either manually through the touch screen or automatically through the balance port.

Auto Sample ID Controls:
Accesses sub-menu for controlling the automatic assignment of sample identification numbers.



Automatic Sample ID:
When set to ON the unit will automatically assign sample identification numbers in accordance with parameters set by the other three keys on this menu. When set to OFF, the user manually enters each sample ID when prompted to do so.

Auto Sample ID Prefix:
An entry here will be used as a prefix for all sample IDs, if the Automatic Sample ID is set to ON. Press this key to access a sub-menu for entering an alphanumeric prefix.

Next Auto Sample ID Number:
Establishes the initial sample number for a series of tests and then shows the next sample ID which will be assigned. Used when the Automatic Sample ID is set to ON. Press this key to access a sub-menu for entering a numeric increment.

Auto Sample ID Increment:
Establishes the increment between sample numbers; used when the Automatic Sample ID is set to ON. Press this key to access a sub-menu for entering a numeric increment.

Sample Weight Warning Above:
This key displays and leads to a sub-menu used to set the maximum allowable sample weight (including spike) in grams. A warning will be given if sample weights above this value are entered.

Spike Weight Entry Mode:
This key steps through the options for entering spike weights either manually through the touch screen, balance port or network.

Sulfur Entry Mode:
This key steps through the options for entering the sulfur correction value either manually through the touch screen or automatically through the balance port

Moisture Entry Mode:
This key steps through the options for entering the moisture correction value either manually or through the touch screen or automatically through the balance port.

Auto Preweigh ID Controls:
Accesses sub-menu, used to automatically assign Sample ID numbers when a series of samples are preweighed ahead of the time they are actually tested.

Automatic Preweigh ID:
ON/OFF toggle for this feature.

Automatic Preweigh ID Prefix:
An entry here will be used as a prefix for all pre-weigh sample IDs.

Next Automatic Preweigh ID Number:
Shows the next Sample ID which will be assigned and is used to enter the beginning Sample ID of any series

Automatic Preweigh ID Increment:
Establishes the increment between samples.

Reporting Controls Menu

Report Width:
Toggle this key to set the column width of the printer to either 40 or 80 columns. Select 40 when the 1757 Printer is used.

Automatic Reporting:
Toggles the automatic reporting ON/OFF. When ON, preliminary reports will be generated at the conclusion of the test and final reports will be generated as soon as all of the thermochemical corrections are available. When OFF, reports will only be generated by selecting the Report key.

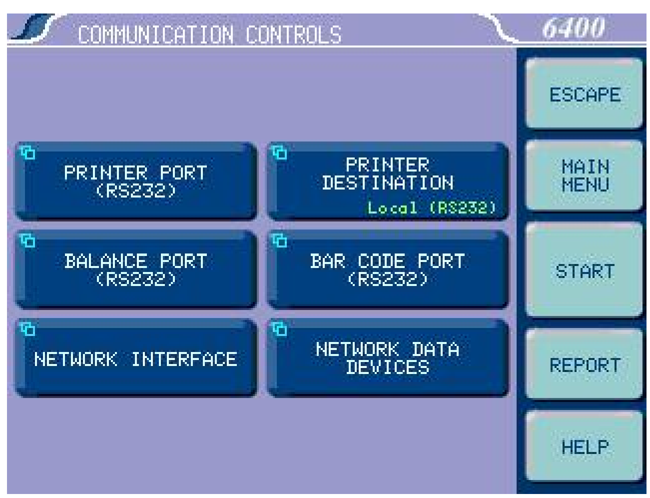
Automatic Report Destination:
Toggles to direct the reports to the Printer port or the screen display.

Individual Printed Reports:
When set to ON, will generate header information for each report printed. In the OFF position, only one header will be printed for a series of tests.

Edit Final Reports:
When set to ON, enables the user to revise sample weight and thermochemical corrections of finalized reports from the report menu.

Recalculate Final Reports:
When set to ON, causes a recalculation of stored final reports using calibration data and menu settings currently in the Calorimeter.

Use New EE Value in Recalculation:
When set to ON, any recalculation made will use the most recent EE value in the calculations. In the OFF position, all calculations will be made using the EE value which was effective when the test was originally run.

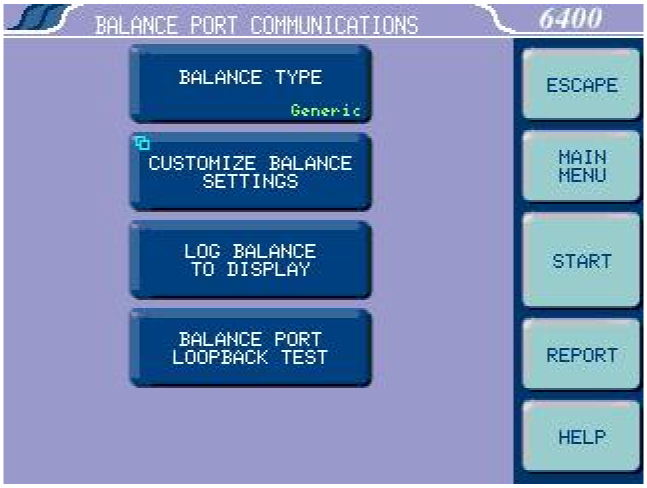


Communication Controls Menu

Accesses sub-menus which set the communications protocols for the printer and balances.

Printer Port (RS232):
Accesses sub-menu, Printer Port Communications. Sets the communication parameters for the RS232 ports used for the printer port. Standard options for data bits, parity, stop bits, handshaking, baud rate and balance type are provided to match any devices that might be connected to these ports.

- Number of Data Bits. Standard options for data bits. Toggles between 7 and 8.
- Parity. Standard options for parity. Choose from None, Odd or Even.
- Number of Stop Bits. Standard options for stop bits. Toggles between 1 and 2.
- Handshaking. Standard options for handshaking. Choose from Xon/Xoff, RTS/CTS and None.
- Baud Rate. Standard options for baud rate. Choose from 19.2K, 9600, 4800, 2400, 1800, 1200, 600, 300, 150, 134.5, 110, and 75.
- Printer Type. Toggles between a Parr 1757 and a generic printer. When set for the 1757 Printer, all of the features of this printer, such as bold printing, will be activated.
- Printer Port Loop Back Test. Used for factory testing of the printer port.



Balance Port (RS232):
Accesses sub-menu, Balance Port Communications.

Balance Type
Toggles through the available balance templates.

Customize Balance Settings
Sets the communication parameters for the RS232 port used for the balance port. Standard options for data bits, parity, stop bits, handshaking, baud rate and balance type are provided to match any devices that might be connected to these ports.

- Number of Data Bits. Standard options for data bits. Toggles between 7 and 8.

- Parity. Standard options for parity. Choose from None, Odd or Even.
- Number of Stop Bits. Standard options for stop bits. Toggles between 1 and 2.

- Handshaking. Standard options for handshaking. Choose from Xon / Xoff, RTS/CTS and None
- Baud Rate. Standard options for baud rate. Choose from 19.2K , 9600, 4800, 2400, 1800, 1200, 600, 300, 150, 134.5, 110, and 75.
- Data Characters from Balance. This setting is only used when the generic balance format is selected. This value determines the number of numeric data characters (0-9 . + -) to accept. Any additional characters after this value and before the string terminating <CR> are discarded.
- Data Precision. This key allows the user to establish the number of digits to the right of the decimal point that are passed from the balance handler.
- Transfer Timeout (seconds). This value determines how long the interface will wait before giving up on a weight transfer. The value is entered in seconds.
- Balance Handler Strings. This key leads to a submenu that allows balance template to be customized for unique balances or needs.

Log Balance to Display

Directs the incoming data stream from the balance to a display buffer. This function can be used to determine the data format from an unknown balance type. The display buffer is 40 characters in length. The balance must be forced to issue at least 40 characters before the contents of the buffer are displayed.

Balance Port LoopBack Test

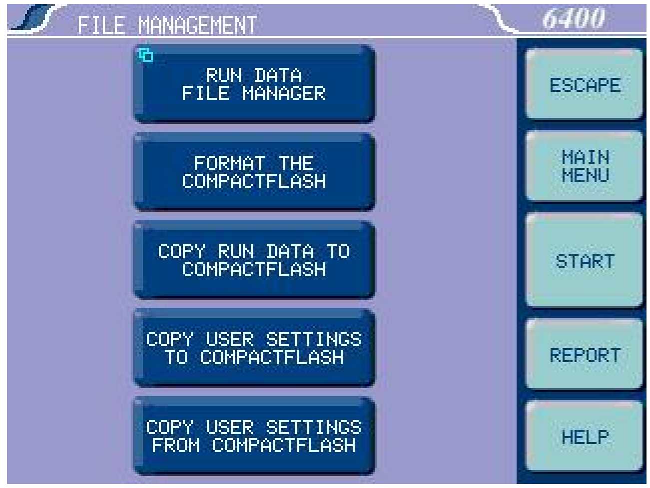
Initiates a loopback test on the port. A special loopback plug is required in order to perform this test.

Parr offers the following communication cables:

25 pin D (male)		
A1837E	9-pin DP	25-pin DP S-T
A1838E	9-pin DP	25 pin DP Null
9 pin D (male)		
A1892E	9-pin DP	9-pin DP S-T
A1893E	9-pin DP	9-pin DP Null

Further information on establishing communications for the Printer Port, Balance Port, Network Interface, Bar Code Port and other Network Data Devices can be found in the Installation section of this manual.

File Management Menu



Run Data File Manager:
This key activates the File Manager. The File Manager is used to delete or rename test report files. It is also used to convert file types.

Format the CompactFlash:
This key allows the user to format an installed CF card in a manner that is compatible with the calorimeter.

> ***Note:*** *Formatting will erase all files on the card!*

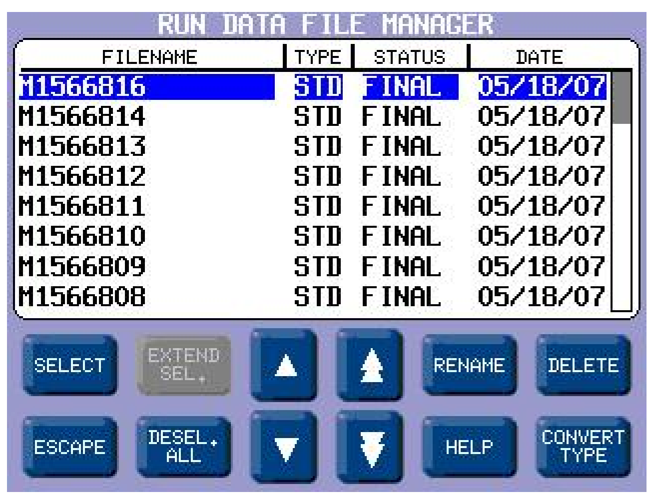
Copy Run Data to CompactFlash:
This key copies all test data to a Compact Flash (CF) card inserted into the rear of the calorimeter controller. This feature is used as a means of either archiving data or transferring it to a PC.

> ***Note:***
> *Subsequent use of the same CF card will overwrite the data currently on the card.*

Copy User Settings to CompactFlash:
This key copies all previously saved user setups to CF.

Copy User Settings From CompactFlash:
This key copies all user setups previously saved to CF back to the calorimeter controller memory. This feature can be used to configure multiple calorimeters in an identical manner.

Run Data File Manager



The white upper portion of the Run Data File Manager screen presents all tests in memory in a scrollable window. Test attributes include filename (sample ID), test type, status, and date. Touching anywhere in the column related to a given test attribute will sort the file list by that attribute. Successive touches will toggle between an ascending and descending sort.

Select:
This key is used to begin the file selection process. The up / down and page up / page down keys are used to scroll up and down the file list. Pressing the select key when a file is highlighted blue will highlight the file with a cyan color. This indicates that it is selected. Multiple files throughout the list can be selected in this fashion.

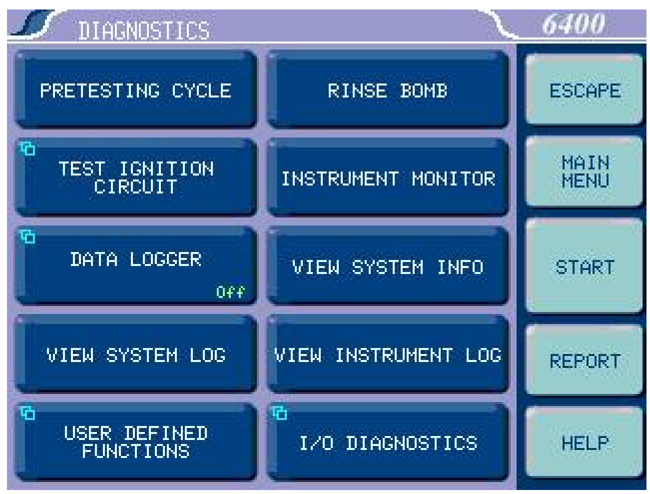
Extend Sel:
This key selects all files between the last file selected and the file that is highlighted in blue.

Desel All:
This key deselects all files previously selected.

Rename:
This key allows the user to rename the blue highlighted filename.

Delete:
This key deletes all selected files.

Convert Type:
This key allows one or more selected tests to be converted from determinations to standardizations and vice versa.



Diagnostics Menu

Provides the user with the means to test many of the components and subsystems of the calorimeter. These capabilities should be used in conjunction with this instruction manual in order to obtain the maximum benefits from these capabilities.

Pretesting Cycle:
This key initiates a pretest to run the calorimeter through the fill and cool/rinse cycles. This function is used to pre-condition the calorimeter if it has been sitting idle for an extended period of time (greater than 15 minutes).

Test Ignition Circuit:
Activates the ignition circuit. A volt meter can be placed across the firing connections to ensure that the actual firing charge is reaching these contacts.

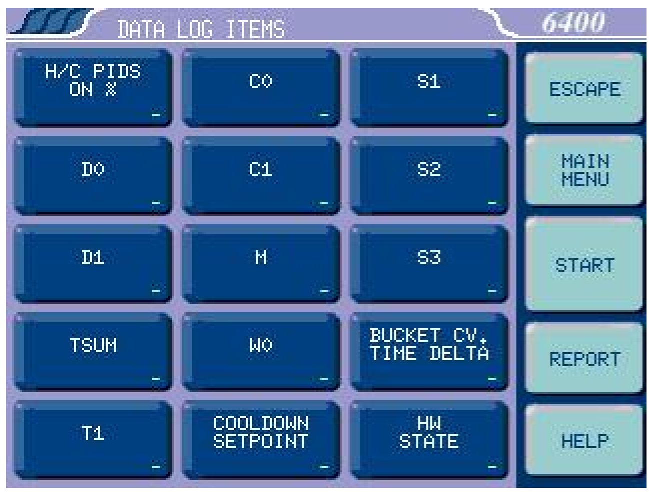
Data Logger:
Displays ON/OFF status and accesses the Data Logger Controls Menu for setting the specific logging controls.

Data Logger:
This key toggles the data logging function ON / OFF.

Data Log Interval:
This key displays the interval of which the selected data is logged. The interval in seconds is defined in the Select Data Items sub-menu (normally 12 seconds). This roughly matches the update interval for the bucket temperature.

Data Log Destination:
Options are logfile, printer or both. When the logfile option is selected, the logfile is located at /flash/datalog.csv. The maximum allowed size for this file is roughly one megabyte. If the file reaches this size, logging is halted.

Select Data Log Items:
Press this key to access the Data Log Items sub-menu, which provides keys for fifteen items that can be individually selected for logging. By default, both the bucket and jacket temperatures are logged. All records are date and time stamped. Helpful items to log are:



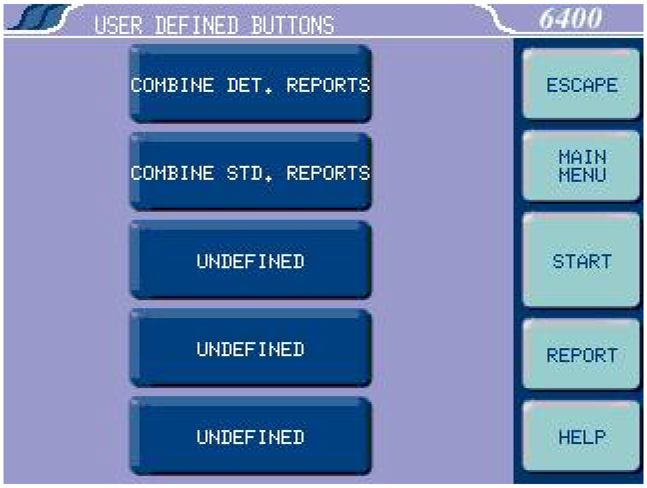
D0 - Corrected calorimeter drift rate
Tsum - Accumulated temperature rise
T1 - Extrapolated temperature rise
C0 - Temperature conversion counter

Data Log Format:
Toggles between Text Format and Data Format (csv). Data is either logged with the supporting tag information (text) or in a comma separated variable (csv) data format as selected by the user. The text setting is useful if the data log destination is a printer. The data (csv) format is especially useful if the data is ultimately transferred to another computer for post processing, graphing, etc. The log file can be transferred to another computer via FTP.

Delete Data Log File:
When this key is pressed the contents of the data log file are deleted.

View System Log:
This key accesses its sub-menu which displays the contents of /flash/log/messages. This file is used primarily to log application program debug messages. Press the Print key to print these messages.

User Defined Functions:
This key leads to a sub-menu that offers five special purpose user/ factory definable function keys.



Combine Det. Reports:
Pressing this key combines all determination reports into a single file named /tmp/bigdetfile.txt.

Combine Std. Reports:
Pressing this key combines all determination reports into a single file named /tmp/bigstdfile.txt.

Rinse Bomb:
This key initiates a bomb rinse. This function can be used to clean out the cylinder in the event a sample is spilled inside the cylinder.

Instrument Monitor:
This key accesses its sub-menu screen which provides a summary of most of the important instrument parameters. This screen is used to detail the course of a test or to observe the heating / cooling performance of the calorimeter.

View System Info:
Press this key to display a screen with current operating system information / statistics such as:

- Processes and their associated PIDs
- Memory
- Mass Storage
- Network

Press the Print key to print this information.

View Instrument Log:
Press this key to display a screen with contents of the /tmp/instlog file. It contains a sequential log of the instrument's processing. Press the Print key to print this log.

I/O Diagnostics:
Press this key to display the I/O Diagnostics sub-menu, which allows the user to manipulate digital outputs for troubleshooting. The I/O Diagnostics screen is used to display the digital outputs at a basic level for troubleshooting. Both the bucket and jacket temperatures are also displayed on this screen. Any output can be selected using the left and right arrow keys. The selected output is turned ON (1) or OFF (0) using the 1 and 0 keys. Prior to entering the Diagnostics Menu, the controller stores the present state of the outputs. This state is restored when you exit this screen. Digital outputs cannot be manipulated while a test is in progress.

CALCULATIONS

Corrections

Fuse Correction

There are two components to the fuse correction in the 6400 Calorimeter:

- The heat introduced by heating the wire used to ignite the cotton thread.
- The heat of combustion of the cotton thread used to ignite the sample.

e_3 = (fuse value) (fuse multiplier)

The semi-permanent heating wire is heated by dissipating an electrical charge from a capacitor. Since this charge is controlled by the size of the capacitor and the charging voltage, and because the capacitor is fully discharged for each test, the energy released can be calculated. In the 6400 Calorimeter this is a correction of 10 calories per test.

Cotton has a heat of combustion of 4000 calories per gram. The actual thread being used should be weighed to see how much is being burned. Ten centimeters of a fine thread will weigh approximately 0.003 grams which would release 12 calories as it burns. Heavier threads weigh up to 0.010 grams per 10 centimeters and increase this correction to 40 calories per test. The finer the thread, the smaller errors will be if the thread is not exactly ten centimeters in length. Polyester thread is not recommended for use in the bomb because it has a tendency to melt and fall away from the heating wire before it ignites.

Using the fine thread mentioned above, the fuse correction for the calorimeter would be the 10 calories from electrical heating plus 12 calories from the burning thread for a total of 22 calories per test. The thread supplied by Parr has a mass of approximately 1 milligram per centimeter. This results in a total fuse correction of 50 calories. Fuse corrections can be entered when Fixed Fuse in the Thermochemical Corrections menu, is set to OFF. Total errors of more than 5 calories will seldom occur when using a fixed fuse correction and the fuse wire and cotton thread supplied by Parr.

Spiking Correction

It is sometimes necessary to add a spiking material to samples which are very small, have a low heat of combustion, or have a high moisture content to add sufficient heat to drive the combustion to completion. Benzoic acid is an excellent material for spiking for all of the same reasons it is a good standard material. White oil is also an excellent material. The 6400 calorimeter can automatically compensate for the addition of spiking materials to these samples. The calculations are modified in these cases as follows:

$$H_c = \frac{WT - e_1 - e_2 - e_3 - (H_{cs})(m_s)}{m}$$

Where:

H_{cs} is equal to the Heat of combustion of the spiking material in calories per gram

m_s is equal to the mass of the spiking material

This factor is added to the calculations when Spike Controls, Use Spiking is set to ON. The Heat of Combustion of the spiking material is entered as calories per gram. The calorimeter will prompt the user to enter the weight of spiking material. Fixed spikes can also be used. To do this, set Use Fixed Spike to ON and enter the mass of the spike (Weight of Fixed Spike key).

Nitric Acid Correction
In the high pressure oxygen environment within the oxygen bomb, nitrogen that was present as part of the air trapped in the bomb is burned to nitric oxide which combines with water vapor to form nitric acid. All of this heat is artificial since it is not a result of the sample burning. The nitric acid correction removes this excess heat from the calculation. Users may find it convenient to enter a fixed value for the acid correction and avoid the need to determine this correction for each test. Use of a fixed value for the acid correction is highly recommended. Fixed acid corrections can be entered when Fixed Acid - Thermochemical Corrections, is set to ON. A correction of 8 calories is a good number for the fixed nitric acid value. For most work, it is recommended to set "Acid Value is Nitric Acid Only", in Calculation Factors to ON. Total errors of more than 3 calories will seldom occur when using fixed nitric acid corrections.

Sulfur Correction
In the oxygen rich atmosphere within the bomb, sulfur in the sample is oxidized to sulfur trioxide which combines with water vapor to form sulfuric acid. This liberates additional heat over the normal combustion process which converts sulfur to sulfur dioxide. The sulfur correction removes this excess heat from the calculation. Fixed sulfur corrections can be entered if a series of samples contain a constant amount of sulfur. Fixed sulfur corrections can be entered when Fixed Sulfur - Thermochemical Corrections, is set to ON. Enter percent sulfur as indicated on this line. Any errors will be proportional to the difference between the actual and assumed value for sulfur.

> ***Note:***
> *For ordinary work where benzoic acid is used for standardizing the calorimeter, the Fixed Sulfur Correction for Standardizations should be ON applying a fixed value of 0.0 to all standardization tests. Benzoic acid contains no sulfur.*

Manual vs. Fixed Corrections

If fixed values for fuse, acid and sulfur are turned OFF on the Thermochemical Corrections Menu, then the user must manually enter the values at the prompt. Final reports for each test can be obtained whenever the operator is prepared to enter any required corrections for fuse, acid and sulfur. When entering corrections, the user can choose either of two methods. These are:

- Manual Entry
- Fixed Corrections

Manual Entry
During the reporting process, the controller will prompt the user to enter the following values:

- Fuse Correction — Key in the Fuse/Heat Wire Correction and press the ENTER key. The default setting for this value is to be entered in calories.
- Acid Correction — Key in the Acid Correction and press the ENTER key. The default setting for this value is to be entered in milliliters of standard alkali required to titrate total acid or calories.
- Sulfur Correction — Key in the Sulfur Correction and press the ENTER key. The default setting for this value is to be entered as percent sulfur in the sample.

Note: *If the Spiking Correction is used, a spiking correction must be entered before obtaining a Final Report. After the last entry has been made, the Calorimeter will automatically produce a Final Report. If values for these corrections are not available, the operator can use the SKIP key to bypass any of the corrections, however, a Final Report will not be printed until an entry is made for fuse, acid and sulfur.*

Fixed Corrections
In many cases, fixed values for fuse and acid can be used without introducing a significant error since the corrections are both relatively small and constant. Fixed sulfur corrections can also be used whenever a series of samples will be tested with a reasonably constant sulfur content. Any value set-up as a fixed correction will be automatically applied and the calorimeter will not prompt the user for this value.

Definitions

Total acid is the amount of base required to titrate the bomb washings (milliliters).

Nitric acid is that portion of the total acid in the bomb washings that result when the nitrogen in the air that is trapped in the bomb is burned at high pressure. Since this nitric acid does not result from the sample, and the combustion conditions are reasonably constant from test to test, the amount of nitric acid formed is also constant.

Acid multiplier is multiplied by the user entered acid value to arrive at the number of milliequivalents of acid. This value is usually the concentration (normality) of the base in equivalents per liter.

Percent sulfur is the concentration of sulfur in the sample (weight %).

Molecular weight of sulfur is 32.06.

Equivalent weight of sulfur in H_2SO_4 is 16.03 (one half of the molecular weight).

Heat of formation of nitric acid is 14.1 calories / milliequivalent.

Heat of formation of sulfuric acid (from SO_2) is 36.1 calories / milliequivalent.

Sample mass is the mass of sample burned in the bomb (grams).

Sulfur multiplier is multiplied by the product of the user entered sulfur value and the sample mass to arrive at the number of milliequivalents of sulfuric acid in the bomb washings.

Example 1:

The following assumptions are made for the factory default settings of the calorimeter

1) The acid value is that of nitric acid only
2) The concentration of the titrant used is 0.0709N
3) 1 calorie (I.T.) is equal to 4.184 Joules
4) The heat of formation of HNO_3 is 14.1 cal/meq
5) The heat of formation of H_2SO_4 is 36.1 cal/meq

$$e_1 = (\text{ml of acid})(\text{acid mult})(\text{H}_\text{f}\ \text{HNO}_3)$$
$$= (9.6ml)\left(\frac{0.0709meq}{ml}\right)\left(\frac{14.1cal}{meq}\right)$$
$$= 9.6\ \text{cal}$$

$$e_2 = (\text{\%S2})(\text{sample mass})(\text{sulfur mult})(\text{Hf H}_2\text{SO4})$$
$$= (2.16)(0.5087\text{g})\left(\frac{0.6238\text{meq}}{cal}\right)\left(\frac{36.1cal}{meq}\right)$$
$$= 24.7439\ \text{cal}$$

$$\text{H}_\text{c} = \frac{\text{Wt - e}_1\text{ - e}_2\text{ - e}_3}{m}$$
$$= \frac{\left(\frac{937.5971cal}{^\circ C}\right)(5.6468^\circ C) - 9.6cal - 24.7439cal - 50.0000cal}{0.5087g}$$
$$= 10241.9489\ \text{cal/g or } 42.8523\ \text{MJ/kg}$$

Example 2:

The following assumptions are made and outlined in D240.

1) The acid value is ALL nitric
2) The concentration of the titrant used is 0.0866M
3) 1 calorie (I.T.) is equal to 4.1868 Joules
4) The heat of formation of HNO3 is 57.8 kJ/mol
5) The heat of formation of H2SO4 is 301.4 kJ/mol

The calorimeter must be set accordingly. (The calorimeter performs ALL calculations in calories, milliequivalents and degrees Celsius)

1) Set "Acid Value is Nitric Acid Only" to OFF
2) Set the "Acid Multiplier" to 0.0866
3) Set the "Sulfur Value is Percent" to ON
4) Set the "Sulfur Multiplier" to 0.6238
5) Leave the Fixed Fuse correction set to ON (50 cal = 209.34 J)

$$e_1 = [\text{(ml of acid)(acid mult) - (\%S2)(Sample Mass)(sulfur mult)}] \times 14.1$$
$$= \left[(9.6ml)\left(\frac{0.0866meq}{ml}\right) - (2.16\%)(0.5087g)(0.6238meq)\right] \times 14.1$$
$$= 2.0cal$$

$$e_2 = \text{(\%S2)(sample mass)(sulfur mult)(Hf } H_2\text{SO4)}$$
$$= (2.16)(0.5087\text{g})\left(\frac{0.6238\text{meq}}{cal}\right)\left(\frac{36.0cal}{meq}\right)$$
$$= 24.675 \text{ cal}$$

$$H_c = \frac{\text{Wt - e}_1\text{ - e}_2\text{ - e}_3}{m}$$
$$= \frac{\left(\frac{937.5971cal}{{}^\circ C}\right)(5.6468^\circ C) - 2.0cal - 24.675cal - 50.0000cal}{0.5087g}$$
$$= 10257.0244 \text{ cal/g or } 42.9441 \text{ MJ/kg}$$

ASTM, ISO and Other Methods

Current ASTM, ISO, and British Standard Methods differ on their treatment of the nitric and sulfuric acid thermochemical corrections. ASTM Methods call for titrating the bomb washings to determine the total acid present. This is assumed to be all nitric acid with a heat of combustion of -14.1 Kcal per mole. The amount of sulfur is then determined and converted to equivalents of sulfuric acid. The difference between the heat of formation of sulfuric acid (-72.2 Kcal per mole or -36.1 calories per milliequivalent) and nitric acid is then subtracted as the sulfur correction.

Most other test methods treat nitric and sulfuric acid corrections as entirely separate values instead of combined values. This eliminates the requirement for a total acid determination and permits the nitric acid correction to be handled in a variety of ways, including the assumption of a fixed nitric acid correction.

The 6400 Calorimeter can be set up to apply the acid correction by either the ASTM or ISO convention, as the user prefers. Care must be used to ensure the proper corrections are applied, and the calculations made are consistent with the procedure used. See Table 4.

ASTM

In the ASTM treatment, the correction for acid formation assumes that all the acid titrated is nitric acid. Obviously, if sulfur is present in the sample, which in turn produces sulfuric acid, part of the correction for the sulfuric acid formed is already included in the ASTM nitric acid correction (e_1). This is adjusted by a separate computation based upon the sulfur content of the sample. An additional correction of 1.37 Kcal must be applied for each gram of sulfur converted to sulfuric acid from sulfur dioxide. This is based upon the heat of formation of sulfuric acid, from sulfur dioxide, under bomb conditions, which is -72.2 Kcal per mole or -36.1 calories per milliequivalent. But remember, a correction of 14.1 calories per milliequivalent of sulfuric acid is already included in the ASTM nitric acid correction (e_1). Therefore the additional correction which must be applied for sulfur will be the difference between 36.1 and 14.1 or 22.0 calories per milliequivalent (44.0 Kcal per mole). For convenience, this is expressed, in the ASTM e_2 formula, as 13.7 calories (44.0/32.06) for each percentage point of sulfur per gram of sample.

ISO

Both the ISO 1928 and BSI 1016: Part 5 methods for testing the calorific value of coal and coke, deal with acid and sulfur corrections in a manner which is somewhat different than ASTM procedures. The analysis of bomb washings in these methods call for a titration, first using 0.1N barium hydroxide (V2) followed by filtering, and a second titration using 0.1N HCl (V1) after 20 ml of a 0.1N sodium carbonate has been added to the filtrate. Table 3 gives the settings which allow the results of the two titrations, V1 and V2, to be entered into the controller directly for the calculation of the total acid correction. V1 should be entered at the prompt for acid and V2 is entered at the prompt for sulfur. The settings in Table 3 assume that the same procedure is carried out for both standardization and determination. The offset value is the product of -1, the Heat of Formation of Nitric Acid, the acid multiplier, and the 20 ml of 0.1 N sodium carbonate used in the analysis. The formula used to get the total correction in calories is as follows:

V1 (Acid Multiplier) (Heat of Formation of Nitric Acid) V2 (Sulfur Multiplier) (Heat of Formation of Sulfuric Acid) + offset value

The values for fixed acid and sulfur, which are used in preliminary reports, will reflect a sulfur correction of 0, and a nitric acid correction of 10 calories.

Conversion to Other Moisture Bases

The calculations described previously 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 D3180 and in other references on fuel technology.

Conversion to Net Heat of Combustion

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_{net} Btu per pound can be calculated as follows:

$$H_{net} = 1.8Hc - 92.7H$$

(Solid fuels, ASTM D2015)

$$H_{net} = 1.8Hc - 91.23H$$

(Liquid fuels, ASTM D240)

REPORTS

The 6400 Calorimeter can transmit its stored test data in either of two ways. The Auto Report Destination key on the Reporting Controls Menu toggles the report destination between the display and an optional printer connected to the RS232 printer port of the calorimeter. This menu also selects the type of reports that are generated automatically by the calorimeter.

There are two kinds of reports: Preliminary and Final.

- Preliminary Reports are generated at the conclusion of a test. They are intended to confirm to the operator that the results of the test fell within the expected range.
- Final reports are generated once all of the thermochemical corrections have been entered into the file. If fixed corrections are used for all of the thermochemical corrections, a preliminary report will not be generated. The report will automatically become finalized.

Thermochemical corrections are entered by using the following steps to select and edit preliminary reports. Test results are stored as files using the sample ID number as the file name. A listing of the stored results is accessed by pressing the REPORT command key. The REPORT command key brings up a sub-menu on which the operator specifies.

<u>Select From List</u> This key displays the stored results specified with the following two keys.

<u>Run Data Type</u> This key enables the operator to display only determination runs, only standardization runs, only solution runs (not applicable to the 6400) or all runs.

<u>Run Data Status</u> This key enables the operator to display only preliminary reports, only final reports, only preweighed samples reports, all stored reports or preliminary and final reports.

<u>Prompt For Final Values</u> When turned on, the controller will prompt the operator to enter any missing corrections for fuse, sulfur and acid in any selected preliminary reports. When turned off preliminary reports will be displayed as entered.

The displayed files can be sorted by sample ID number, by type, by status or by date of test by simply touching the appropriate column. Individual files can be chosen by highlighting them using the up and down arrow keys to move the cursor. Press the SELECT key to actually enter the selection. Once selected the highlight will turn from dark blue to light blue. A series of tests can be selected by scrolling through the list and selecting individual files. The double up and down keys will jump the cursor to the top or bottom of the current display. If a range of tests is to be selected, select the first test in the series, scroll the selection bar to the last test in the series and press EXTEND SEL to select the series.

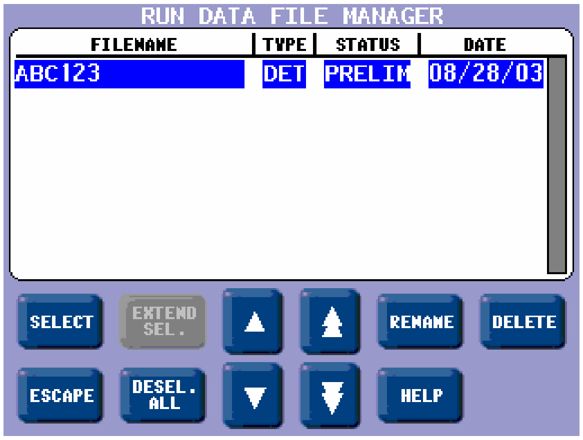
The DESEL ALL key is used to cancel the current selection of files.
To bring the selected report or series of reports to the display, press the DISPLAY key. To send the reports to the printer press the PRINT key.

The EDIT key brings up a sub-menu which enables the operator to edit any of the data in the report or add thermochemical corrections to convert preliminary reports to final reports. Final reports can only be edited if Edit Final Reports on the Reporting Controls Menu is turned on.

To have the Net Heat of Combustion print as part of preliminary and final reports, go to the Thermochemical Corrections Menu and turn ON Calculate Net Heat of Combustion. During the reporting process, the controller will prompt for the hydrogen (H) value.

MEMORY MANAGEMENT

The 6400 Calorimeter will hold data for 1000 tests in its memory. These tests may be pre weights, preliminary or final reports for either calibration or determination runs. Once the memory of the controller is filled, the controller will not start a new analysis until the user clears some of the memory.



The FILE MANAGEMENT key on the main menu leads to the file management sub-menu. The RUN DATA FILE MANAGER key leads to a listing of the files. Single files can be deleted by highlighting the file and pressing the DELETE key. The controller will then ask the user to confirm that this file is to be deleted. A series of files can be deleted by selecting the first file in the series and then the last file in the series using the EXTEND SEL key and then pressing the DELETE key.

The controller of the 6400 Calorimeter can accept compact flash memory cards. These cards can be used to:

- Copy test file data for transfer to a computer
- Copy user settings for back up
- Reload user settings to the controller to restore or update the controller's operating system.

Compact flash memory cards are inserted into the slot on the back of the control section of the Calorimeter. Keys are provided on the FILE MANAGEMENT sub-menu to initiate each of the above three actions.

MAINTENANCE

Note:
Some of the following manual sections contain information in the form of warnings, cautions and notes that require special attention. Read and follow these instructions carefully to avoid personal injury and damage to the instrument. Only personnel qualified to do so, should conduct the maintenance tasks described in this portion of the manual.

Caution – Risk of Electrical Shock
Disconnect the electrical power before servicing or replacing any components!

Inspection of Critical Sealing Surfaces

The sealing grooves and related surfaces for most of the Parr bombs are machined to tolerances as small as +/- 0.001" (0.03mm). As a result, any imperfection in a sealing surface resulting from either normal use or carelessness in handling the bomb can cause the bomb to leak. If the damage or accumulated wear is much less than 0.001" (0.03mm), then careful polishing will restore the bomb sealing to an as new condition. Imperfections that penetrate the sealing surface more than one or two thousandths of an inch (0.03-0.06mm) may render the seal surface unserviceable.

Any surface that comes in contact with an elastomer seal should be carefully examined for imperfections that would compromise its ability to seal. A freshly sharpened pencil can be used to probe the metal sealing surfaces for significant imperfection. If the pencil point hangs up in the imperfection, further attention is warranted. An attempt should be made to polish (remove) any significant imperfections. This operation generally requires the use of a lathe in order to guarantee that the sealing surface to be repaired remains concentric with the mating surface. Knowledge of the dimensional tolerances and the ability to accurately measure or gauge the affected area is required in order to insure that too much polishing (metal removal) has not taken place. **We recommend that bombs with significant imperfection of this nature be serviced at Parr.**

Caution! *Do not pry elastomer seals (o-rings and quad-rings) from seal grooves with metallic tools.*

The use of dental picks and other metallic tools to pry the seals from their grooves must be avoided. These hard steel tools, if misused, can leave permanent tool marks on the sealing surface, which are difficult or impossible to remove. These blemishes are hidden by the seal during normal use and as a result, are not readily apparent as the cause of a leaking bomb.

Larger size seals (0.8" or 20 mm O.D.) typically used to seal the bomb head can be extracted from its groove using either of the following two methods:

1. Grasp the outer circumference of the seal with the thumb and forefinger and slide them together while applying sufficient pressure on the seal to cause it to pucker out of the groove. With the other hand, grab the exposed, pinched section and pull the seal from the groove.

2. Use a non-metallic object, such as the rounded corner of a plastic credit card, to simply pry the seal from its groove.

Smaller diameter seals usually require a different approach. A portion of the seal should be carefully pulled, not pried, from the groove with a small pair of pliers or a hemostat. The exposed portion of the seal can then be cut, or pulled further to remove the seal. The pliers or hemostat should never contact the sealing surface, only the seal.

Bomb Removal

To service or remove the bomb cylinder from the bucket assembly, remove the 668DD Check Valve from the bomb cylinder. Remove the 941DD Wedge with needle nose pliers. Remove the two SA1632RD18 Machine Screws (see Figure 27), then remove the 942DD plastic bushings and the 1071DD Quad-ring.

The entire bucket can be removed by disconnecting the bucket probe at the quick disconnect. Carefully lift the bucket and bomb assembly out of the air chamber and position horizontally on the calorimeter to remove the 925DD Oxygen Bomb Retainer Nut (see Figure 23). Now the cylinder can be removed from the bucket assembly. Note the position of the locating pin.

To replace, follow these steps in reverse.

Fuses

Lines protective fuses rated: Fast-Acting 15A, 250VAC (Parr number 139E23).
The replacement of protective fuses for the 6400 Calorimeter should be performed by qualified personnel.

Daily Maintenance

Clean the 1444DDJB o-ring that seals the bomb head and cylinder by wiping with a tissue. Wet this sealing area with water prior to starting a series of tests. Clean the corresponding sealing area in the cylinder in a similar fashion. Both surfaces must be free of any accumulated foreign matter, such as unburned sample material or combustion by-products. Wet the hole in the center of the head which contains the check valve.

With a tissue, clean the head where the large bucket quad-ring (1071DD) contacts the head perimeter. Wet this sealing area with water prior to starting a series of tests.

Remove, inspect and clean the cylinder check valve (668DD) and corresponding sealing area in the bomb cylinder. In extreme cases, i.e. a spilled sample, use soap and water to clean the area.

Quarterly Maintenance

Change water in the Water Tank and replace the 149C water filters.

Periodic cleaning may be performed on the exterior surfaces of the instrument with a damp cloth. All power should be disconnected when cleaning the instrument.

50 to 100 Test Maintenance

Replace the heating wire, with 2” of 840DD2. Wind the wire 360 degrees clockwise around screws. Clean the 986DD Electrode Contact Pins with a mild abrasive, such as a pencil eraser. Clean the bomb head electrode points in a similar fashion and tighten the screws holding the heating wire in place.

500 Test Maintenance

Clean the ignition contacts.

Under normal usage Parr oxygen bombs will give long service if handled with reasonable care. However, the user must remember that these bombs are 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.

For your convenience, these parts may be purchased as kit number 6038, Firing Maintenance Kit.

Parr recommends that the following parts on the oxygen bomb be changed every 500 tests or six months whichever comes first: 840DD2, 1374HCJV (2), 394HC, 821DD (1), 1071DD, 1444DDJB, 659DD, 519AJV, 694DD. See Figures 10 and 26 for parts locations. When reassembling the bomb head, take care not to roll the 694DD o-ring as this will cause an oxygen leak.

> ***Note:***
> *Samples that contain chlorine or are abrasive may require this maintenance to be performed on a more frequent interval such as every 250 tests*.

The 882DD and 969DD o-rings should also be replaced. See Figures 23 and 24 for o-ring locations.

The 1140DD Seal/Release mechanism should be serviced with the same frequency as the bomb head. This includes the replacement and lubrication of the 659DDJU (2), 1138DD, 1143DD and 357HCJB o-rings with 811DD lubricant. See Figure 24 and Figure 25 for o-ring locations. The tools required are: screwdriver, snap ring pliers and needle nose pliers.

1. Turn off the gas supply to the calorimeter. Disconnect the oxygen connection to the back of the calorimeter. Go to the Diagnostics Screen and turn on the bomb seal command. These steps are necessary to release the gas pressure in the seal/release mechanism before disassembling.
2. Turn off the calorimeter.
3. Insert the bomb head into the cylinder and lock into place.
4. Disengage the screws, SA163X2RD018 that hold the bucket in the air can. Remove the 941DD plastic wedge that secures the front of the air can assembly.
5. Lift the bomb and bucket as a unit from the calorimeter air can chamber and disconnect the bucket thermistor probe. Set this unit aside.
6. Remove the vessel spacer, 964DD and the associated o-ring, 969DD.

7. Remove the cylinder spacer, 1141DD, which sits on top of the snap ring, 1137DD.
8. Remove the snap ring that retains the cylinder insert in the release mechanism at the bottom of the air can. Withdraw both the insert and the release pin as a unit using needle nose pliers.
9. If present, remove any scoring on the 966DD2 release pin, above the smaller o-rings, with crocus cloth. Replace the 659DDJU o-rings on the release pin as well as the 1138DD o-ring that seals the cylinder insert. Replace the 659DDJU and 357HCJB o-rings.
10. Reverse the above procedure to reinstall the cylinder insert/pin as well as the bomb bucket assembly. Make sure that the large side hole in the 1139DD insert is oriented toward the left side of the instrument. The insert is keyed to the cylinder and can not be fully inserted unless it is properly oriented

5000 Test Maintenance

To deal with the realities of today's test loads and cycle times, the ASTM Committee recommends in method E144-94 that "all seals and other parts that are recommended by the manufacturer be replaced or renewed after each 5000th firing or a more frequent interval if the seals or other parts show evidence of deterioration." Oxygen bombs returned to Parr for service will be tested in accordance with ASTM E144-94. A test certificate is provided with each repair.

This service includes:

- Disassembly, cleaning and inspection of all parts
- Re-polishing of the inner surfaces of the bomb
- Re-assembly with new insulators, and seals, sealing rings, and valve seats
- Proof testing
- Hydrostatic testing

The hydrostatic and proof testing of the oxygen bomb should be performed after every 5000 firings or if:

- The bomb has been fired with an excessive charge
- The ignition of any of the internal components has occurred
- There have been any changes in the threads on the bomb cylinder
- The bomb has been machined by any source other than Parr Instrument Company

After repeated use with samples high in chlorine (over 1%), the inner surfaces of the bomb will become etched to the point where appreciable amounts of metal salts will be introduced during each combustion. Any bomb which is being used for chlorine determination should be polished at more frequent intervals to prevent the development of deep pits. If the interior of the bomb should become etched or severely pitted, the resistance of the metal to further attack can be improved by restoring the surface to its original polished condition.

There are no user serviceable parts inside the product other than what is specifically called out and discussed in this manual. Advanced troubleshooting instructions beyond the scope of this manual can be obtained by calling Parr Instrument Company in order to determine which part(s) may be replaced or serviced.

TROUBLESHOOTING

Bomb Exhaust Troubleshooting

The bomb exhaust and sealing is controlled by movement of the 966DD2 piston inside of the 1140DD bomb seal/release cylinder. This assembly is mounted on the bottom of the calorimeter air can. The piston is driven to the up position (bomb exhaust) by applying oxygen at 30 atm to the 1/8 male connector (344VB). The piston is driven down (bomb seal) by applying pressure to the 376VB elbow. The application of the oxygen pressure is controlled by the A1251DD three station solenoid valves. There is a flow restrictor, part 527VB, on the inlet side of this solenoid which limits the maximum flow rate of oxygen and in turn creates a gradual increase in pressure at the 1140DD bomb seal/release cylinder when the solenoid is turned on. Failure of the bomb to exhaust in a timely fashion can have more than one cause. Certain causes can be eliminated systematically by checking the bomb exhaust diffuser, at the end of the bomb exhaust line, for any restrictions in the six small cross drilled holes. This fitting should be removed from the tubing, inspected thoroughly and cleaned as required.

Confirm Correct Operation of the A1251DD Solenoid Valve

If the piston does not move, it is worthwhile at this point to confirm that both sections of the A1251DD are working properly (Figure 15). For the location of the A1251DD assembly, Figure 12.

Disconnect the 1/8 nylon pressure hose at the elbow connection nearest the back panel by using a 7/16 wrench. Apply power to the unit and re-enter the I/O diagnostics. Turn the exhaust output on. Oxygen should flow from the elbow connection on the A1251DD. Turn the bomb exhaust output off and re-connect the nylon pressure hose. Disconnect the 1/8 nylon pressure hose at the middle connection. Activating the bomb seal output should produce a flow of oxygen at the elbow.

Turn off the bomb seal output and reconnect the nylon pressure hose. If neither solenoid produces a flow of gas when activated and the O2 Fill key does not produce a flow of gas, then, in all likelihood, the 527VB flow restrictor is plugged and should be replaced. If only one of the solenoids sources gas when activated, then the problem must be further diagnosed as either being electrical (I/O board, solenoid coil or external wiring) vs. mechanical (in the valve) and dealt with in an appropriate manner.

If either solenoid sources gas when it is off (i.e. leaks) then replacement of the entire A1251DD solenoid assembly is indicated. For reference purposes, the normal upward thrust generated by the 966DD2 piston is 50 pounds. The downward thrust is 20 pounds. Far less than 20 pounds are required to move the piston in either direction when the bomb is not pressurized.

Service the O-rings on the 966DD2 Piston

This process is described in the 500 test maintenance section.

Confirm function of the 966DD2 piston

In order to reduce the amount of time it takes to duplicate and troubleshoot this type of situation, the I/O diagnostics can be used to pressurize and exhaust the bomb without having to run lengthy combustion or pre-tests.

> **WARNING**: *This screen allows unconditional and arbitrary output control for testing purposes. Be aware that all user and instrument protection is disconnected while on this screen. This is very important and you should take proper pre-caution.*

1. Make sure the 668DD check valve is installed at the bottom of the cylinder.
2. Lock the head into the cylinder and close the calorimeter lid.
3. Confirm bomb release is off.
4. Turn bomb seal on then off to retract the 966DD2 piston.
5. Turn on O_2 fill to begin filling the bomb. The bomb will be completely filled in one minute, at which time O_2 fill should be turned off. This seats the check valve in the head which in turn seals the contents of the bomb.
6. The calorimeter lid can be unlocked at this time.
7. Activating bomb release should initiate a bomb exhaust within two seconds. If it takes much longer than two seconds before the bomb begins to vent, then at least one of the two following conditions outlined below exist.
8. If the bomb exhaust is initiated in a timely manner but fails to complete in 10 seconds, a blockage or restriction in the bomb exhaust circuit is indicated. This must be investigated and corrected.

If the bomb fails to exhaust, the 1454DD funnel and 1453DD bomb cap adapter can slowly be removed to release the pressure in the bomb. See Figure 26.

If the piston moves properly with no applied bomb pressure, but still fails to initiate an exhaust of a pressurized bomb in a timely fashion, at least one of the following conditions exist:

1. The 527VB restrictor is partially blocked.
2. The exhaust line is blocked.
3. There is a gas leak between the outlet of the solenoid and the 1140DD cylinder. This also includes the 357HCJB o-ring seal on the piston inside of the cylinder.

The first condition can be eliminated by cleaning or replacing the 527VB restrictor.

The second condition can be eliminated by replacing the tubing and clearing all connections.

The third condition can be eliminated by following the procedure outlined in the section servicing the o-rings on the 966DD2 piston and carefully inspecting the 1/8 nylon pressure hose and associated compression fittings for leaks while this circuit is maintained at operating pressure, using the calorimeter I/O diagnostics. A minute leak will result in a significant reduction in upward thrust.

Jacket Temperature Troubleshooting

The jacket temperature is monitored with the use of a thermistor installed in the A1448DD temperature control assembly. This assembly is heated by a heater cartridge, A1459DD. In the Diagnostics Menu, select Instrument Monitor. If the heater PID is ON and reading 100%, yet the jacket is at ambient temperature, check the following possible causes.

If the heater PID is OFF, the heater and pump must be turned on in the Calorimeter Operation screen to perform the troubleshooting steps.

WARNING: *Turn off the power to the calorimeter prior to attempting to reset the thermostat. The temperature control assembly can become very hot. Use caution when servicing this area of the calorimeter.*

If line voltage (115V or 230V) is present across the heater cartridge connection, check the resistance across the heater cartridge. Approximately 70 ohms will be seen with a 115V calorimeter. Approximately 140 ohms will be seen with a 230V calorimeter. If the resistance is not correct the heater may have failed.

If the voltage is not present, then examine the 2040E thermostat reset button. If the reset button extrudes this means that the temperature in the temperature control assembly has exceeded 75ºC. Confirm that water is flowing through the system, turn off the power and then reset the switch by depressing the button. If the thermostat continues to trip even though water is flowing through the system, refer to the error code "There Is A Problem With The Jacket Thermistor" for further troubleshooting.

If there is no voltage present, and the reset button on the thermostat is not tripped, refer to the error code "There Is A Problem With The Jacket Thermistor" for further troubleshooting. There may also be a problem with the calorimeter controller, A1250DD, and Parr service should be contacted.

Error List

The calorimeter will run a number of diagnostic checks upon itself and will advise the operator if it detects any error conditions. Most of these errors and reports will be self-explanatory. The following list contains errors that are not necessarily self-evident and suggestions for correcting the error condition.

A Misfire Condition Has Been Detected

This error will be generated in the event the total temperature rise fails to exceed 0.5°C after the first minute of the post-period. Possible causes for this error are listed below.

- Fuse wire not intact, too long or improperly formed
- Breakdown of the insulator and o-ring on the insulated electrode assembly
- Bomb not filled with oxygen
- A bomb leak
- Poor bucket stirring
- Ignition lead wires have broken internal strands

A Preperiod Timeout Has Occurred

The calorimeter has failed to establish an acceptable initial temperature, prior to firing the bomb, within the time allowed. Possible causes for this error are listed below:

- A bomb leak
- Poor bucket stirring
- Lid not tight

The Current Run Has Aborted Due To Timeout

The calorimeter has failed to establish an acceptable final temperature within the time allowed. Possible causes for this error are listed below:

- A bomb leak
- Poor bucket stirring

There Is A Problem With The Bucket Thermistor

Possible electrical open. These errors will result if the temperature probe response is not within the expected range. Probe substitution can be useful in determining the cause of the problem (probe or electronics). The valid working range of the probe resistance is 1000 to 5000 ohms

- Check connection to the board
- Check quick disconnect
- Replace probe
- Replace board

There Is A Problem With The Jacket Thermistor

Possible electrical open. These errors will result if the temperature probe response is not within the expected range. Probe substitution can be useful in determining the cause of the problem (probe or electronics). The valid working range of the probe resistance is 1000 to 5000 ohms

- Check connection to the board
- Check quick disconnect
- Replace probe
- Replace board

A/D Initialization Failed

Shortly after power is applied to the calorimeter controller and the operating system has started, the CPU attempts to read the unique I/O board calibration information from the I/O board. If the I/O board is not connected to the CPU, or the information on the board is not valid, this error will be issued.

Bomb ID – Has Been Fired – Times Which Exceeds the Bomb Service Interval

The calorimeter controller keeps track of how many times the bomb has been fired. When this count exceeds a preset limit (usually 500) this message will be issued each time the bomb is used for a test. Perform bomb maintenance and reset counter on Calibration Data and Control page for appropriate bomb number.

You Have Exceeded the Run Data File Limit (1000 Files)
The memory set aside for test runs has been filled. Use the memory management techniques to clear out non-current tests.

Bomb EE Standard Deviation Warning
The relative standard deviation for the calibration runs in memory for the indicated bomb exceeds the preset limit.

Sample Weight Warning
The entered sample mass exceeds the value entered via the Sample Weight Warning Above key on the Data Entry Controls page. This warning threshold is normally 2 grams.

The Lid has Failed to Lock or is not Closed Properly.
This error will be reported when the controller fails to detect continuity through the bomb ignition circuit. The most probable cause will be either a poor electrical connection between the bomb's internal electrodes and the fuse wire, carbon build up on the electrodes or a fuse wire that has burned out. This will also occur if the lid is not down before the pretest or test is initiated.

The heater loop break limit has been detected. The heater will now be shutdown
This error means that the calorimeter is trying to heat the water in the unit for an extended period of time. The calorimeter suspects that there is a short and shuts the system down in order to "save" itself. This is a fairly normal occurrence if the lab temperature is very cool at night. It is acceptable practice to ignore the warning and re-start the unit. However, if this error occurs more than three times in a row, then it may be a true thermistor problem. Please refer to the Jacket Temperature Troubleshooting section of the manual.

Could Not Cool the Bomb Successfully
The calorimeter has failed to establish the desired cool down temperature within the time allowed. Check the flow of cooling water to see that it is not restricted or the filter plugged. It may also be that the water is not cold enough.

Rinse Tank Level May Be Low
The controller decrements the rinse tank counter each time the bomb is rinsed. This message will be issued when the counter is at or below zero when the bomb rinse sequence is executed. This message is a reminder that the rinse tank needs refilling, followed by a manual resetting of the bomb rinse counter.

TECHNICAL SERVICE

Contact Us

Should you need assistance in the operation or service of your instrument, please contact the Technical Service Department.

Telephone: **(309) 762-7716**
Toll Free: **1-800-872-7720**
Fax: **(309) 762-9453**
Email: **parr@parrinst.com**

Any correspondence must include the following basic information:

1. The model and serial # of the instrument.
2. Date purchased.
3. Software version(s) shown on the “Software and Hardware Information” page.
4. Help system revision. This is displayed by pressing the <MAIN MENU> key and then the <HELP> key.

When calling by phone, it is helpful if the person is close to the instrument in order to implement any changes recommended by the Technical Service Department.

Return for Repair

To return the instrument for repair, please call the Technical Service Department for shipping instructions and a RETURN AUTHORIZATION NUMBER. This number must be clearly shown on the outside of the shipping carton in order to expedite the repair process.

If you have not saved the original carton and traps, please request a packaging return kit.

We prefer the calorimeter to be shipped in our cartons and traps to prevent shipping damage.

Ship repair to:

Parr Instrument Company
Attn: Service Department, RMA# XXXX
211- 53rd Street
Moline, Illinois 61265

PARTS LIST

Principal Assemblies in Calorimeter

Item	Description
1138/1138CL	Oxygen Combustion Vessel
1795E	Power Supply, 24V
1796E	Power Supply, 5/12V
897E	Capacitor, Ignition
A1250DD	Controller Assembly
A1251DD	Oxygen Solenoid Assembly
A1447DD	Water Solenoid Assembly
A1448DD	Temp Control Sub-Assy
A1449DD	Water Chiller Assembly
A1254DDEB	Pump Assembly Circulating 115V
A1254DDEE	Pump Assembly Circulating 230V
A1255DD	Propeller Assembly, stirrer
1433DD	Tank, Water (A1435DD / 6400)
A1449DD	Water Chiller Assembly
A1456DD	Rinse Valve Assembly
A1275DDEB	Cartridge, Heater Assembly, 120V
A1275DDEE	Cartridge, Heater Assembly, 230V
139E23	Fuse Fast/ Act 15 AMP 250V

WARNING: For continued protection against possible hazard, replace fuses with same type and rating of fuse.

A1250DD Controller Assembly Parts List

Item	Description
1217DD	Gasket for Display
1803E	Backlight, Inverter
A1876E	Touchscreen LCD w/ cable
A1792E	Transition Display Board
A1933E6	CPU Board 6400
A1794E	Input / Output Board
A1806E	Cable, Backlight Inverter

A1457DD Accessory / Installation Kit Parts List

Item	Description
231C2	Container, PP 10L Foldable
3415	Benzoic Acid Pellets 100 Gram Bottle O-Ring,
356HCW	Pliers for Snap Ring
43AS	Capsule, SS
811DD	Lube/Sealant
840DD2	Heating Wire
845DD2	Ignition Thread, 4”
876DD	Cutter, Plastic Tubing
1005DD	Forceps
A1006DD	Waste Tube Assembly
A38A	Bomb Head Support Stand
TX03SK	1/32 Socket Screw Key
TX14SK	9/64 Socket Screw Key
149C	In-line Filter
1344DD	LCD Stylus
1889E	LCD Screen Protector
356HCW	Pliers for Snap Ring (356HC)
HJ0025TB035	Tubing, Nylon 1/4 OD X .35W
HX0012TB024	1/8 OD Tubing, Pressure Nylon

A1265DD Bucket and Stirrer Tube Assembly Parts List

Item	Description
944DD	O-Ring, Buna-N .237 ID
946DD	Seal ¼ SS
1129DD	Pin, Anti-rotating (A940DD)
1416E	Thermistor, Bucket
1462E2	Thermistor Cable
A940DD	Tube Assy, Bucket; Soldered
A1255DD	Bucket Stirrer Assembly

A1255DD Bucket Stirrer Assembly Parts List

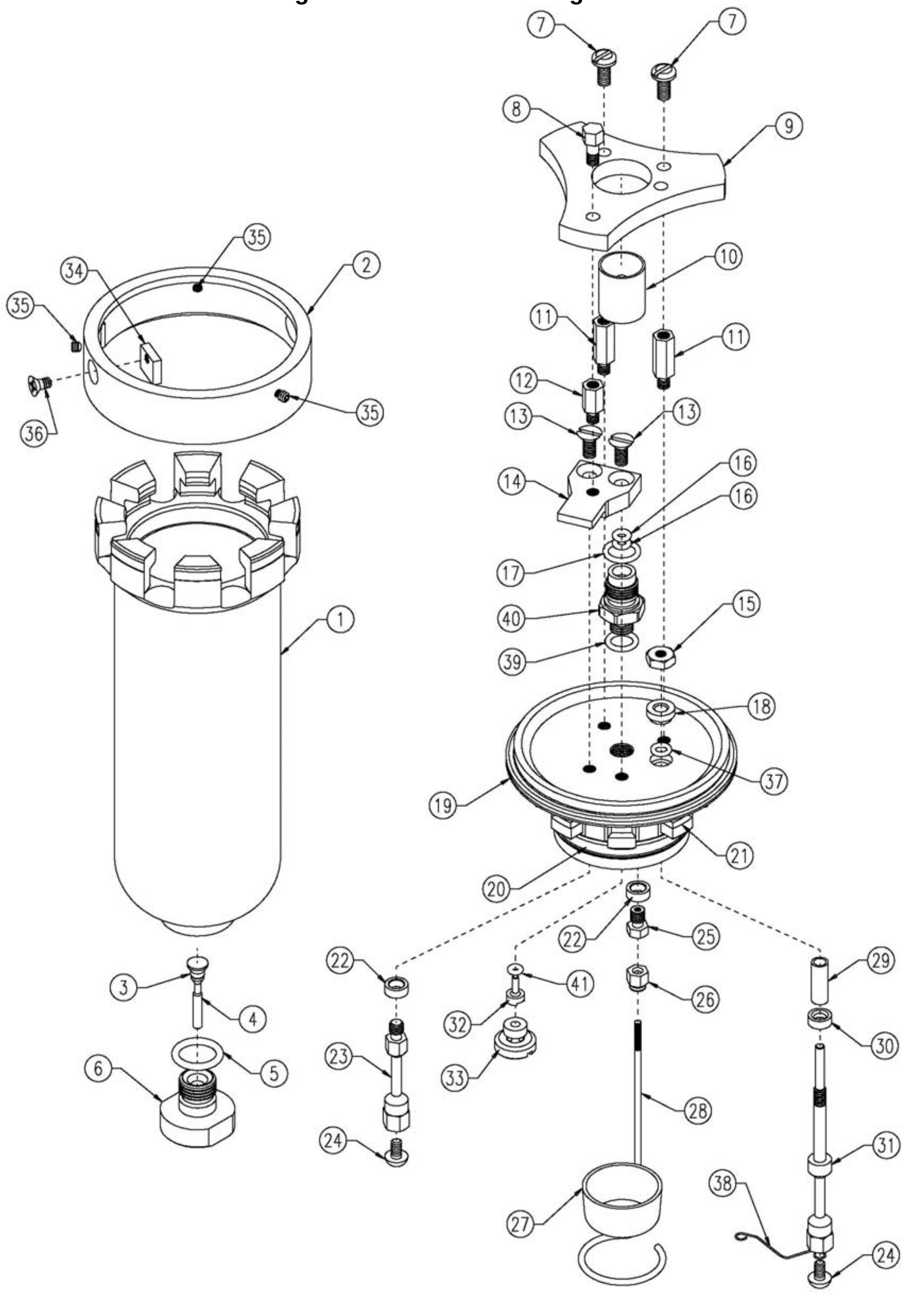
Item	Description
682DD	Snap Ring, Internal .50
683DD	Wave Spring, .50 OD
684DD	Ball Bearing, .50 OD
690DD	V-Seal, Nitrile
715HC	O-Ring NBR 1-1/4 ID
954DD	Propeller
1029DD	Baffle Assembly
1242DD2	Pulley, Timing
SA1140RD04	4-40 X ¼ RHMS 18-8 SS
SN1140HLHJ	Nut, 4-40 Hex Lock

A1266DD Cover Assembly Parts List

Item	Description
Contact Pin Assembly	
986DD	Pin Contact
987DD	Block Contact Pin
988DD	Spring Compression
989DD	Snap Ring External .219"
1021DD	Bushing 0.125 ID
SN1332HX	6-32 Hex Nut 18-8 SS
A1400DD	Plunger / Knob Assembly
1218DD	Cover, Air
1229DD3	Plate
1237DD2	Friction Hinge
1396DD	Latch Block
1324DD2	Post, Latch Locking
A1230DD2	Cover / Tubing Assembly

DRAWINGS

Figure 10 - 1138 Parts Diagram



1138 Parts Diagram Key

Key No.	Item	Description
1	888DD2	Cylinder
2	889DD	Outer Ring
3	821DD	O-Ring 1/16 ID NBR
4	668DD	Check Valve
5	882DD	O-Ring 1/2 ID NBR
6	925DD	Bomb Retainer
7	SA1632RD06 (2)	8-32 x 3/8 RHMS
8	902DD	Ground Stud Head
9	899DD	Head Handle
10	1454DD	Funnel
11	904DD (2)	Standoff 8-32 x 5/8 M-F
12	905DD	Standoff 8-32 x 3/8 M-F
13	SA1632FT06 (2)	8-32 x 3/8 FHMS
14	898DD	Locator Cap
15	SN1632HX	8/32 Hex Nut
16	1374HCJV (2)	O-Ring 1/8 ID FKM
17	394HC	O-Ring 3/8 ID FKM
18	663DD	Contact Bushing
19	1071DD	Quad Ring 2.88 ID NBR
20	1444DDJB	O-Ring 1-5/8 ID NBR
21	1452DD	Head
	1452DDCL	Head for Chlorine Service
22	655DD (2)	Electrode Spacer
23	1095DD	Electrode
24	PA1332RD04 (2)	6-32 x 1/4 RHMS
25	656DD	Reducer Bushing
26	653DD	Electrode Nut
27	43AS	Capsule
28	906DD	Capsule Holder
29	658DD	Insulator
30	654DD	Electrode Washer
31	1094DD	Electrode
32	643DD	Check Valve
33	645DD	Water Diffuser
34	647DD	Anti-Rotator
35	SC1332SC02 (3)	6-32 x 1/8 SHSS
36	SA1332FP04	6-32 x 1/4 FHMS
37	659DD	O-Ring 5/32 ID NBR
38	840DD2	60” Ignition Wire (2” per use)
39	694DD	O-Ring 5/16 ID NBR
40	1453DD	Adapter, Bomb Cap
41	519AJV	O-Ring, FKM 5/64 ID x 1/16CS
	Item	**Complete Assemblies**
	A1450DD	Oxygen Bomb Head Assembly
	A1450DDCL	Oxygen Bomb Head Assembly for Chlorine Service
	A890DD2	Cylinder Assembly
	A890DD2CL	Cylinder Assembly for Chlorine Service

Figure 11 - 6400 Cutaway Right

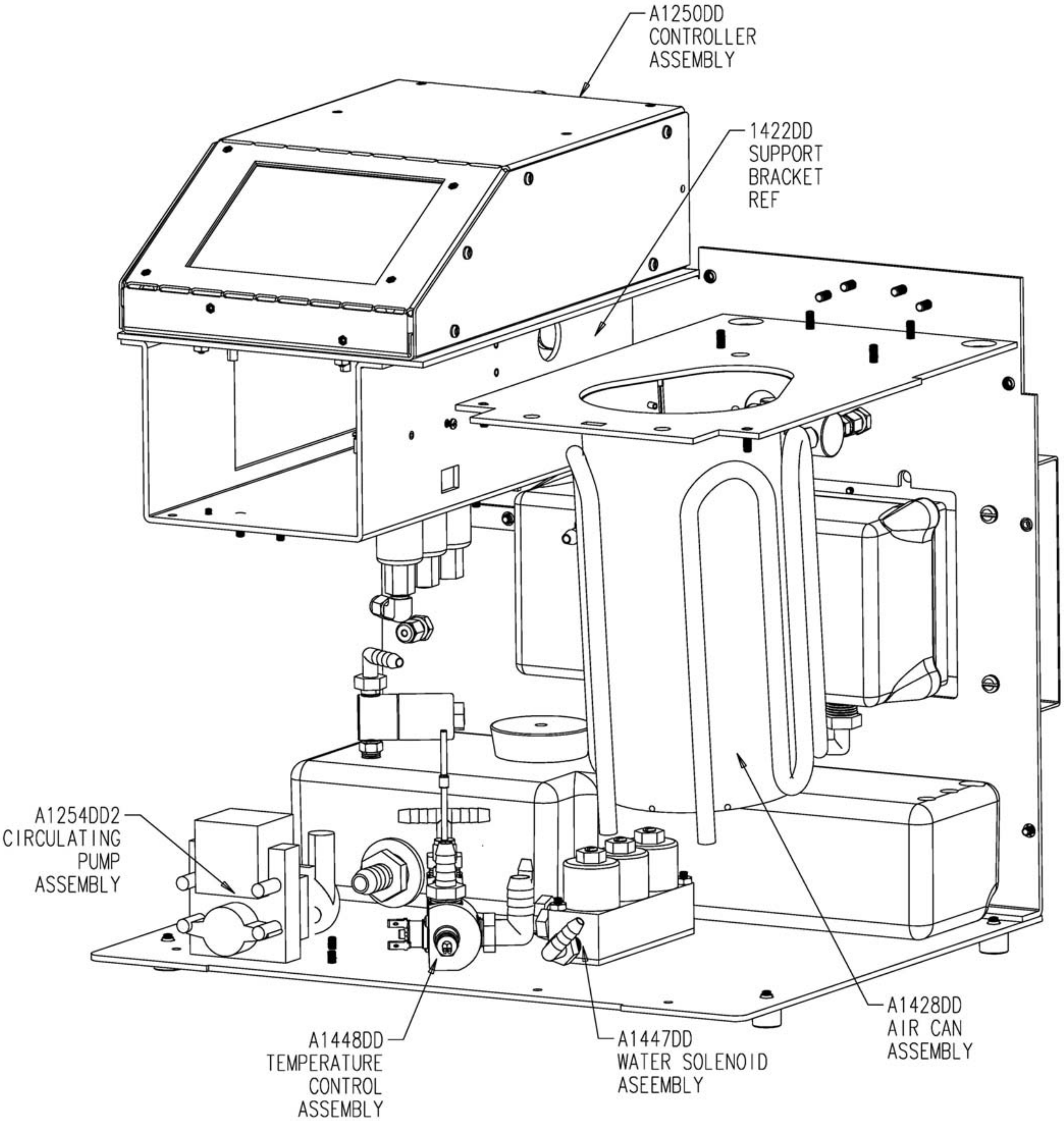


Figure 12 - 6400 Cutaway Left

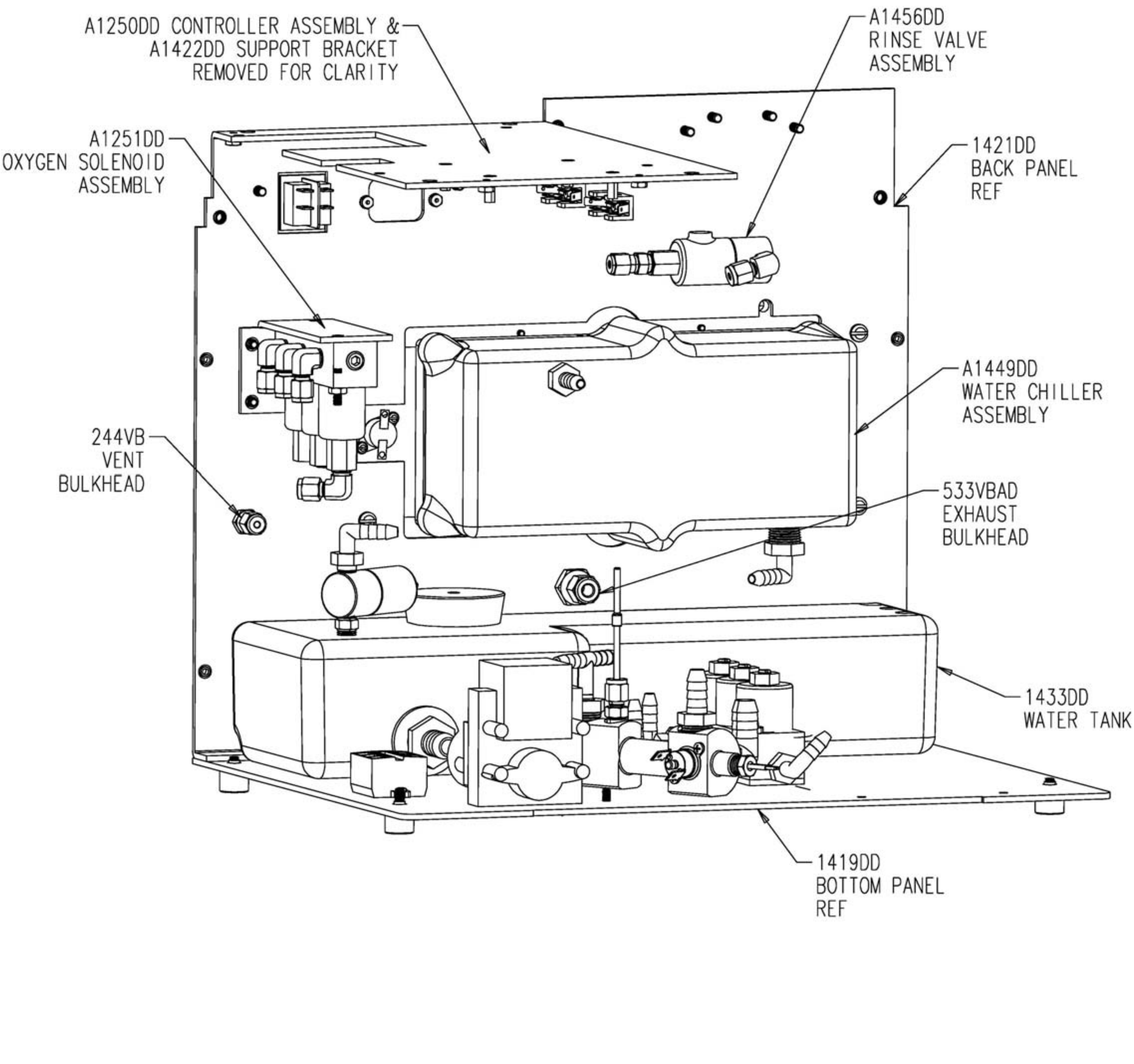


Figure 13 - 6400 Cover Open

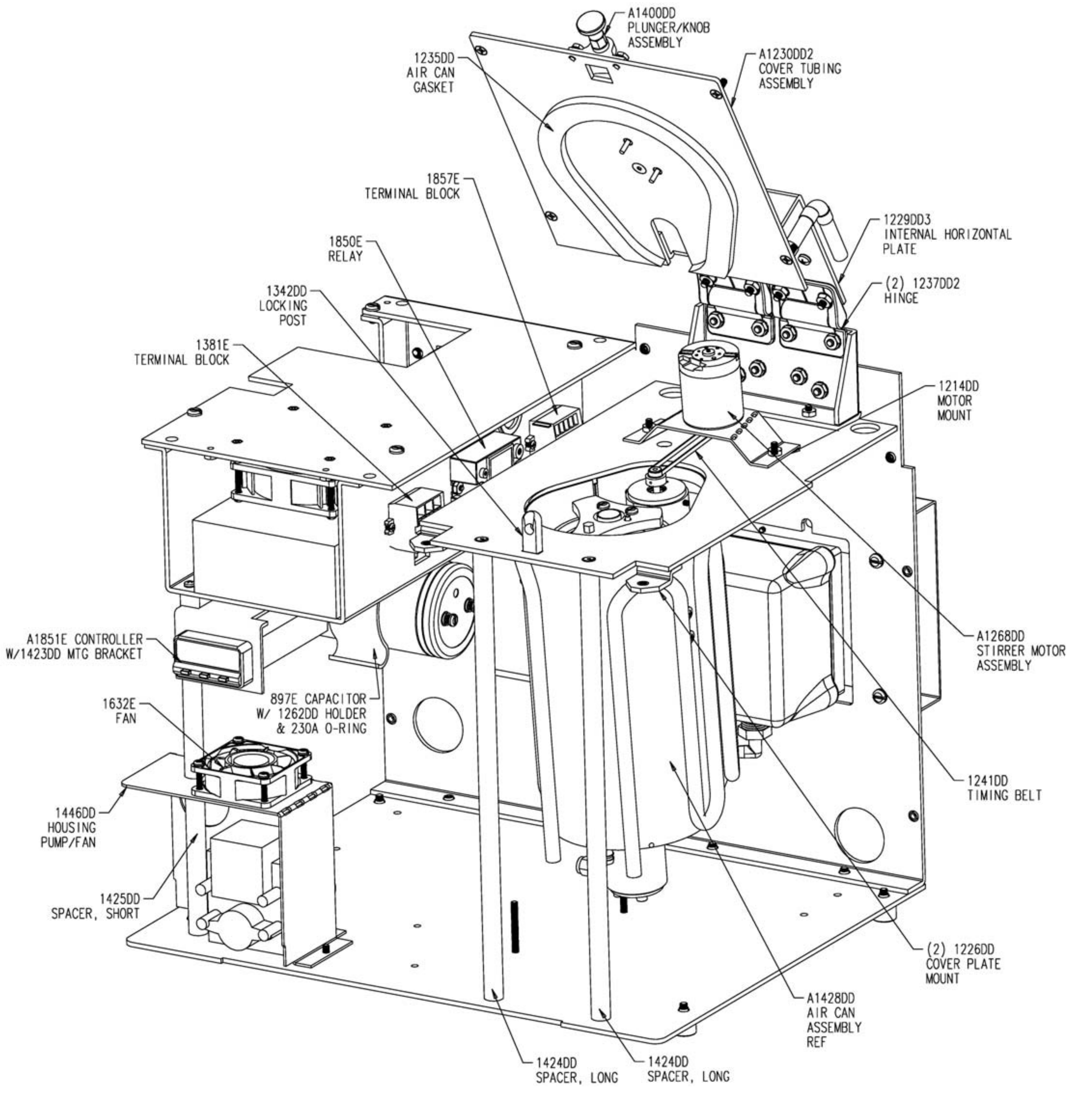


Figure 14 - A1250DD Control Schematic

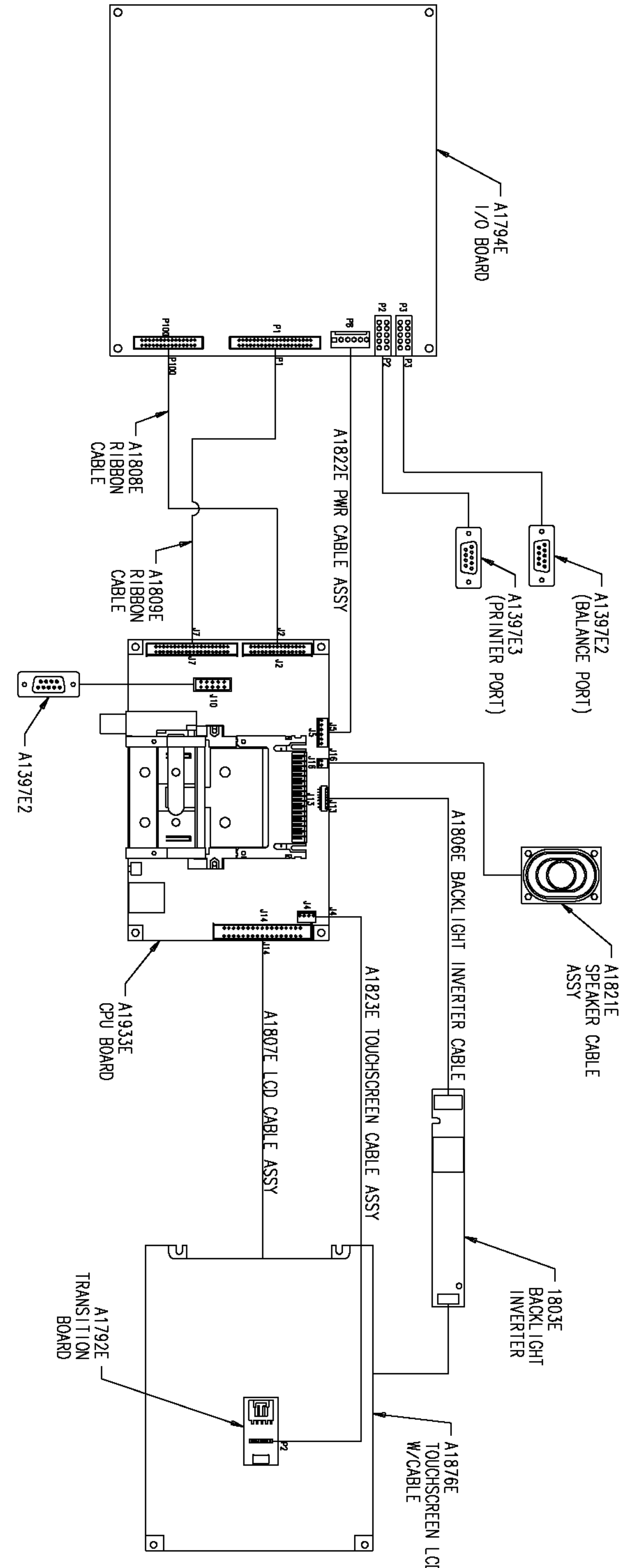


Figure 15 - A1251DD Oxygen Solenoid Assembly

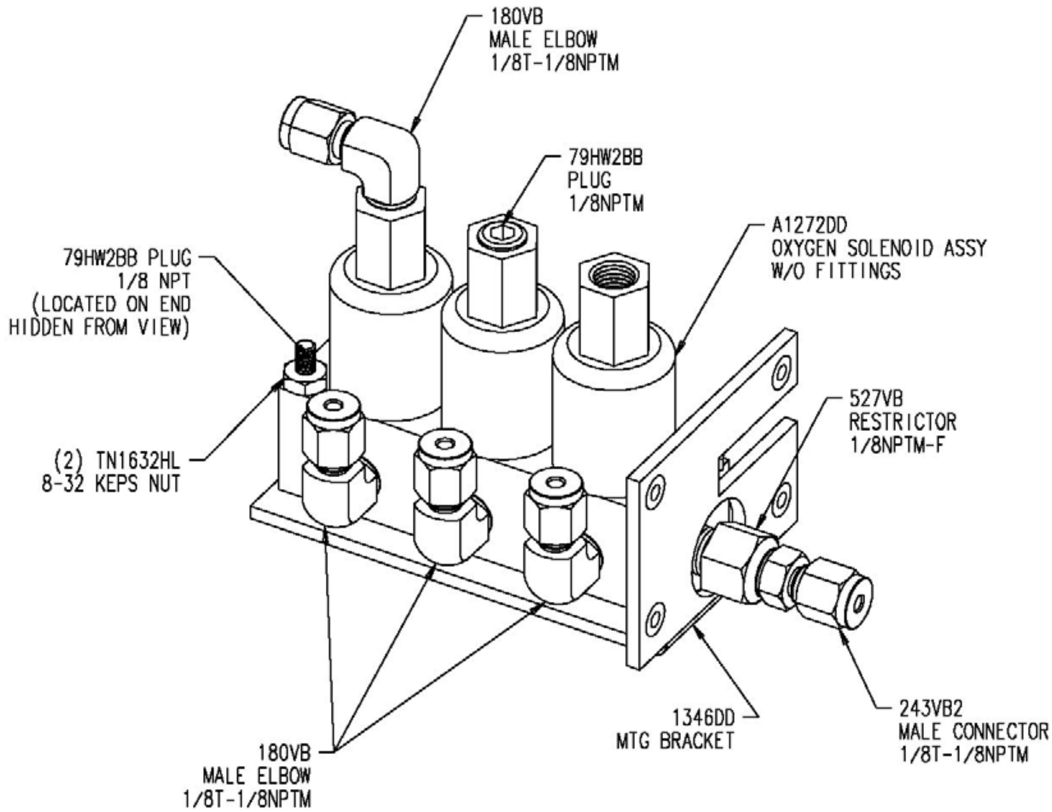


Figure 16 – A1447DD Water Solenoid Assembly

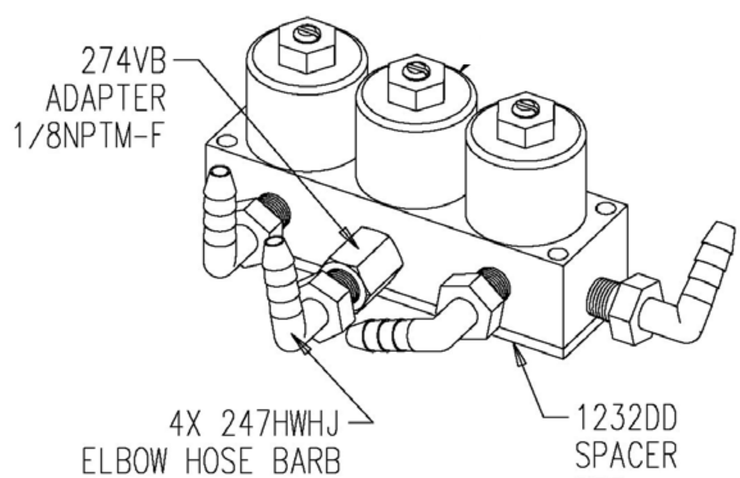


Figure 17 – A1435DD Rinse Valve Assembly

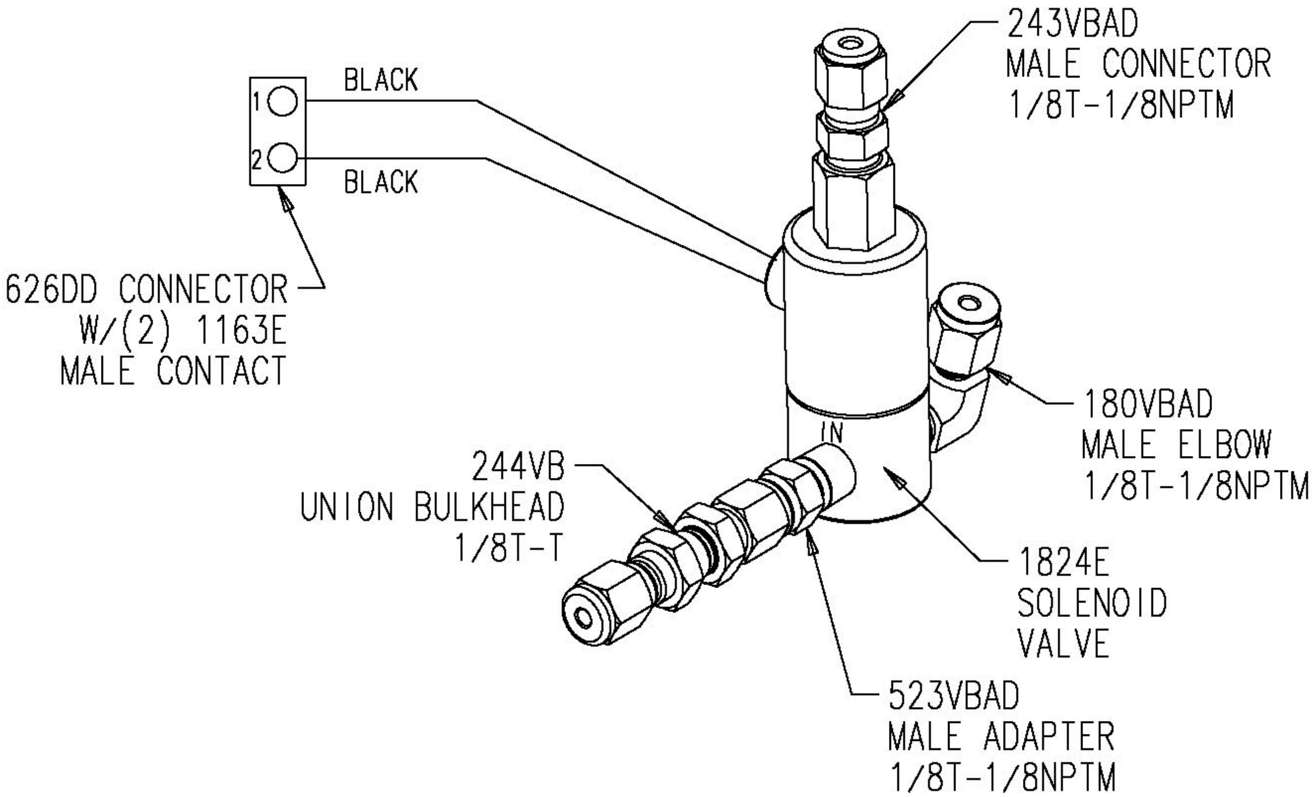


Figure 18 - 6400 Internal Plumbing Diagram

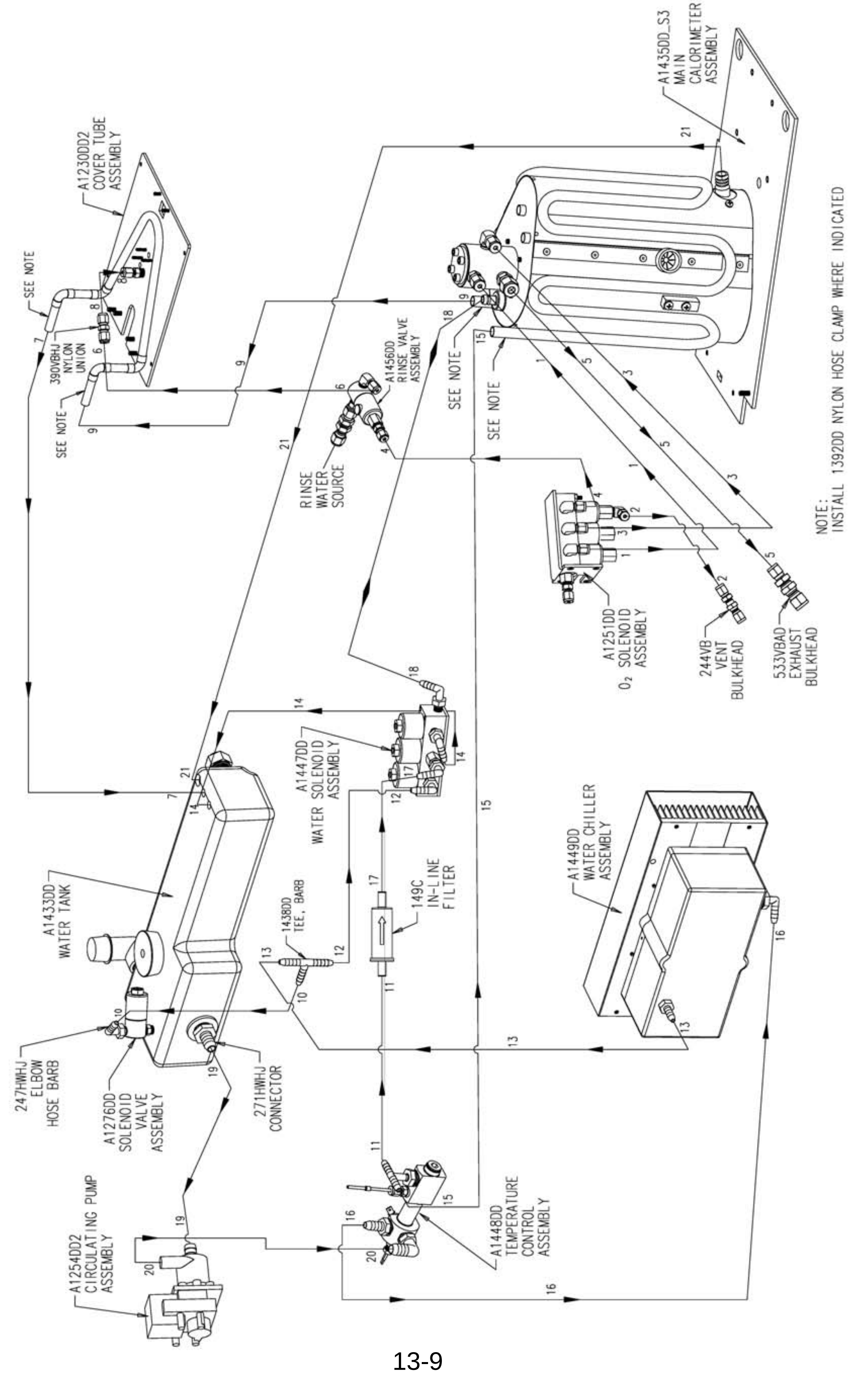


Figure 19 - 6400 Water Tank and Jacket Cooling Solenoid

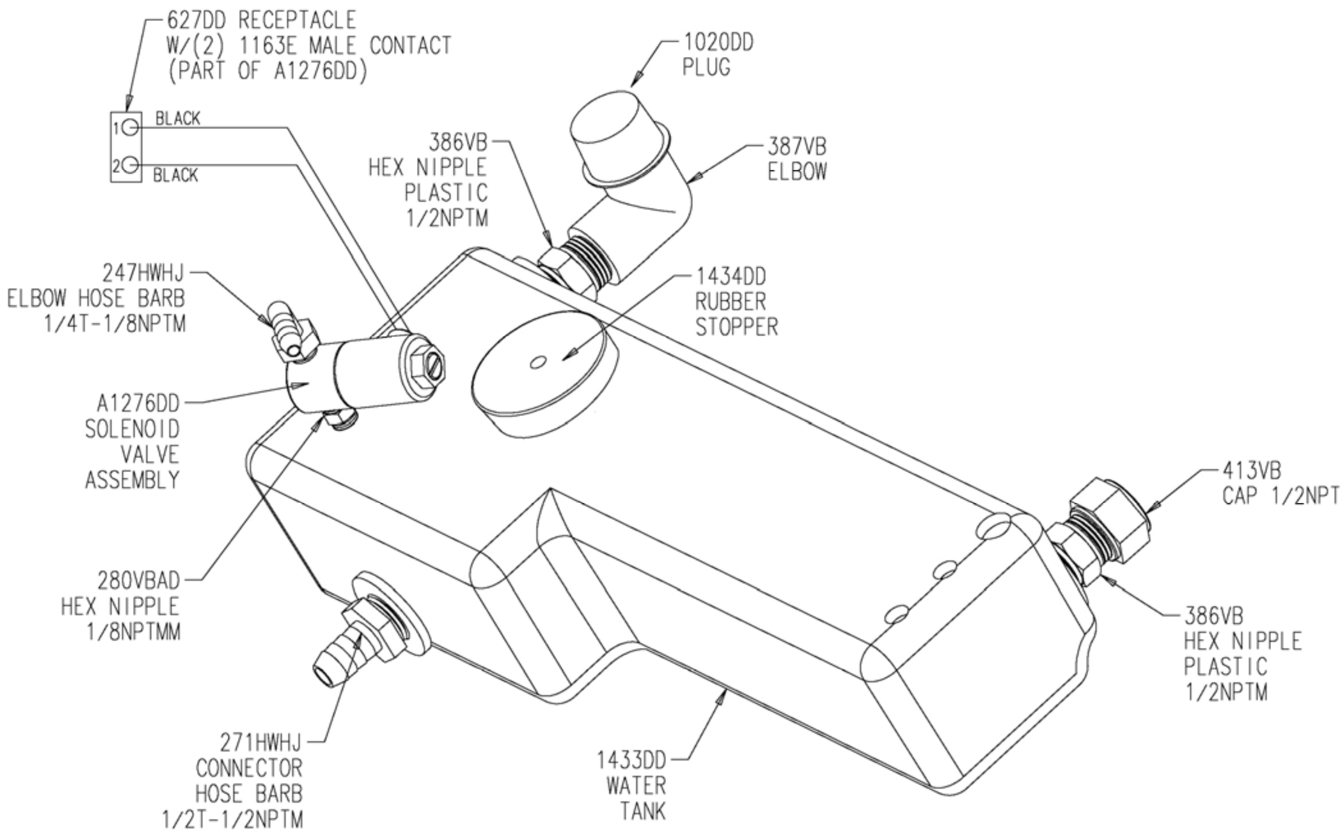
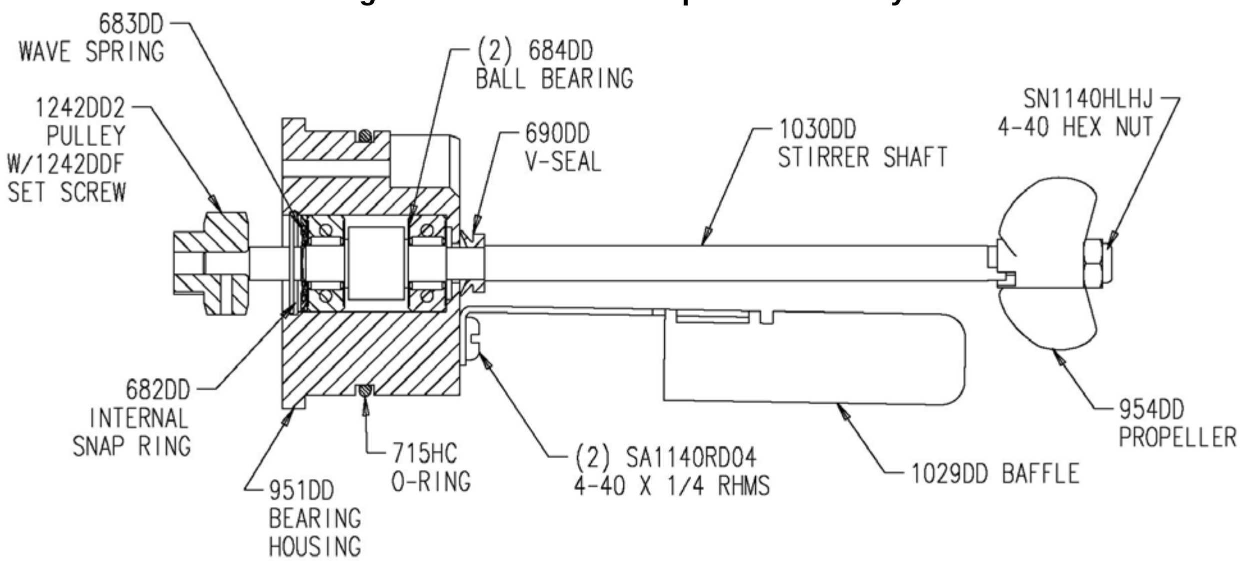


Figure 20 – A1455DD Propeller Assembly



Apply thread sealant (Locktite or equivalent) to set screw in 1242DD2 pulley before installing.

Figure 21 – A1448DD Temperature Control Assembly

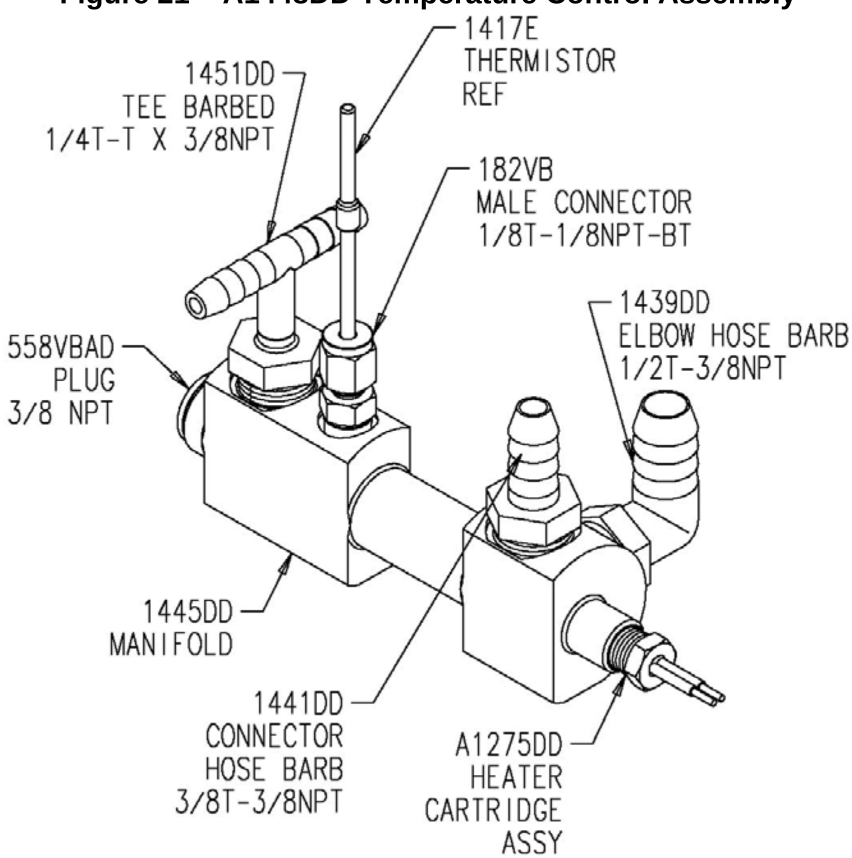


Figure 22 - A1268DD Stirrer motor and Mount

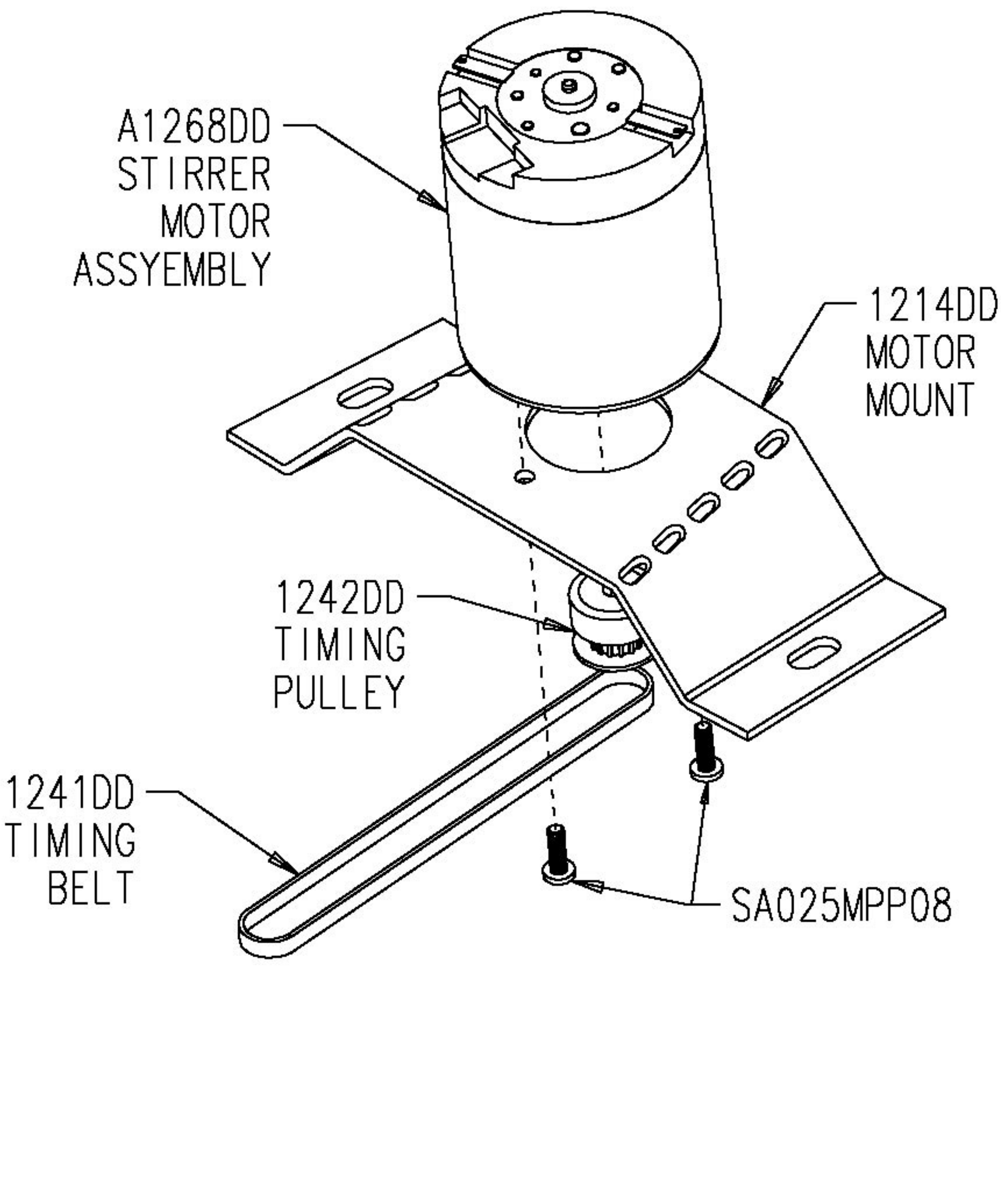


Figure 23 – 6400 Bucket Assembly

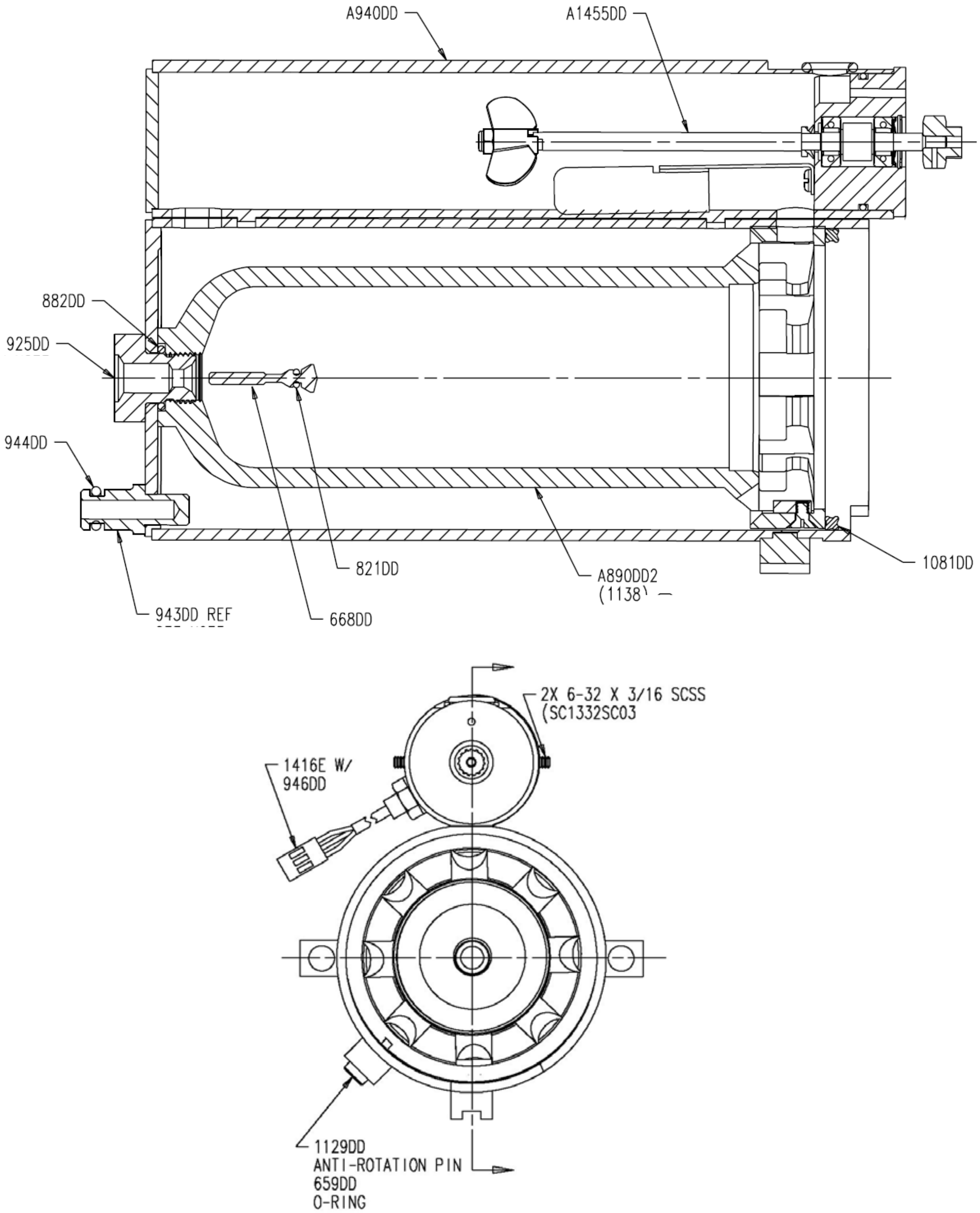


Figure 24 - 6400 Air Can Assembly, Cutaway Left

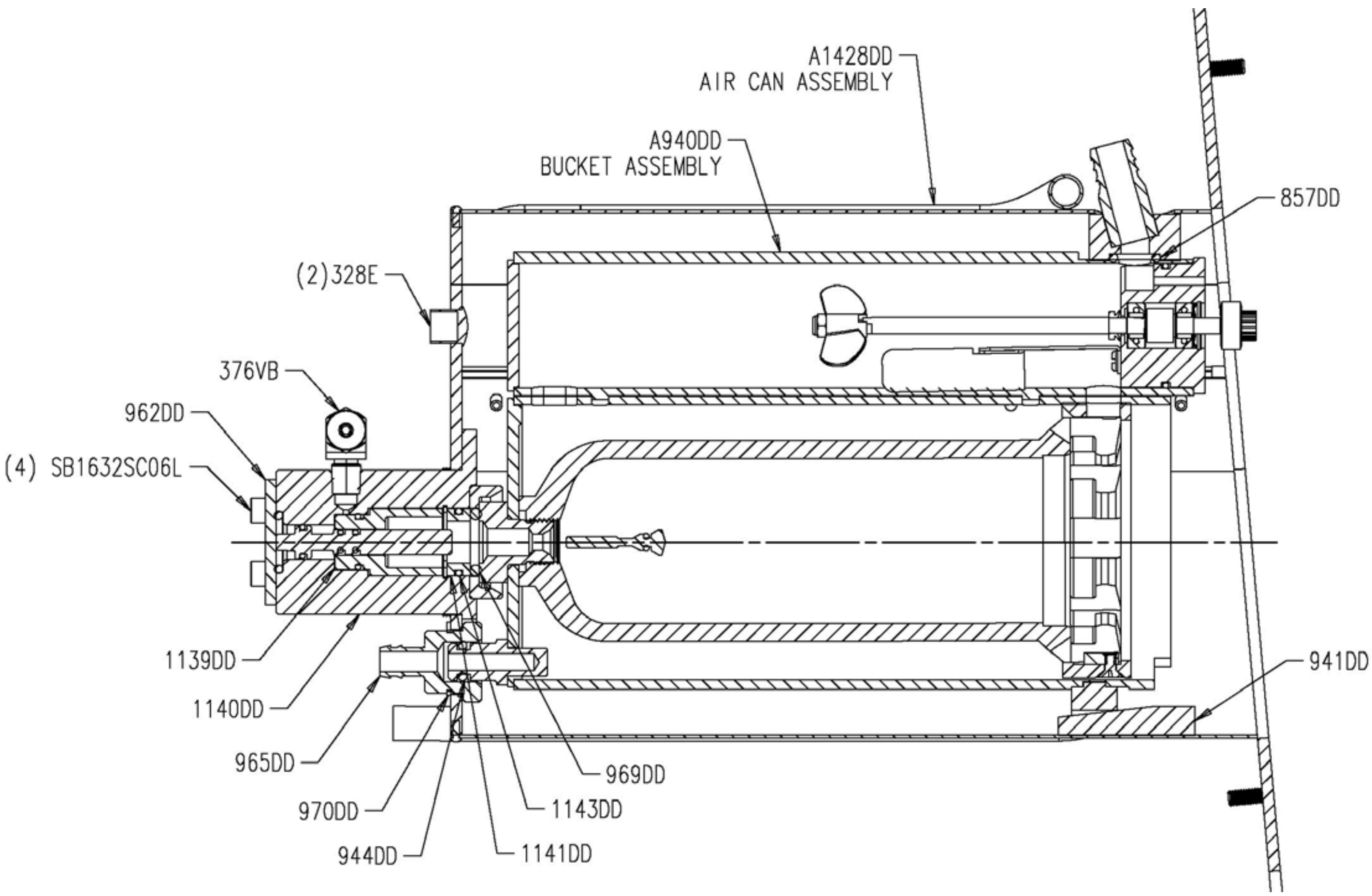


Figure 25 - Air Can Assembly, Cutaway Front

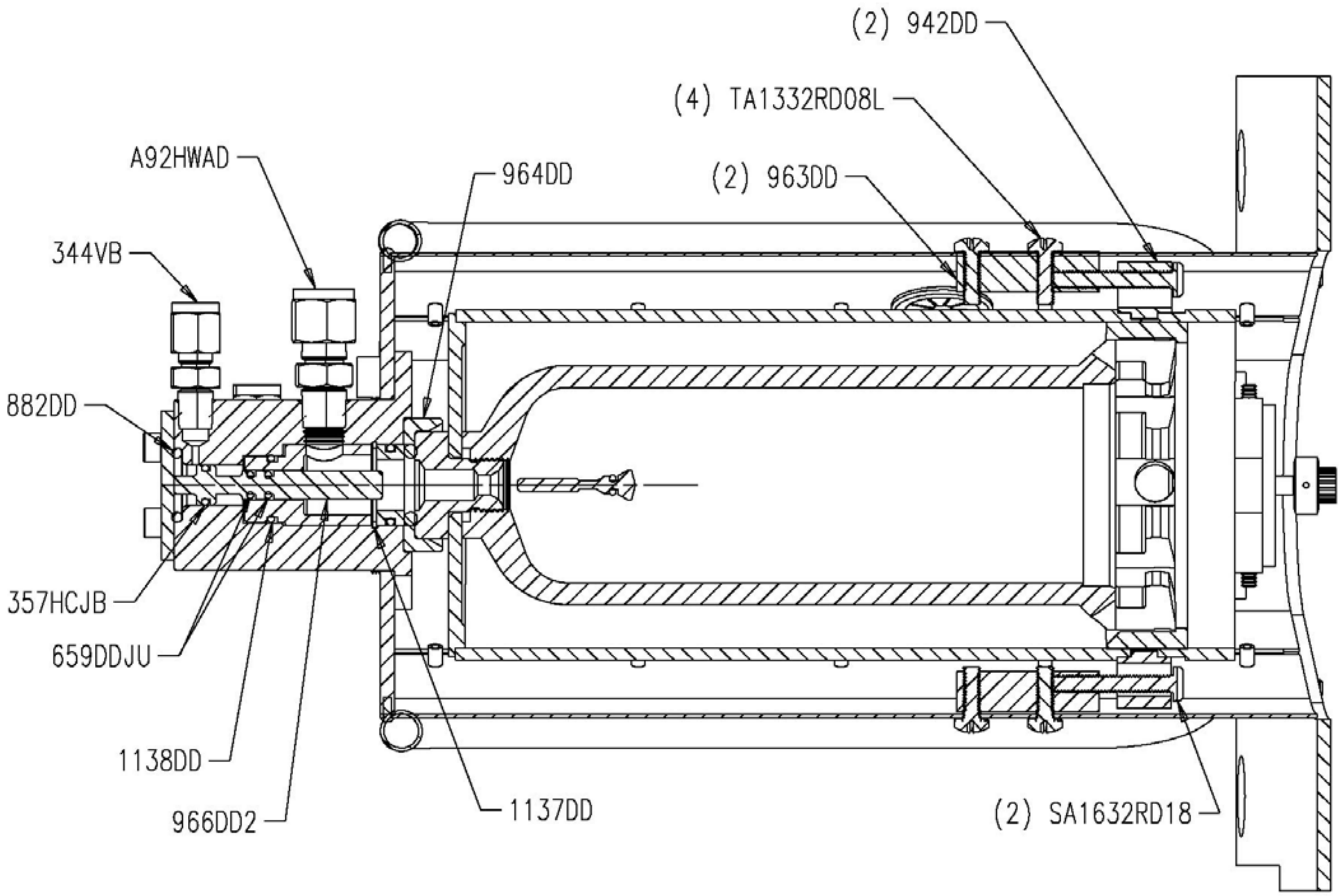


Figure 26 - A1450DD Bomb Head Assembly, View 1

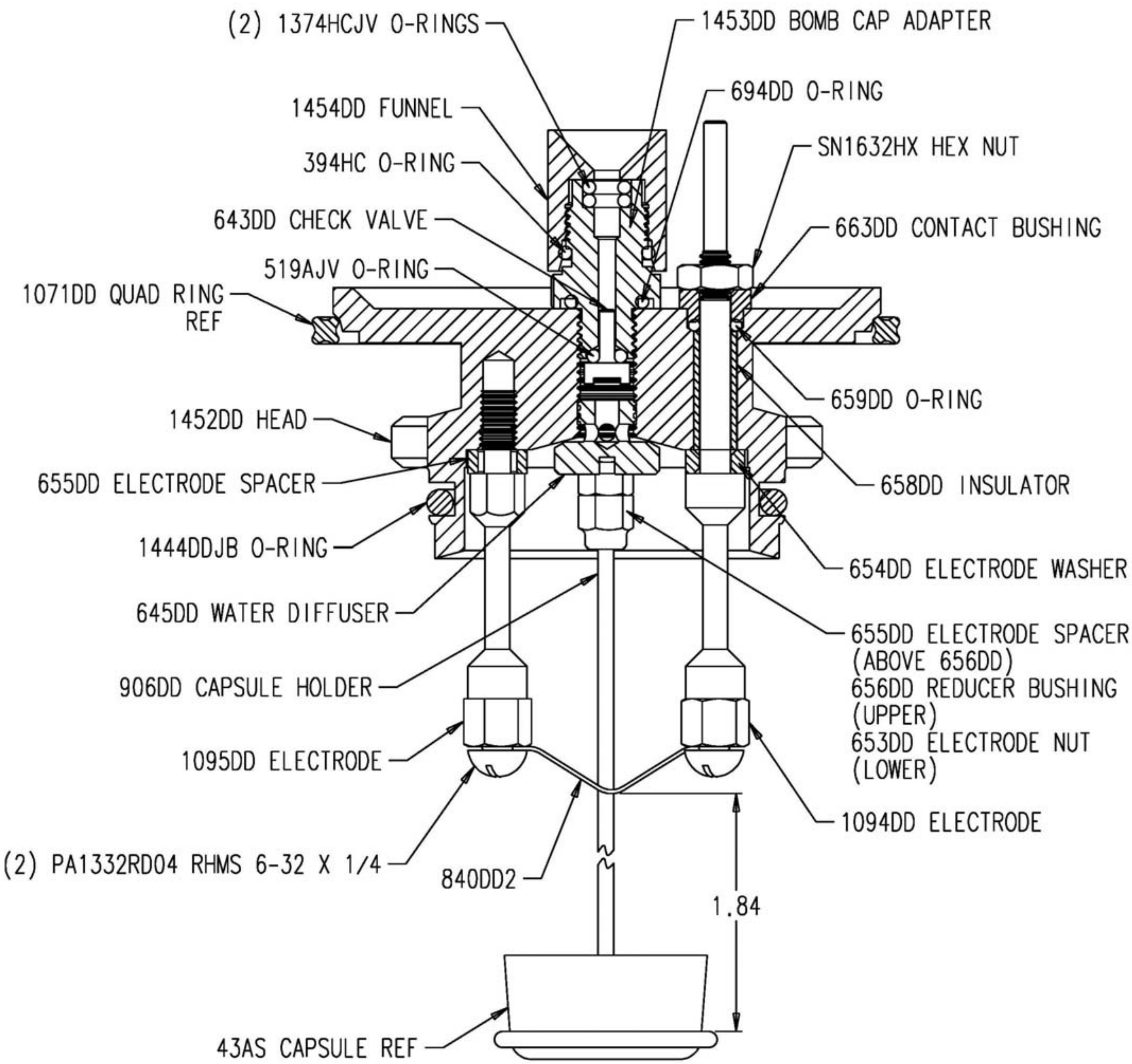


Figure 27 - A1450DD Bomb Head Assembly, View 2

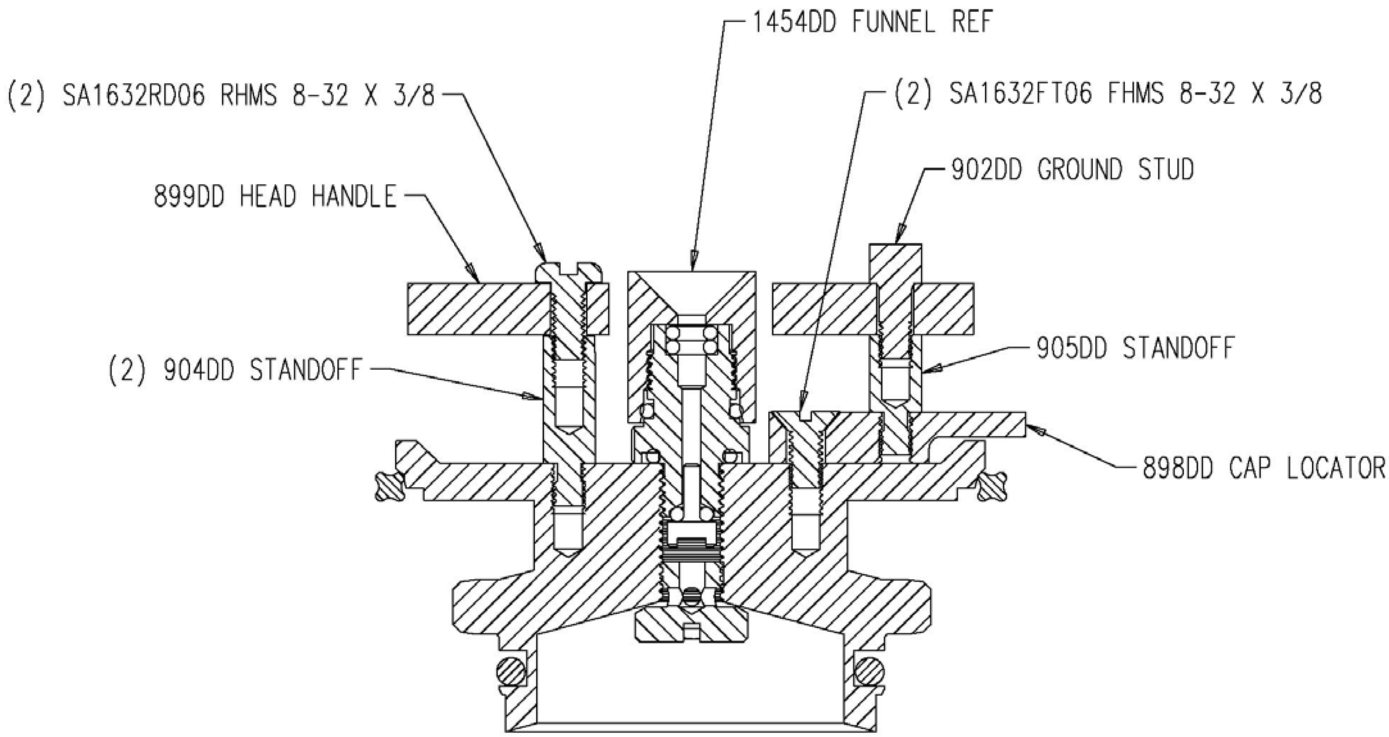


Figure 28 – A1050DD Rinse Collection Assembly

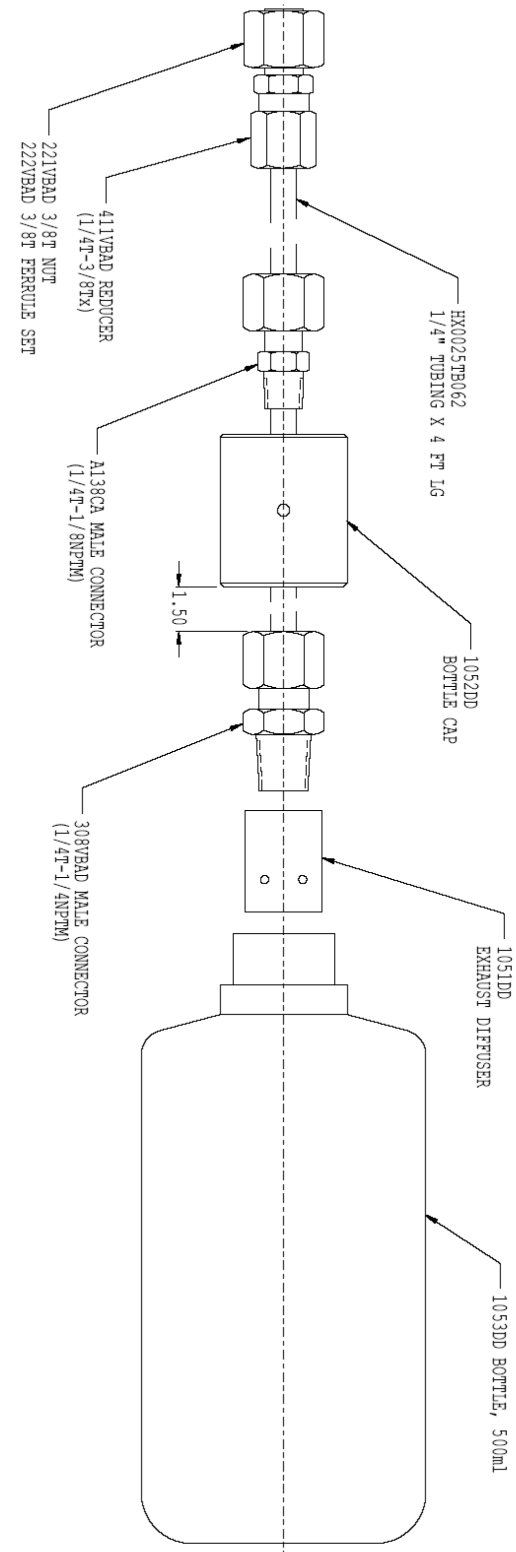


Figure 29 - Wiring Diagram

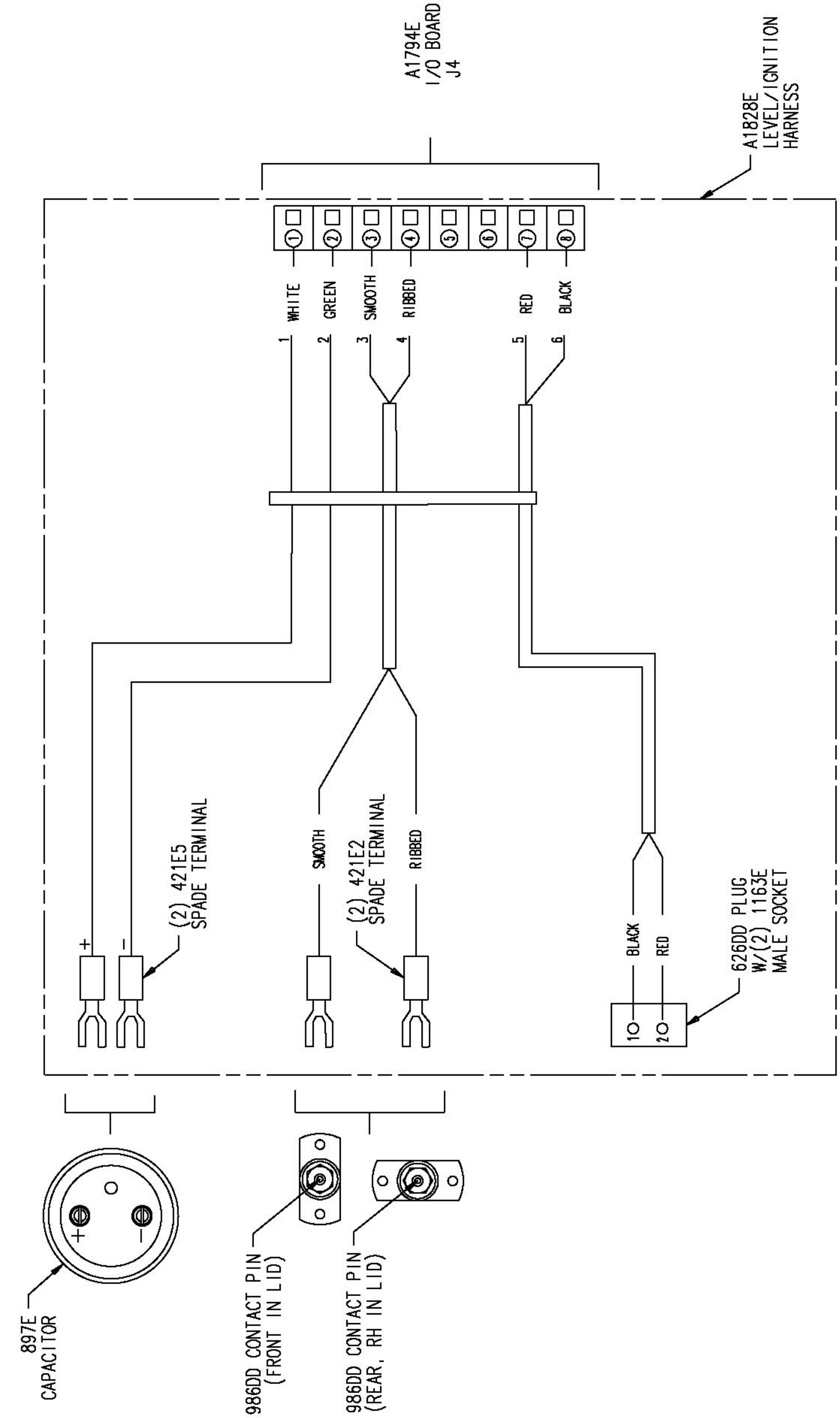


Figure 30 - Wiring Diagram

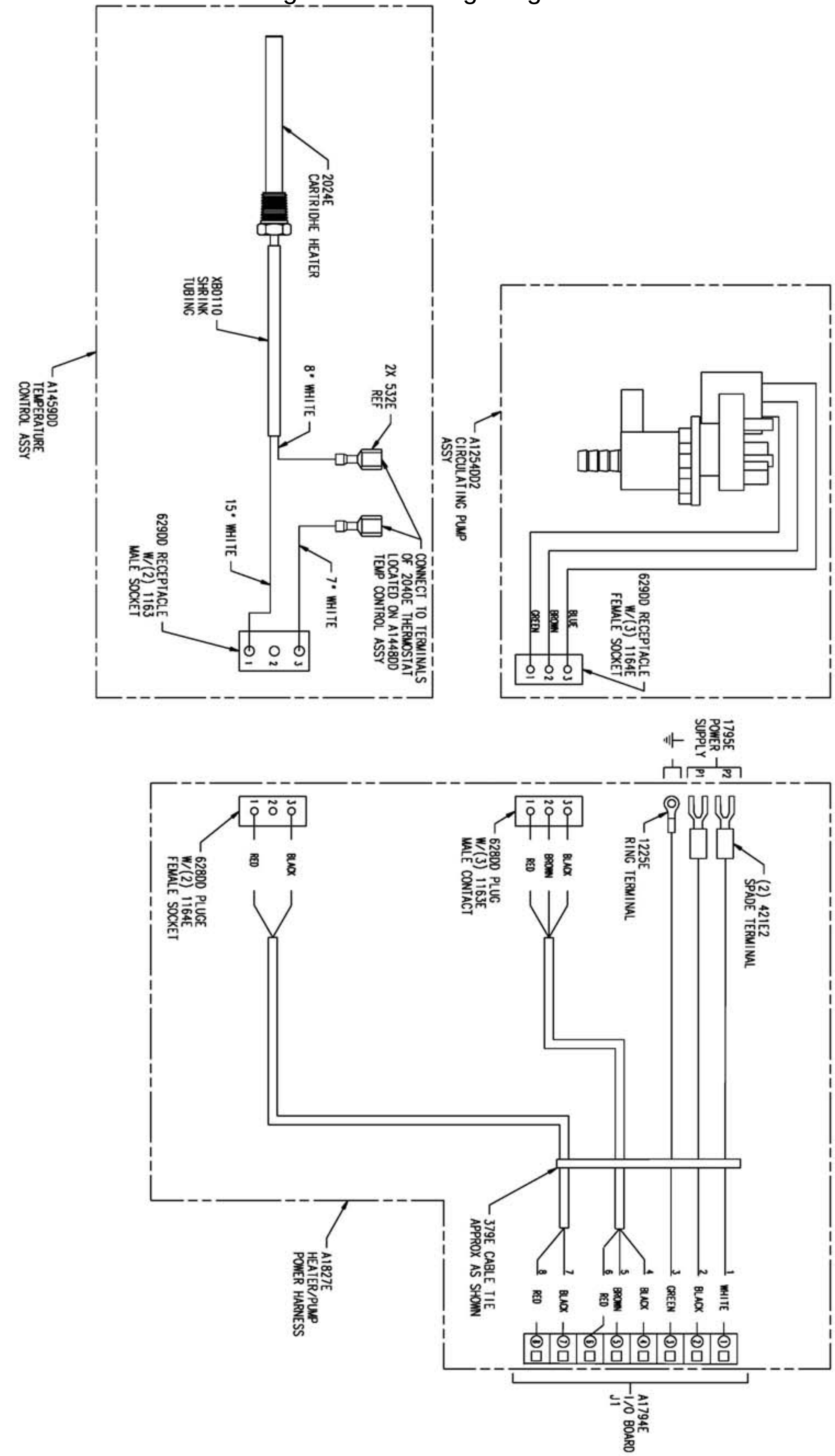
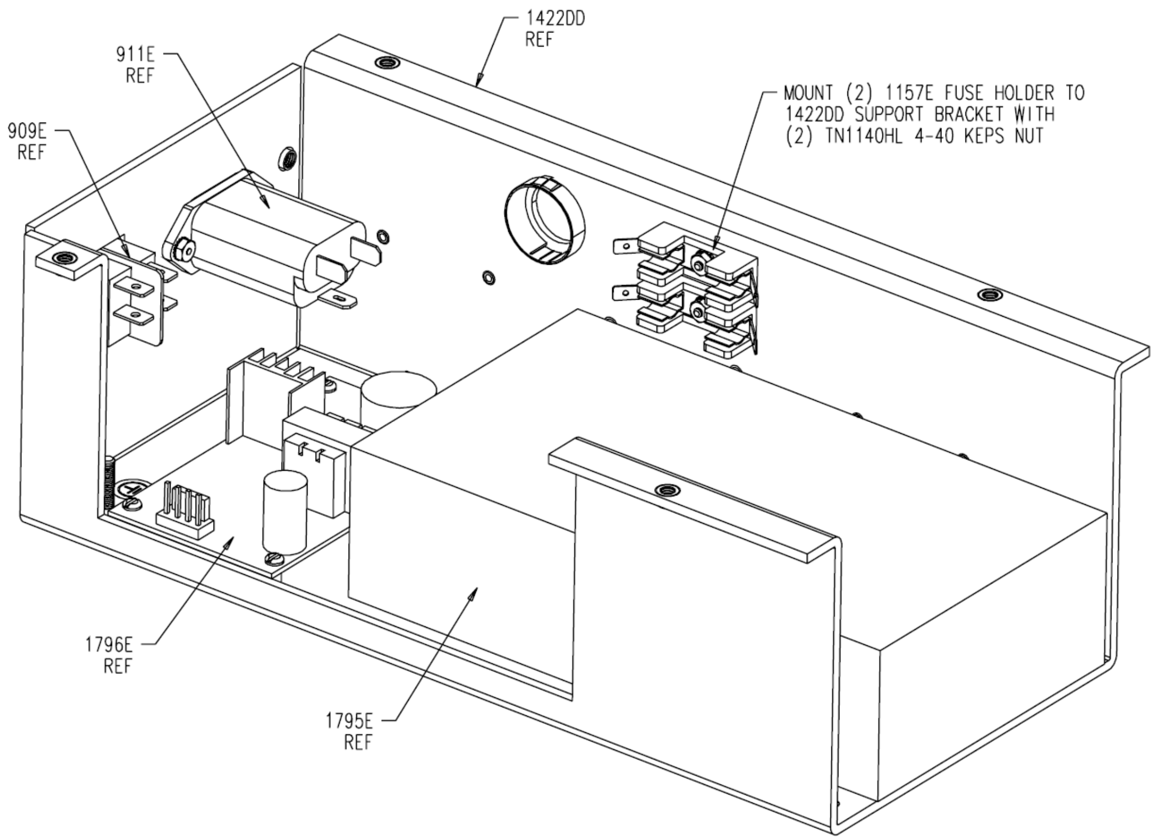


Figure 31 – Fuse Diagram



TABLES

Table 2 - Default Settings

Calorimeter Operation

Operating Mode	Determination
Bomb Installed/EE	1/940
Heater and Pump	OFF

Operating Controls

Method of Operation	Dynamic
Reporting Units	btu/lb
Use Spiking Correction	OFF
“OTHER” Multiplier	4.1868
Calibrate Touchscreen	
LCD Backlight Timeout(s)	1200 S
LCD Contrast	30%
Print Error Messages	ON
Language	English

Spike Controls

Use Spiking	OFF
Heat of Combustion of Spike	6318.4
Use Fixed Spike	OFF
Weight of Fixed Spike	0.0
Prompt for Spike before Weight	OFF

Bomb Rinse Tank Control

Report Rinse Tank Empty	ON
Rinse Tank Capacity	150
# Rinses Left	150
Reset Rinse Tank Counter	
Rinse Time	25
Rinse Flush Time	20
Clear Time	100
# of Rinse Cycles	3

Program Information and Controls

Date	XX/XX/XXXX
Time	XX:XX
Software and Hardware Info	
Settings Protect	OFF
User/Factory Settings	
Feature Key	
Bomb Type Select	
User Function Setup	
Cold Restart	

User Factory Settings

User Setup ID	64-1138
Reload Factory Default Settings	
Reload User Default Settings	
Save User Default Settings	

Calibration Data & Controls

Calibration Run Limit	10
EE Max Std Deviation	0.0
Heat of Combustion of Standard	6318.4
Bomb Service Interval	500
Use Bomb	1

Bomb 1 Through 4

EE Value	940
Protected EE Value	OFF

Thermochemical Corrections

Standardization

Fixed Fuse Correction	ON 50.0
Fixed Acid Correction	ON 8.0
Fixed Sulfur Correction	ON 0.0

Determination

Fixed Fuse Correction	ON 50.0
Fixed Acid Correction	ON 8.0
Fixed Sulfur Correction	OFF 0.0
Calculate Net Heat of Combustion	OFF

Calculation Factors

Acid Value is Nitric Acid Only	ON
Acid Multiplier	0.0709
Sulfur Value is Percent	ON
Sulfur Multiplier	0.6238
Fuse Multiplier	1.0
Use Offset Correction (ISO)	OFF
Offset Value	0.0
Fixed Hydrogen	OFF 0.0
Hydrogen Multiplier	50.68
Dry Calculation	OFF

Dry Calculation

Dry Calculation	OFF
Fixed Moisture	OFF 0.0%
Moisture Multiplier	5.83

Table 3 - Default Settings

Data Entry Controls	
Prompt for Bomb ID	ON
Weight Entry Mode	Touch Screen
Acid Entry Mode	Touch Screen
Hydrogen Entry Mode	Touch Screen
Auto Sample ID Controls	ON
Sample Weight Warning above	2.0
Spike Weight Entry Mode	Touch Screen
Sulfur Entry Mode	Touch Screen
Moisture Entry Mode	Touch Screen
Auto Preweigh Controls	ON

Auto Sample ID Controls	
Automatic Sample ID	ON
Automatic Sample ID Number	1
Automatic Sample ID Increment	1

Auto Preweigh Controls	
Automatic Preweigh ID	ON
Automatic Preweigh ID increment	1
Automatic Preweigh ID Number	1

Reporting Controls	
Report Width	40
Automatic Reporting	ON
Auto Report Destination	Printer
Individual Printed Reports	OFF
Edit Final Reports	OFF
Recalculate Final Reports	OFF
Use New EE Values in Recalculation	OFF

Communication Controls	
Printer Port (RS232)	
Balance Port (RS232)	
Network Interface	
Printer Destination	Local (RS232)
Bar Code Port (RS232)	
Network Data Devices	

Printer Port Communications	
Number of Data Bits	8
Parity	None
Number of Stop Bits	1
Handshaking	Xon/Xoff
Baud Rate	9600
Printer Type	Parr 1757

Balance Port Communications	
Balance Type	Generic
Customize Balance Settings	

Balance Port Settings	
Number of Data Bits	8
Parity	None
Number of Stop Bits	1
Handshaking	None
Baud Rate	9600
Data Characters from Balance	8
Data Precision	4
Transfer Time Out	10
Balance Handler Strings	

Data Logger	
Data Logger	OFF
Interval in Seconds	12
Data Log Destination	Log File and Printer
Data Log Format	Text
Data Log Format	Text

Table 4 - ISO & BSI Method Settings

Page	Line	Setting	Value
Thermochemical Corrections	Fixed Acid STD	Off	13
	Fixed Sulfur STD	Off	7
	Fixed Acid DET	Off	13
	Fixed Sulfur DET	Off	7
Calculation Factors	Acid is Nitric Only	On	
	Acid Multiplier		0.154
	Sulfur Value is Percent	Off	
	Sulfur Multiplier		0.1
	User Offset Correction	On	
	Offset Value		-43.5

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