



Parr Instrument Company

6300

Oxygen Bomb Calorimeter

Operating Instruction Manual

For models produced after October 2010



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PREFACE

SCOPE

This manual contains instructions for installing and operating the Parr 6300 Calorimeter. For ease of use, the manual is divided into nine chapters.

Concept of Operation

Installation

Instrument Description

Program Installation & Control

Operating Instructions

Corrections & Final Reports Reporting

Instructions

File Management

Maintenance & Troubleshooting

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 6300 Calorimeter have been included and made a part of these instructions.

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

Additional instructions for the printer, cooler, and water handling systems are found in the respective package and should be made a part of this book.

Note:

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

Customer Service:

Questions concerning the installation or operation of this instrument can be answered by the Parr Customer Service Department:

1-309-762-7716 • 1-800-872-7720 • Fax: 1-309-762-9453
E-mail: parr@parrinst.com • <http://www.parrinst.com>

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. 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.

CLEANING & MAINTENANCE

Periodic cleaning may be performed on the exterior surfaces of the instrument with a lightly dampened cloth containing mild soap solution. All power should be disconnected when cleaning the instrument.

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.

GENERAL SPECIFICATIONS

Electrical Ratings

120VAC, 6.0 Amps, 50/60 Hz

240VAC, 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: 15°C to 30°C; maximum relative humidity of 80% non-condensing. Installation Category II (over voltage) in accordance with IEC 664. Pollution degree 2 in accordance with IEC 664. Altitude Limit: 2,000 meters.

Storage: -25°C and 65°C; 10% to 85% relative humidity.

Provisions for Lifting and Carrying

Before moving the instrument, disconnect all connections from the rear of the apparatus. Lift the instrument by grabbing underneath each corner.

GETTING STARTED

These steps are offered to help the user become familiar with, install, operate and develop the full capabilities of the Parr 6300 Calorimeter.

1. Review the *Concept of Operations*, Chapter 1, to get an understanding of the overall capabilities of the calorimeter and microprocessor control.
2. Unpack and install the calorimeter in accordance with *Installation*, Chapter 2. This simple, step-wise procedure will acquaint the user with the various parts of the calorimeter and make it easier to understand the operating instructions which follow.
3. Turn the power switch ON (located on the back). Turn to the *Instrument Description*, Chapter 3, to review the touch screen controls.
4. Review the *Program Installation and Control*, Chapter 4, to match the factory settings to the intended mode of operation. Any required changes can be made to the program parameters located in the Main Menu.
5. Review the *Reporting Instructions*, Chapter 7, to become familiar with the manner in which calorimetry corrections are entered. Also discussed are generating final reports, editing and clearing memory.
6. Turn to the *Menu Operating Instructions*, Appendix A, to review the menu functions used to modify the program contained in the 6300 Calorimeter. A review of the menus will provide a good idea of the capabilities and flexibility designed into this instrument.
7. Review the *Calculations*, Appendix B. This provides information about calculations performed by the 6300 Calorimeter.
8. Review *Standardization*, Appendix C. This will serve two important functions. First, it provides instructions on generating the energy equivalent factor required to calculate the heat of combustion of unknown samples. Secondly, it will give the user the opportunity to run tests on a material with a known heat of combustion to become familiar with the instrument and confirm that the instrument and operating procedures are producing results with acceptable precision. Most 6300 Calorimeters will have an energy equivalent of approximately 940 calories per °C with an 1138 oxygen bomb (800 calories per °C with an 1136 oxygen bomb.) The runs for standardization and determinations are identical, except for the setting of the instrument to the standardization or determination mode.
9. Review the *Communication Interfacing*, Appendix D, for the correct installation of any peripherals connected to the 6300 Calorimeter.
10. After successful standardization, the 6300 Calorimeter should be ready for testing samples.

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CHAPTER 1

CONCEPT OF OPERATION

A HIGHLY AUTOMATED PROCEDURE

Parr proudly introduces a new Oxygen Bomb Calorimeter, No. 6300, in which new technology is combined with time-proven calorimetric techniques to produce a completely automatic system for measuring the heat of combustion of solid and liquid fuels, combustible wastes, foods, feeds and other oxygen combustible materials. This new approach to bomb calorimetry results in a remarkable simplification of the steps required for a calorimetric test without compromising the need for complete combustion, rapid heat flow and precise thermometry which are essential in a combustion calorimeter.

In the 6300 Oxygen Bomb Calorimeter most of the manual operations in conventional bomb calorimetry have been eliminated by a new technology centered around a semi-automatic bucket handling mechanism and an automatic bomb filling, venting and rinsing design. To perform a test the user simply loads a sample into a holder, attaches a short auxiliary fuse, places the head into the cylinder, seals with a 1/16 of a turn, closes the cover and presses the START key to begin the procedure.

NEW CONVENIENCE AND NEW TECHNOLOGY

The 6300 Calorimeter represents a blending of some new unique design features with some long proven Parr calorimetric technology to dramatically simplify the user's tasks during a calorimetric determination.

In this new design the bomb cylinder and bucket are mounted in the calorimeter. The bomb is completely surrounded by a bucket chamber, sealed co-axially with the bomb head. After the bomb and bucket are closed and sealed, the bomb is filled with oxygen, the bucket chamber is filled with water, initial equilibrium is established, the bomb is fired and the temperature rise is monitored and recorded - all under automatic microprocessor control. Then, at the completion of a test, automatic control releases the residual pressure in the bomb, rinses the bomb, cools the system and empties the bucket.

These new mechanical features support an established technology in which water is circulated around the bomb to bring all inner parts of the calorimeter to a uniform temperature rapidly, while true isoperibol operating conditions are maintained by an outer water jacket. Microprocessor based, real time heat leak corrections are applied to implement the isoperibol jacketing method and to support the Parr rapid dynamic method for predicting the final temperature rise. Precise temperature measurements are made with thermistor thermometry providing 0.0001°C resolution over the operating range of the calorimeter.

In addition to handling all test sequence operations, the microprocessor makes all calculations and reports and stores all results, as provided in earlier Parr isoperibol and adiabatic calorimeters. A bright, backlit liquid crystal display, prompts the operator through all setup and operating steps with on-screen menus which make user training quite simple.

ISOPERIBOL OPERATION

In Isoperibol operation, the calorimeter jacket is held at a constant temperature while heat from the burning sample causes the bomb and bucket temperature to rise. The small heat flow between the bucket and its surroundings during a test is monitored by a microprocessor in the calorimeter, which continuously determines the effect of any heat leak and applies the necessary correction automatically. This system differs from adiabatic operation in which the jacket temperature must be adjusted continuously to match the bucket temperature in an attempt to maintain a zero temperature differential with no heat leaks between the bucket and its surroundings. Calorimetrists have long recognized the advantages of simplification and better precision obtainable with a well designed and executed Isoperibol system as opposed to the rapidly changing jacket temperature required in an adiabatic calorimeter.

DYNAMIC OPERATION

In its Dynamic Operating Mode, the calorimeter uses a sophisticated curve matching technique to compare the temperature rise with a known thermal curve to extrapolate the final temperature rise without actually waiting for it to develop. Repeated testing, and over 20 years of routine use in fuel laboratories, has demonstrated that this technique can cut the time required for a test by one-half without significantly affecting the precision of the calorimeter.

FULL MICROPROCESSOR BASED PROCESS CONTROL

The microprocessor controller in this calorimeter has been pre programmed to automatically prompt the user for all required data and control input and to:

- Generate all temperature readings in the calorimeter.
- Monitor jacket as well as bucket temperature.
- Confirm equilibrium conditions.
- Fire the bomb.
- Confirm that ignition has occurred.
- Determine and apply all necessary heat leak corrections.
- Perform all curve matching and extrapolations required for dynamic operation.
- Terminate the test when it is complete.
- Monitor the conditions within the calorimeter and report to the user whenever a sensor or operating condition is out of normal ranges.

- Complete freedom for reagent concentrations and calculations.
- Unlimited choice of reporting units.
- Automatic bomb usage monitoring and reporting.
- A choice of Equilibrium or Dynamic test methods.
- Automatic statistical treatment of calibration runs.
- Enhanced testing and trouble shooting procedure.

The 6300 Calorimeter is equipped with a USB connection plus an Ethernet port for direct communication with attached peripherals and a computer or network.

FULL MICROPROCESSOR BASED DATA ACQUISITION AND HANDLING

In addition to its process control functions, the microprocessor in the calorimeter has been pre programmed to:

- Collect and store all required test data.
- Apply all required corrections for combustion characteristics.
- Compute and report the heat of combustion for the sample.

FLEXIBLE PROGRAMMING

The fifth generation software built into this calorimeter and accessed through the screen menus permit the user to customize the operation of the calorimeter to meet a wide variety of operating conditions including:

- A large selection of printing options.
- Choice of accessories and peripheral equipment.
- Multiple options in regard to handling thermochemical corrections.
- Choice of ASTM or ISO correction procedures.
- A variety of memory management and reporting procedures.

CHAPTER 2

INSTALLATION

REQUIRED CONSUMABLES, UTILITIES AND POWER REQUIREMENTS

The 6300 Calorimeter System requires availability of Oxygen, 99.5% purity, with appropriate connection, 2500 psig, maximum.

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 $\pm 10\%$ of marked nominal voltage on the apparatus. The supply voltage receptacle must have an earth ground connection.

Approximately 4 liters of tap water, with a total hardness of 85 ppm or less, are required for filling the calorimeter jacket reservoir. This water is provided via the tap water connection at the rear of the calorimeter. The inlet pressure should be in the range of 20 to 60 psig. The required flow rate is on the order of 0.5 liters/minute. This connection also supplies cooling water for the calorimeter. As a result, the temperature of the water should not exceed 25 °C. The speed at which the calorimeter will recycle between tests is a function of the temperature of the incoming tap water. (The performance will slow noticeably above 20 °C and will become sluggish above 25 °C). Water consumption is dependent on the incoming water temperature and shouldn't normally exceed 1.5 liters per test.

An open water drain connection is required.

The power requirements for the subassemblies of the 6300 Calorimeter are:

- **Calorimeter**

5A @ 120VAC

3A @ 230VAC

- **Printer**

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

- **Printer Supplies**

334C Printer Paper

335C Printer Ribbon

INSTALLING THE CALORIMETER

Each Parr 6300 Calorimeter was completely assembled and thoroughly tested prior to shipment. The following stepwise procedure will guide the user through the installation process.

- 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. The calorimeter needs to be located near a water drain. A cold-water tap water supply, oxygen and an electrical outlet are also required. Set the calorimeter on a sturdy, level, bench or table, free from drafts, vibration and sources of radiant heat.
- Make the calorimeter drain connection using the provided 7/8" Tygon tubing (assembly A1336DD). The calorimeter must be located so that the drain tubing is always lower than the drain port at the rear of the calorimeter. Failure to meet this requirement will cause water to back up inside the calorimeter.
- Make the tap water connection at the rear of the calorimeter using 1/4" Nylon tubing (HJ0025TB035). The inlet pressure should not exceed 60 psig. Refer to figure 2-1 and 2-2. The inlet connection incorporates a water filter, 1245DD, just behind the inlet connection. When making the water connection, a back-up wrench should be placed on the water filter to insure a secure connection and to prevent over tightening the filter.

NOTE:



During extended periods of inactivity (overnight or longer), shut off the tap water supply to the calorimeter.

- Make the connection to the rinse water source using 3/8" Tygon tubing (JT0038TB062A). A barbed fitting is provided at the rear of the calorimeter for this connection. A 10 liter carboy (231C2) is provided as a distilled water rinse reservoir. Place a 149C in-line water filter at the end of the water line that is inserted into the carboy.

INSTALLING THE CALORIMETER (CONTINUED)

- Make the connections to the oxygen supply at this time. Refer to figure 2-2 and 2-3. 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 at 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).

Note:

The cause of any leaks must be corrected before proceeding.

Note:

During extended periods of inactivity (overnight or longer), close the tank valve to prevent depleting the tank in the event of a leak. When changing tanks, close the tank valve prior to re-moving the regulator. 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.

- 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.

Note:

This step is optional for use with A1050DD. See Appendix G for A1050DD Bomb Rinse Container Installation and Use.

- Connect the printer USB cable between the 1758

printer and the connections at the rear of the calorimeter. Install the printer ribbon and printer paper at this time. Apply power to the calorimeter and turn on the printer.

SWAGELOK TUBE FITTINGS

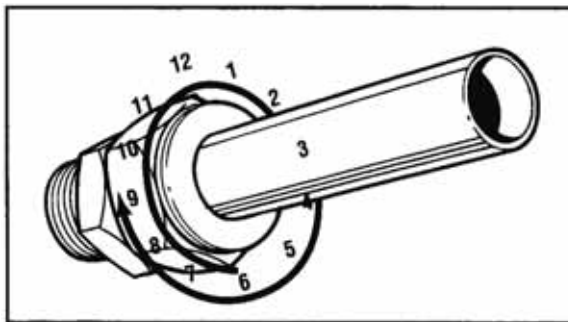
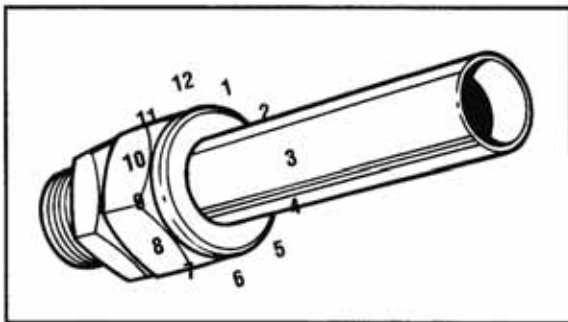
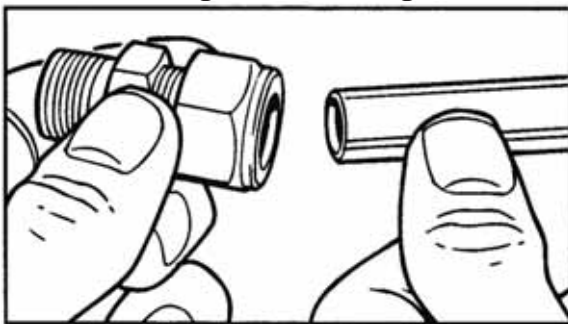
When Swagelok Tube Fittings are used, the instructions for installation are:

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.

Figure 2-1
Swagelok Tube Fittings



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.

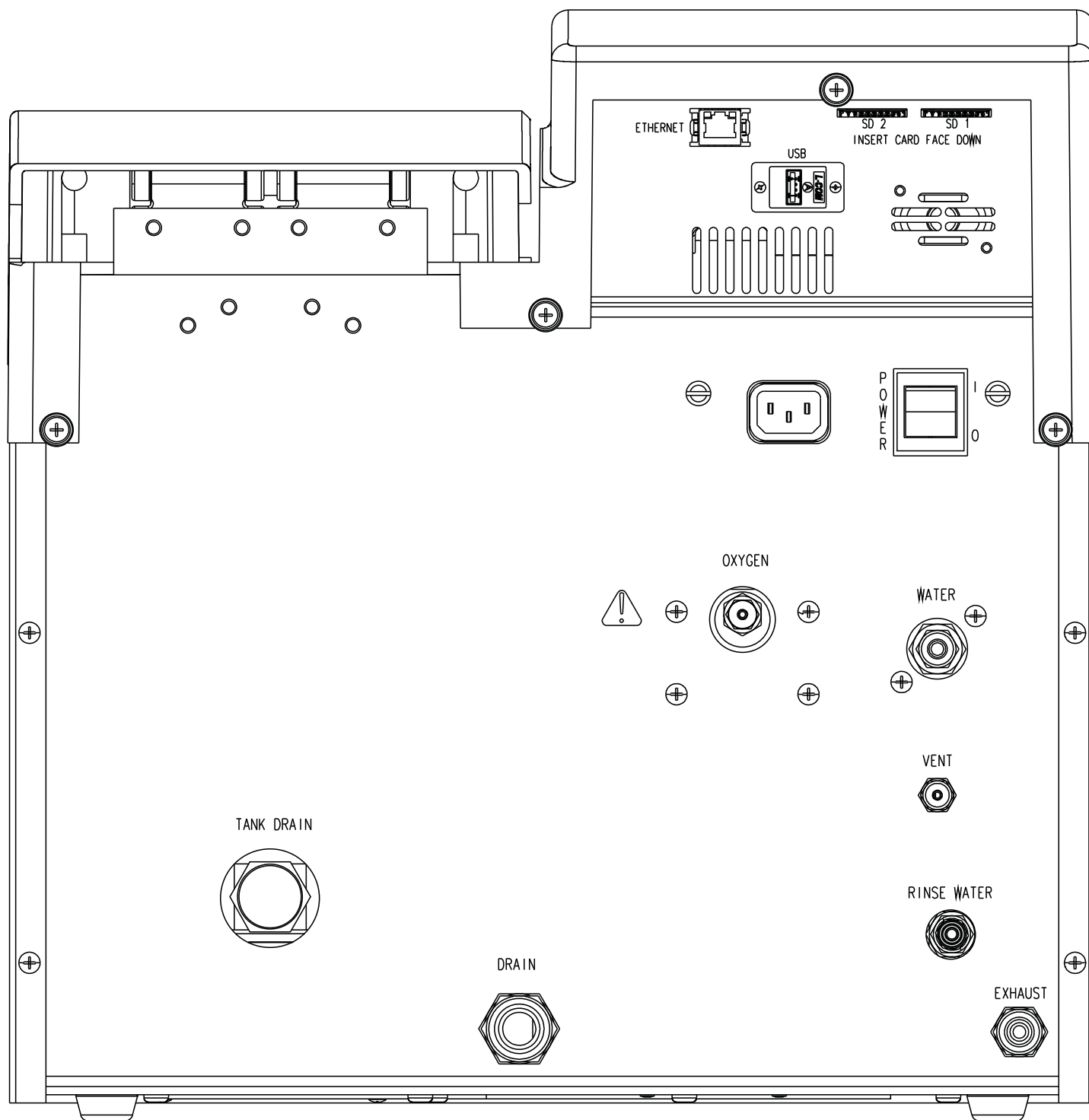
The type of tubing and the wall thickness also has an effect on the amount of tightening required. Plastic tubing requires a minimal amount of additional tightening while heavy wall metal tubing may require somewhat more tightening. In general, the nut only needs to be tightened about 1/8 turn beyond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

Over tightening the nut should be avoided. 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.

RETIGHTENING SWAGELOK TUBE FITTINGS

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.

Figure 2-2
6300 Calorimeter Back Panel



6300 CALORIMETER EXTERNAL PLUMBING

Figure 2-3
Closed Loop Configuration with 6520A

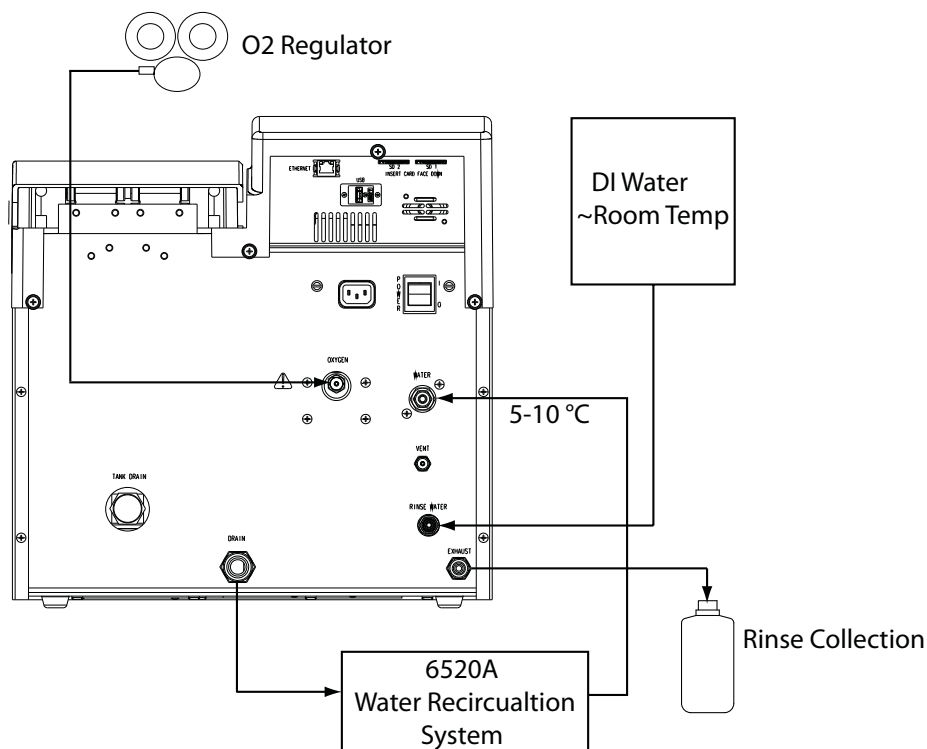
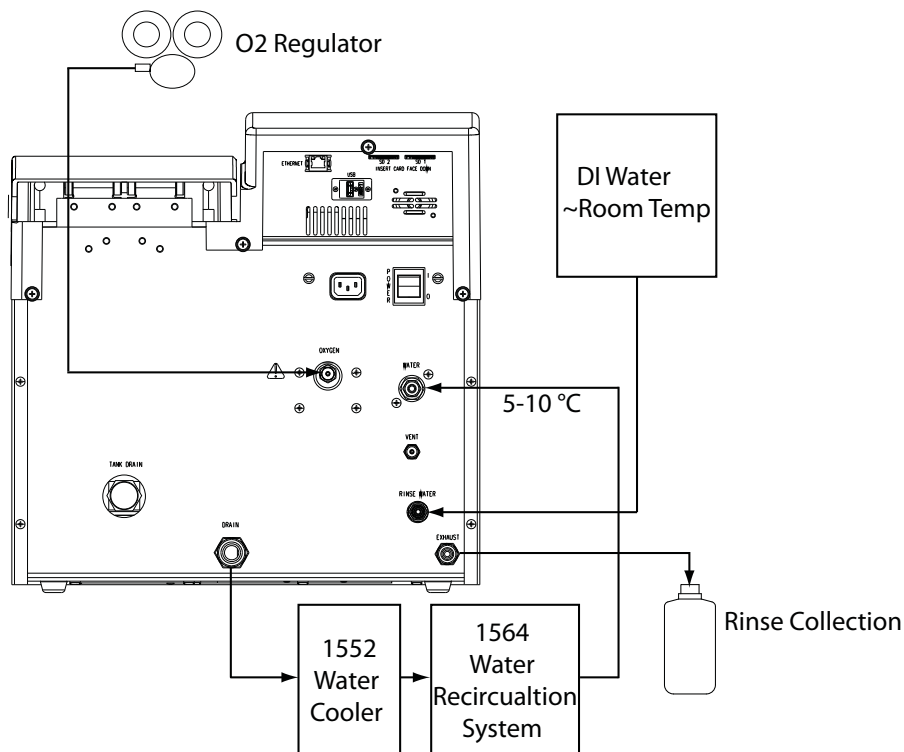


Figure 2-4
Closed Loop Configuration with 1564



Line 1 & 2 – Maximum length of 10 feet, 1/4" OD, Polyurethane (Part Number HJ0025TB035)

Line 3 - Maximum length of 25 feet, 1/8" OD, Nylon (Part Number HX0012TB024)

6300 CALORIMETER EXTERNAL PLUMBING (CONTINUED)

Figure 2-5
Open Loop Configuration with 1552

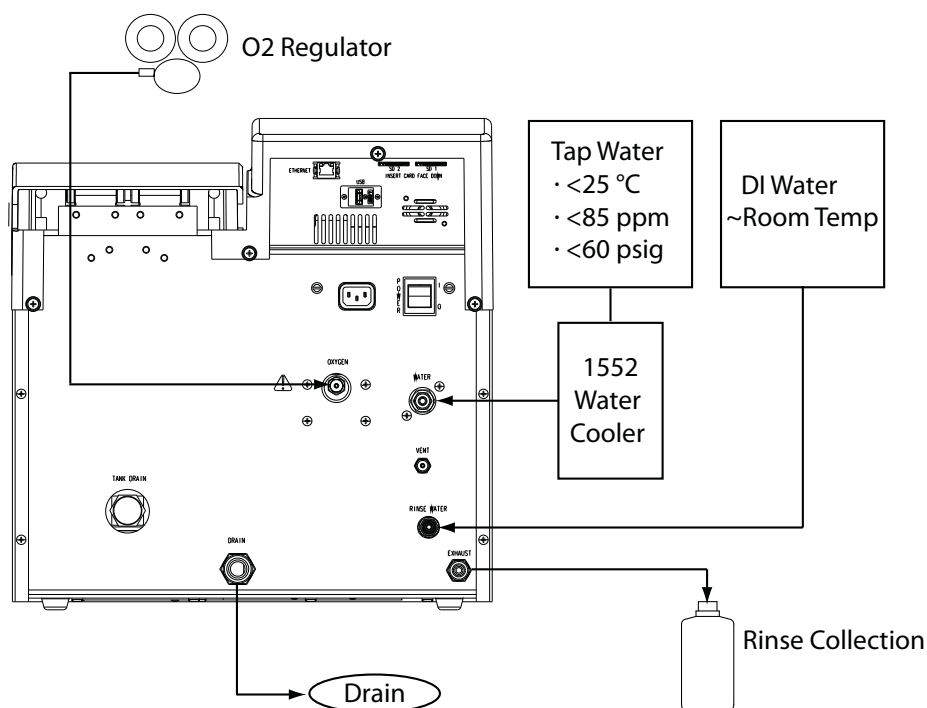
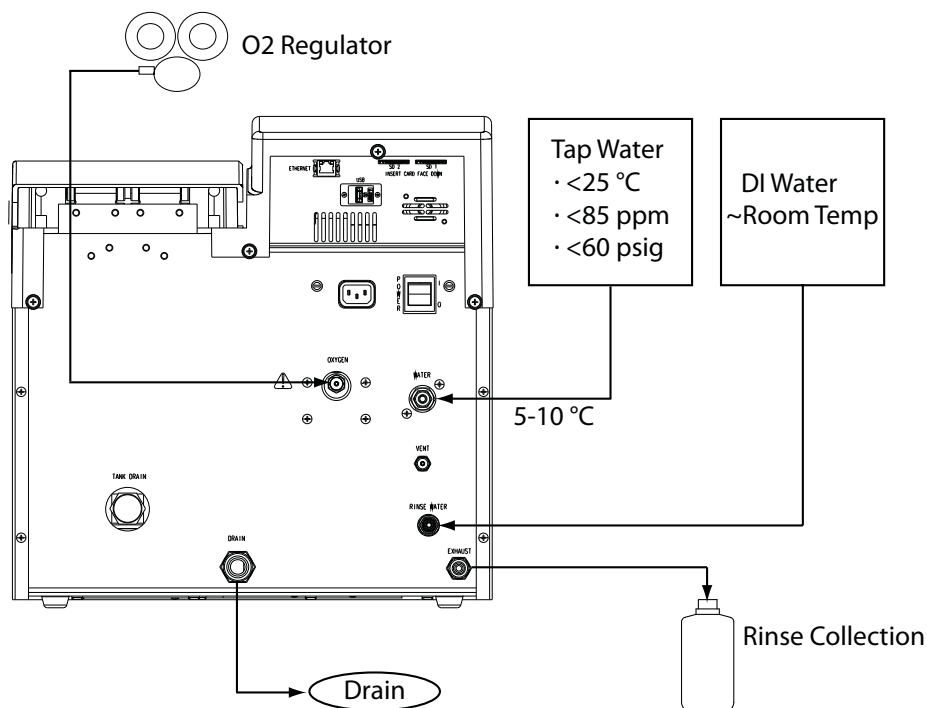


Figure 2-6
Open Loop Configuration



Line 1 & 2 – Maximum length of 10 feet, 1/4" OD, Polyurethane (Part Number HJ0025TB035)

Line 3 - Maximum length of 25 feet, 1/8" OD, Nylon (Part Number HX0012TB024)

INSTALLING THE CALORIMETER (CONTINUED)

- After the calorimeter displays the main menu, press the Calorimeter Operation button. This screen should indicate that the jacket is filling with water. The initial fill can take as long as 8 to 10 minutes to complete. If the jacket filling process times out, simply acknowledge the timeout message to resume the jacket filling process. After the jacket is filled press the Heater and Pump button in order to toggle the heater and pump on.
- Wait for the calorimeter jacket temperature to stabilize within a half a degree of 30 °C. (When the pump and heater are turned on after being off for an extended period of time, it may take longer than 10 minutes for the calorimeter warm up. This may cause an error. Simply restart the heater and pump.) While waiting for the jacket temperature to stabilize, raise the calorimeter lid and remove the bomb head by twisting 1/16 turn counterclockwise and pulling straight up. Examine the bomb release pin at the bottom of the combustion cylinder. If it has become dislodged during shipping, position it correctly using the long forceps supplied in the calorimeter accessory kit. Refer to figure F-19.
- Lock the head in the bomb cylinder (see section 5-7), close the lid and while applying a slight downward pressure. Press the CALORIMETER OPERATION key on the main menu followed by the Pretesting Cycle button to initiate a pre-test cycle. (This button will not be available until the jacket temperature has been stable for 15 minutes.) During the initial portion of this cycle, check to see that the oxygen supply pressure is set to 450 psig. Adjust as required. The calorimeter should complete the pre-test cycle with no errors.
- Assemble the bomb head stand (A38A), located in the accessory kit. Remove the head from the calorimeter and place it on the stand. Place a 1 gram pellet of benzoic acid in a combustion capsule and place this unweighed sample on the capsule holder of the bomb head. Attach 10 cm of fuse thread as shown in figure 5-3. Install the bomb head in the calorimeter and close the cover. Apply a slight downward pressure on the cover and press the START key to begin the test sequence. Press the ENTER key to accept the displayed sample ID number. At the sample weight prompt press 1 then

ENTER to store the default sample mass of 1 gram. This test should go through Fill, Pre-period, Post-period and Cool/Rinse Cycles without error. The calorimeter is now ready to be standardized.

- The calorimeter must be accurately standardized prior to actually performing calorimetric tests on sample materials. Review Appendix C – Standardization, in order to become familiar with the general procedure and calculations. The user should configure the calorimeter at this time to accommodate the desired sample weight entry mode. The calorimeter can be placed into the standardization mode on the Calorimeter Operation Page, by pressing the Operating Mode button. If two bomb heads are being used with the calorimeter to maximize sample throughput, the calorimeter can be configured to prompt for a Bomb ID at the start of each test. The Bomb ID can also be selected on the Calorimeter Operations Page by pressing the Bomb Installed/EE key. Both bomb heads will need to be standardized separately. The end result of a standardization test is an energy equivalent value, or the amount of energy required to raise the calorimeter one degree. Repeated standardization with any given bomb head should yield an energy equivalent value with a range of up to 4 calories per degree, centered on the mean value for all tests using that bomb head. The calorimeter is ready for testing samples after a suitably constant energy equivalent value has been obtained.



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CHAPTER 3

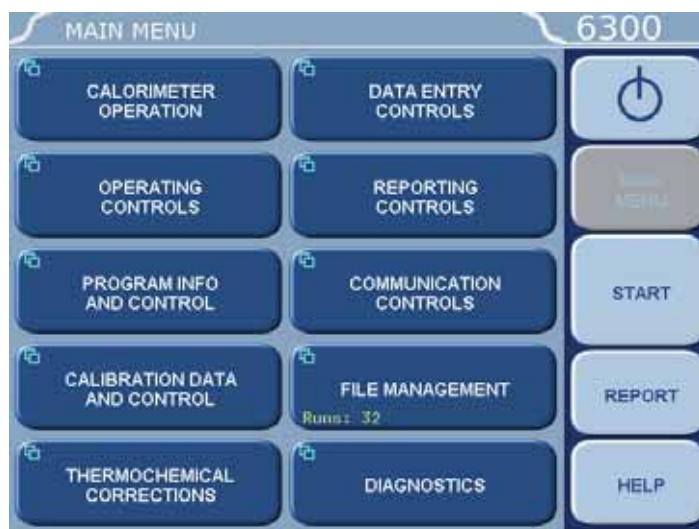
INSTRUMENT DESCRIPTION

TYPES OF CONTROLS

All calorimeter configurations and operations are handled by a menu-driven system operated from the bright touch screen display. The settings and controls are organized into nine main sections or pages which comprise 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 revert 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 calorimeter test.
4. **Report.** This key is used to access the test results stored in the calorimeter, to enter thermochemical corrections and to initiate 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.
6. **Abort.** This key appears in the Start key location while a test or pretest is running. Pressing this key will abort the test or pretest in progress.
7.  This key appears in the Escape key location when the main menu is displayed. This key is used to shut down the calorimeter program before turning off the power.



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CHAPTER 4

PROGRAM INSTALLATION & CONTROL

SOFTWARE INSTALLATION

The program in the 6300 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 described in Appendix A of this manual. Any of these items can be individually entered at any time to revise the operating program.

DEFAULT SETTINGS

Units are pre programmed with DEFAULT SETTINGS. See pages 24 and 25 for a listing of the factory default settings.

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 Information and Control Menu, then to User/Factory Settings, and then 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 its default settings.

REVISING DEFAULT SETTINGS

The default parameters of the 6300 Calorimeter can be changed to guarantee that the 6300 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 settings, go to the Program Info and Control Page, User/Factory Settings, Re-load User Default Settings, and YES.

Figure 4-1
6300 Factory Default Settings

Calorimeter Operations	
Operating Mode	Determination
Bomb Installed/EE	1/940.0
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 Backlight Intensity	70%
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 Control	
Date & Time Settings	
Volume Level Adjust	85%
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	63-1138
Reload Factory Default Settings	
Reload User Default Settings	
Save User Default Settings	
Compare settings with Factory Defaults	
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
Control Chart Parameters	
Charted Value	HOC Standard
Process Sigma	0.1
Temp Rise High Warning	8.5
Temp Rise Low Warning	5.1
Bomb 1 Through 4	
EE Value	800.0
Protected EE Value	OFF
Thermochemical Corrections Standardization	
Fixed Fuse	ON 50.0
Acid Correction	Fixed HNO ₃ 8.0
Fixed Sulfur	ON 0.0
Determination	
Fixed Fuse	ON 50.0
Acid Correction	Fixed HNO ₃ 8.0
Fixed Sulfur	OFF 0.0
Net Heat/Dry Factors	
Calculation Factors	
Nitric Acid Factor	1.58

*Factory Default Settings
Continued*

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
Net Heat/Dry Factors	
Fixed Hydrogen	OFF 0.0
Fixed Oxygen	ON 0.0
Fixed Nitrogen	ON 0.0
Calculate Net Heat of Combustion	OFF
Fixed Moisture as Determined	OFF 0.0
Fixed Moisture as Received	OFF 0.0
Dry Calculation	OFF
Data Entry Controls	
Prompt for Bomb ID	ON
Weight Entry Mode	Touch Screen
Acid Entry Mode	Touch Screen
Net Heat Entry Modes	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 Modes	Touch Screen
Auto Prewweigh Controls	ON
Auto Sample ID Controls	
Automatic Sample ID	ON
Automatic Sample ID Increment	1
Automatic Sample ID Number	1
Auto Prewweigh Controls	
Automatic Prewweigh ID	ON
Automatic Prewweigh ID Increment	1
Automatic Prewweigh 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
Report Schedule	End of Postperiod
Communication Controls	
Printer Type	Parr 1758
Balance Port	
Network Interface	
Printer Destination	Local USB
Bar Code Port	
Network Data Devices	
Balance Port Communications	
Balance Type	Generic
Balance Port Device	
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 Timeout (seconds)	10
Balance Handler Strings	
Data Logger	
Data Logger	OFF
Data Log Interval	12s
Data Log Destination	Log File and Printer
Select Data Log Items	
Data Log Format	Text Format



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CHAPTER 5

OPERATING INSTRUCTIONS

TO BEGIN A TEST

1. Weigh the sample to 0.1 mg.
2. Gently tap the 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.)
3. Carefully place the capsule into the capsule holder.
4. Attach 10 cm of ignition thread (see Figure 5-2).
5. Install bomb head in calorimeter.
6. Close calorimeter cover making certain the latch is engaged
7. Select determination or standardization as appropriate on Calorimeter Operations Page, Operating Mode.
8. Press START to begin the test. Calorimeter will prompt operator for Cal ID number, Sample ID numbers and weights in accordance with operating modes set into the instrument.

OPERATING THE OXYGEN BOMB

Combustion with oxygen in a sealed bomb is a very effective and reliable method for releasing all heat energy obtainable from a sample, and for preparing hydrocarbon compounds and carbonaceous materials for analysis.

Note:

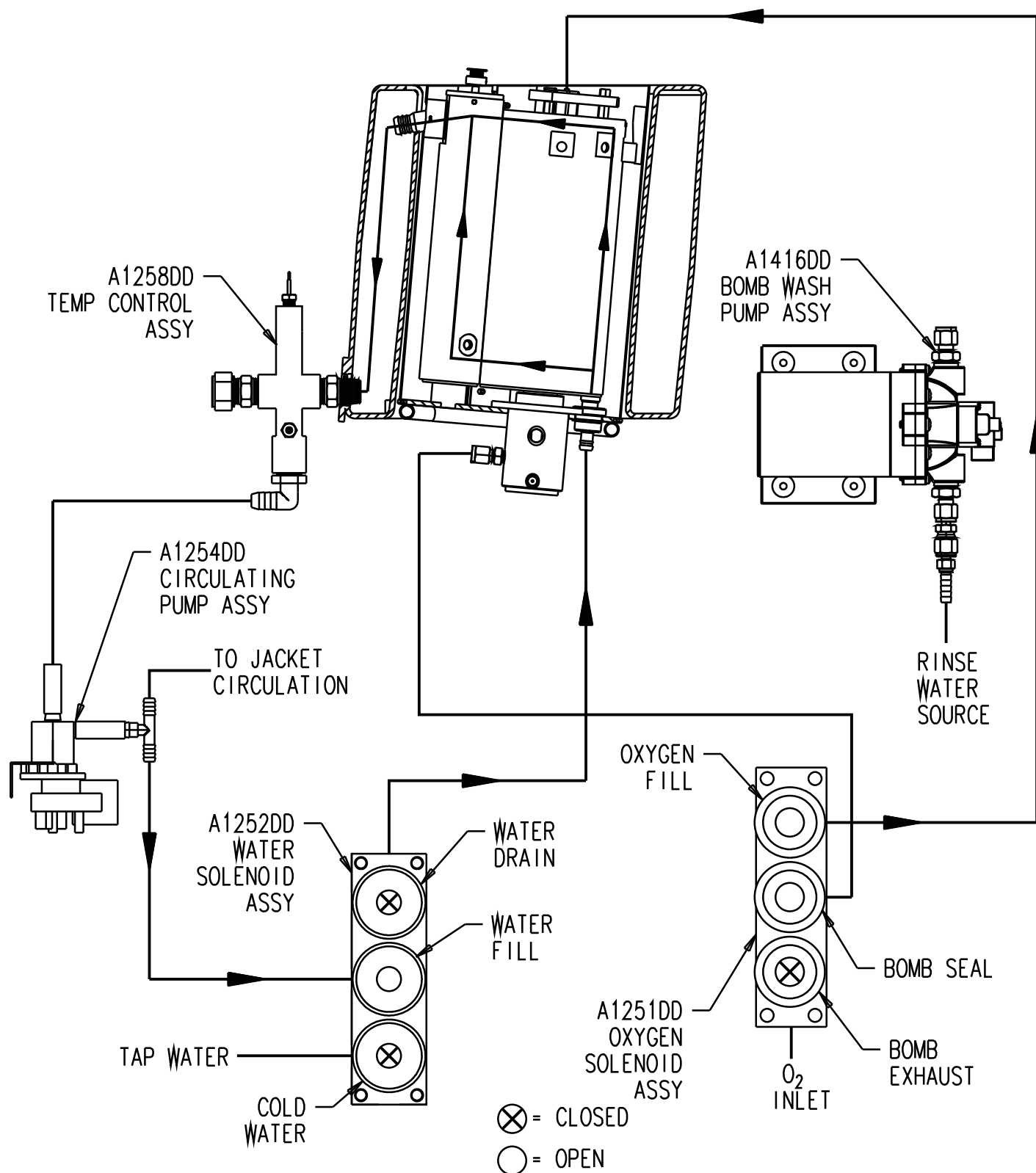
The following precautions must always be observed when using this equipment:

1. Do not overcharge the bomb with sample or with a sample which might react with explosive violence.
2. Do not overcharge the bomb with oxygen. The initial charging pressure should not exceed 40 atm (600 psig).
3. Do not fire the bomb if there is any indication that it is leaking.
4. Stand away from the calorimeter during firing and for at least 20 seconds after firing.
5. Keep the bomb in good condition at all times. Any parts that show signs of weakness or deterioration must be replaced promptly.
6. Read the maintenance and safety instructions before starting to use the bomb, and urge all operating personnel to read these instructions often.

Note:

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

*Figure 5-1
Fill Flow Diagram*



ALLOWABLE SAMPLE SIZE

To stay within safe limits, the bomb should never be charged with a sample which will release more than 8000 calories when burned in oxygen.

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.

When starting tests with new or unfamiliar materials, it is always best to use samples of less than .7 gram with the possibility of increasing the amount if preliminary tests indicate no abnormal behavior and sample will not exceed the 8000 calorie limit.

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.

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.

ATTACHING THE COTTON THREAD

Remove any moisture from the heating wire prior to attaching the cotton thread.

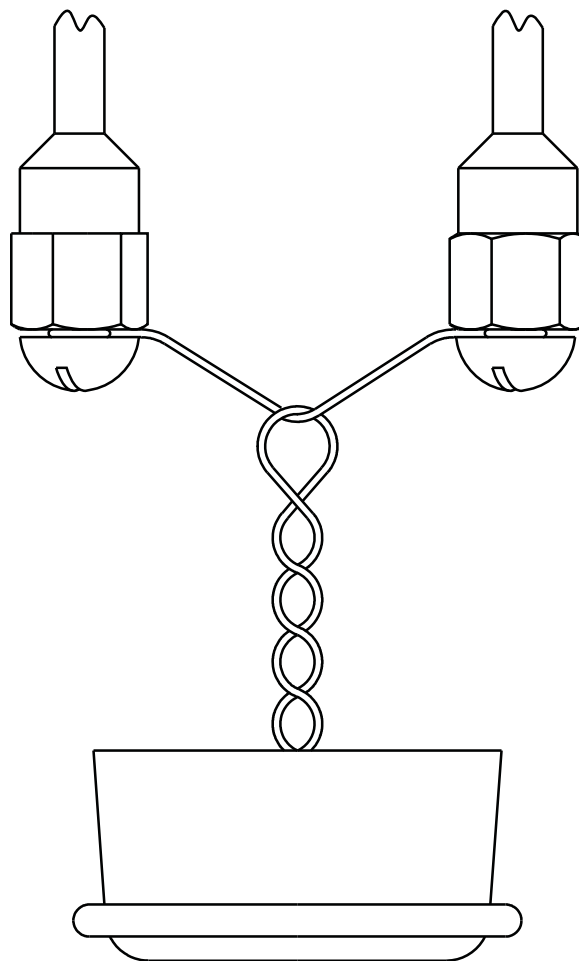
A cotton thread (845DD) is used as an auxiliary fuse to ignite the sample (See Figure 5-2).

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.

Figure 5-2
Cotton Thread Assembly

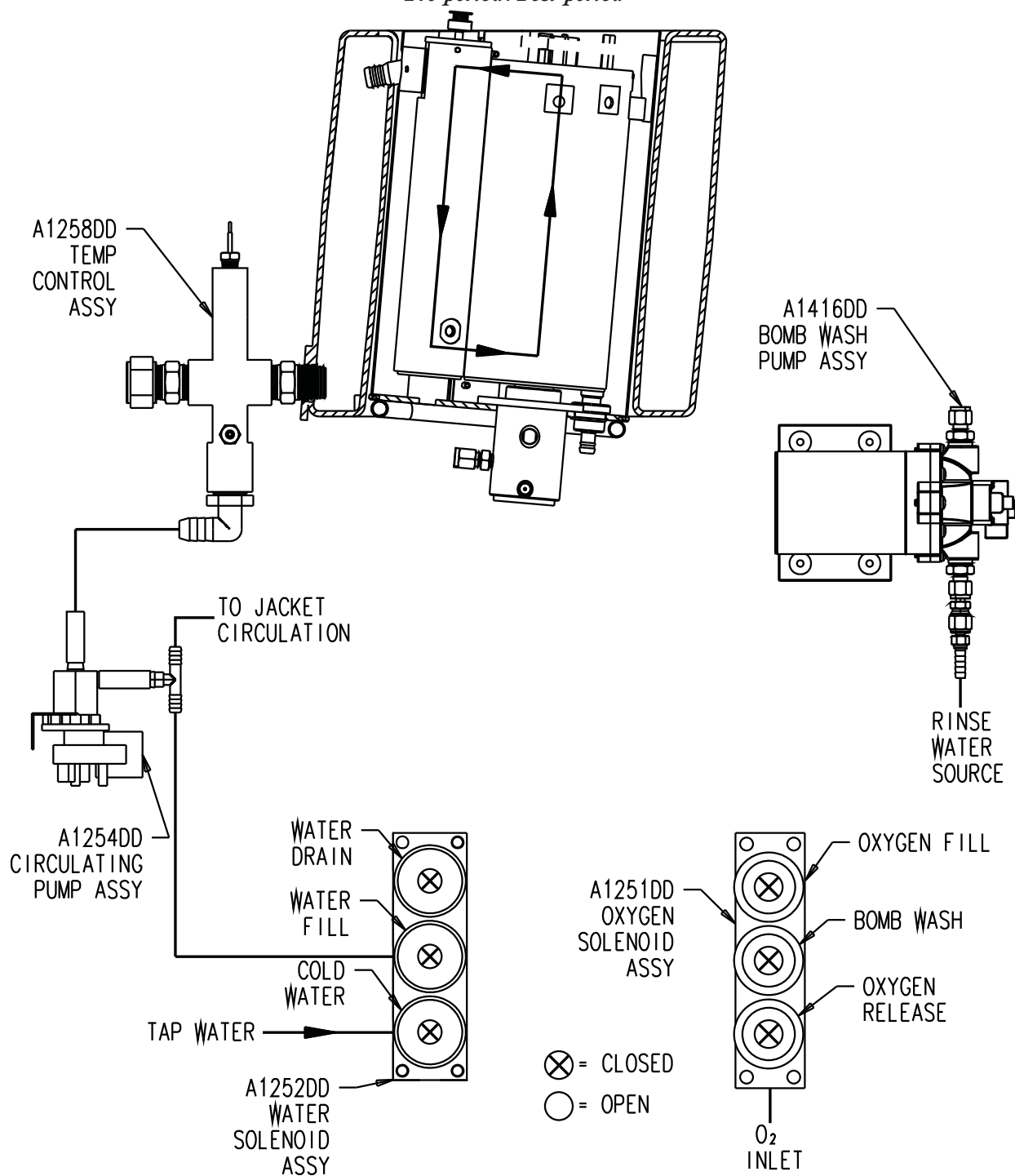


WARNING - DO NOT OVERFILL THE BOMB



The safety relief valve on the regulator should protect the system from an over fill. If for any reason, the bomb should accidentally be charged to more than 600 psig (40 atm), do not fire the bomb. The dangerous pressures which might develop under such conditions could damage the bomb and injure the operator. If there is any reason to believe that the bomb has been over-filled, stop the filling operation immediately, exhaust the bomb and open it to check for any loss of sample before repeating the filling procedure.

Figure 5-3
Pre-period / Post-period



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 (see Figure 5-1).

1. The calorimeter checks the bomb ignition circuitry for continuity.
2. The water fill solenoid opens and water is pumped from the closed water supply 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 (see Figure 5-3).

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.

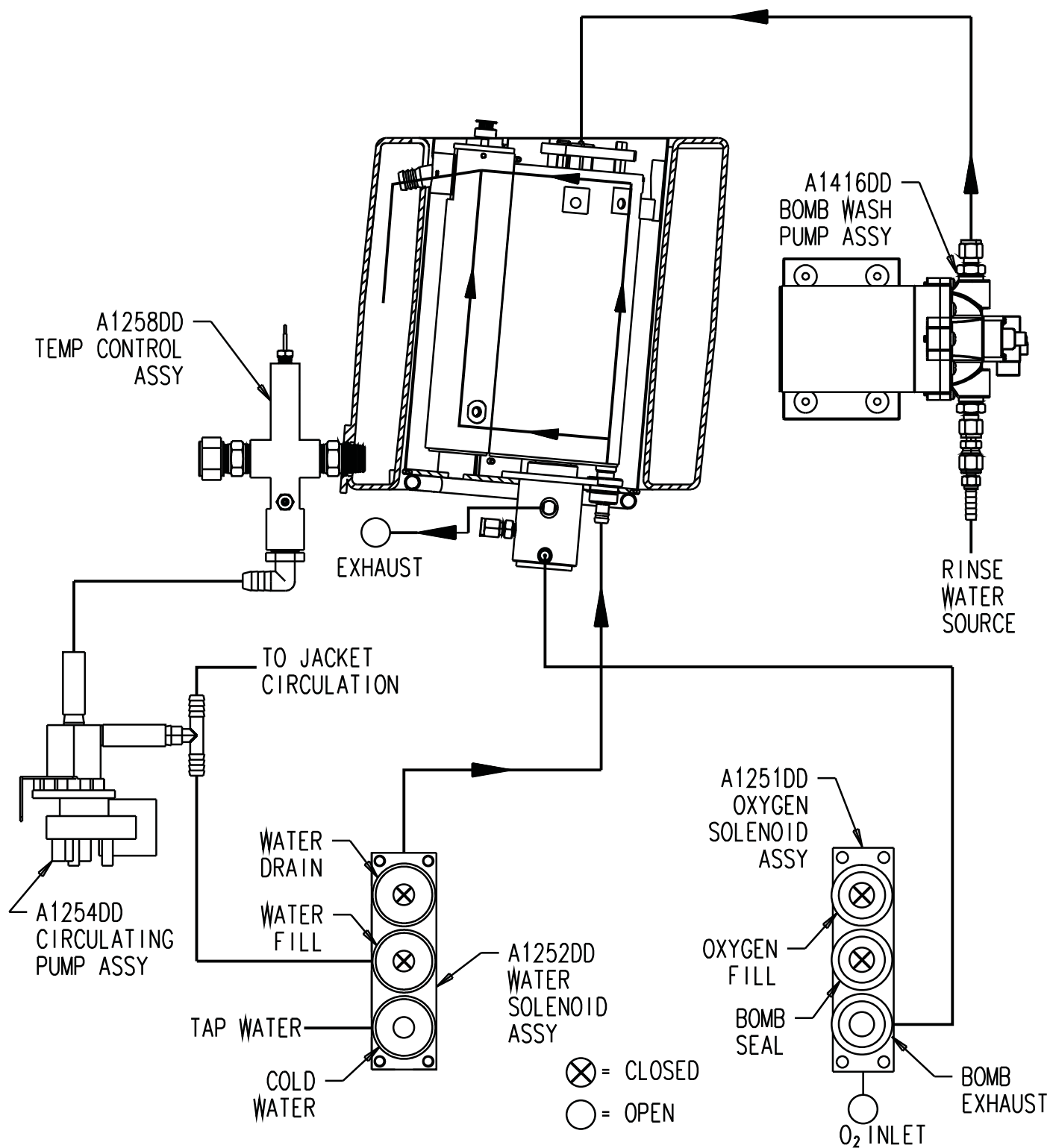
BOMB FIRING

Once the initial equilibrium is confirmed, the controller initiates the firing sequence.

There are no changes to the circulation pattern, as shown in Figure 5-3, from the pre-period through the bomb firing and post-period.

1. A warning of five short beeps is sounded indicating the bomb is about to be fired.
2. Current is passed through the electrical leads to ignite the ignition thread.
3. The controller monitors the temperature in the bucket to establish that a temperature rise actually occurs. If no temperature rise occurs, the misfire message is displayed on the controller and the abort sequence is initiated.

Figure 5-4
Rinse & Cool Flow Diagram



POST-PERIOD

After firing is confirmed, the post-period begins (see Figure 5-3).

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 (see Figure 5-4).

1. Tap 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 wash water from the carboy of the rinse water tank is admitted through the bomb wash solenoid valve and the check valve at the top of the bomb. The bomb wash water is released to the wash bottle.

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

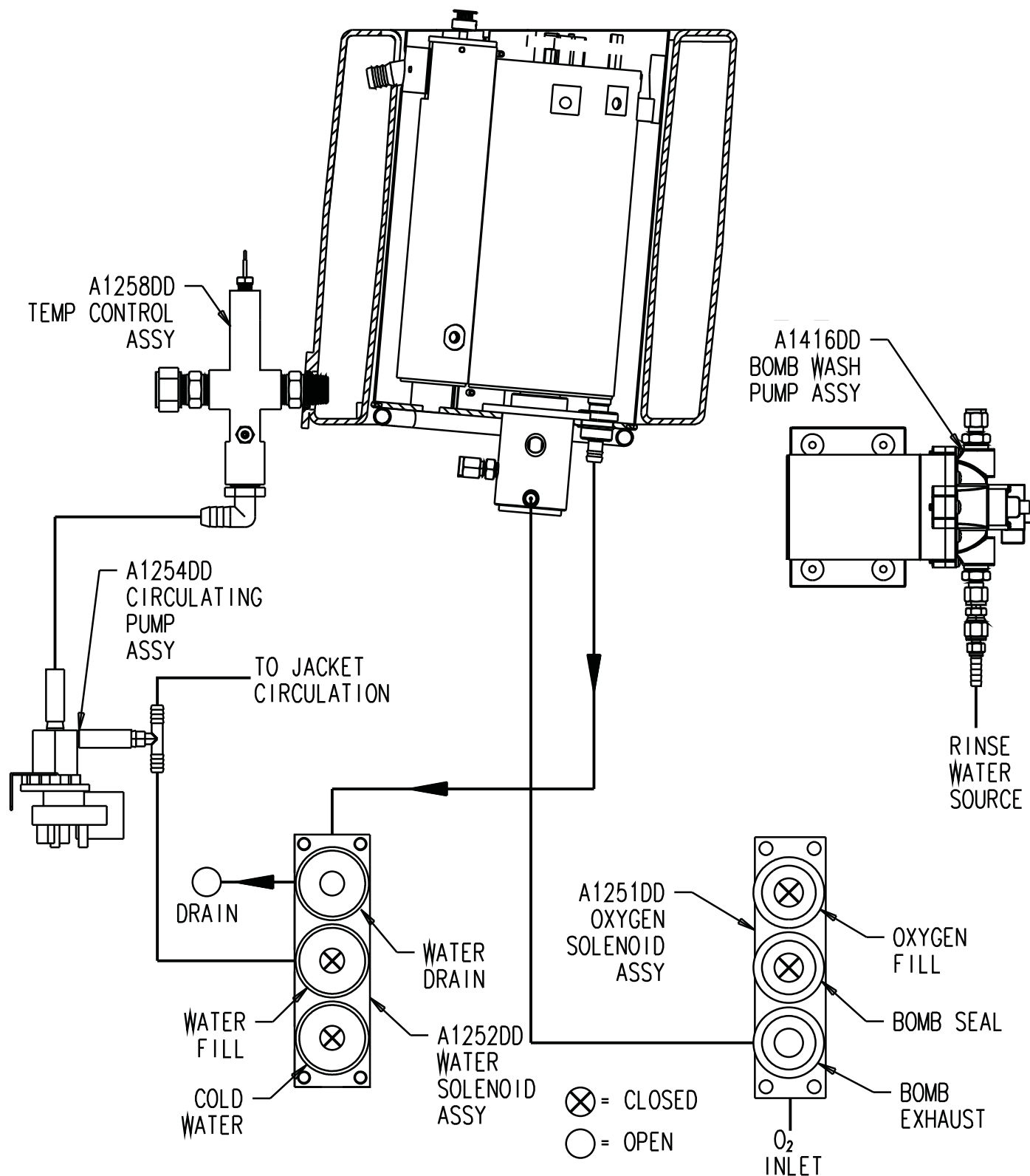
The bomb is filled one more time with oxygen to help flush the water residue from the interior of the bomb.

DRAIN

At the completion of the bomb rinse sequence, the drain sequence begins (see Figure 5-5).

1. The water in the bucket is drained out of the bucket and routed to the drain connection.
2. Once the bucket is drained, the calorimeter may be opened to remove the bomb head and load the next sample.
3. The test result will be printed or displayed in accordance with the setting of the Report Schedule on the Reporting Controls Submenu.

Figure 5-5
Drain Flow Diagram



SAMPLES

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 pelletizing of samples 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 water directly into a loose sample or onto a pellet after the sample has been weighed. Then let the sample stand for awhile to obtain uniform distribution.

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 powdered benzoic acid, white oil or any other combustible material of known purity can be mixed with the sample. Ethylene glycol, butyl alcohol or decalin may also be used for this purpose.

Note:

It must be remembered, however, 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.

Also, when benzoic acid is combusted for standardization runs or for combustion aid purposes, 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

OXYGEN CHARGING PRESSURE

The 6300 Calorimeter has been designed to operate with an oxygen filling pressure of 30 atm. Significant changes from this value are not recommended.

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. **When combusting samples that contain metal particles such as aluminum or magnesium, the non-metallic (fused silica) 43A3 Capsule or 43A3KQ Fused Quartz is required.** When superior corrosion resistance is needed, the Platinum Rhodium 43A5 Capsule or 43A3KQ Fused Quartz is required.

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.

Capsules should be monitored for wear. Do not use the capsule if the wall or base thickness is less than 0.025".

New capsules are heated in a muffle furnace at 500°C 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.

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.

FOODSTUFFS AND CELLULOSIC MATERIALS

Fibrous and fluffy materials generally require one of three modes of controlling the burn rate. Fibrous materials do not pelletize readily and generally require either moisture content or 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.

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

1138 Oxygen Combustion Bomb

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 G30™ 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.

1136 Oxygen Combustion Bomb

The 1136 bomb has been used extensively in our model 1281 calorimeter for the past ten years. It will safely burn samples liberating up to a maximum 8000 calories per charge using oxygen charging pressures up to 40 atm.

The 1136 bomb, like the 1138 bomb, is made of alloy 20 and is also available in Hastelloy G30™ as part number 1136CL. These bombs are 340 mL in volume and are rated to a maximum working pressure of 2000 psi. Like the 1138, 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 Pressure Oxygen Bomb designed specifically for these types of samples.

VOLATILE SAMPLE HOLDERS

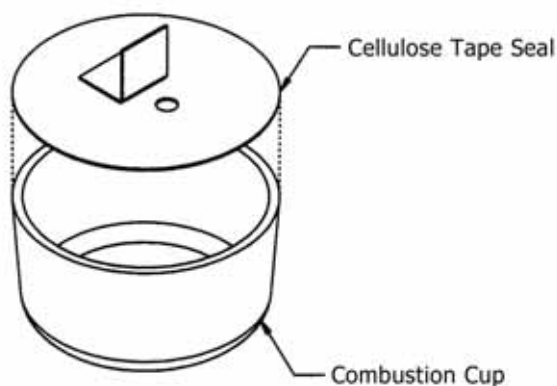
Volatile samples can be handled in a Parr 43A6 Platinum Capsule with a spun rim, or in a Parr 43AS Alloy Capsule which has a sturdy wall with a flat top rim. These holders can be sealed with a disc of plastic adhesive tape prepared by stretching tape across the top of the cup and trimming the excess 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, Line 2 - Heat of Combustion of Spike.

Note:

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

Figure 5-6
Combustion Capsule with Adhesive Tape Seal



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

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 5-7).

- Puncture the tape at a point below the flag, then re-weigh the empty cup with its tape cover.
- Add the sample with a hypodermic syringe; close the opening with the flag and re-weigh the filled cup.
- Set the cup in the capsule holder and arrange the auxiliary fuse so that it touches the center of the tape disc.
- 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.
- Fill the bomb with the usual oxygen charging pressure.
- The calorimeter will fire the bomb and complete the test in the usual manner.

Volatile samples are defined as one with an initial boiling point below 180°C per ASTM D-2.

Low volatile samples with a 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.

POOR COMBUSTION

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.
- Loose or powdery condition of the sample which will permit unburned particles to be ejected during a violent combustion.
- The use of a sample containing coarse particles which will not burn readily. Coal particles which are too large to pass a 60 mesh screen may not burn completely.
- The use of a sample pellet which has been made too hard or too soft. Either condition can cause spalling and the ejection of unburned fragments.
- Insufficient space between the combustion cup and the bottom of the bomb. 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.
- Excessive moisture or non-combustible material in the sample. If the moisture, ash and other non combustible material in the sample amounts to 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.

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CHAPTER 6

CORRECTIONS & FINAL REPORTS

ENTERING CORRECTIONS AND OBTAINING THE FINAL REPORT

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

Refer to the *Reporting Instructions*, Chapter 7, for the steps necessary to initiate a report from the controller.

MANUAL ENTRY

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

Fuse Correction

Key in the Fuse Wire Correction and press the ENTER key. The default setting for this value is to be entered in calories. The fuse correction has two components and these are explained in Appendix B.

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.

If fixed values for fuse, acid and sulfur are turned OFF on the Thermochemical Corrections Page, then the user must manually enter the values at the prompt.

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.

Details for applying fixed corrections are found in Appendix B, *Thermochemical Calculations*.

Any value set-up as a fixed correction will be automatically applied and the controller will not prompt the user for this value.

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CHAPTER 7

REPORTING INSTRUCTIONS

REPORT OPTION SECTION

The 6300 Calorimeter can transmit its stored test data in either of two ways. The REPORT DESTINATION key on the Reporting Controls Page toggles the report destination between the display and an optional printer connected to the USB port of the calorimeter. This page also selects the type of reports that are generated automatically by the calorimeter.

REPORT GENERATION



There are two kinds of calorimeter reports: Preliminary and Final.

Preliminary Reports are generated at the conclusion of a test. They will not contain the thermochemical corrections for sulfur, fuse, or acid. 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.

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.

Select From List

This key displays the stored results specified with the following two keys:

Run Data Type

This key enables the operator to display only determination runs, only standardization runs and all runs. (The choice of solution data type is not applicable to this calorimeter.)

Run Data Status

This key enables the operator to display only preliminary reports, only final reports, both preliminary and final reports, only pre weighed samples reports or all stored reports.

Prompt For Final Values

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.

REPORT GENERATION (CONTINUED)

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 control page is turned on.

NET HEAT OF COMBUSTION

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

CHAPTER 8

FILE MANAGEMENT

The 6300 Calorimeter will hold data for 1000 tests in its memory. These tests may be pre weights, preliminary or final reports for either standardization 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.

RUN DATA FILE MANAGER			
FILENAME	TYPE	STATUS	DATE
6300-12	DET	PRELIM	07/03/10
6300-11	DET	PRELIM	07/02/10
6300-10	STD	FINAL	07/02/10
6300-9	STD	FINAL	07/02/10
6300-8	STD	FINAL	07/02/10
6300-7	STD	FINAL	07/02/10
6300-6	STD	FINAL	07/02/10
6300-5	STD	FINAL	07/02/10

SELECT	EXTEND SEL.	▲	▲	RENAME	DELETE
ESCAPE	DESEL. ALL	▼	▼	HELP	CONVERT TYPE

CLEARING 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.

REMOVABLE SD MEMORY

The controller of the 6300 calorimeter can accept SD 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.

SD memory cards are inserted into the slots 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 with the exception of restoring or updating the controllers operating system.

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CHAPTER 9

MAINTENANCE & TROUBLESHOOTING

ROUTINE MAINTENANCE

Note:

See the corresponding water system manuals for information on the maintenance of those systems.

To service or remove the bomb cylinder from the bucket assembly, remove the 941DD Wedge with needle nose pliers. Remove the 668DD Check Valve from the bomb cylinder. Remove the two SA1632RD18 Machine Screws (see Figure F-20). Then remove the 942DD Bushings and the 1081DD Quad-ring.

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 F-19). 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

The replacement of protective fuses for the 6300 Calorimeter should be performed by qualified personnel.

All fuses except Parr part # 139E23 are located on the A2140E I/O board located inside the instrument. Contact Parr Customer Service for instructions on how to access the fuses.

Note:

Check the labels on the instrument for correct fuse rating.

Parr Part #	Description	Type	Ratings
139E23	Lines Protective Fuses	Fast-Acting	15 Amps, 250Vac
1641E2	Heater Fuse (F2)	Fast-Acting	2.5 Amps, 250VAC
1641E	Pump Fuse (F1)	Fast-Acting	1 Amp, 250VAC
997E5	Bomb Rinse (F5)	Slo-Blo	5 Amps, 250VAC

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 corresponding sealing area in 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 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.

50 to 100 Tests

Replace the heating wire, with 2.5" of 840DD2 360 degrees clockwise around screws. Clean 986DD Electrode Contact Pins with mild abrasive, such as a pencil eraser, clean bomb head electrode points in a similar fashion, tighten screws holding heating wire in place.

Quarterly

Change water in the Water Tank and replace the 1245DD and 149C water filters.

500 Tests

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.

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 (2), 1071DD, 1444DDJB, 659DD, 519AJV, 694DD. When reassembling the bomb head, take care not to roll the 694DD O-ring as this will cause an oxygen leak. Samples that contain chlorine or are

500 Tests (Continued)

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 and is positioned between the bucket and the air-can of the calorimeter. For your convenience, these parts may be purchased as kit number 6038, Firing Maintenance Kit. See Figures F-1 and F-2 for O-ring locations.

Lubricate the 659DDJU and 357HCJB O-rings in the bomb release cylinder with 811DD lubricant. See Figures F-20 for O-ring locations. Clean the ignition contacts.

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, 1138DD, 969DD, 1143DD and 357HCJB O-rings with 811DD lubricant. Tools required are: screwdriver, snap ring pliers and needle nose pliers.

1. Turn off the gas supply to the calorimeter. Go to the Diagnostics Screen and turn on the bomb seal command. Raise the lid and turn ON the O₂ Fill Command. These steps are necessary to release the gas pressure in the seal/release mechanism before disassembling.
2. Turn off the calorimeter, once the oxygen has depressurized.
3. Insert the bomb head into the cylinder and lock into place.
4. Disengage the screws, SA1632RD018 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.
9. Withdraw both the insert and the release pin as a unit using needle nose pliers.
10. Remove any scoring on the release pin, above the smaller O-rings, 659DDJU in present with crocus cloth. Replace the O-rings on the 966DD2 release pin as well as the 1138DD O-ring that seals the cylinder insert. Lubricate 659DDJU and 357HCJB O-rings with 811DD lubricant.

11. 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 Tests

To deal with the realities of today's test loads and cycle times, the ASTM Committee recommends in method E144 - 94(2006)e1 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(2006)e1. 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.

6300 MAINTENANCE CHECKLIST**Quarterly Maintenance**

	Date	Date	Date	Date
Change water				
Replace 149C				
Clean grill on heat sink.(6520A)				

50 to 100 Test Maintenance

	Date	Date	Date	Date
Replace 8400DD2 Heating Wire				
Clean 986DD Electrode Contact Pins				

500 Test Maintenance

Replace the following:				
	Date	Date	Date	Date
Head:				
1374HCJV (2) O-rings				
394HC O-ring				
694DD O-ring				
519AJV O-ring				
659DD O-ring				
1444DDJB O-ring				

Cylinder:				
1071DD Quad ring				
821DD O-ring				
882DD O-ring				

Bucket and Bomb Seal/Exhaust area:				
659DD O-ring				
1143DD O-ring				
1138DD O-ring				
659DDJU (2) O-rings				
357HCJB O-ring				

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!



Avoid prying 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 is strongly discouraged. 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 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 (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.

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 the Exhaust is off.
4. Turn Bomb Seal on then off to retract the 966DD2 piston.
5. Turn on O2 Fill to begin filling the bomb. The bomb will be completely filled in one minute, at which time O2 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 the Exhaust 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 899DD Head Handle and SN1632HX 8/32 Hex Nut can slowly be removed to release the pressure in the bomb. See Figure 13-1.

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.

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 F-7). For the location of the A1251DD assembly (Figure F-4).

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. The solenoid should click and oxygen should flow from the elbow connection on the A1251DD. Turn the 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 click from the solenoid and 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.

JACKET FILL AND COOLING PROBLEMS

Jacket Fill Error – A problem has been detected while attempting to fill the bomb jacket; please correct the problem and press ‘OK’ to continue...

Error – Could not cool the bomb successfully; check water supply and drain.

Jacket Fill Error: When the water level in the jacket drops a level switch closes. This tells the CPU board (through the I/O board) that more water is needed in the jacket. If the jacket is still low after 10 minutes this error will be generated. Pressing ‘OK’ will restart the jacket fill process. To troubleshoot this error start with step 3 below.

Cooling Error: At the end of the test cool water is pumped into the bucket to bring down the temperature of the cylinder quickly. If it takes too long to cool then this error is generated.

There are several things to check for this cooling error.

The drain tubing should go straight to the water handling system or sink. The drain works by gravity. If water backs up in the drain it can cause cooling problems.

The cooling water could be too warm. The cooling water should be between 10 and 25 °C. The higher the temperature the longer it can potentially take to cool the bomb. The following steps assume that there is a water handling system present.

1. Look at the display and note the temperature. The temperature should not read higher than 25 °C.
2. Take the top off the water handling system and look inside. There are two tubes. The shorter tube should have water coming out of it in a good stream into the top of the reservoir. If it doesn't then the recirculation pump in the water handling system is bad (A308HWEE). The other tube should go to the bottom of the reservoir.
3. Turn off the water handling system.
4. Remove the tube from the water input at the back of the 6300.
5. Get a bucket or point the hose into a sink or something.
6. Turn on the water handling system. Water should be pumped out very quickly (faster than 2 liters/min).
7. If water does not come out or is not very fast then the A1418DD output pump on the water handling system is bad. Turn off the water handling system.
7. Remove the 1245DD water filter. This is the fitting that attaches to the tubing that you took off in step 4. See the A1257DD Water Regulator Assembly figure in Appendix F of the manual for a picture.
8. Attach the tube from the water handling system to 1245DD water filter.
9. Turn on the water handling system. Water should be pumped out at the same rate that you saw in step 6. If the water flow is less than the 1245DD needs to be cleaned or replaced.
10. Turn off the water handling system and re-attach the water filter and tube to the back of the 6300. Refill the water handling system if it needs it. Turn the water handling system back on.
11. Remove the lower cover of the 6300. This will give a view of all the solenoids and much of the tubing.
12. Find the A1275DD Cold Water Solenoid assembly. It is located on the bottom left of the calorimeter as you face the front. Coming out of the front of this solenoid is a right angle hose barb and a hose. Follow the hose to the other end (another right angle hose barb that goes into the jacket reservoir) and disconnect the tubing there.
13. Get a beaker capable of measuring 600 ml.
14. Go to the Diagnostics Menu and then to the I/O Diagnostics Menu.
15. Put the head into the calorimeter and lock it in place.
16. Turn on the H2O Fill solenoid. After a few seconds (10 seconds or so) water should start to flow through the disconnected tube.
17. Time the water flow into the beaker. The water should flow at a rate of 400 – 600 ml/min. If the flow is less than 400 ml/min the water regulator needs to be adjusted. Loosen the 1343DD retaining nut on the 1244DD regulator (note that this is black plastic on black plastic and may be hard to see initially). Turn the black plastic slotted screw clockwise (to the right) to increase the water flow. Adjust the water flow for approximately 500 ml/min. Tighten the 1343DD retaining nut (this helps keep the setting from shifting over time).
18. Reattach the hose to the hose barb going into the jacket reservoir.
19. Reinstall the cover.

BOMB REMOVAL AND REPLACEMENT

To service or remove the bomb cylinder from the bucket assembly, remove the 941DD Wedge with needle nose pliers. Remove the 668DD Check Valve from the bomb cylinder. Remove the two SA1631RD018 Machine Screws (see Figure F-20). Then remove the 942DD Bushings and the 1081DD Quad-ring.

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 F-19). Now the cylinder can be removed from the bucket assembly. Note the position of the locating pin.

To replace, follow these steps in reverse.

6300 CALORIMETER 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.

Start and Pretest buttons dim.

The Start and Pretest buttons will be dim (not lit) when the calorimeter is not ready to begin a test or pretest. When the heater and pump are first turned on the jacket temperature will be less than 30 °C. Once the jacket temperature reaches 30 °C $\pm$.5 °C it will be another 10 minutes before the Start and Pretest buttons light up. This is to make sure that all of the jacket water is stable at the correct temperature.

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 and the test run is aborted.

If the cotton thread is gone when the bomb head is extracted, the thread simply failed to ignite the sample. In most cases, the sample can be re-run with a new thread.

If the thread is present, make sure it is dry. If the thread is dry, it is best to check and/or change the metal heating wire. If it is wet, replace it and re-run the sample if no sample has adhered to the wet thread.

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.

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 and the user should contact Parr Technical Service.

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:

- 907DD failing (check valve head)
- Poor jacket water circulation due to a kinked hose or insufficient water in the tank.
- A bomb leak.
- Poor bucket stirring.
- Leaking bucket water solenoid.
- Metal to metal contact between the bucket and the jacket.
- Lid not tight may be high in back.

Could Not Cool the Bomb Successfully.

The calorimeter has failed to establish the desired cool down temperature within the time allowed. See Jacket Fill and Cooling Problems section on page 47.

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:

- Poor jacket water circulation due to a kinked hose or insufficient water in the tank.
- A bomb leak.
- Poor bucket stirring.
- Leaking bucket water solenoid.

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.

There Is A Problem With The Bucket Thermistor.

Possible electrical open. (1416E left side of bucket at bottom):

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

There Is A Problem With The Jacket Thermistor.

Possible electrical open or short. 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.

Jacket Fill Error.

A timer is started whenever the jacket water tank needs to be filled or topped off. If the jacket fill time exceeds ten minutes, this error or warning will be issued and the jacket fill process is halted. If the heater and pump are on, they are turned off. Pressing the OK key on the warning/error prompt box will restart the jacket filling process. The heater and pump can't be started until the jacket water tank is completely filled. Repeated or persistent errors or warnings are an indication of a problem. See Jacket Fill and Cooling Problems section on page 47.

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.

APPENDIX A

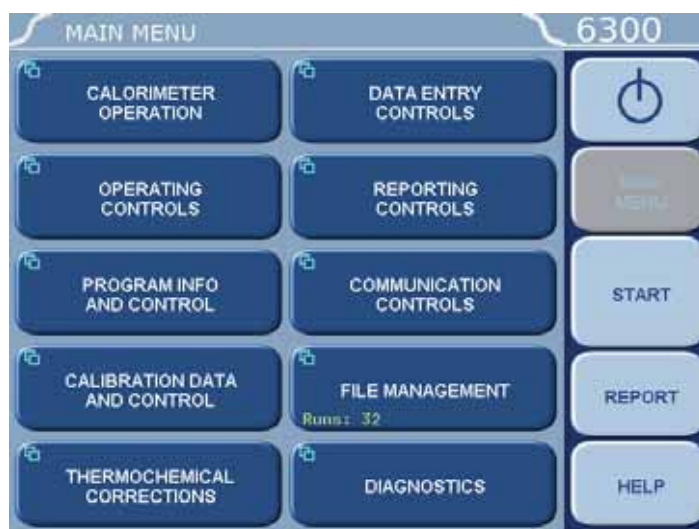
MENU OPERATING INSTRUCTIONS

The settings and controls are organized into nine main sections or pages which comprise the Main Menu. This appendix describes all pages of the menu-based operating system of the 6300 Calorimeter.

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.

Abort:

This key appears in the START key location while a test or pretest is running. Pressing this key will abort the test or pretest in progress.



This key appears in the Escape key location when the main menu is displayed. This key is used to shut down the calorimeter program before turning off the power.

CALORIMETER OPERATION MENU

The calorimeter will normally be operated from the Calorimeter Operating Page, although tests can always be started from any menu page.



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.

CALORIMETER OPERATION MENU (CONTINUED)

Start Prewrite:

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



Setup:

Press this 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

TEMPERATURE PLOT SETUP		
ENABLE BUCKET On	BUCKET PLOT SYMBOL No Point	BUCKET PLOT COLOR Red
BUCKET AUTOSCALE Off	BUCKET MIN VALUE 29.0	BUCKET MAX VALUE 39.0
ENABLE JACKET On	JACKET PLOT SYMBOL No Point	JACKET PLOT COLOR Black
JACKET AUTOSCALE Off	JACKET MIN VALUE 29.0	JACKET MAX VALUE 31.0
TIME MODE Window	TIME WINDOW 15.0	TIME MINIMUM 0.0
ESCAPE	TIME UNITS minutes	TIME MAXIMUM 30.0

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

Bucket Min Value:

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

Jacket Plot Symbol:

Toggles between: (same as Bucket Plot Color, p. 54).

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, p. 54).

Jacket Max Value:

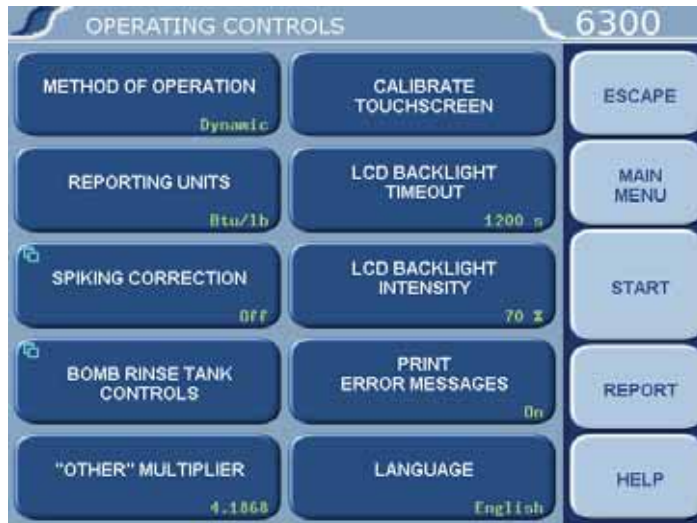
Press this key to access its numeric dialog box to set a maximum jacket value.

Time Minimum:

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

Time Maximum:

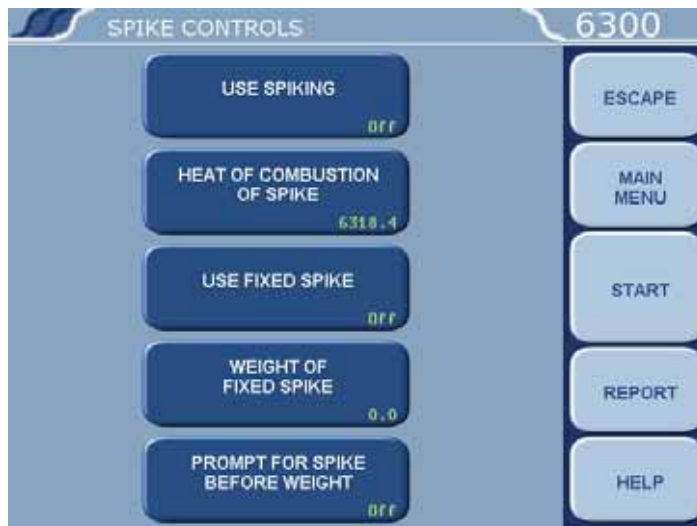
Press this key to access its numeric dialog box to set the greatest amount of time for the display.

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.

OPERATING CONTROLS MENU (CONTINUED)

- 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 sub-menu - Bomb Rinse Tank Control. Wash water for the bomb is drawn from the Bomb Rinse Tank. Users who wish to collect the bomb washings for acid titrations or further analysis will want to fill it with distilled or other suitable water.



- 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 full container. If the rinse timing controls have been changed, then the value must be changed proportionally.
- # 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.
- 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.
- 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 pre-defined 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 backlight 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 Backlight Intensity:

This key accesses a sub-menu with a slide control which adjusts the brightness 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 & Time:**

Accesses a sub-menu to set the current date and time.

- 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.
- Time Zone. Displays the selected time zone in relation to Greenwich Mean Time. Pressing this key will step through the time zones and automatically adjust the time setting.

Volume Level Adjust:

Opens a window with a slide adjustment to set the volume of the key clicks and alarms of the calorimeter. Default is 85%.

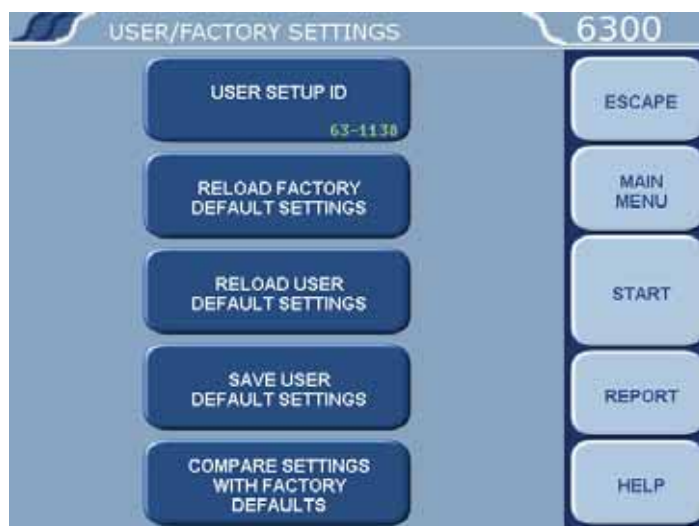
**Software and Hardware Info:**

This screen displays important information such as the main software version, I/O board hardware information, CPU type, I/O firmware revision, and Controller IP address.

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.

PROGRAM INFORMATION AND CONTROL MENU (CONTINUED)



User/Factory Settings:

This key leads to a sub-menu that allows the user to save or recall user defined instrument settings. Additionally, factory pre installed 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.
- 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 last saved 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.
- Compare Settings With Factory Defaults. This button will bring up a screen that will show the differences in the current settings of the calorimeter with the factory defaults.

Feature Key:

Unique Feature Keys obtained from Parr allow the user to access capabilities on the instrument such as bar code interfacing, remote operation of the calorimeter, or Samba Server.

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).

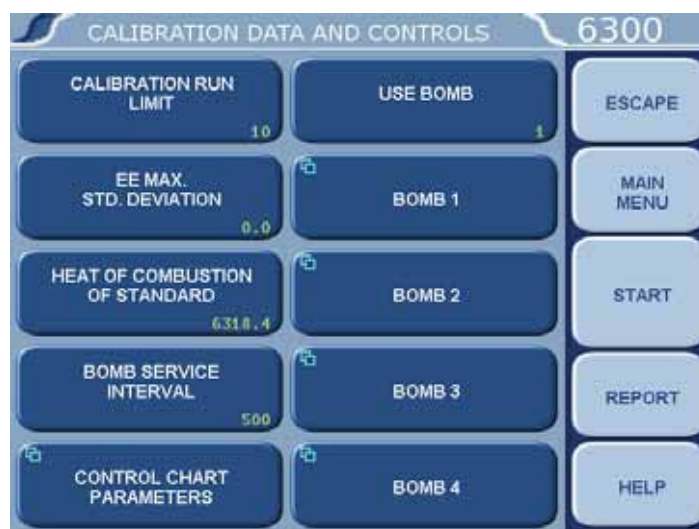
User Function Setup:

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 AND DATA 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 calcu-

lated 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 tool which can assist the user in determining whether or not their process is in control. Many standard methods will dictate that a reference sample be measured periodically and the results plotted on a graph. Limits for acceptable values are defined and the process is assumed to be in control as long as the results stay within these limits. Since results are expected to scatter with a normal distribution within established limits, systematic trends or patterns in the data plots may also be an early warning of problems.

- **Charted Value:** Toggles the charted value between the HOC Standard (Heat Of Combustion of Standard) and Energy Equivalent.
- **Process Sigma:** In relation to calorimetry, sigma is used as the classification of the instrument. The higher the process sigma the higher the limits for acceptable values for precision control.

The 6300 is a .1 Process Sigma calorimeter.

- **Temp. Rise High Warning:** Sets a limit for the temperature rise during a test. If the temperature rise exceeds the limit the user will be warned.
- **Temp. Rise Low Warning:** Sets a lower limit warning for the temperature rise during a test. If the temperature rise is lower than this setting the user will be warned.

Use Bomb:

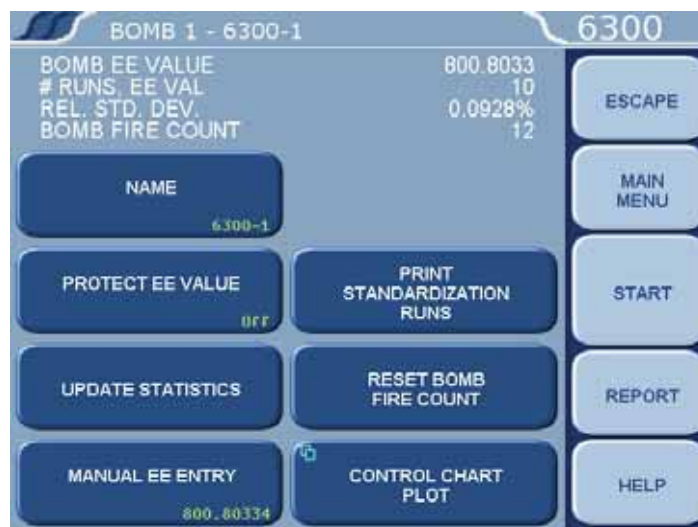
Displays the bomb number of the bomb currently installed in the calorimeter and toggles through the four possible bomb numbers. The left and right arrow keys are used to toggle through the bomb identification numbers available for each bomb.

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.

CALIBRATION DATA AND CONTROLS MENU (CONTINUED)

- **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.

Name.

Enables the operator to assign a unique alpha-numeric label for the bomb ID. The ID can be up to 8 characters.

Protect EE Value:

Toggles between OFF and ON. When set to OFF, the 6300 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:

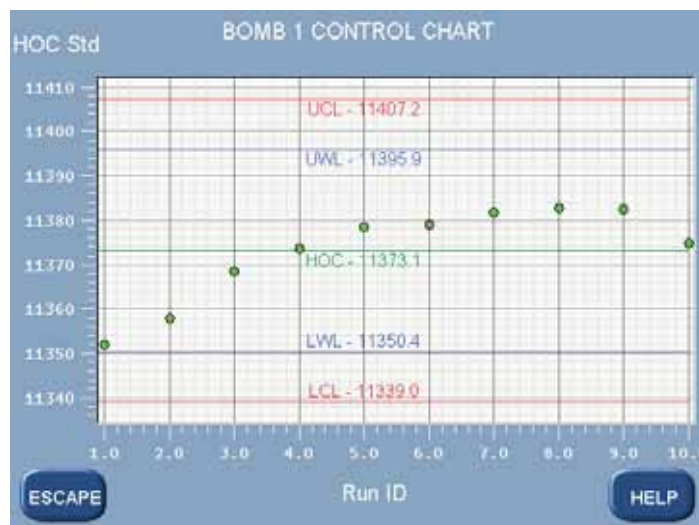
After bomb service, press this key to reset the fire count to zero.

Control Chart Plot:

Displays the current standardization runs being used to calculate the Bomb EE Value. The display will either

chart the value of the Heat of Combustion (HOC) of the Standard or the Energy Equivalent (EE) depending on the selection on the Control Chart Parameters menu (see Calibration Data and Controls menu).

You can display the information used for each test by selecting the appropriate dot.



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.

Bomb 2. Accesses sub-menu, Bomb 2. Provides the same controls as described for Bomb 1.

Bomb 3. Accesses sub-menu, Bomb 3. Provides the same controls as described for Bomb 1.

Bomb 4. Accesses sub-menu, Bomb 4. Provides the same controls as described for Bomb 1.

THERMOCHEMICAL CALCULATIONS MENU

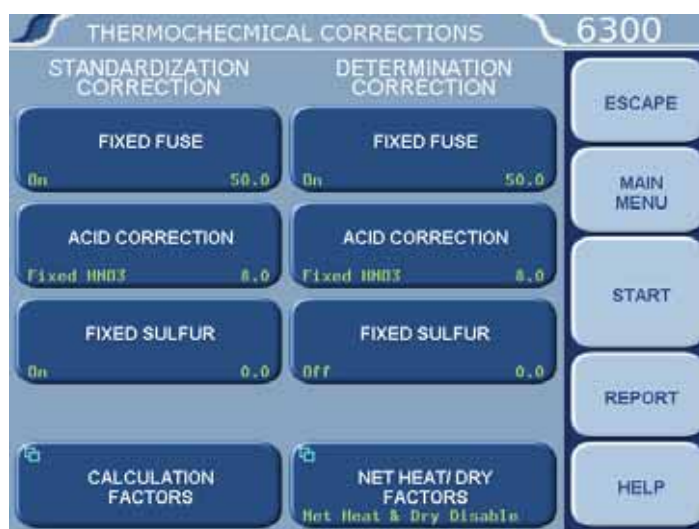
The Thermochemical Corrections Menu permits three types of fixed corrections for standardization (instrument calibration) runs, and the same three types for determination (test) runs. Pressing the LEFT side of each key toggles the correction ON or OFF. Press the RIGHT side of each key to access the specific 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 6300 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.

Acid Correction:

Displays both the ON/OFF of the fixed acid corrections for standardization runs and the value of the correction. This key toggles the options for treatment of the acid correction and accesses a sub-menu on which the value is set. An appropriate fixed HNO_3 value is 8 calories when one-gram benzoic acid pellets are used to calibrate the instrument.

Options for the Acid Correction:

Fixed HNO_3
Calculated HNO_3
Entered Total
Entered HNO_3
Fixed Total

These options are discussed further in Appendix B - Calculations.

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.

Acid Correction:

Displays both the ON/OFF of the fixed acid corrections for standardization runs and the value of the correction. This key toggles the options for treatment of the acid correction and accesses a sub-menu on which the value is set. An appropriate fixed HNO_3 value is 8 calories when one-gram benzoic acid pellets are used to calibrate the instrument.

Options for the Acid Correction:

Fixed HNO_3
Calculated HNO_3
Entered Total
Entered HNO_3
Fixed Total

These options are discussed further in Appendix B - Calculations.

THERMOCHEMICAL CALCULATIONS MENU (CONTINUED)

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.

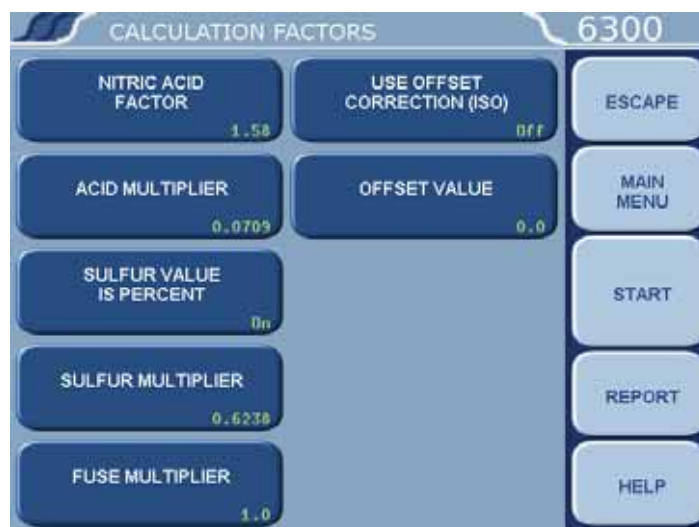
Calculation Factors:

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

Net Heat/Dry Factors:

Accesses the Net Heat/Dry Factors sub-menu, which provides for setting the net heat of combustion and Dry Factors Thermochemical Corrections.

CALCULATION FACTORS MENU



CALCULATION FACTORS		6300
NITRIC ACID FACTOR 1.58	USE OFFSET CORRECTION (ISO) OFF	ESCAPE
ACID MULTIPLIER 0.0709	OFFSET VALUE 0.0	MAIN MENU
SULFUR VALUE IS PERCENT ON		START
SULFUR MULTIPLIER 0.6238		REPORT
FUSE MULTIPLIER 1.0		HELP

Nitric Acid Factor:

Ratio of the nitric acid correction when the released energy corresponds to 6318 calories. The default is 1.58 calories per 1000 calories of released energy.

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:

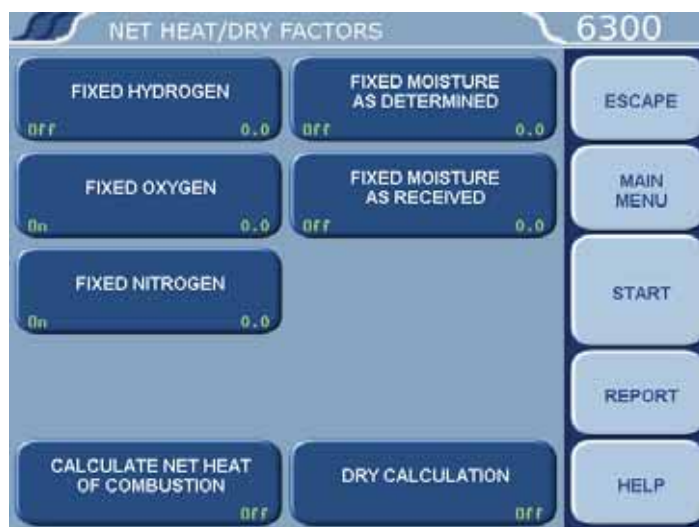
The fuse corrections represent the number of calories liberated by the burning fuse wire used to ignite the sample. If another measurement is used, the correction factor must be entered here. Press this key to access the Fuse Multiplier numeric dialog box and enter this multiplier value.

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.

NET HEAT/DRY HEAT FACTORS**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.

Fixed Oxygen:

ON/OFF and value entry.

Fixed Nitrogen:

ON/OFF and value entry.

Calculate Net Heat of Combustion:

ON/OFF. Turn On to have the calorimeter calculate the net heat of combustion.

Fixed Moisture as Determined:

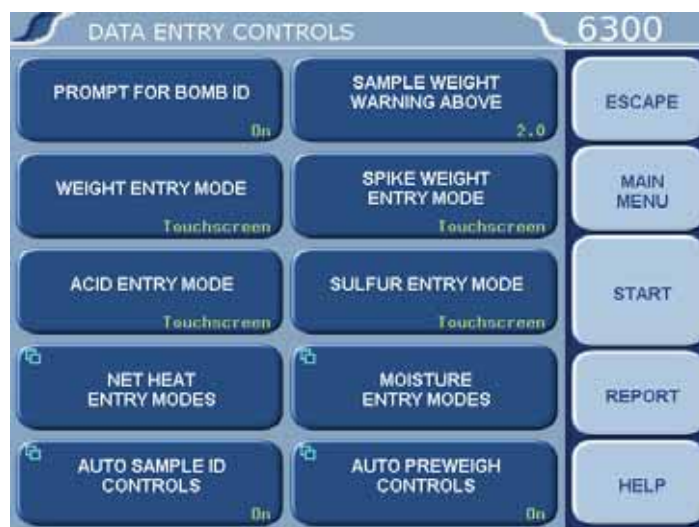
Press the LEFT side to toggle ON or OFF whether to use the entered moisture correction. Press the RIGHT side to access the Fixed Moisture as Determined numeric dialog box and set the value. Units are weight %.

Fixed Moisture as Received:

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 as Received numeric dialog box and set the value. Units are weight %.

Dry Calculation:

Toggles the dry calculation ON or OFF.

DATA ENTRY CONTROLS MENU**Prompt for Bomb ID:**

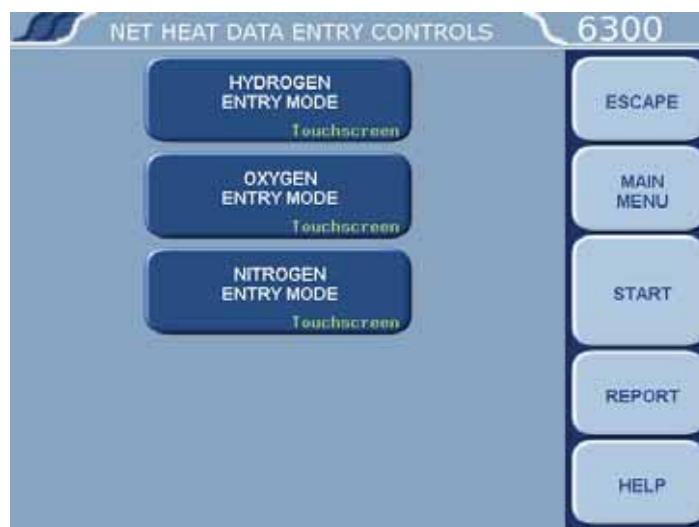
Toggles ON or OFF. In the ON position the controller will prompt for a Bomb ID (1-4) when a test is started.

Weight Entry Mode:

This key steps through the options for entering sample weights either manually through the touch screen, balance (USB) 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 (USB) port.



DATA ENTRY CONTROLS MENU (CONTINUED)

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 (USB) port.

Oxygen Entry Mode:

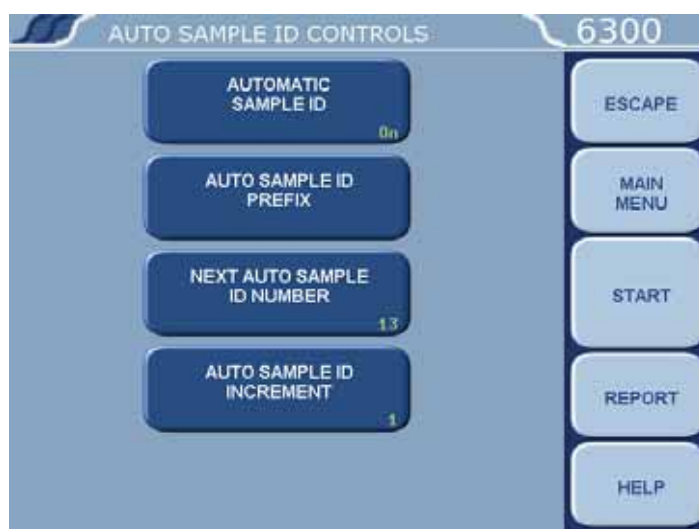
This key steps through the options for entering oxygen content for calculating the net heat of combustion either manually through the touch screen or automatically through the balance (USB) port.

Nitrogen Entry Mode:

This key steps through the options for entering nitrogen content for calculating the net heat of combustion either manually through the touch screen or automatically through the balance (USB) 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:

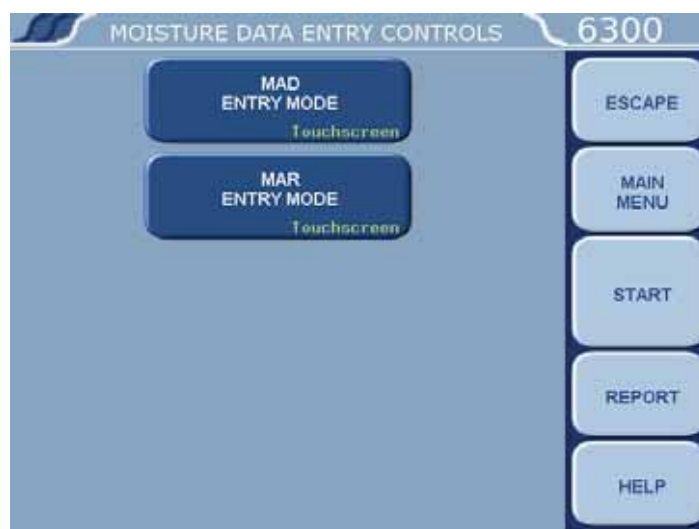
This key displays and leads to a sub-menu used to set the maximum allowable sample weight (including spike) in grams. A warning will be given if sample weights above this value are entered.

Spike Weight Entry Mode:

This key steps through the options for entering spike weights either manually through the touch screen, balance (USB) 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 (USB) port.

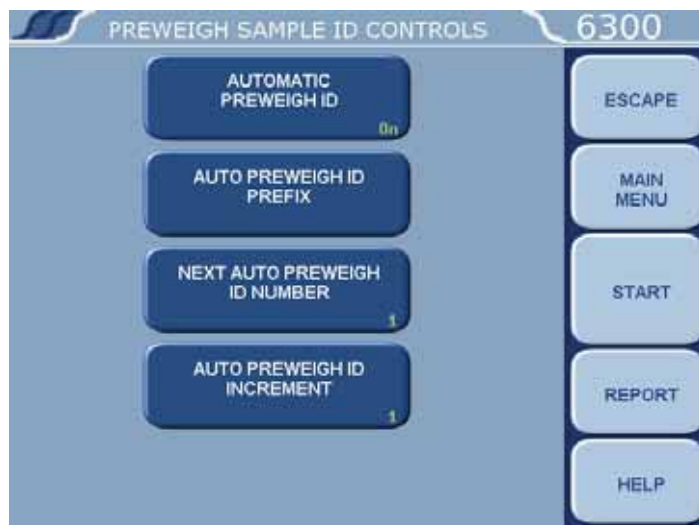


Moisture as Determined Entry Mode:

This key steps through the options for entering the moisture as determined correction value either manually or through the touch screen or automatically through the balance (USB) port.

Moisture as Received Entry Mode:

This key steps through the options for entering the moisture as received correction value either manually or through the touch screen or automatically through the balance (USB) port.

**Auto Prewrite ID Controls:**

Accesses sub-menu, used to automatically assign Sample ID numbers when a series of samples are pre weighed ahead of the time they are actually tested.

- Automatic Prewrite ID. ON/OFF toggle for this feature.
- Automatic Prewrite ID Prefix. An entry here will be used as a prefix for all pre-weigh sample IDs.
- Next Automatic Prewrite ID Number. Shows the next Sample ID which will be assigned and is used to enter the beginning Sample ID of any series
- Automatic Prewrite 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 1758 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 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.

REPORTING CONTROLS MENU (CONTINUED)

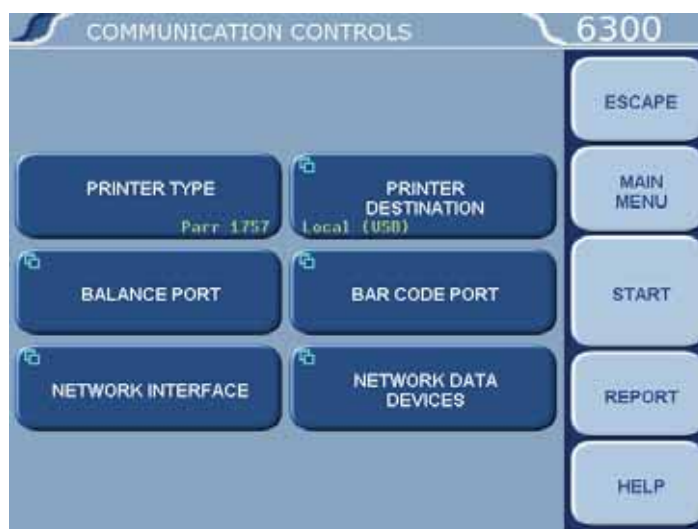
Use New EE Value in Recalculation:

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.

Report Schedule:

Toggles between End of Post Period and end of Cool/Rinse. This setting determines when the report will be printed or displayed.

COMMUNICATION CONTROLS MENU



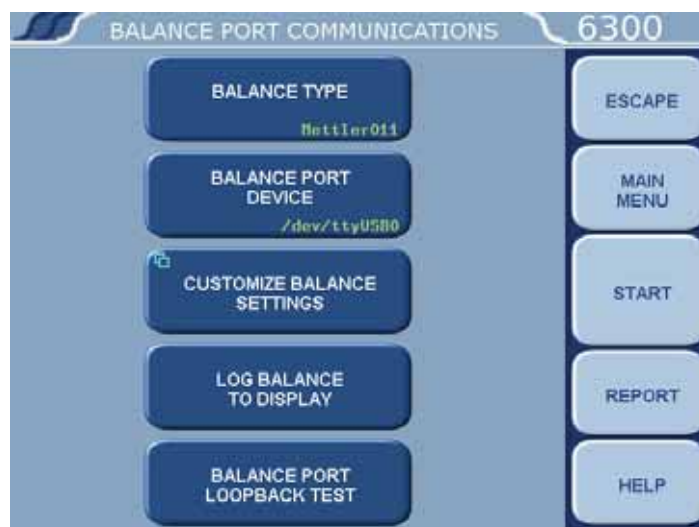
Accesses sub-menus which set the communications protocols for the printer and balances.

Printer Port Type:

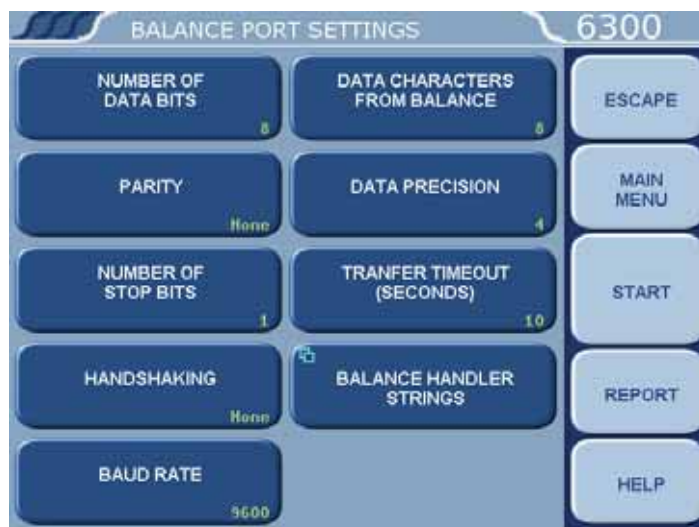
Toggles between Parr 1758 and Generic.

Balance Port:

Accesses sub-menu, Balance Port Communications.



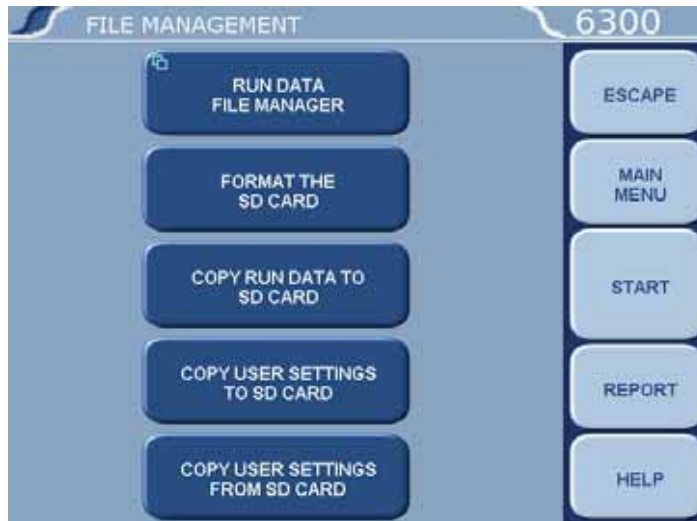
- Balance Type. Toggles through the available balance templates.
- Balance Port Device. This key displays a screen which allows the user to specify the balance port device. The default (dev/ttyUSB0) is the designation for the first USB to serial converter cable assigned by the calorimeter upon power up.
- Customize Balance Setting. Sets the communication parameters for the balance. 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.

Further information on establishing communications for the Printer, Balance, Network Interface, Bar Code and other Network Data Devices can be found in Appendix D, *Communication Interfaces*, of this manual.

FILE MANAGEMENT



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 SD Card:

This key allows the user to format an installed SD card in a manner that is compatible with the calorimeter.

Note:

Formatting will erase all files on the card!

Copy Run Data to SD Card:

This key copies all test data to an SD 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 SD card will overwrite the data currently on the card.

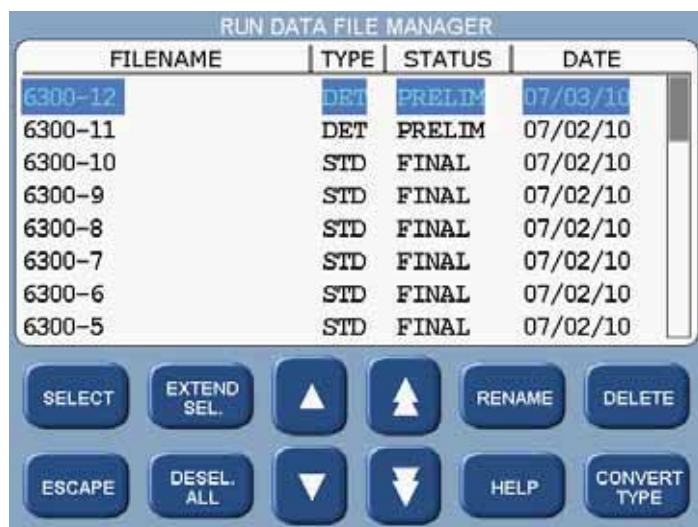
Copy User Settings to SD Card:

This key copies all previously saved user setups to SD.

Copy User Settings From SD Card:

This key copies all user setups previously saved to SD 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 (single arrow) and page up/page down (double arrow) 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.

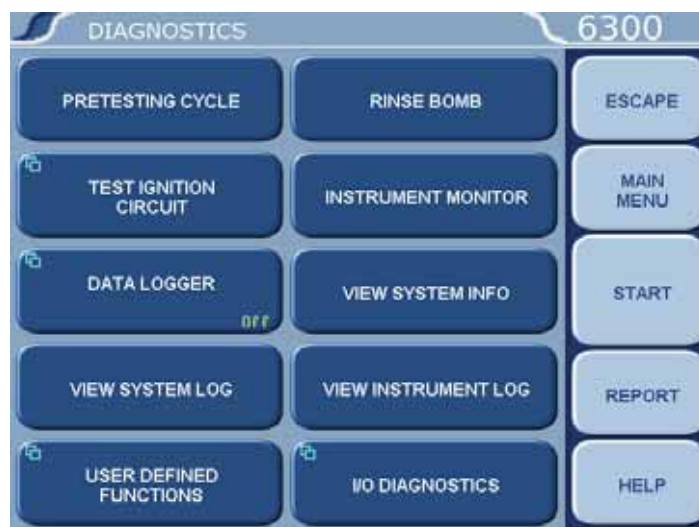
Delete:

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/log/datalog.csv. The maximum allowed size for this file is roughly one megabyte. If the file reaches this size, logging is halted.

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:

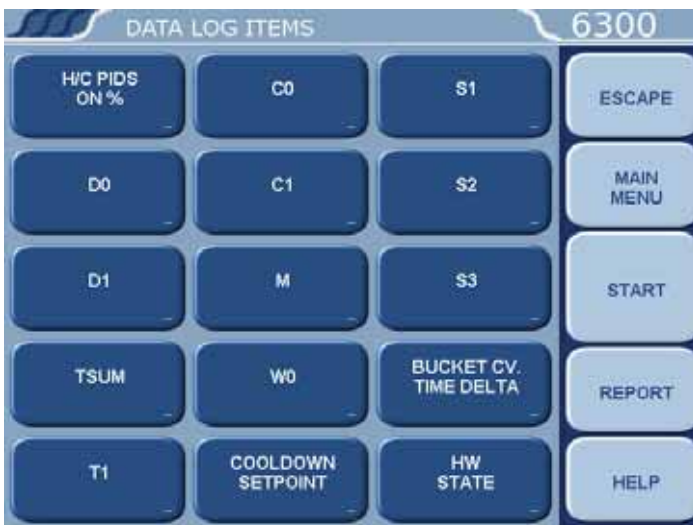
- » D0 - Corrected calorimeter drift rate
 - » Tsum - Accumulated temperature rise
 - » T1 - Extrapolated temperature rise
 - » C0 - Temperature conversion counter
- Data Log Format. Toggles between Text Format and Data Format (csv). Data is either logged with the supporting tag information (text) or 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.



- Select Data Log Items. Press this key to access the Data Log Items sub-menu, which provides

DIAGNOSTICS MENU (CONTINUED)

- 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.

APPENDIX B

CALCULATIONS

CALCULATING THE HEAT OF COMBUSTION

The 6300 Calorimeter will automatically make all of the calculations necessary to produce a gross heat of combustion for the sample. However, it is important that the user understand these calculations to ensure the instrument is set up so the calculations match the procedures and the units are consistent throughout the process.

GENERAL CALCULATIONS

The calculation for the gross heat of combustion is done by:

$$H_c = \frac{WT - e_1 - e_2 - e_3}{m}$$

Where:

- H_c = Gross heat of combustion.
 T = Observed temperature rise.
 W = Energy equivalent of the calorimeter being used.
 e_1 = Heat produced by burning the nitrogen portion of the air trapped in the bomb to form nitric acid.
 e_2 = The heat produced by the formation of sulfuric acid from the reaction of sulfur dioxide, water and oxygen.
 e_3 = Heat produced by the heating wire and cotton thread.
 m = Mass of the sample.

These calculations are made in calories, grams, and degrees Celsius and then converted to other units if required.

Temperature Rise.

The 6300 Calorimeter produces a corrected temperature rise reading automatically. Corrections for heat leaks during the test are applied. (For a complete discussion of this process see *Introduction to Bomb Calorimetry*, Manual No. 483M).

Energy Equivalent.

The energy equivalent (represented by W in the formula, or abbreviated as EE) is determined by standardizing the calorimeter as described in Appendix C - *Standardization*. It is an expression of the amount of energy required to raise the temperature of the calorimeter one degree. It is commonly expressed in calories per degree Celsius. Since it is directly related to the mass of the calorimeter, it will change whenever any of the components of the calorimeter (i.e. the bomb, bucket or amount of water) is changed.

THERMOCHEMICAL CORRECTIONS

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.

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.

Fuse Correction.

The fuse correction in the 6300 Calorimeter is significantly different than the correction used in earlier model Parr calorimeters where the correction was made to compensate for the amount of fuse wire burned in the test. There are two components to the fuse correction in the 6300 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.

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

THERMOCHEMICAL CORRECTIONS (CONTINUED)

be calculated. In the 6300 Calorimeter this is a fixed 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.

ASTM AND ISO METHODS DIFFER

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 6300 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.

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 Acid Correction - Thermochemical Corrections, is set to Fixed HNO₃. 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.

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 and then enter percent sulfur as indicated on this line. Any errors will be proportional to the difference between the actual and assumed value for sulfur.

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.

Please note that the values entered into the test report appear as entered in the report. Values for e_1 , e_2 and e_3 are calculated and used as energy corrections in accordance with the formulas and settings given above. The formulas used above to arrive at e_1 or e_2 are not the same as the formulas used for e_1 and e_2 which appear in most ASTM bomb calorimetric procedures. However, the sum of e_1 and e_2 , above, is equal to the sum of the ASTM treatment of e_1 and e_2 .

Note:

Please review the following section on Acid and Sulfur Corrections. Different standard test methods use different values for the heat of formation of sulfuric acid. These differences are generally insignificant. The 6300 Calorimeter uses the most recent, published values for all thermochemical data.

Thermochemical Calculation Details

Traditionally, standard solutions and procedures have been established to simplify the calculations related to

the thermochemical corrections. The 6300 Calorimeter has been programmed to permit the user to use standard solutions and units which are most convenient, since the microprocessor can easily apply any conversion factors required.

FUSE CORRECTION

The fuse correction applied by the calorimeter is calculated as:

$$e_3 = (\text{fuse value})(\text{fuse multiplier from calculation factors page})$$

$$= (\text{entered value})(\text{fuse multiplier from thermochemicals page})$$

"Fuse Value" is the number entered by the user and the value which appears in the test report.

Note:

Calculation Factors,- Fuse Multiplier is normally set to 1.0 so the entered value is in calories.

Users may find it convenient to enter a fixed value for the fuse correction and avoid the need to determine this correction for each test.

Fixed fuse corrections can be entered when Thermochemical Corrections, is set to ON. By default a fixed fuse correction of 50 calories is applied to all tests. Total errors of more than 5 calories will seldom occur when using a fixed fuse correction and the ignition thread supplied by Parr.

ACID AND SULFUR CORRECTIONS

- **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 normally the concentration (normality) of the base in equivalents per liter (N).
- **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.

Sulfur Correction:

$$e_2 = (\text{percent sulfur})(\text{sample mass})(\text{sulfur multiplier})$$

(heat of formation of H_2SO_4).

Acid Correction:

In the 6300 there are a number of settings for the acid correction.

e_1 is the nitric acid portion of the correction.

Fixed HNO_3 : The Acid Correction is a fixed value set by the operator.

The calculation is:

$$e_1 = (\text{nitric acid value})(\text{acid multiplier})(\text{heat of formation of nitric acid})$$

For an 1138 bomb the default nitric acid value is 8 and acid multiplier is .0709. The heat of formation of nitric acid is 14.1 calories/milliequivalent so the calculation is:

$$e_1 = (8)(.0709)(14.1) \text{ or } e_1 = 7.9975 \text{ calories (rounds to 8)}$$

When the Acid Correction is set to Fixed HNO_3 the value is considered a final value and the operator is not prompted for an acid value when reporting the results.

Entered HNO_3 : The Acid Correction is entered by the operator when reporting the results.

The calculation is the same as Fixed HNO_3 above. The value listed on the Acid Correction button is used for preliminary calculations. When finalizing the report the operator will be prompted for the acid value.

ACID AND SULFUR CORRECTIONS (CONTINUED)

Fixed Total: The Acid Correction represents the total base required to titrate the bomb washings (in milliliters). This includes both nitric and sulfuric acid. The correction is a fixed value set by the operator.

The calculation is:

$$e1 = [((\text{total acid})(\text{acid multiplier})) - (\% \text{ sulfur})(\text{sample mass})(\text{sulfur multiplier})](\text{heat of formation of nitric acid})$$

Using the default acid and sulfur multipliers as well as a heat of formation of nitric acid of 14.1 cal/milliequivalent a 1 gram sample with 25 ml of washings and 2 % sulfur would result in the following calculation:

$$\begin{aligned} e1 &= [((25)(.0709)) - (2)(1)(.6238)] 14.1 \\ e1 &= [(1.7725) - (1.2476)] 14.1 \\ e1 &= [.5249] 14.1 \\ e1 &= 7.40109 \text{ calories} \end{aligned}$$

When the Acid Correction is set to Fixed Total the value is considered a final value and the operator is not prompted for an acid value when reporting the results.

Entered Total: The Acid Correction represents the total base required to titrate the bomb washings (in milliliters). This includes both nitric and sulfuric acid. The correction is entered by the operator when reporting the results.

The calculation is the same as the Fixed Total above.

The value listed on the Acid Correction button is used for preliminary calculations. When finalizing the report the operator will be prompted for the acid value.

Calculated HNO₃: In ASTM D5865 there are provisions for calculating the nitric acid contribution.

For test samples that contain no nitrogen, the quantity of nitric acid formed during the combustion process is a function of the volume of the bomb, the oxygen filling pressure, and the quantity of energy released.

For the calculated nitric acid method:

$$e1 = (\text{nitric acid factor}/1000)(\text{Energy Equivalent})(\text{corrected temperature rise})$$

Example: For a test run with energy equivalent of 927.4022 and a corrected temperature rise of 6.892 would result:

$$\begin{aligned} e1 &= (1.58/1000)(927.4022)(6.892) \\ e1 &= 10.0988 \text{ calories} \end{aligned}$$

The calculated nitric acid method can be applied to samples containing up to 2% nitrogen without introducing a significant error in the resulting heat of combustion value.

Table B-1
Settings for ISO & BSI Methods

Page	Line	Setting	Value
Thermochemical Corrections	Acid Correction (STD)	Entered HNO ₃	13
	Fixed Sulfur STD	Off	7
	Acid Correction (DET)	Entered HNO ₃	13
	Fixed Sulfur DET	Off	7
Calculations Factors	Acid Multiplier		0.154
	Sulfur Value is Percent	Off	
	Sulfur Multiplier		0.1
	Use Offset Correction	On	
	Offset Value		-43.5
	Offset Value		-43.5

ASTM TREATMENT FOR ACID AND SULFUR

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 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 CALCULATIONS

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. Provision has been made in the 6300 Controller for dealing with these different 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 B-1 gives the settings which allows 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 B-1 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.

SPIKING SAMPLES

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, particularly for liquid samples. The 6300 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} = Heat of combustion of the spiking material (cal/g)

M_s = Mass of spiking material

This factor is added to the calculations when Spike Controls, Use Spiking is set to ON. Heat of Combustion of Spike is entered as calories per gram. The controller will prompt the user to enter the weight of spiking material. Fixed spikes can be used when, Use Fixed Spike is set to ON and entering the mass of the spike on - Weight of Fixed Spike.

CONVERSION TO OTHER MOISTURE BASES

The calculations described above give the calorific value of the sample with moisture as it existed when the sample was weighed. For example, if an air-dried coal sample was tested, the results will be in terms of heat units per weight of air-dry sample. This can be converted to a moisture free or other 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:

$$H_{net} = \frac{1.8H_c - 91.23H}{(\text{Liquid fuels, ASTM D240})}$$

To calculate H_{net} for solid fuels please refer to ASTM D5865.

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APPENDIX C

STANDARDIZATION

STANDARDIZING THE CALORIMETER

The Energy Equivalent Factor.

The term “standardization”, as used here, denotes the operation of the calorimeter on a standard sample from which the energy equivalent or effective heat capacity of the system can be determined. The energy equivalent, W or EE of the calorimeter is the energy required to raise the temperature one degree, usually expressed as calories per degree Celsius. Standardization tests should be repeated after changing any parts of the calorimeter, and occasionally as a check on both the calorimeter and operating technique.

Standardization Procedure.

The procedure for a standardization test is exactly the same as for testing a fuel sample. Use a pellet of calorific grade benzoic acid weighing not less than 0.9 nor more than 1.1 grams. The corrected temperature rise, T, is determined from the observed test data and the bomb washings are titrated to determine the nitric acid correction. The energy equivalent is computed by substituting the following equation:

$$W = \frac{Hm + e_1 + e_2 + e_3}{T}$$

Where:

- W = Energy equivalent of the calorimeter in calories per °C.
- H = Heat of combustion of the standard benzoic acid sample in calories per gram.
- m = Mass of the standard benzoic acid sample in grams.
- T = Temperature rise in °C.
- e₁ = Correction for heat of formation of nitric acid in calories.
- e₂ = Correction for sulfur which is usually 0.
- e₃ = Correction for heating wire and combustion of cotton thread.

STANDARD MATERIALS

A bottle of 100 one-gram benzoic acid pellets (Part No. 3415) is furnished with each calorimeter for standardizing purposes. The Parr benzoic acid has been calibrated against NIST benzoic acid. Additional benzoic acid pellets can be obtained from Parr. For very high precision measurements, a primary standard benzoic acid powder can be purchased from the National Institute of Standards & Technology, Washington, D.C.

It is not common to have sulfur in standard materials, or to use spikes in standardizations, but the capabilities have been included in this calorimeter.

Users should take great care to ensure that the conditions during standardization runs and determinations are as identical as possible.



Caution:

Benzoic acid must always be compressed into a pellet before it is burned in an oxygen bomb to avoid possible damage from rapid combustion of the loose powder. This is best accomplished by using a Parr 2811 Pellet Press.

AUTOMATIC STATISTICAL CALCULATIONS

The 6300 Calorimeter includes a provision for calculating and using a mean energy equivalent for each of up to 4 separate bomb and bucket combinations. ASTM procedures recommend that the energy equivalent be determined by averaging ten tests. The 6300 Calorimeter automatically determines and uses up to ten tests in its memory and will update the EE Value as additional standardizations are run. Only Final Tests will be used in determining and updating EE values. These values, the number of tests, and the relative standard deviation for the tests used in determining the EE value are stored in the Calibration Data Page under the EE Value for each bomb.

The user can chose to turn off the automatic averaging and updating procedure and protect the EE Values by turning ON the protection feature for the appropriate bomb on the Calibration Data and Control Page using Protected EE Value.

AUTOMATIC STATISTICAL CALCULATIONS (CONTINUED)

Any outliers or other tests which should not be included in the average EE Value must be deleted from the memory using the memory management procedures (see Chapter 8). A list of all tests associated with any Cal ID can be printed from the Calibration Data Page using Print Standardization Runs.

The user can elect to have any number of stored standardization runs used in determining the EE value by entering this number on Calibration Data & Controls Page - Calibration Run Limit.

EE Max Std Deviation on this same page establishes the maximum allowable standard deviation for the EE Value before an error condition is reported. The default value is zero which turns off this limit. But the user should enter a value appropriate for the test being made.

Table C-1
Calorimeter Control Limit Values in J/g When Benzoic Acid is Used as a Test Sample

Accepted heat of combustion taken as 26454 J/g.

Instrument precision 0.10%.

Control limits based on 99% confidence (3 sigma) values.

Values are in J/g.

NUMBER OF OBSERVATIONS IN A GROUP	UCL FOR THE RANGE (HIGH – LOW) WITHIN THE GROUP	UCL FOR THE RSD WITHIN THE GROUP	MAXIMUM PERMISSIBLE DEVIATION OF THE GROUP MEAN FROM THE ACCEPTED VALUE OR GRAND MEAN
1			79.4
2	97.5	0.261%	56.1
3	115.3	0.228%	45.8
4	124.3	0.209%	39.7
5	130.1	0.196%	35.5
6	134.3	0.187%	32.4
7	137.6	0.181%	30.0
8	140.4	0.175%	28.1
9	142.7	0.171%	26.5
10	144.7	0.167%	25.1
11	146.4	0.164%	23.9
12	147.9	0.161%	22.9
13	149.4	0.159%	22.0
14	150.7	0.156%	21.2
15	151.8	0.154%	20.5
16	153.0	0.153%	19.8
17	154.0	0.151%	19.2
18	154.9	0.150%	18.7
19	155.8	0.148%	18.2
20	156.7	0.147%	17.7
21	157.4	0.146%	17.3
22	158.2	0.145%	16.9
23	158.9	0.144%	16.5
24	159.5	0.143%	16.2
25	160.2	0.142%	15.9

Table C-2***Calorimeter Control Limit Values in cal/g When Benzoic Acid is Used as a Test Sample***

Accepted heat of combustion taken as 6318 cal/g.

Instrument precision 0.10%.

Control limits based on 99% confidence (3 sigma) values.

Values are in cal/g.

NUMBER OF OBSERVATIONS IN A GROUP	UCL FOR THE RANGE (HIGH – LOW) WITHIN THE GROUP	UCL FOR THE RSD WITHIN THE GROUP	MAXIMUM PERMISSIBLE DEVIATION OF THE GROUP MEAN FROM THE ACCEPTED VALUE OR GRAND MEAN
1			19.0
2	23.3	0.261%	13.4
3	27.5	0.228%	10.9
4	29.7	0.209%	9.5
5	31.1	0.196%	8.5
6	32.1	0.187%	7.7
7	32.9	0.181%	7.2
8	33.5	0.175%	6.7
9	34.1	0.171%	6.3
10	34.6	0.167%	6.0
11	35.0	0.164%	5.7
12	35.3	0.161%	5.5
13	35.7	0.159%	5.3
14	36.0	0.156%	5.1
15	36.3	0.154%	4.9
16	36.5	0.153%	4.7
17	36.8	0.151%	4.6
18	37.0	0.150%	4.5
19	37.2	0.148%	4.3
20	37.4	0.147%	4.2
21	37.6	0.146%	4.1
22	37.8	0.145%	4.0
23	37.9	0.144%	4.0
24	38.1	0.143%	3.9
25	38.3	0.142%	3.8

Table C-3
Calorimeter Control Limit Values in BTU/lb When Benzoic Acid is Used as a Test Sample

Accepted heat of combustion taken as 11373 BTU/lb.

Instrument precision 0.10%.

Control limits based on 99% confidence (3 sigma) values.

Values are in BTU/lb.

NUMBER OF OBSERVATIONS IN A GROUP	UCL FOR THE RANGE (HIGH – LOW) WITHIN THE GROUP	UCL FOR THE RSD WITHIN THE GROUP	MAXIMUM PERMISSIBLE DEVIATION OF THE GROUP MEAN FROM THE ACCEPTED VALUE OR GRAND MEAN
1			34.1
2	41.9	0.261%	24.1
3	49.6	0.228%	19.7
4	53.4	0.209%	17.1
5	55.9	0.196%	15.3
6	57.8	0.187%	13.9
7	59.2	0.181%	12.9
8	60.4	0.175%	12.1
9	61.3	0.171%	11.4
10	62.2	0.167%	10.8
11	62.9	0.164%	10.3
12	63.6	0.161%	9.8
13	64.2	0.159%	9.5
14	64.8	0.156%	9.1
15	65.3	0.154%	8.8
16	65.8	0.153%	8.5
17	66.2	0.151%	8.3
18	66.6	0.150%	8.0
19	67.0	0.148%	7.8
20	67.4	0.147%	7.6
21	67.7	0.146%	7.4
22	68.0	0.145%	7.3
23	68.3	0.144%	7.1
24	68.6	0.143%	7.0
25	68.9	0.142%	6.8

APPENDIX D

COMMUNICATIONS INTERFACES

USB PORT

USB Connection

The 6300 Calorimeter is equipped with a USB port for connection to either a 40 or 80 column printer and/or a computer.

The default parameters for the 6300 Calorimeter are set up for use with the Parr 1758 Printer.

BALANCE AND PORT INPUT DRIVER SPECIFICATIONS

The 6300 Calorimeter supports input from multiple balance types. Additionally, a generic input driver is provided for communications with balances that do not conform to the 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 BALANCE INTERFACE

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

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.

SARTORIUS BALANCE INTERFACE

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

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]

Note:

The automatic data output option should not be used.

The calorimeter will send this command string once every few seconds after the ENTER key has been pressed during a mass entry sequence. The ENTER key should only be pressed when the mass reading is stable. However, unstable readings will be rejected and a warning will be issued. Acknowledging the warning by pressing the CLEAR ENTRY key will re-issue the command string to the balance on a periodic basis.

GENERIC INTERFACE

Field	Length
data	9
CR	1

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.

Table D-1
6300 Data File Naming Convention

Test data files are named with the following convention.

Test Type	Filename
Preliminary Standardization	<ID>.std.plim.csv
Final Standardization	<ID>.std.finl.csv
Preliminary Determination	<ID>.det.plim.csv
Final Determination	<ID>.det.finl.csv
Pre-weigh	<ID>.-.-.pwgh.csv

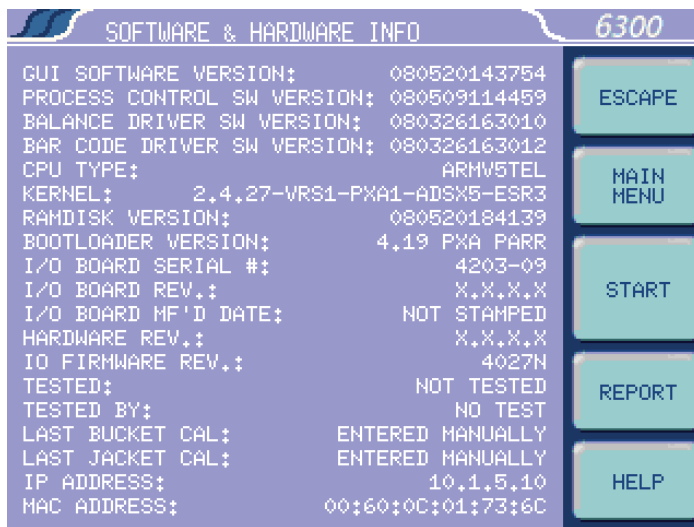
Table D-2
6300 Calorimeter Run Data Template

Field	Description
SampleID	char[16]
Timestamp	MM/DD/YY HH:mm:ss
Mode	0 = determination, 1 = standardization
Method	0 = equilibrium, 1 = dynamic
State	0 = preweigh, 1 = preliminary, 2 = final
Units	0 = MJ/kg, 1 = Btu/lb, 2 = cal/g, 3 = J/kg, 4 = other
UnitMultIfOther	unit multiplier in effect at time of report
BombID	[1,4]
BombEE	bomb energy equivalent
SampleWt	sample weight
SpikeWt	spike weight
Fuse	fuse value

FuseFinal	fuse value is final
Acid	acid value
AcidFinal	acid value is final
Sulfur	sulfur value
SulfurFinal	sulfur value is final
Hydrogen	hydrogen value (net calc option)
HydrogenFinal	hydrogen value is final (net calc option)
MAD	moisture as determined value (dry calc option)
MAD Final	moisture as determined is final
JacketTemp	jacket temperature
InitTemp	initial temperature
DeltaT	temperature rise
HOC	gross heat of combustion
NetHOC	dry net HOC (net calc options enabled)
DryHOC	dry gross HOC (if dry calc option enabled)
DryNetHOC	dry net HOC (if both dry and net calc options enabled)
Oxygen	oxygen value (net calc option)
Oxygen Final	oxygen value is final
Nitrogen	nitrogen value (net calc option)
Nitrogen Final	nitrogen value is final
MAR	moisture as received (dry calc option)
MAR Final	moisture as received value is final
Dry Net HOC_AR	Dry net HOC as received value (if both dry and net calc option enabled)
Bomb Name	bomb name assigned to bomb ID

ETHERNET INTERFACE

Calorimeter 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 or a static IP address can be assigned. The address can be seen on the “software and hardware info” page, under “program information and control”. 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/`

The datalog file can be accessed at:

`ftp://root:rootroot@192.168.0.125/../../flash/log/datalog.csv`

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

SAMBA SERVER FEATURE (OPTIONAL)

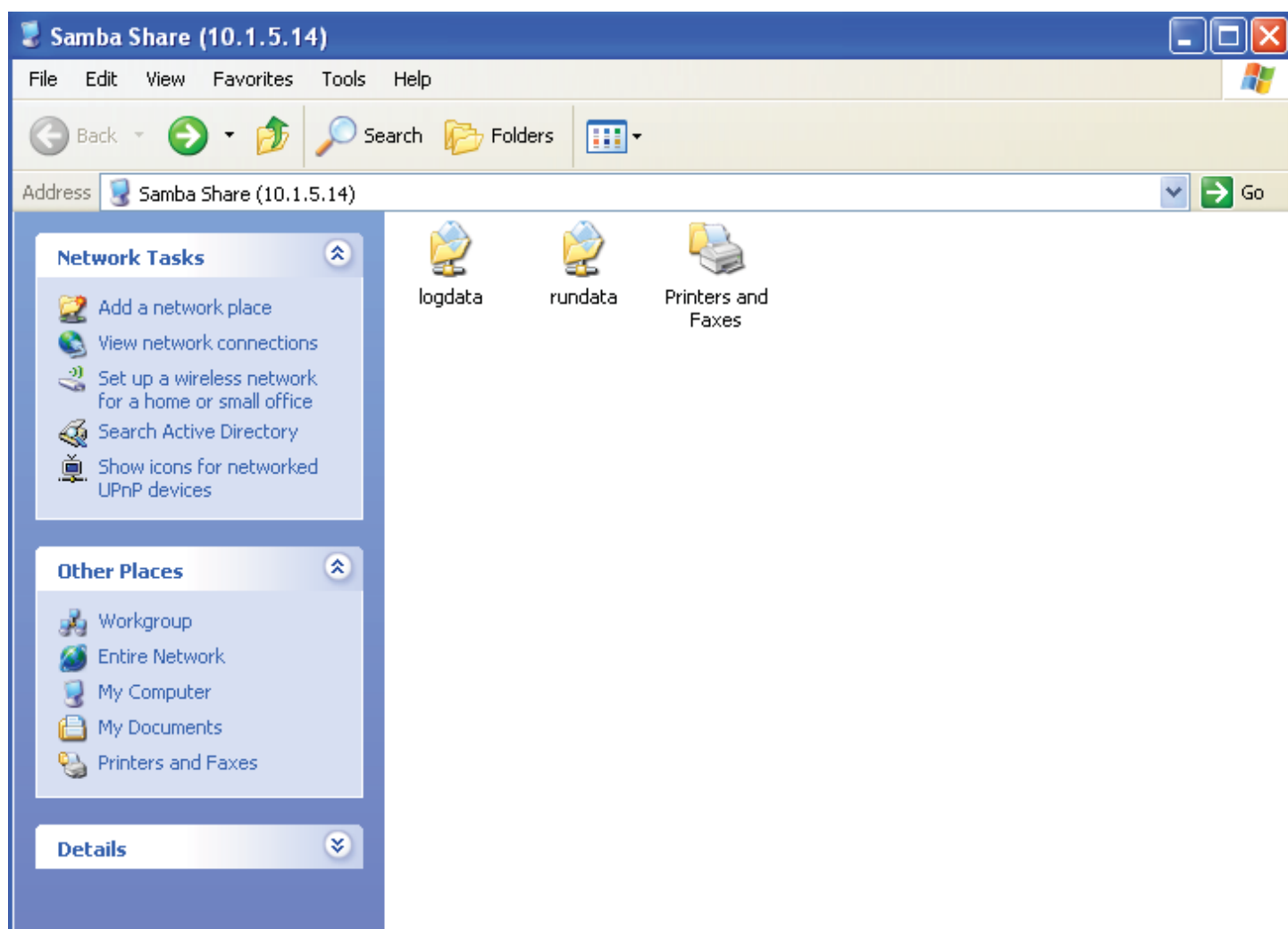
Samba was originally developed as an implementation of the SMB (Server Message Block) protocol. The most common use of SMB is in Microsoft's CIFS (Common Internet File System) implementation. As a result, Samba has become a de facto Microsoft network compatibility tool. In relation to CIFS, Samba allows non-Microsoft operating systems to enjoy effectively seamless server and client operation in networks catering to the needs of Windows computers. It is an "open" standard and defined in IETF RFC1001 and RFC1002.

The Samba server feature option in the Parr 6300 Calorimeter offers seamless file services to Windows based clients. It allows the calorimeter to interact with a Microsoft Windows client as if it is a Windows file server. The Samba server feature can be used to facilitate data file transfer from a calorimeter or proximate interface

to a PC running the Windows operating system. This method of file transfer, for some users, may be less cumbersome and more intuitive than using a web browser as an FTP client program to retrieve or log files.

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.

To access the test data open the run data folder. To access the log file open the log data folder.



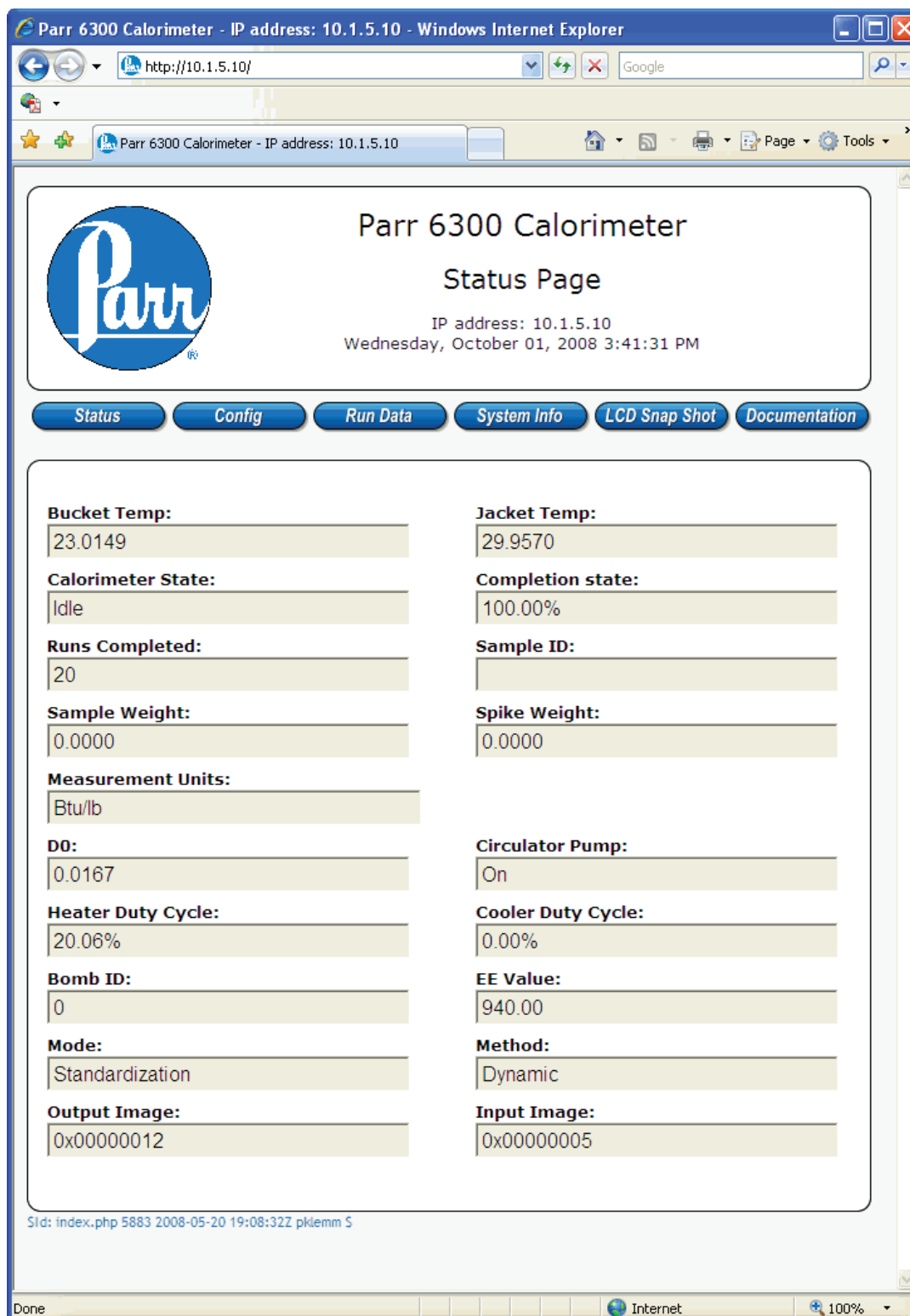
The following screenshot illustrates the contents of the calorimeter data directory as presented by a web browser.



The calorimeter offers a web server service. Test reports can be viewed with a web browser using a URL of the following form.

`http://10.1.5.10`

Where 10.1.5.10 is the IP address of the calorimeter. The following screenshot illustrates the calorimeter home page.



Parr 6300 Calorimeter
Status Page
 IP address: 10.1.5.10
 Wednesday, October 01, 2008 3:41:31 PM

Buttons: [Status](#) [Config](#) [Run Data](#) [System Info](#) [LCD Snap Shot](#) [Documentation](#)

Bucket Temp: 23.0149	Jacket Temp: 29.9570
Calorimeter State: Idle	Completion state: 100.00%
Runs Completed: 20	Sample ID:
Sample Weight: 0.0000	Spike Weight: 0.0000
Measurement Units: Btu/lb	
DO: 0.0167	Circulator Pump: On
Heater Duty Cycle: 20.06%	Cooler Duty Cycle: 0.00%
Bomb ID: 0	EE Value: 940.00
Mode: Standardization	Method: Dynamic
Output Image: 0x00000012	Input Image: 0x00000005

Std: index.php 5883 2008-05-20 19:08:32Z pklemm S

Clicking on the Config button will display the screen below. Changes made on this screen will change the settings in the calorimeter.

The screenshot shows a web browser window titled "Parr 6300 Calorimeter - IP address: 10.1.5.10 - Windows Internet Explorer". The address bar shows "http://10.1.5.10/configure.php". The page features the Parr logo and the title "Parr 6300 Calorimeter Configuration Page". Below the title, it displays the IP address "10.1.5.10" and the date/time "Wednesday, October 01, 2008 4:38:21 PM". A navigation bar contains buttons for "Status", "Config", "Run Data", "System Info", "LCD Snap Shot", and "Documentation". The "Config" button is highlighted. The main content area is divided into three sections: "Error Reporting", "Sample ID & Bomb ID Options", and "Data Entry Options".

Error Reporting

Print Error Messages ☒

Sample ID & Bomb ID Options

Automatic Sample ID Generation	☒	Automatic Prewrite ID Generation	☒
Sample ID Prefix		Prewrite ID Prefix	
Next Sample ID	1	Next Prewrite ID	1
Sample ID Increment	1	Prewrite ID Increment	1
Prompt for Bomb ID	☒		
Sample Weight Warning	2.0		

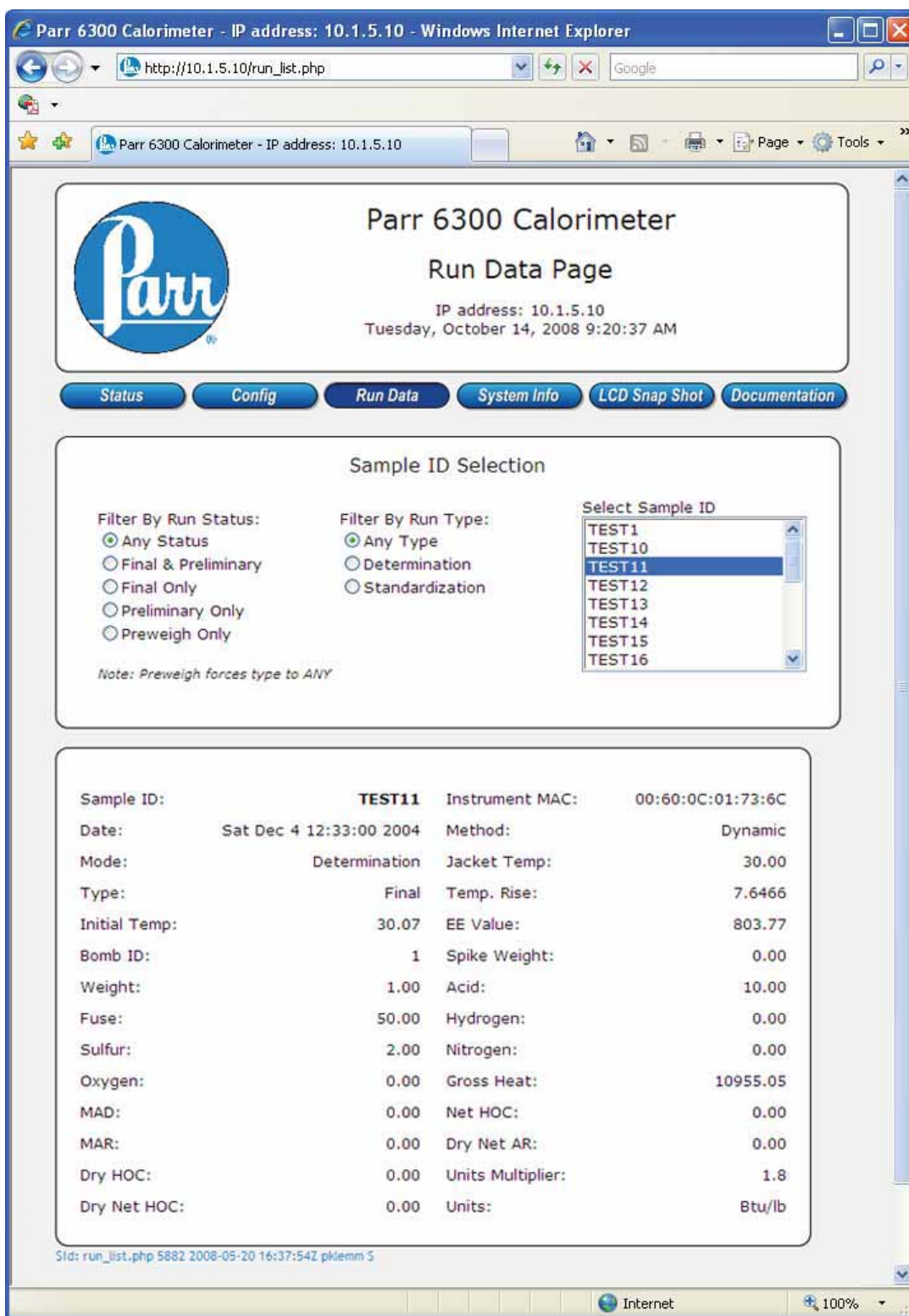
Data Entry Options

Weight Entry Source	Touchscreen	Use Spike Correction	☐
Acid Value Entry Source	Touchscreen	Prompt for Spike Weight	☐
Sulfur Value Entry Source	Touchscreen	Spike Weight Entry Source	Touchscreen

SLid: configure.php 5882 2008-05-20 16:37:54Z pklemm S

Clicking on the Run Data button displays a list of reports currently in the instrument memory.

Clicking on a test under the select sample ID box will display the data for the selected sample ID.



Parr 6300 Calorimeter
Run Data Page
IP address: 10.1.5.10
Tuesday, October 14, 2008 9:20:37 AM

[Status](#)
[Config](#)
[Run Data](#)
[System Info](#)
[LCD Snap Shot](#)
[Documentation](#)

Sample ID Selection

Filter By Run Status:

- ☒ Any Status
- ☐ Final & Preliminary
- ☐ Final Only
- ☐ Preliminary Only
- ☐ Preweigh Only

Filter By Run Type:

- ☒ Any Type
- ☐ Determination
- ☐ Standardization

Select Sample ID

- TEST1
- TEST10
- TEST11**
- TEST12
- TEST13
- TEST14
- TEST15
- TEST16

Note: Preweigh forces type to ANY

Sample ID:	TEST11	Instrument MAC:	00:60:0C:01:73:6C
Date:	Sat Dec 4 12:33:00 2004	Method:	Dynamic
Mode:	Determination	Jacket Temp:	30.00
Type:	Final	Temp. Rise:	7.6466
Initial Temp:	30.07	EE Value:	803.77
Bomb ID:	1	Spike Weight:	0.00
Weight:	1.00	Acid:	10.00
Fuse:	50.00	Hydrogen:	0.00
Sulfur:	2.00	Nitrogen:	0.00
Oxygen:	0.00	Gross Heat:	10955.05
MAD:	0.00	Net HOC:	0.00
MAR:	0.00	Dry Net AR:	0.00
Dry HOC:	0.00	Units Multiplier:	1.8
Dry Net HOC:	0.00	Units:	Btu/lb

Std: run_list.php 5882 2008-05-20 16:37:54Z pklemm S

Clicking on the System Info button will display the screen below.

The screenshot shows a web browser window titled "Parr 6300 Calorimeter - IP address: 10.1.5.100 - Windows Internet Explorer". The address bar shows "http://10.1.5.100/systeminfo.php". The page content includes the Parr logo, the title "Parr 6300 Calorimeter System Information Page", and the IP address "10.1.5.100" with the date "Thursday, August 12, 2010 11:06:07 AM". Below this is a navigation bar with buttons for "Status", "Config", "Run Data", "System Info", "LCD Snap Shot", and "Documentation". The "System Info" button is highlighted. The main content area displays a list of system information items, each with a label and a value. At the bottom, there is a status bar showing "SId: systeminfo.php 117 2010-04-29 02:10:27Z pklemm S".

Label	Value
Ramdisk Version:	100806013327
GUI Software Version:	100805225942
Process Control Software Version:	100806011338
Balance Driver Software Version:	100624144631
Barcode Driver Software Version:	
CPU Type:	armv5tel
Kernel Version:	2.6.26-ads4
Boot Loader Version:	700120-1450c
IO Board Serial Number:	8334
IO Board Version:	NOT SET
IO Board Mfg. Date:	NOT SET
Hardware Revision:	NOT SET
IO Firmware Version:	Jul 17 2010 14:03:05
Test Date:	NOT SET
Tested By:	NOT SET
Bucket Channel Calibration:	NOT SET
Jacket Channel Calibration:	NOT SET
IP Address:	10.1.5.100
MAC Address:	00:60:0C:01:D5:EF

SId: systeminfo.php 117 2010-04-29 02:10:27Z pklemm S

Clicking on the LCD Snap Shot button will display the current menu screen displayed by the calorimeter. If the backlight is not on, this screen will display a blank blue square.

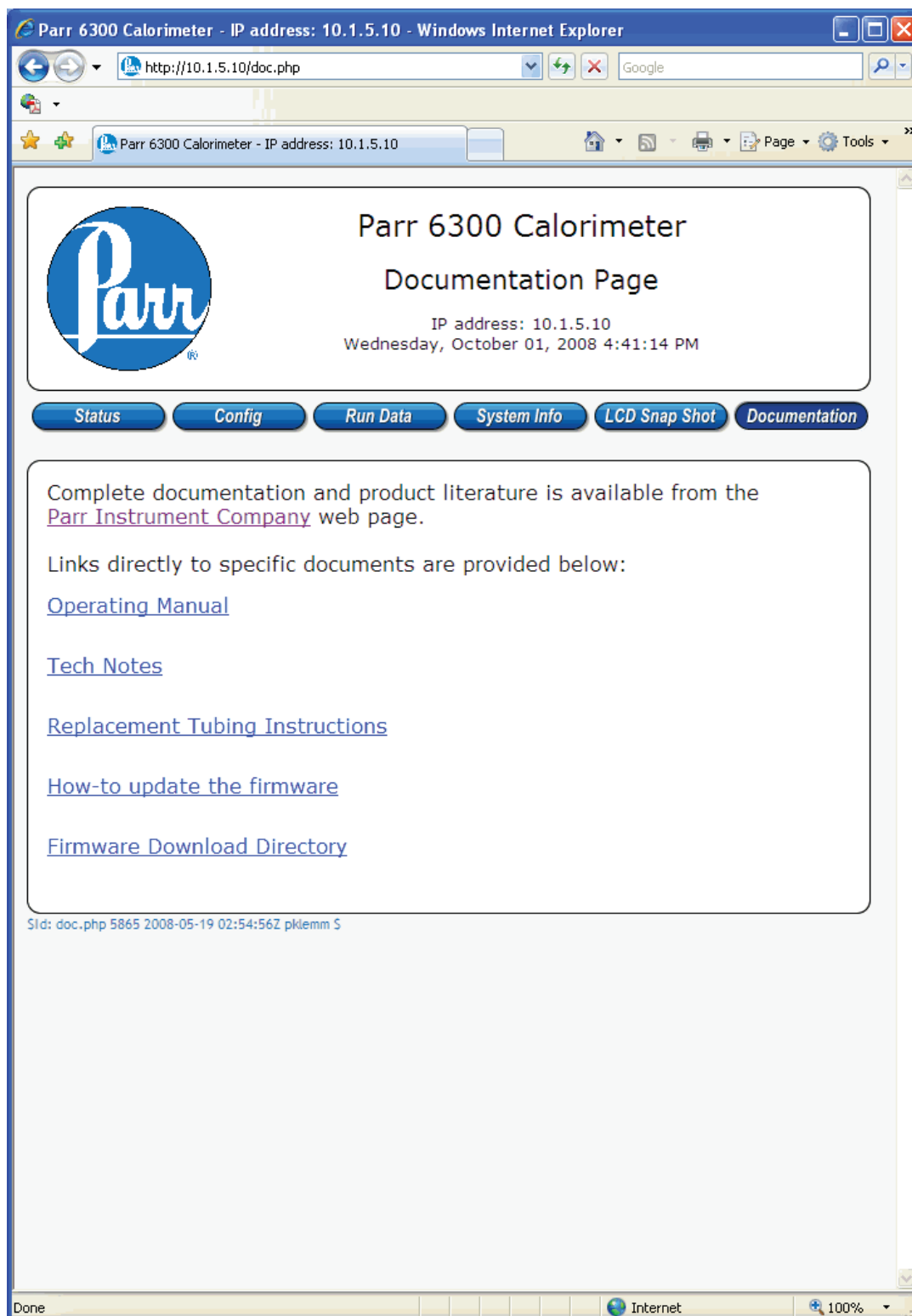
Note: This is a picture only. The calorimeter cannot be remotely operated from this screen. Remote operation requires the appropriate Feature Key.

Please contact Parr Instrument Company for more details about available Feature Keys.



Clicking on the Documentation button will display the screen below. Clicking on any of the links will open the corresponding web page.

Note: Connection to the internet is required for these links.



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 touch pad. Pressing the key labeled “ABC” allows the user to switch from upper case letters, to lower case letters and finally to numerals. 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.

NETWORK DATA SERVICES

These keys allow the user to specify the IP addresses of one or more Balance Interface devices on the network. 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.

APPENDIX E

TECHNICAL SERVICE

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

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

Any correspondence must include the following basic information:

1. The model and serial # of the instrument.
2. Software version(s) shown on the “Software and Hardware Information” page.

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 an A1340DD 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

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APPENDIX F

PARTS LISTS & DRAWINGS

PRINCIPAL ASSEMBLIES IN CALORIMETER

Item	Description
1136/1136CL	O ₂ Combustion Vessel, 340 mL
1138/1138CL	O ₂ Combustion Vessel, 250 mL
1795E	Power Supply, 24V
1796E	Power Supply, 5/12V
379VB2	Barbed Tee, 3/8T X 1/2T X 3/8T
412VB	Union Fitting, drain
533VBAD	Union Reducer, 3/8T – 1/4T
897E	Capacitor, Ignition
909E	Switch Power
911E	Filter 10 amp, interference
A1050DD	Rinse Container Assembly
A1250DD2	Controller Assembly
A1251DD	Oxygen Solenoid Assembly
A1252DD	Water Solenoid Assembly
A1416DD	Wash Pump Assembly
A1254DDEB	Pump Assembly Circulating 115V
A1254DDEE	Pump Assembly Circulating 230V
A1255DD	Propeller Assembly, stirrer
A1256DD	Water Tank Assembly
A1257DD	Water Regulator Assembly
A1260DD	Water Level Assembly
A1264DD	Air Can Assembly 6300
A1265DD	Bucket and Stirrer Tube Assembly
A1275DDEB	Cartridge, Heater Assembly, 120V
A1275DDEE	Cartridge, Heater Assembly, 230V
A1274DD	Wash Pump Divert Valve Assembly
139E23	Fuse Fast/ Act 15 Amp 250V
1641E	Pump Fuse (F1), Fast-Act, 1 Amp, 250V
1641E2	Heater Fuse (F2), Fast-Act, 2.5 Amps, 250V
997E5	Bomb Rinse (F5), Slo-Blo, 5 Amps, 250V



WARNING:

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

A1250DD2 CONTROLLER ASSEMBLY

Item	Description
1926E	Film Guard, LCD 1802E
A1821E	Speaker Assembly with Cable
A1822E	Power Cable Assembly
A1823E	Touchscreen Cable Assembly, 12"
A2140E	I/O Board
A2141E	LCD Transition Board
A2154E	CPU Board (GCM)
A2163E	LCD Cable
A2164E	Backlight Control Cable
A2166E	I/O to CPU USB Cable
A2167E	USB Peripheral Cable
1477DD	Display Kyrocera/Hantronix Gasket
2147E	LCD
1472DD	LCD Encasement - Kyrocera

A1251DD OXYGEN SOLENOID ASSEMBLY

Item	Description
180VB	Elbow, Male, 1/8T X 1/8 NPTM
243VB2	Connector, Male, 1/8T X 1/8 NPTM
527VB	Restrictor, .012" SS 1/8 NPTM-F
79HW2BB	Plug, 1/8" NPT
A1272DD	Oxygen Solenoid Assembly w/o Fittings

A1252DD WATER SOLENOID ASSEMBLY

Item	Description
179VB	Tee, Street, 1/8 NPT
247HWHJ	Hose, Barb, Elbow, Nylon, 1/4T x 1/8M
321VB	Water Solenoid Valve, 1/4T x NPTM
60HWHJ	Hose, Barb, Straight
79HW2BB	Plug, 1/8 NPT
A1276DD	Cold Water Solenoid Assembly, with Connector
A92HWAD	Male Connector, 1/4 T – 1/8 NPT
283VB	Adapter, Male 1/4 T- 1/8 NPT

A1257DD WATER REGULATOR ASSEMBLY

Item	Description
1211DD	Bracket, Water Regulator
1244DD	Regulator, Water, 6300
1245DD	Filter, Water, 1/4 NPT
447VB	Connector, Female, 1/4 T X 1/4 NPTF
60HW3HJ	Hose Barb, Nylon

A1258DD TEMPERATURE CONTROL ASSEMBLY

Item	Description
1249DD	Manifold, Temperature Control
1417E	Thermistor, Jacket
538VB	Connector, Male, 1/8 NPTM-T-BT Nylon
248HWHJ	Elbow, Hose Barb, 1/2 T x 1/2 M, Nylon
386VB	Nipple, 1/2 NPT Nylon
413VB	Cap, 1/2 NPT Nylon
405VB	Nipple, 1/2 NPT Brass
A1275DDEB	Cartridge, Heater Assembly 120V
A1275DDEE	Cartridge, Heater Assembly 240V

A1260DD WATER LEVEL ASSEMBLY

Item	Description
1231DD	Mount Level Sensor
1797E	Switch, Water Level

A1264DD AIR CAN ASSEMBLY

Item	Description
328E	Hole Plug, 3/8"
344VB	Connector, Male 1/8T x 1/16NPTM
357HCJB	O-ring, Buna-N, 1/4 ID
659DD	O-ring, Buna-N .5/32" ID x 1/16 C ID
857DD	O-ring, Buna-N 3/8 ID
882DD	O-ring, Buna-N .487 ID
941DD	Wedge Bucket
942DD	Bushing, Bucket, Retaining
962DD	Cap, Bomb Release Cylinder
963DD	Retainer, Vessel;
964DD	Spacer, Vessel
965DD	Adapter, Air Can
966DD2	Shaft, Pin Release
967DD	Seal, Air Can
969DD	O-ring, Buna-N, 1/2" ID x 1/8 CS
970DD	Retaining Ring SS
1037DD	Baffle, Bucket, Brass
1137DD	Snap Ring, 0.75"
1138DD	O-ring, 1/2" I.D.
1139DD	Insert, Cylinder
1140DD	Cylinder, Bomb Release, 6300
1141DD	Spacer
1143DD	O-ring, Type 316, .614 ID x .07 CS
1235DD	Gasket, Air Can
A1248DD2	Air Can Assembly, 6300
A92HWAD	Male Connector, 1/4T – 1/8NPT
1224DD2	Plate Hinge Support
1226DD	Mount Cover Plate

A1267DD ACCESSORY/INSTALLATION KIT

Item	Description
231C2	Container, PP 10L Foldable
271C	Cap w/ 7/16 Hole for Container
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
1347DD	Elbow
A1006DD	Waste Tube Assembly
A1336DD	Drain Tube Assembly
A38A	Bomb Head Support Stand
A1271DD	Bomb Wash Filter
TX03SK	1/32 Socket Screw Key
TX09SK	3/32 Socket Screw Key
TX12SK	1/8 Socket Screw Key
TX06SK	1/16 Socket Screw Key
TX14SK	9/64 Socket Screw Key
149C	In-line Filter
1344DD	LCD Stylus
1889E	LCD Screen Protector
HJ0025TB035	Tubing, Nylon 1/4 OD X .35W
JT0038TB062A	Tubing, Tygon 3/8 OD X 1/16W

A1265DD BUCKET AND STIRRER TUBE ASSEMBLY

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, 6300

6300 STIRRER MOTOR AND DRIVE

A1255DD BUCKET STIRRER ASSEMBLY

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 (6300)
SA114ORD04	4-40 X 1/4 RHMS 18-8 SS
SN1140HLHJ	Nut, 4-40 Hex Lock

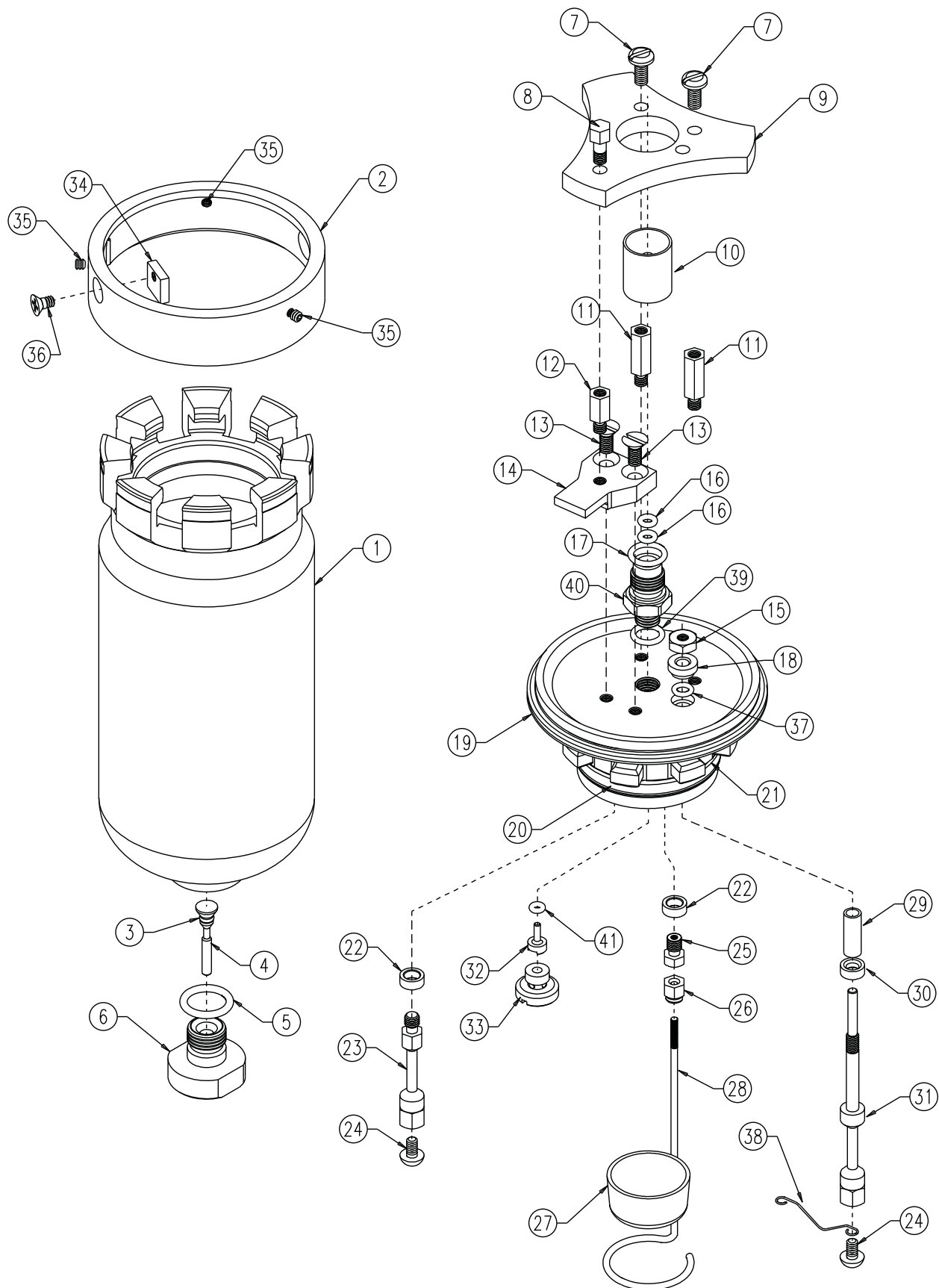
A1266DD COVER ASSEMBLY

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
HA0012TB031	Tubing, PTFE

6309B SPARE PARTS KIT

Item	Description
643DD	Pin, Check Valve
906DD	Capsule Holder (1136)
668DD	Check Valve
845DD2	Ignition Thread, 4" 10g ~1000
3415	Benzoic Acid, 1.0 gram Pellets, Bottle of 100
43AS	Capsule SS
944DD	O-Ring NBR 1/4 ID x 3/32CS
857DD	O-Ring NBR 3/8 ID x 3/32CS
1094DD	Electrode Long (1136)
1095DD	Electrode (1108P/A416A5)
6038	Kit 500 Firing 1136/38 A1450DD
359VB	Filter, In Line 1/8T BR 15 Mic
1235DD	Gasket, Air Can 6300
1241DD	Belt, Timing 6300
A1395DD	Tubing Kit, Water Replace 6300
1245DD	Filter, Water; Brass Mini 1/4M

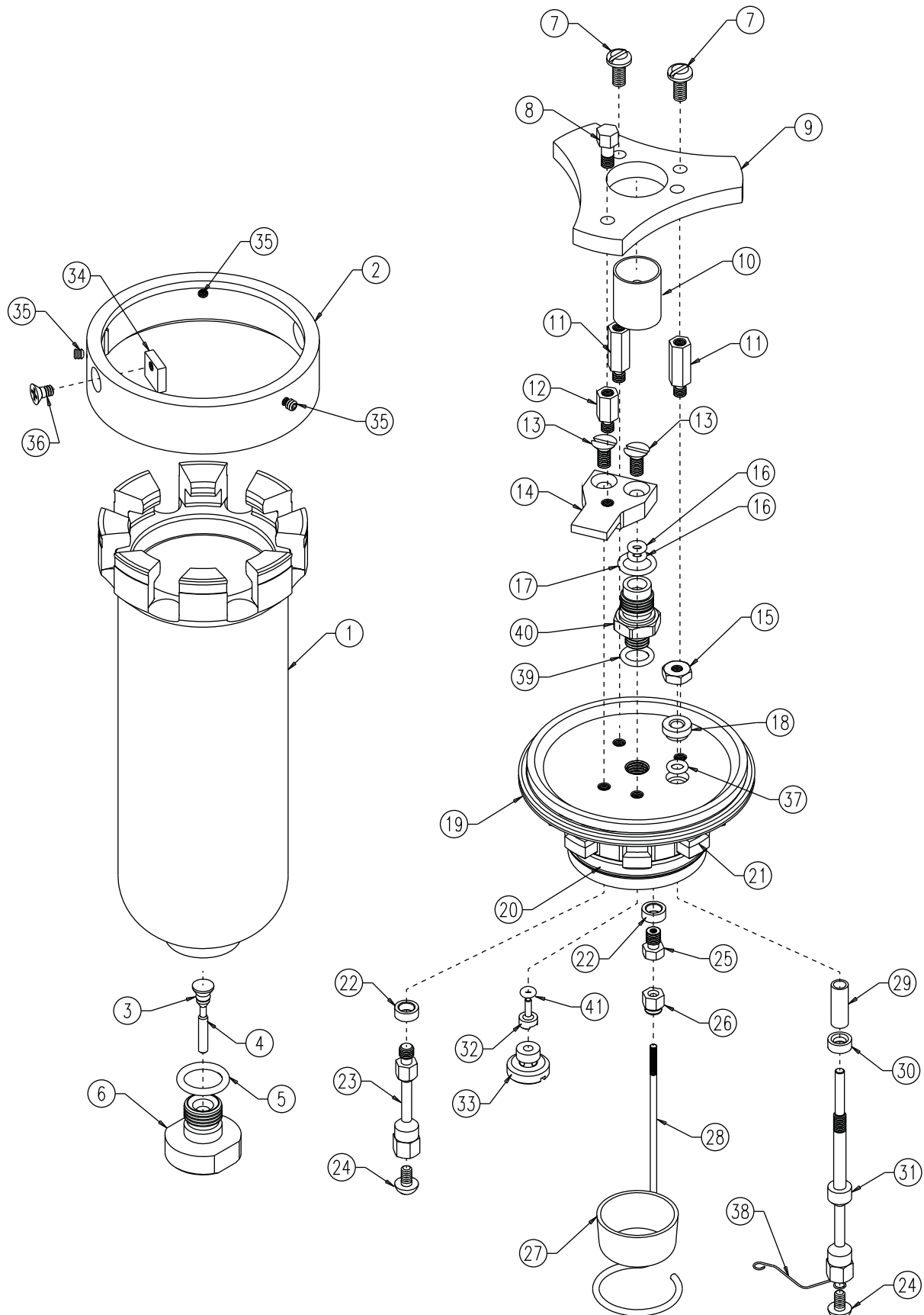
Figure F-1
Parts Diagram for the 1136 and 1136CL Oxygen Bombs



1136 AND 1136CL OXYGEN BOMB

Key No.	Item	Description
1	888DD2	Cylinder
2	889DD	Outer Ring
3	821DD (2)	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	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	1444DDJV	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 1/16 CS
Complete Assemblies		
	A1450DD	Oxygen Bomb Head Assembly
	A1450DDCL	Oxygen Bomb Head Assembly for Chlorine Service
	A890DD	Cylinder Assembly
	A890DDCL	Cylinder Assembly for Chlorine Service

Figure F-2



1138 AND 1138CL OXYGEN BOMB

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 1/16 CS
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 F-3
6300 Oxygen Bomb Calorimeter Cutaway Right

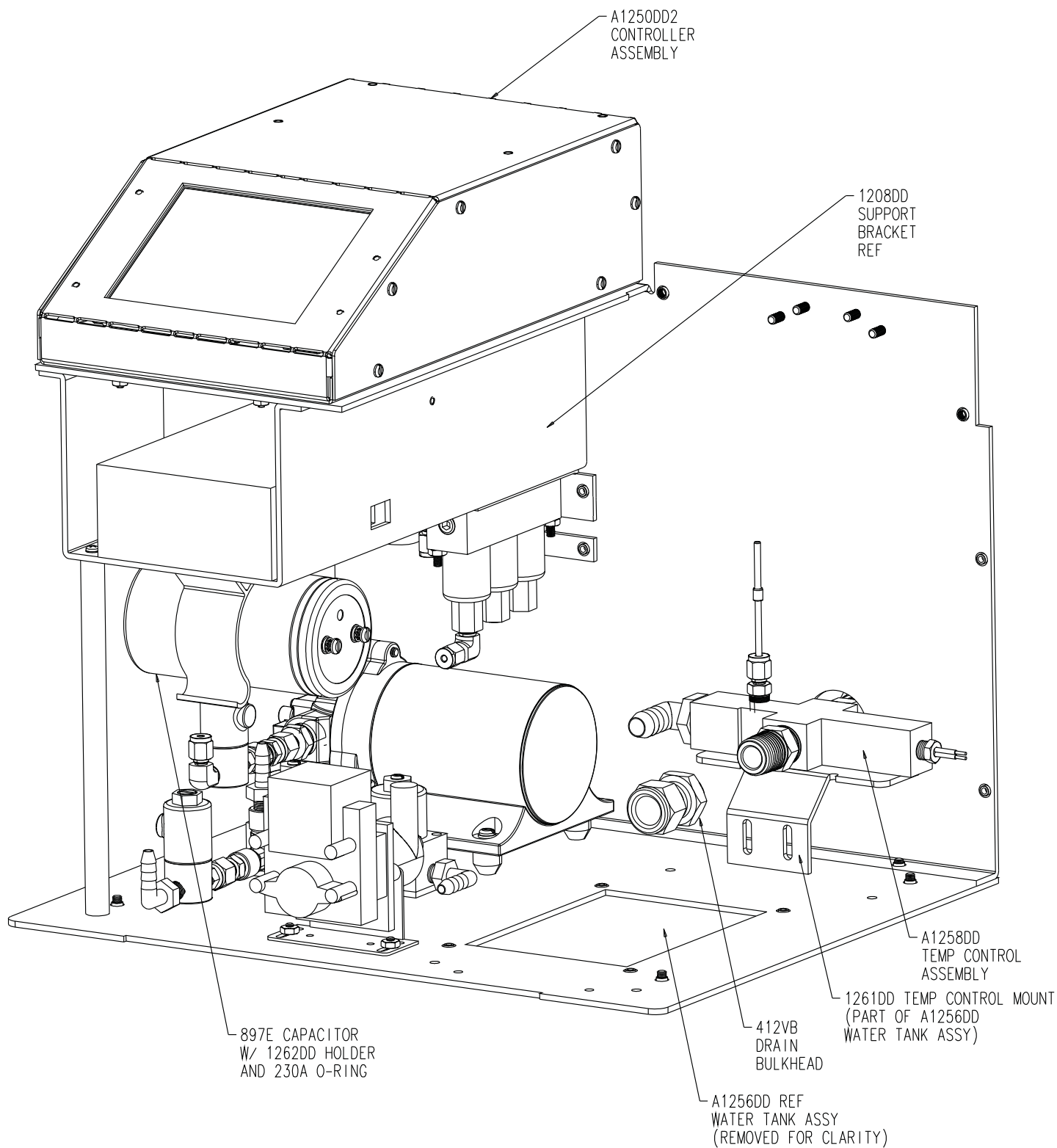


Figure F-4
6300 Oxygen Bomb Calorimeter Cutaway Left

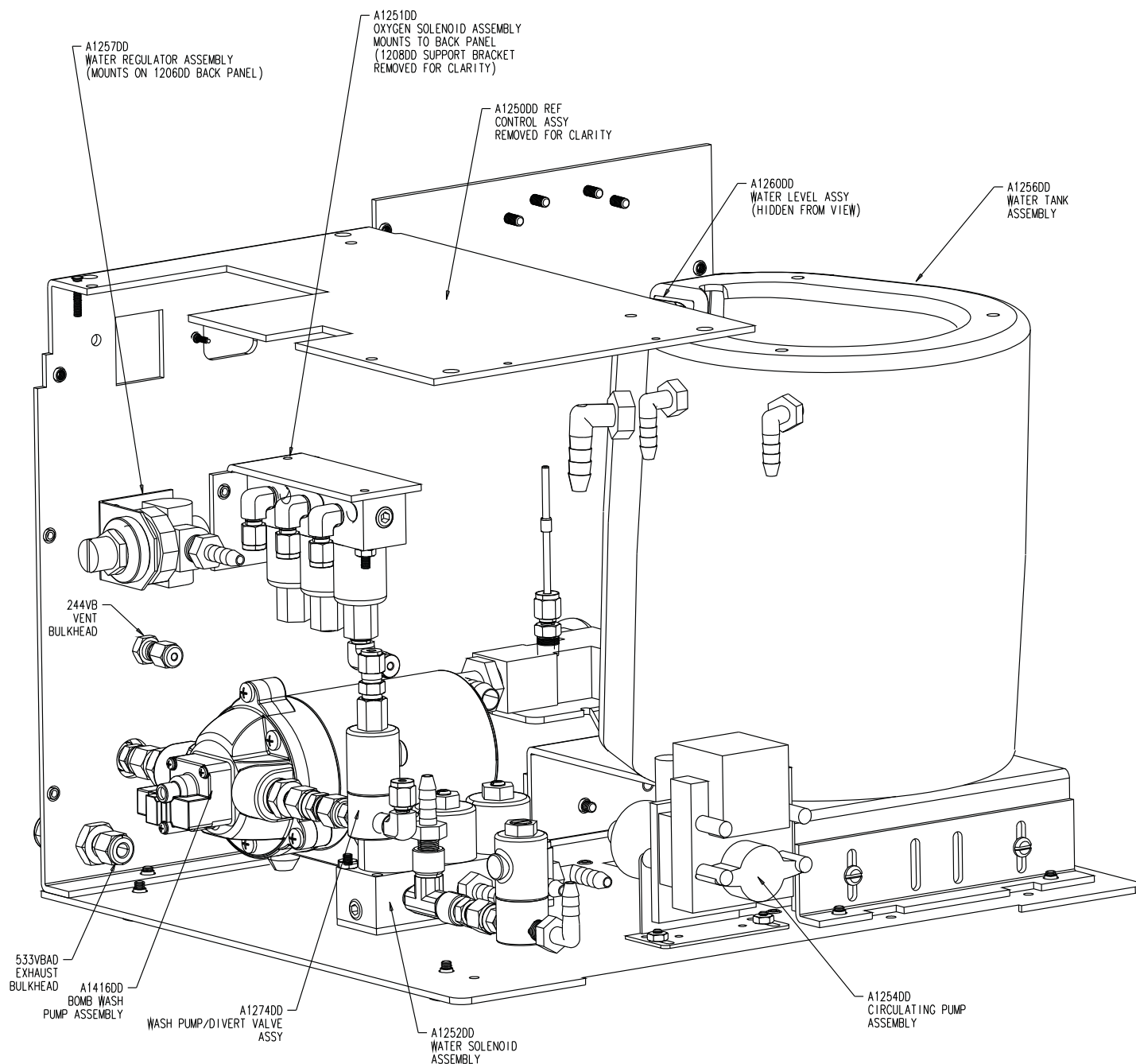


Figure F-5
6300 Oxygen Bomb Calorimeter Cover Open

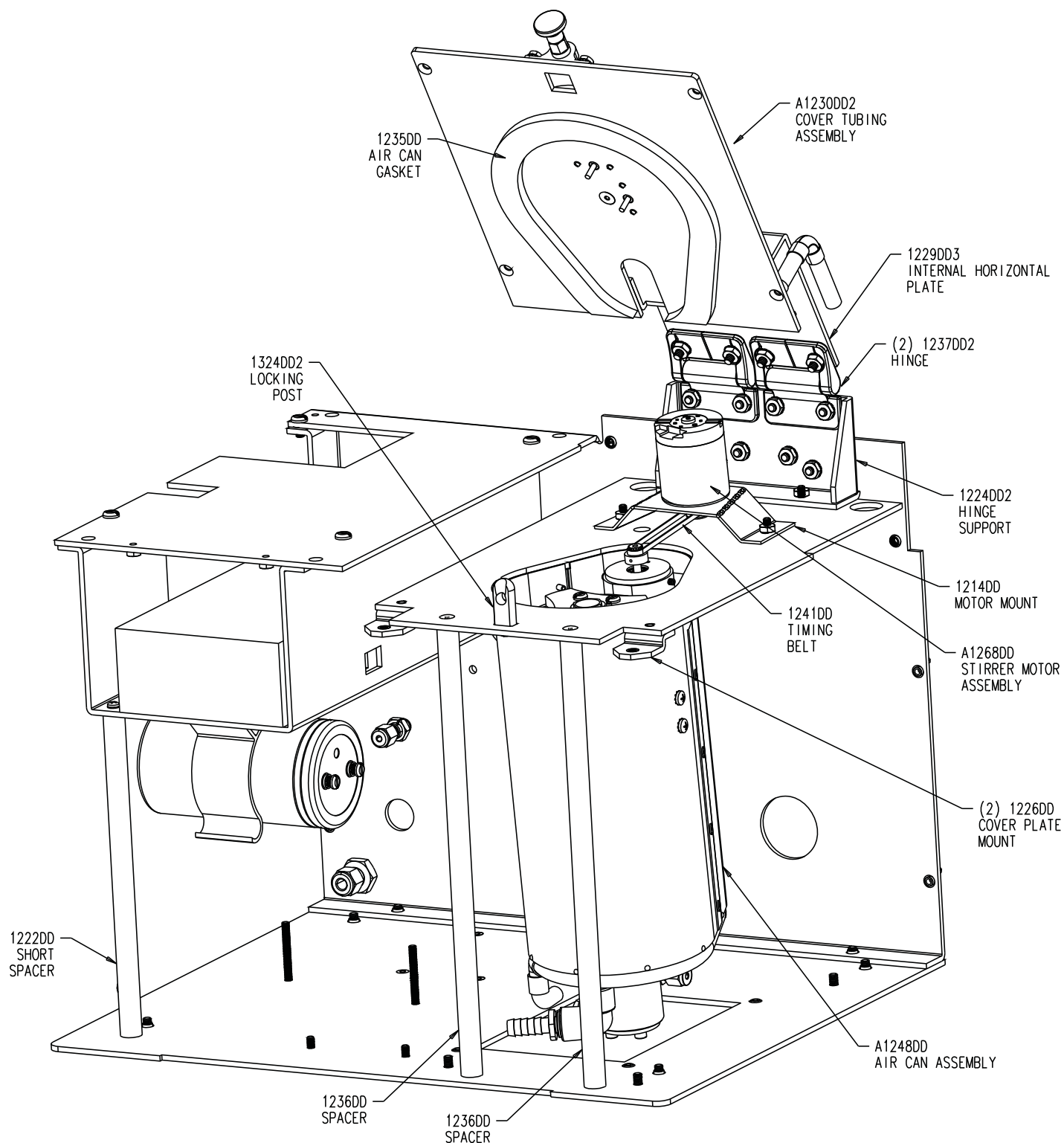


Figure F-6
A1250DD2 Control Schematic

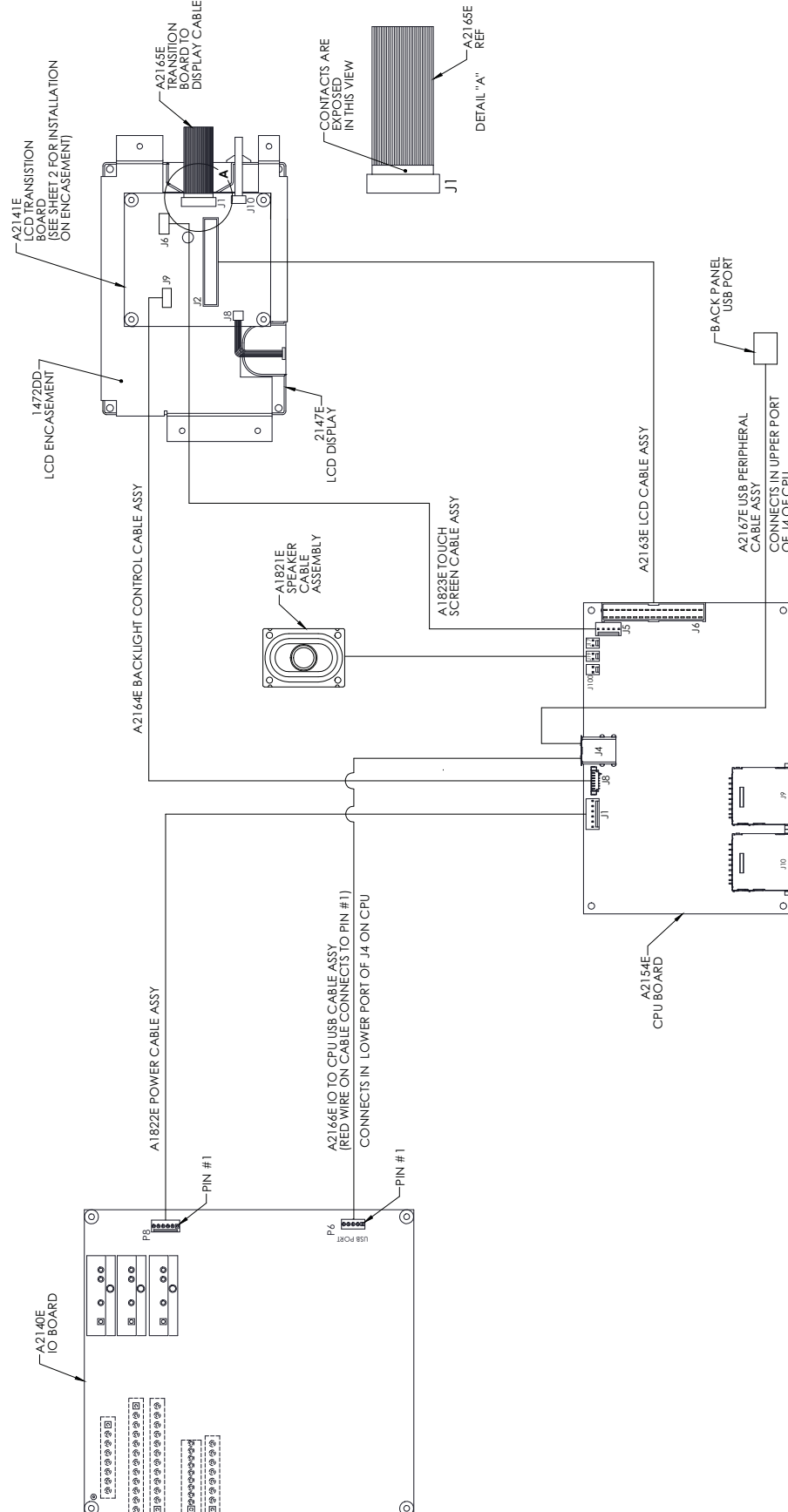


Figure F-7
A1251DD Oxygen Solenoid Assembly

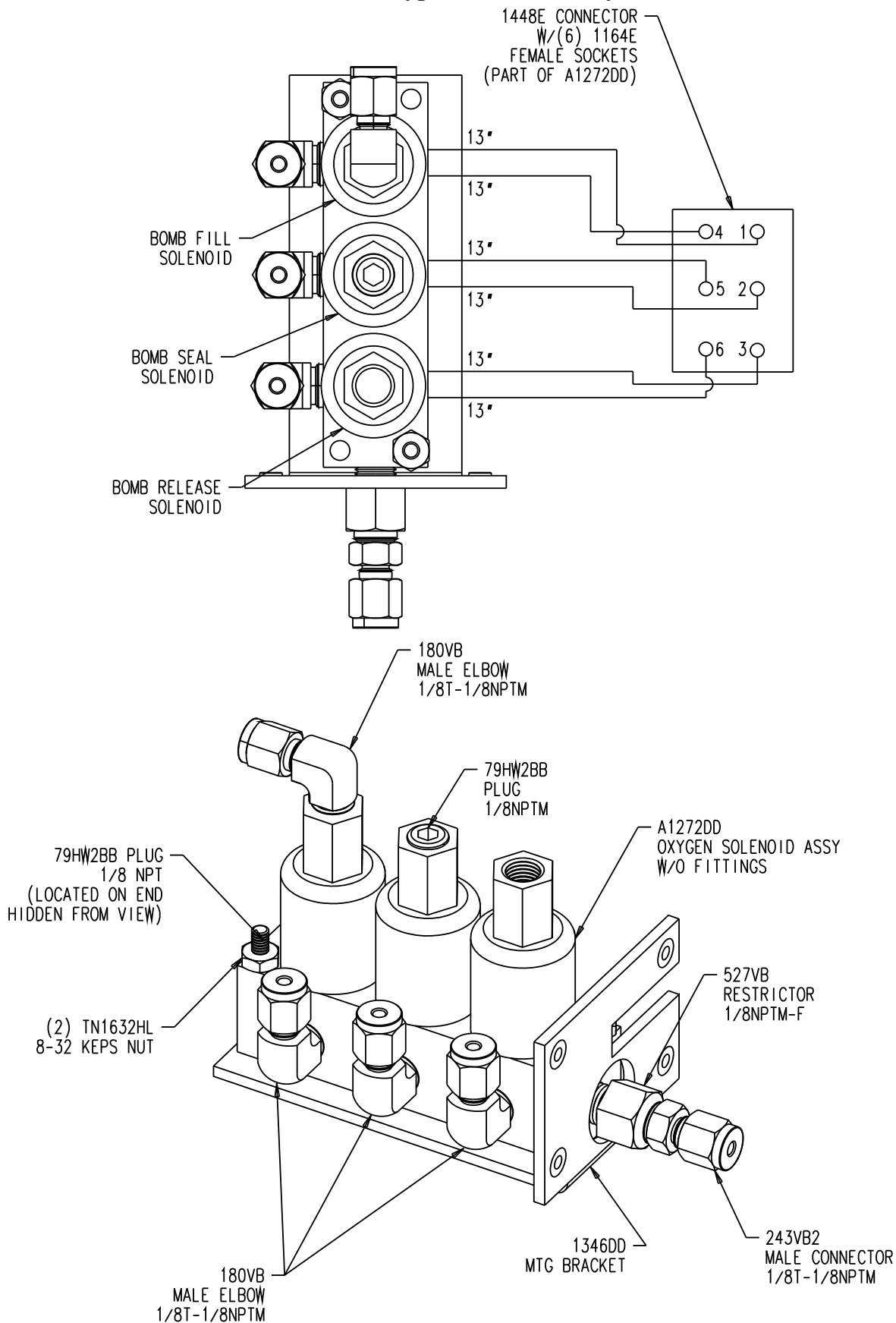


Figure F-8
A1200DD Internal Plumbing Diagram

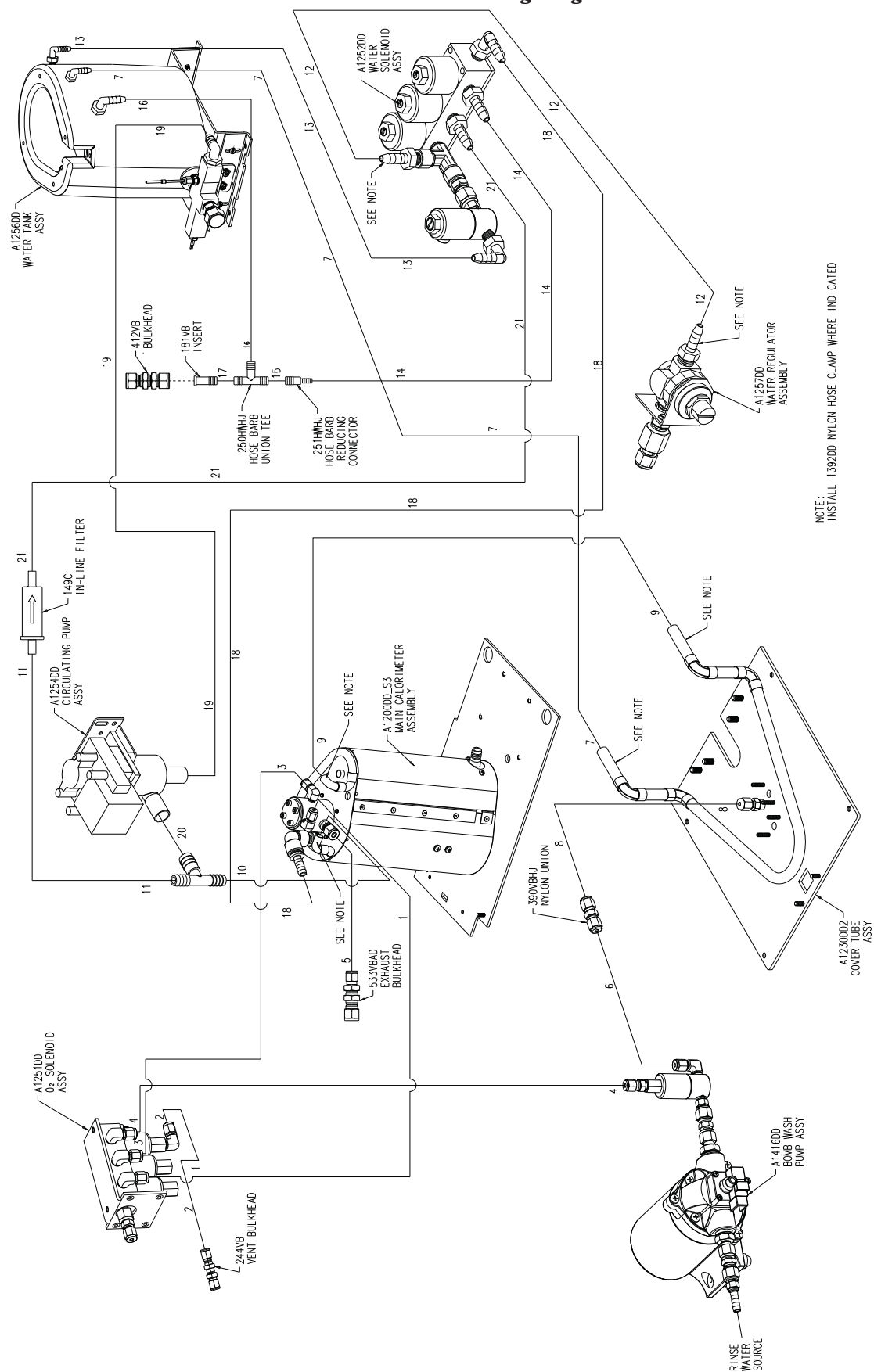


Figure F-9
A1252DD Water Solenoid Assembly

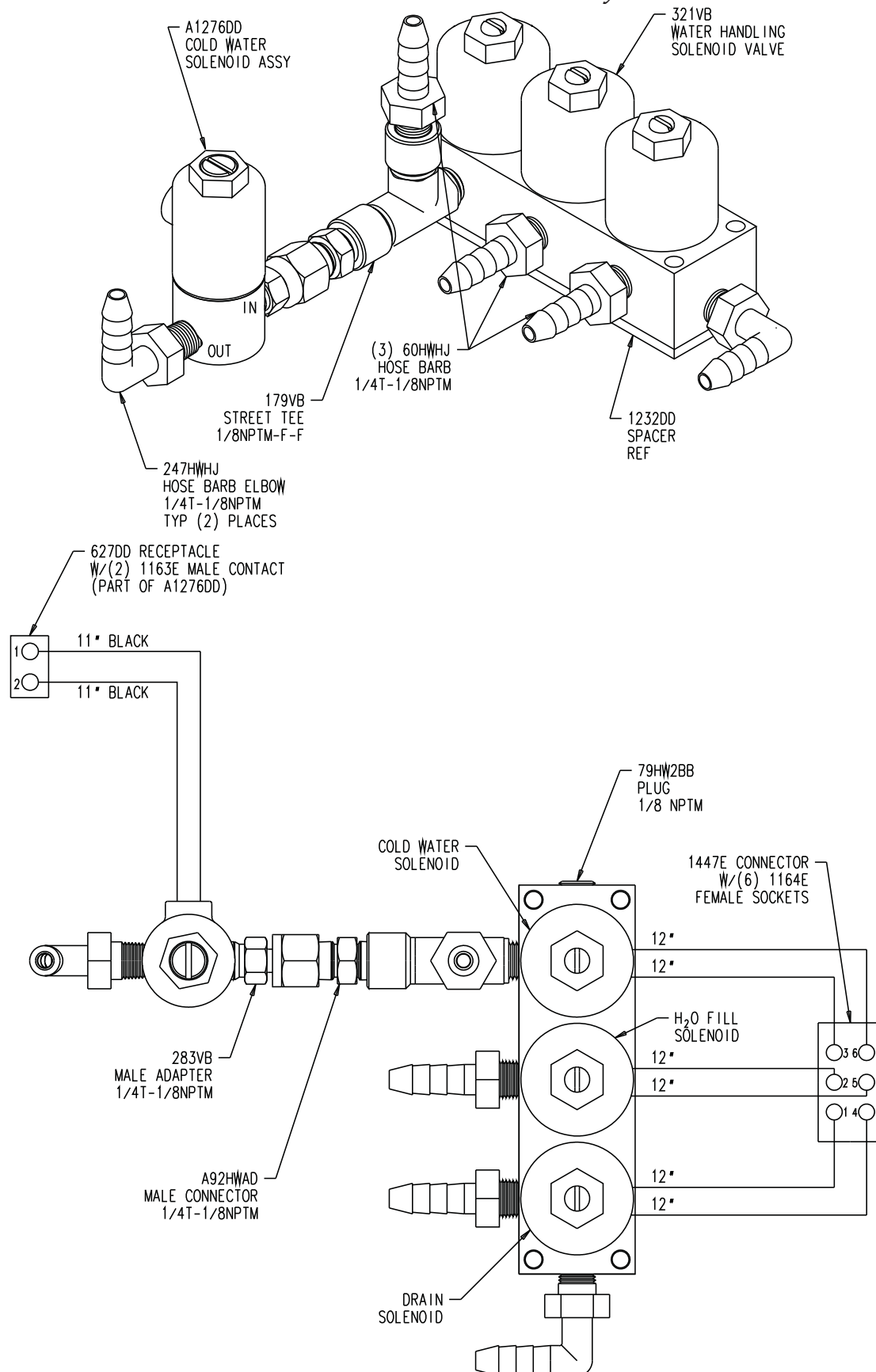


Figure F-10
A1416DD Bomb Wash Pump Assembly and Fittings

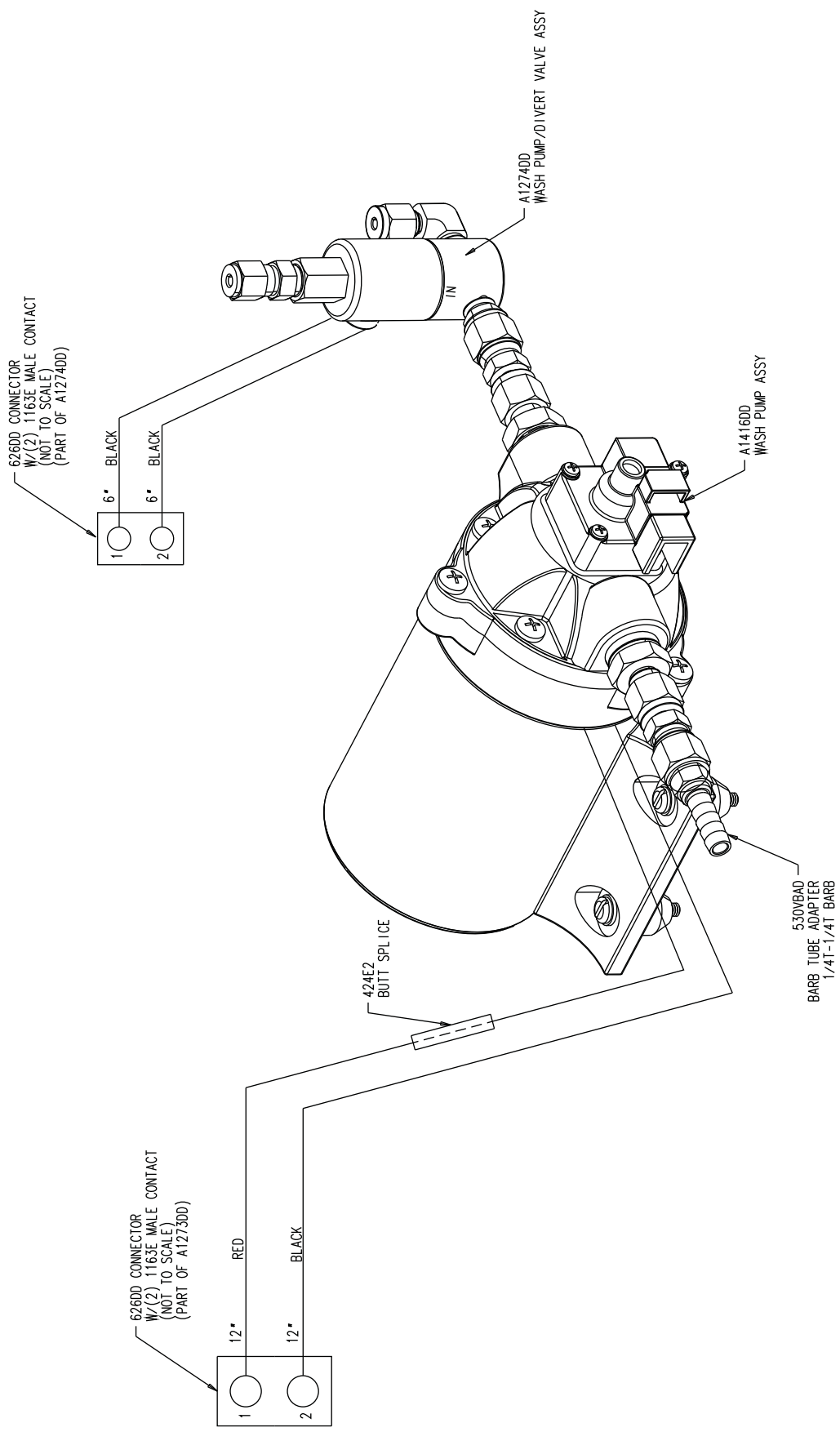


Figure F-11
A1254DD Circulatory Pump Assembly

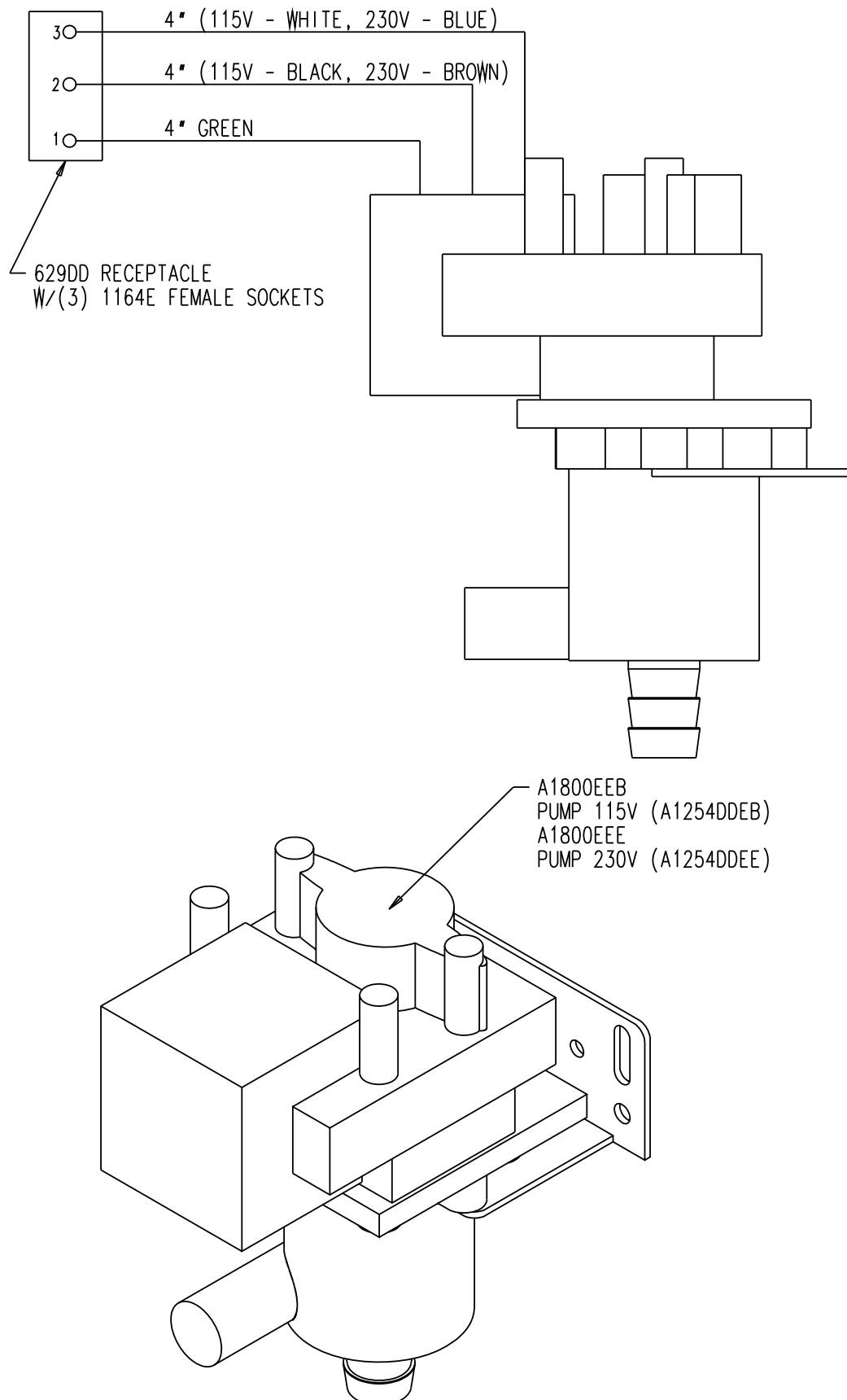
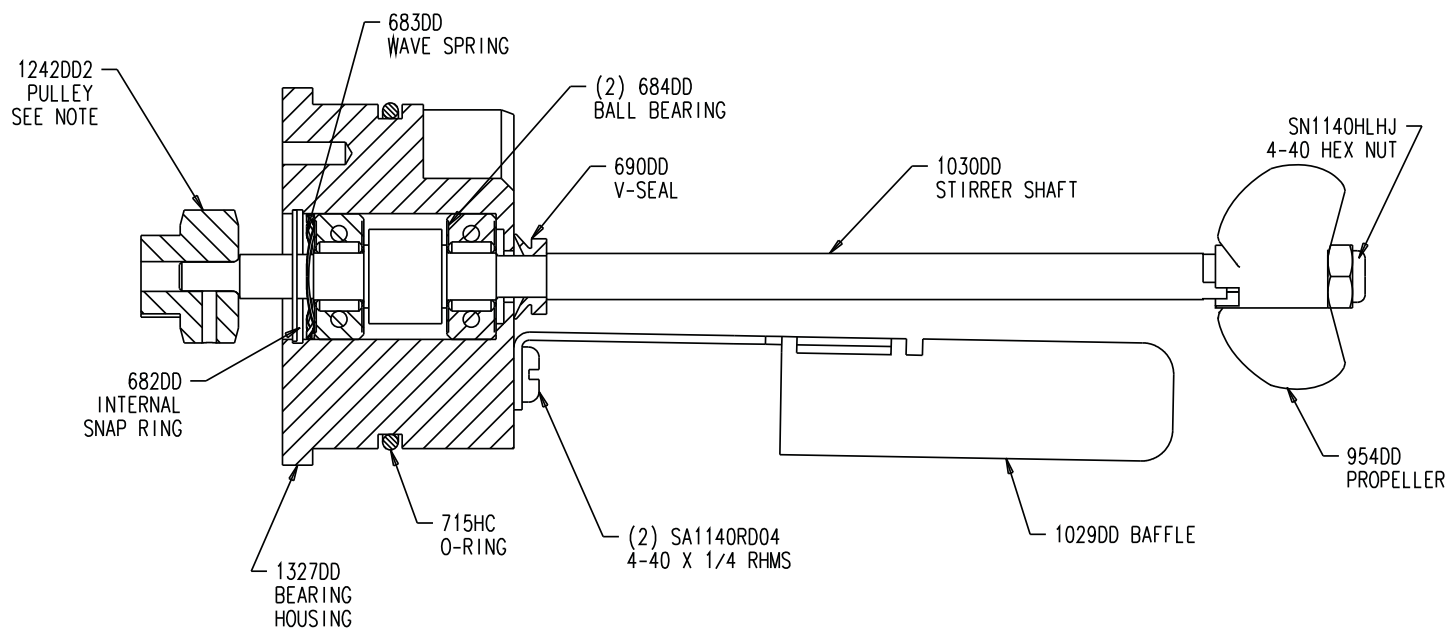


Figure F-12
A1255DD Bucket Stirrer Assembly



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

Figure F-13
A1256DD Water Assembly Tank

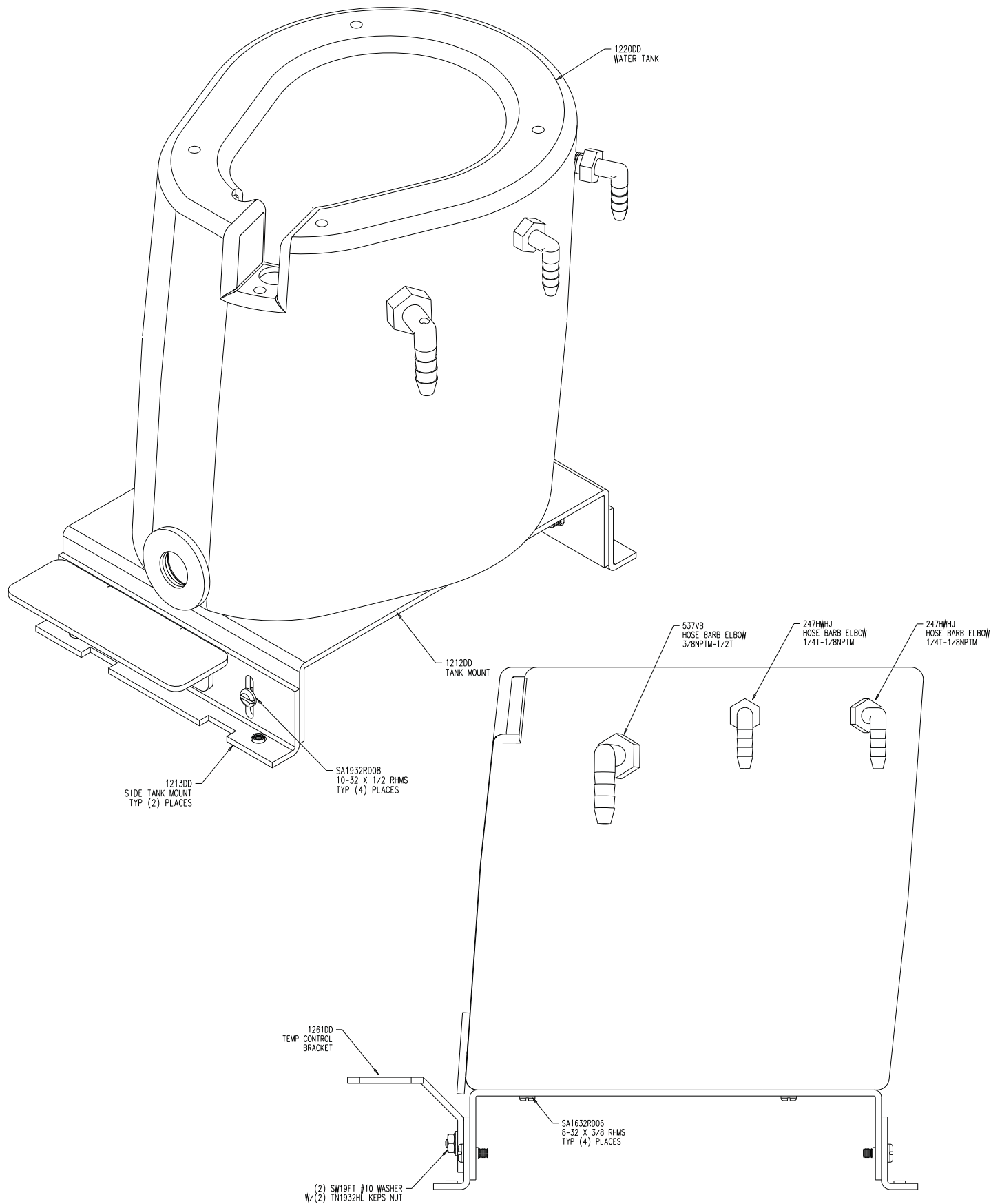


Figure F-14
A1257DD Water Regulator Assembly

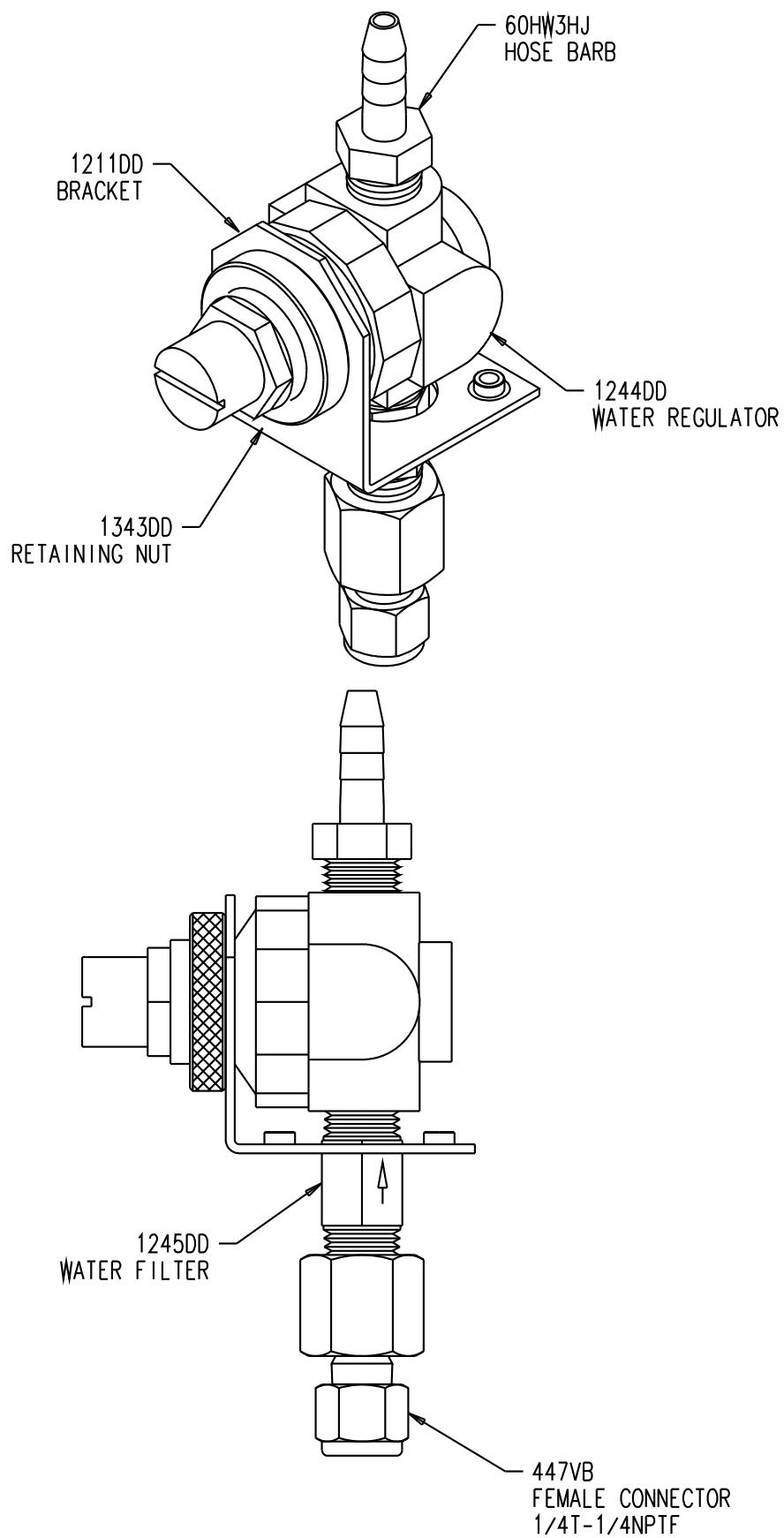


Figure F-15
A1258DD Temperature Control Assembly

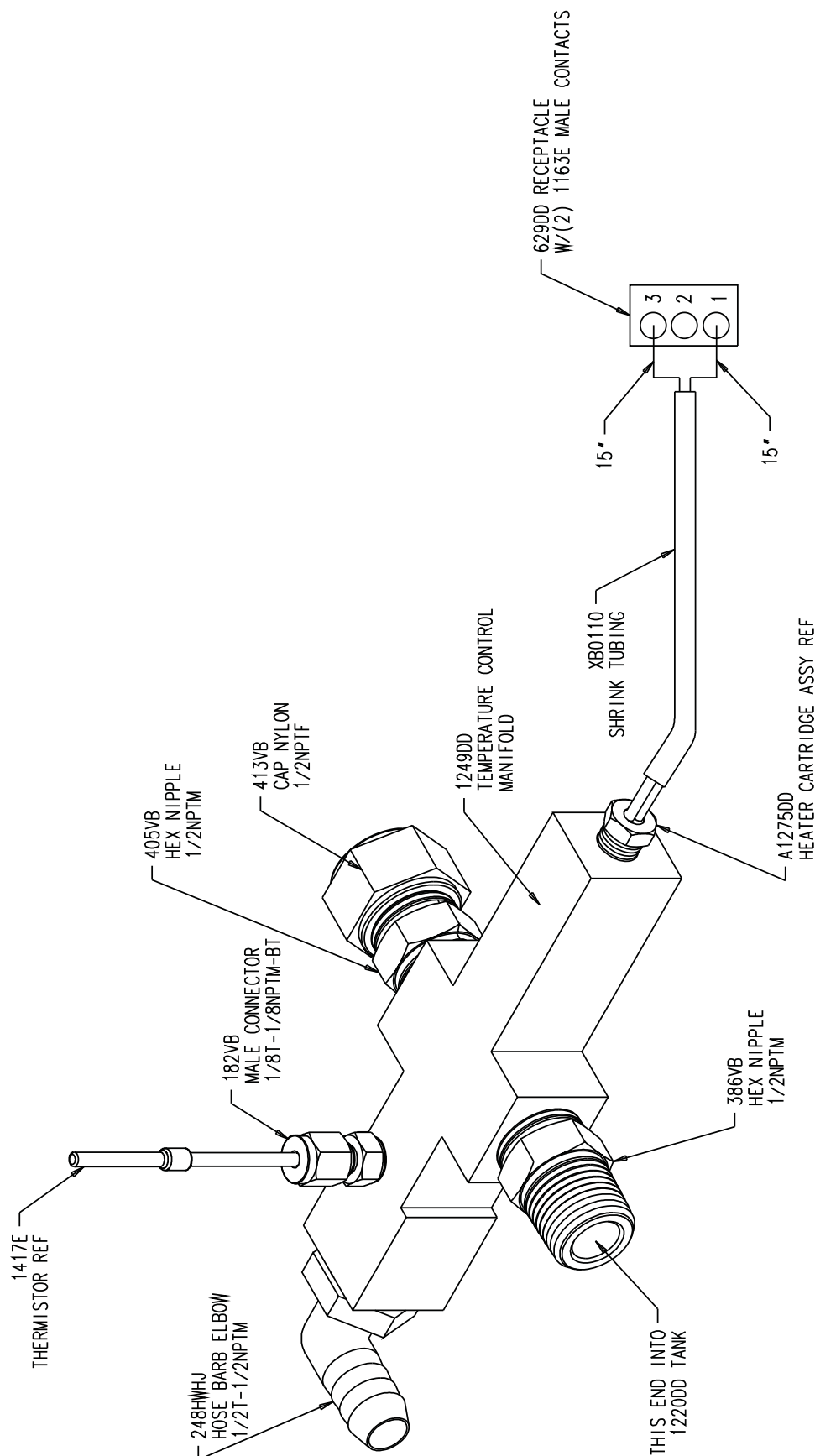


Figure F-16
Cover Contact Pin Assembly

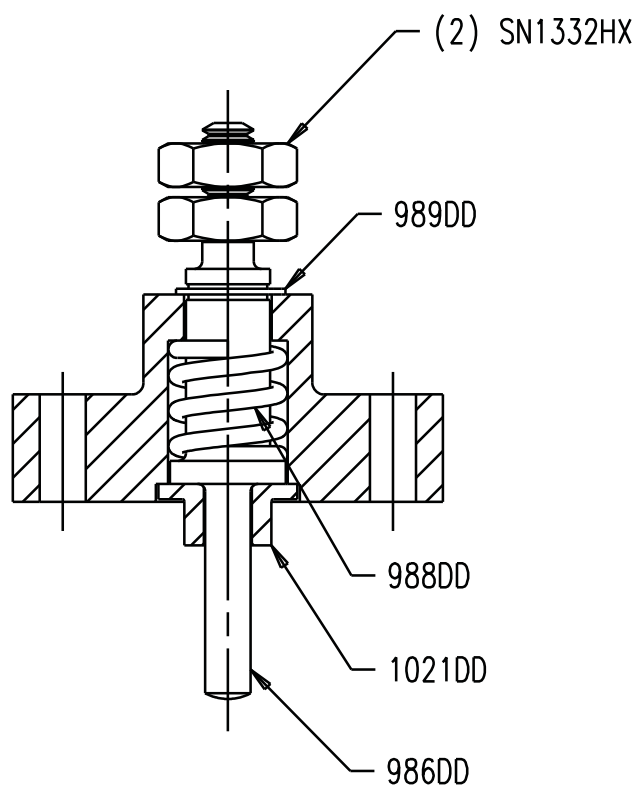
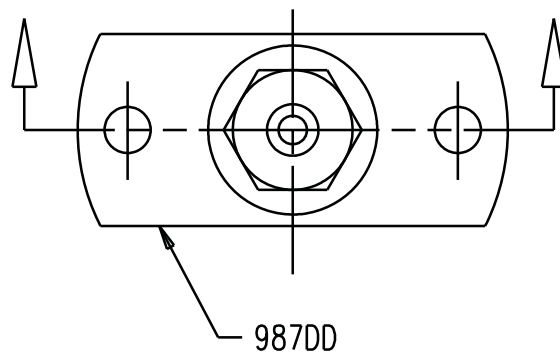


Figure F-17
Stirrer Motor and Mount

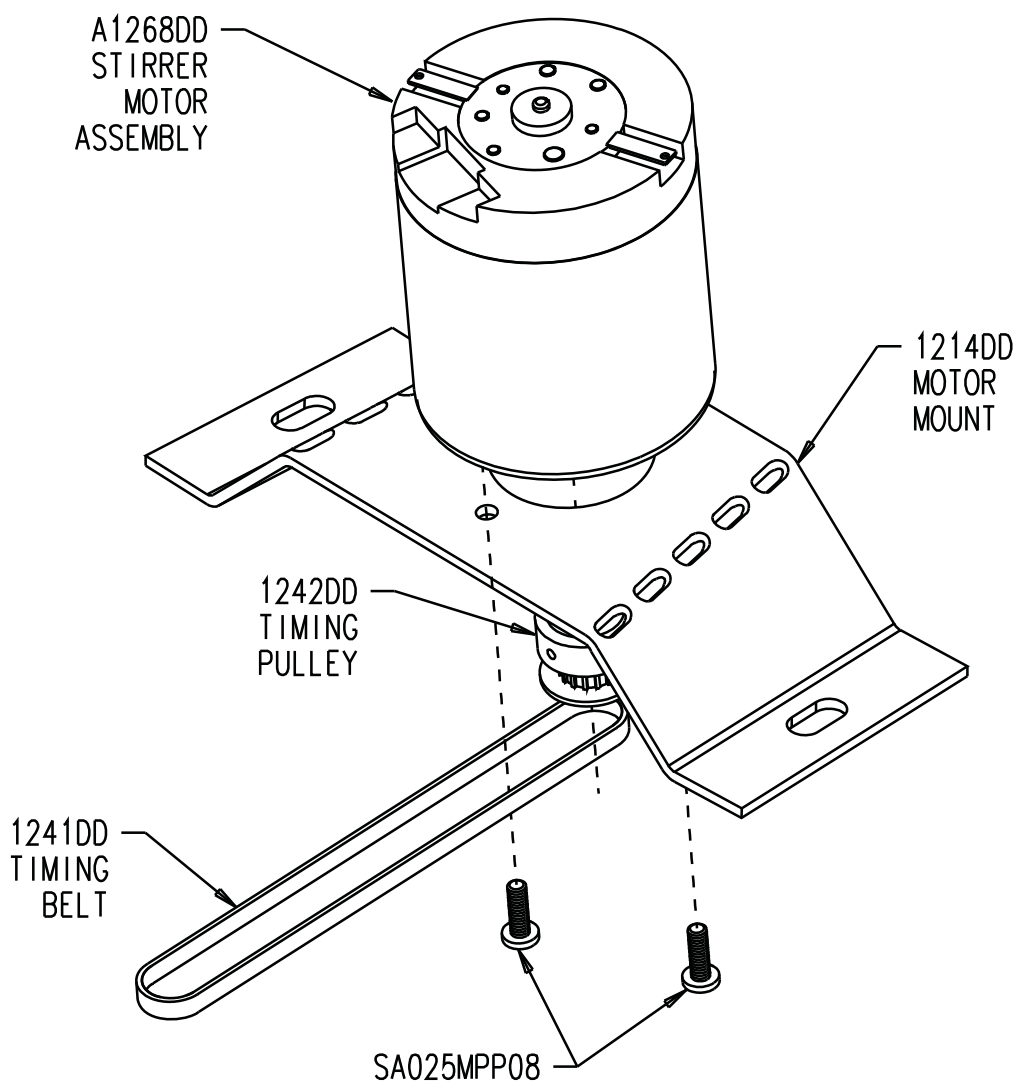


Figure F-18
A1260DD Water Level Control Assembly

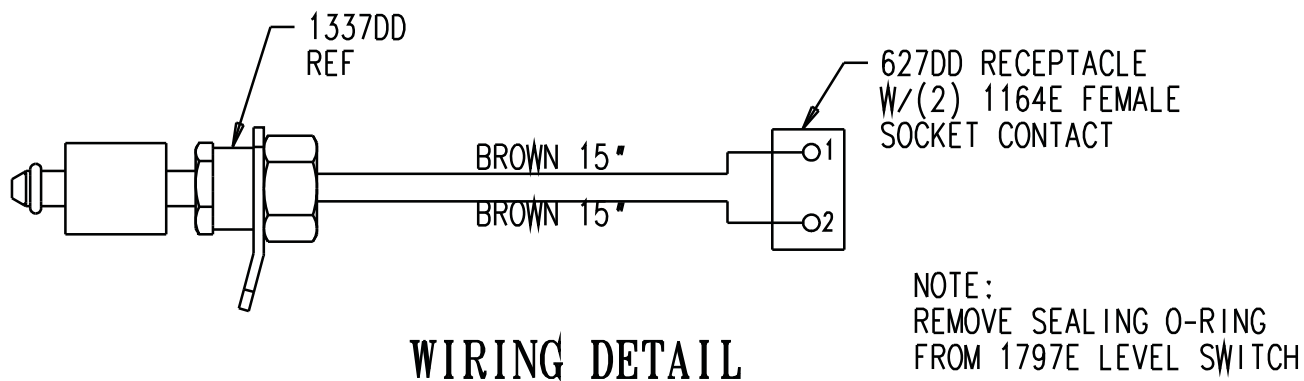
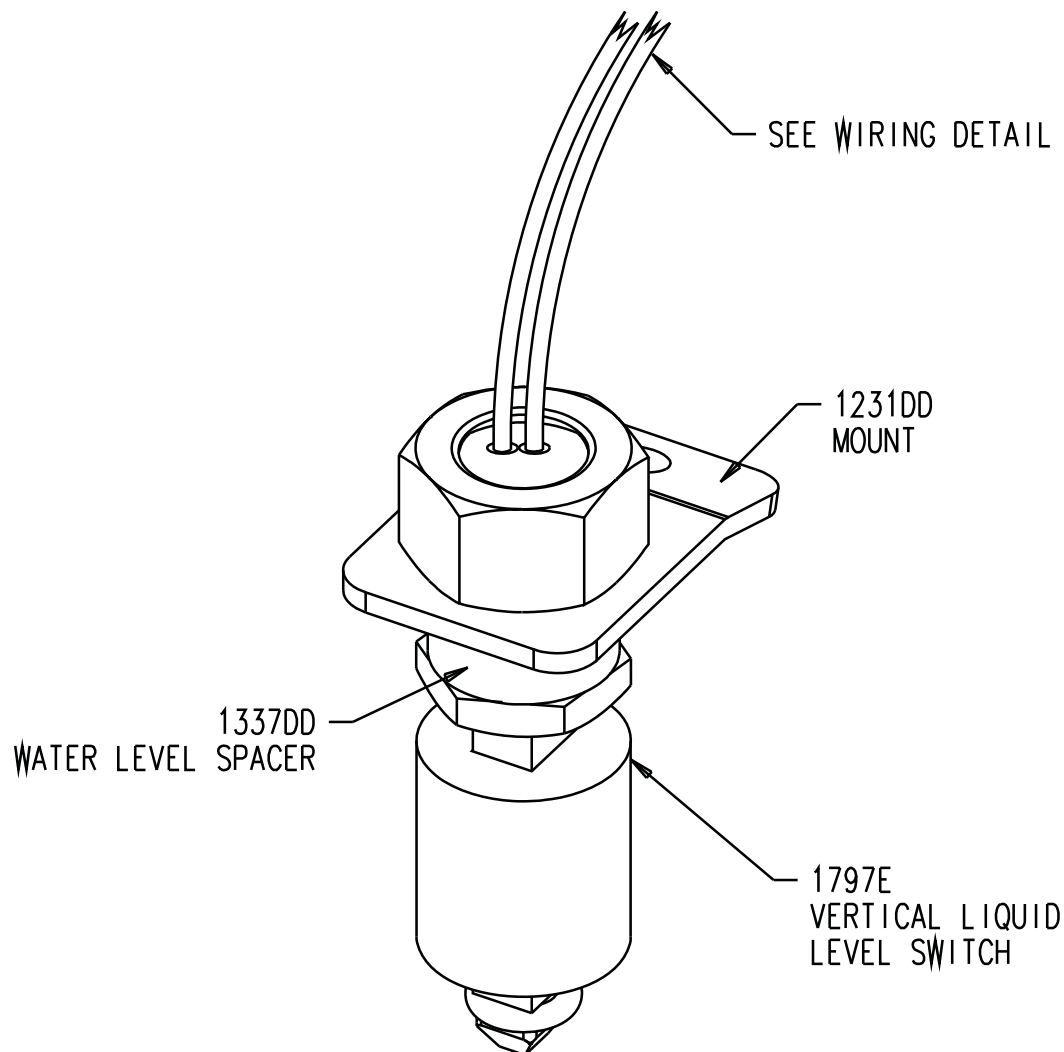


Figure F-19
A1265DD Bucket Assembly

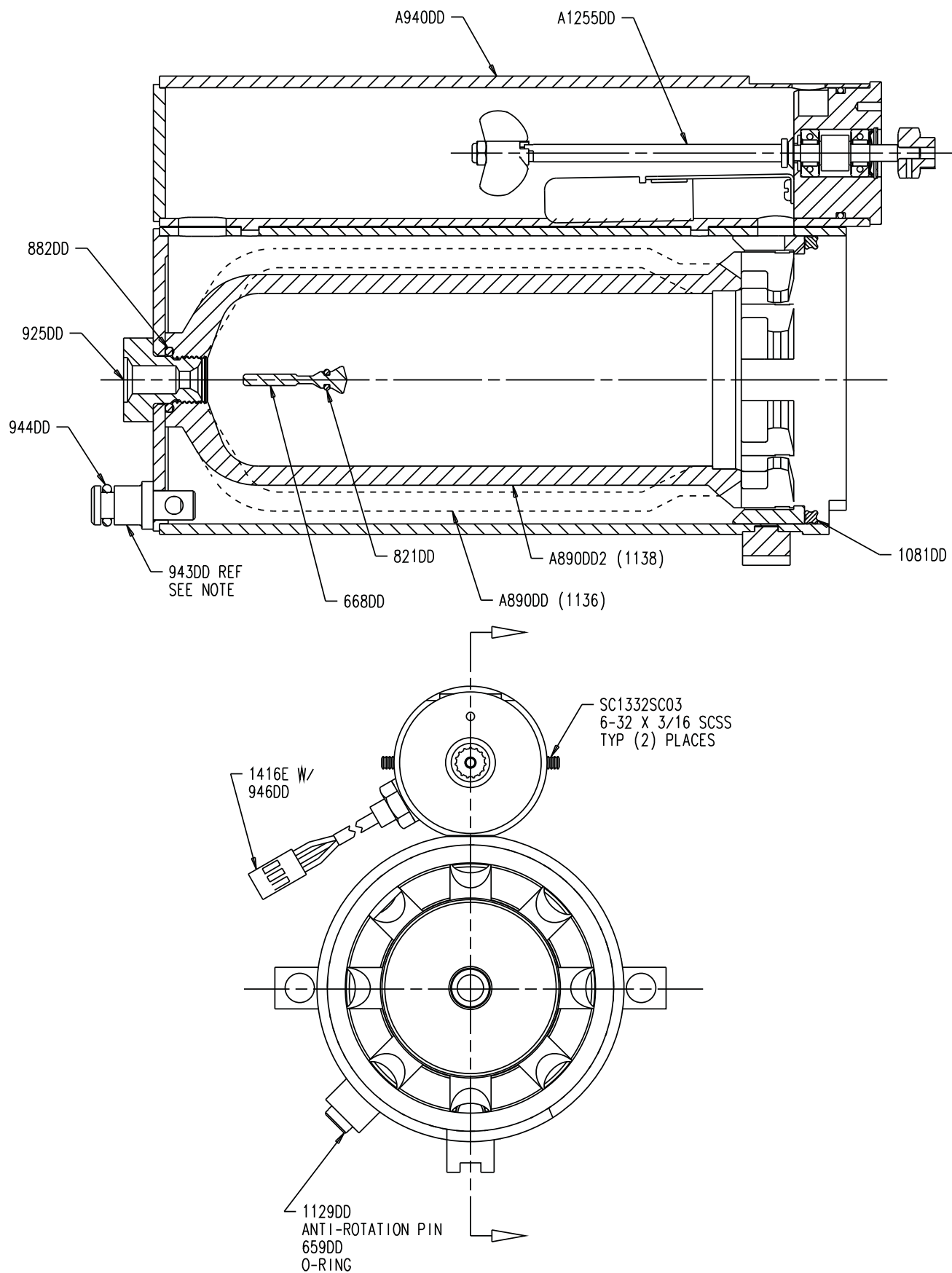


Figure F-20
6300 Air Can Assembly

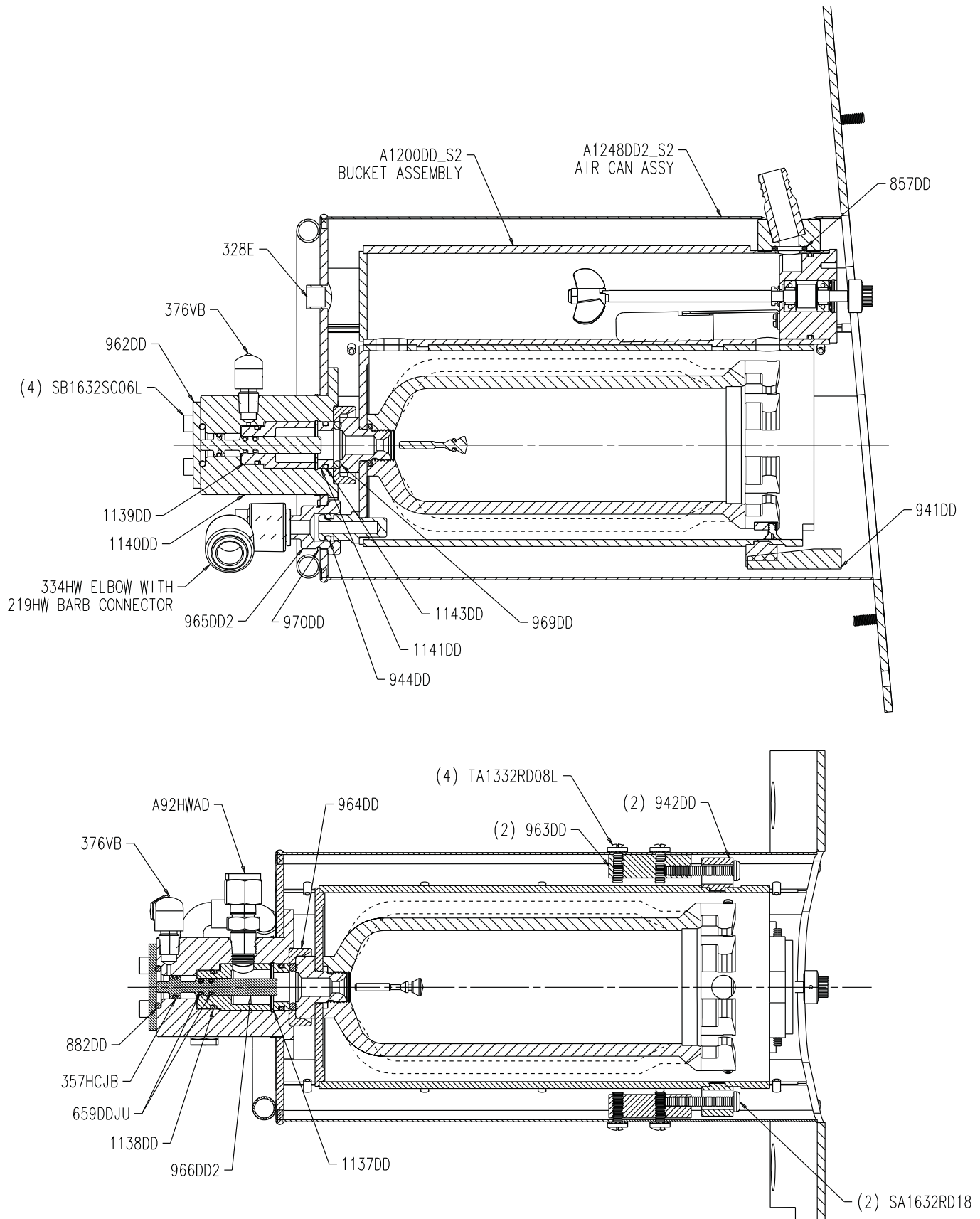


Figure F-21
A1450DD Bomb Head Assembly (1)

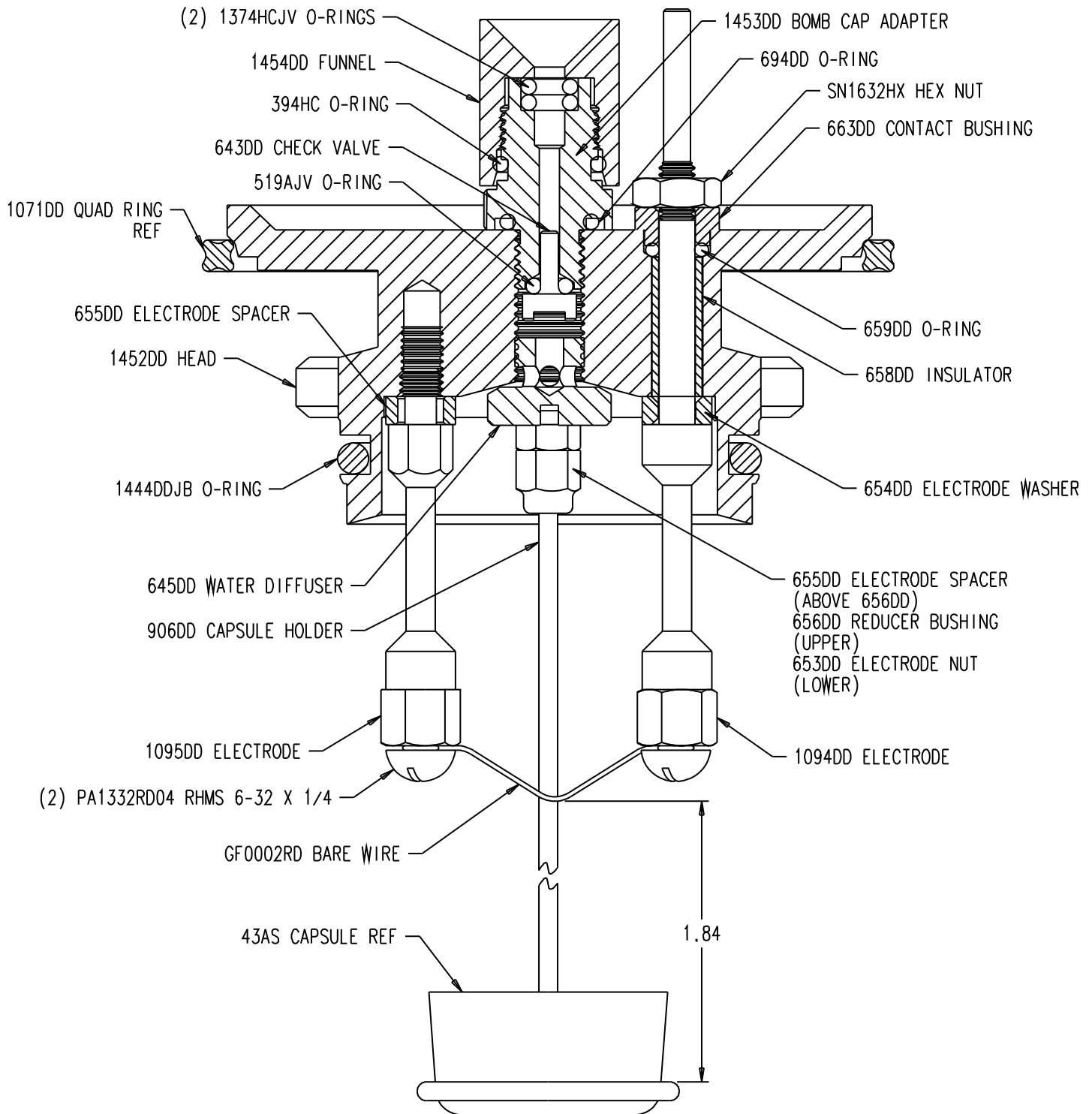
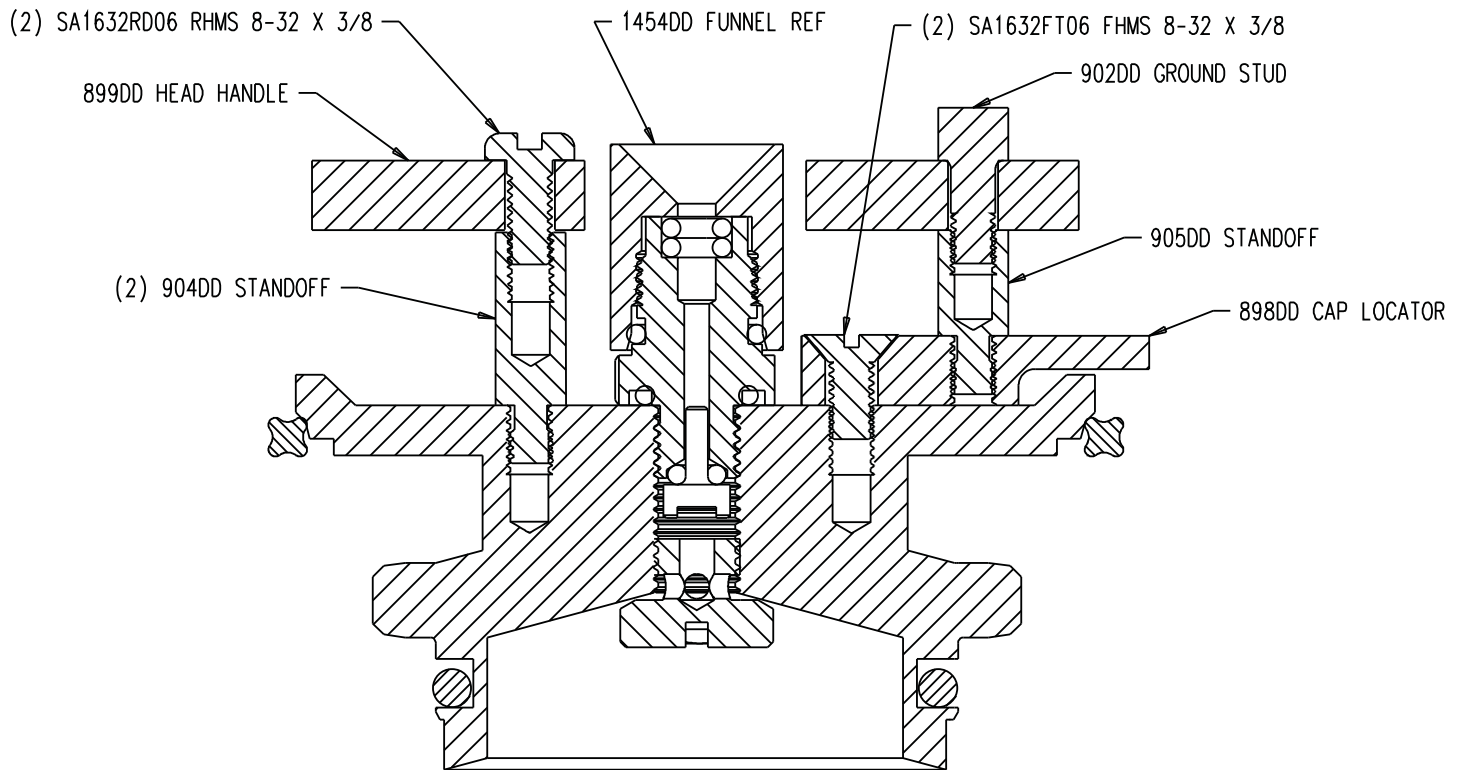


Figure F-22
A1450DD Bomb Head Assembly (2)



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APPENDIX G

BOMB RINSE CONTAINER ASSEMBLY

OVERVIEW

The A1050DD Bomb Rinse Container Assembly is provided as an accessory to the 6300 Calorimeter. This device allows for complete and systematic recovery of the bomb combustion products.

These combustion products include that portion which is released during the initial bomb exhaust, followed by the portion expelled during the bomb rinse cycle.

CONCEPT OF OPERATION

The 6300 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. The bomb exhaust and the bomb washings are collected in the 1053DD, HDPE 500 mL bottle, provided with the A1050DD. The objective is to capture and retain all of the combustion products which have been dissolved in the residual water present in the bomb prior to combustion as well as any water formed during the combustion process. The most common examples of these combustion products include:

Sulfate, derived from any sulfur containing materials in the original sample.

Chloride, from organic samples containing chlorine.

These 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.

CONNECTION

Break the connection at the rear of the 6300 Calorimeter to the bomb exhaust hose. In place of this, connect the “tube and 3/8” compression fitting provided as part of the A1050DD.

Adjusting the Bomb Rinse Parameters

The bomb rinse parameters are factory set to provide approximately 50 ml of bomb washings. This amount was chosen in order to rinse the bomb as quickly as possible in order to prepare the calorimeter for the next test. More quantitative rinsing can be realized, at the expense of time, by increasing the volume of washings. Experience has demonstrated that for a bomb of this volume, wash volumes on the order of 100 ml are required for quantitative recovery. Doubling either the # of rinse cycles or the rinse time on the bomb rinse tank controls menu page of the operating controls menu from the default value of 3 will provide a volume of washing better suited for precise analytical work. The volume of washing can be easily verified by performing a calorimeter pretest sequence.

OPERATION

Place the bottle end of the rinse assembly in the 1054DD Bottle Support and orient in such a way to minimize any undesirable effects by the bomb exhaust discharge from the four holes in the 1052DD bottle cap. Analyze the sample in the calorimeter in the usual fashion, using the following guidelines in order to insure quantitative conversion to the proper ionic form of the analyte:

Samples containing sulfur should contain no more than 5% sulfur as-burned in the calorimeter and have a heat of combustion of at least 9000 Btu/lb.

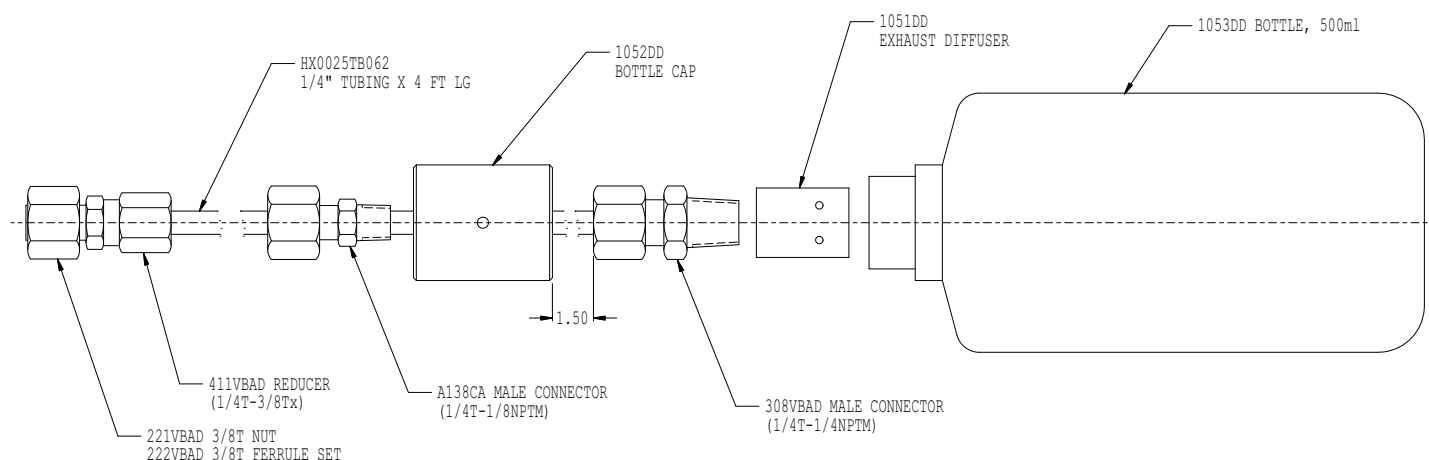
OPERATION (CONTINUED)

Samples to be analyzed for chlorine should be spiked in a manner to insure that the sample as-burned contains less than 10% chlorine and liberates at least 5000 calories. Five thousand calories corresponds to roughly a 6.4 °C temperature rise in a 6300 Calorimeter.

At the completion of the test, the collection bottle should be unscrewed from the 1052DD Bottle Cap and the contents of the combustion capsule added to the collected washings. The bottle then may be capped and labeled until the contents can be analyzed.

Most users find it useful to add the contents of the rinsed combustion capsule to the washings collected in the bottle.

Figure G-1
Vessel Rinse Container





Parr Instrument Company

**211 53rd Street
Moline, Illinois 61265 USA
Phone: 1-309-762-7716 or 1-800-872-7720
Fax: 1-309-762-9453
E-mail: parr@parrinst.com
<http://www.parrinst.com>**