743M

Parr Instrument Company

6200 Isoperibol Calorimeter

Operating Instruction Manual

For models produced after January 2022

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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
parr@parrinst.com • www.parrinst.com

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Register your equipment with us so you can receive:

- Notification of Product Updates
- Free Software Upgrades
- New Product Information

Please complete our Customer Satisfaction Survey so that we may better serve you. Your feedback helps us improve our products and customer service.

FIGURES

Preface

Scope

This manual contains instructions for installing and operating the Parr 6200 Calorimeter. For ease of use, the manual is divided into fourteen chapters.

1. **Concept of Operation**
2. **Installation**
3. **Program Installation & Control**
4. **Operation**
5. **Menu Description**
6. **Standardization**
7. **Standardization (Precision Method)**
8. **Calculations**
9. **Corrections & Final Reports**
10. **Reporting Instructions**
11. **File Management**
12. **Maintenance & Troubleshooting**
13. **Communications Interfaces**
14. **Parts Lists & Drawings**

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.

Note About Nomenclature: Historically, burning a sample enclosed in a high pressure oxygen environment is known as Oxygen Bomb Calorimetry and the vessel containing the sample is known as an Oxygen Bomb. The terms bomb and vessel are used interchangeably.

Related Instructions

Additional instructions concerning the installation and operation of various component parts and peripheral items used with the 6200 Calorimeter have been included and made a part of these instructions.

No.	Description
201M	*Limited Warranty*
205M	*1108 Series Oxygen Combustion Vessel*
207M	*Analytical Methods for Oxygen Bombs*
230M	*Safety in the Operation of Laboratory and Pressure Vessels*
483M	*Introduction to Bomb Calorimetry*

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 calorie, which is equal to 4.1868 absolute joules.

Purpose

Heats of combustion, as determined in an oxygen combustion ("bomb") calorimeter such as the 6200 Isoperibol Calorimeter, are measured by a substitution procedure in which the heat obtained from the sample is compared with the heat obtained from a standardizing material. In this test, a representative sample is burned in a high-pressure oxygen atmosphere within a metal oxygen combustion vessel or "bomb". The energy released by the combustion is absorbed within the calorimeter and the resulting temperature change is recorded.

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.

Explanation of Symbols

I	On Position
O	Off Position
~	Alternating Current
	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.
3. The user shall not open the calorimeter cover while testing is in progress.

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 to 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.

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 contacting Parr Instrument Company in order to determine which part(s) may be replaced or serviced.

Getting Started

These steps are offered to help the user become familiar with, install, operate and develop the full capabilities of the Parr 6200 Calorimeter.

1. Review the ***Chapter 1 - Concept of Operations***, to get an understanding of the overall capabilities of the calorimeter and microprocessor control.

2. Unpack and install the calorimeter in accordance with the ***Chapter 2 - Installation***. 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. Review the ***Chapter 3 - Program Installation & Control***, 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.

4. Turn the power switch ON (located on the back). Turn to the ***Chapter 5 - Menu Description,*** to review the touchscreen controls the menu functions used to modify the program contained in the 6200 Calorimeter. A review of the menus will provide a good idea of the capabilities and flexibility designed into this instrument.

5. Review ***Chapter 6 - Standardization***. 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 6200 Calorimeters will have an energy equivalent of approximately 2400 cal/°C. The runs for standardization and determinations are identical, except for the setting of the instrument to the standardization or determination mode.

6. Review the ***Chapter 8 - Calculations***. This provides information about calculations performed by the 6200 Calorimeter.

7. Review the ***Chapter 10 - Reporting Instructions***. to become familiar with the manner in which calorimetry corrections are entered. Also discussed are generating final reports, editing and clearing memory.

8. Review the ***Chapter 13 - Communication Interfacing,*** for the correct installation of any peripherals connected to the 6200 Calorimeter.

9. After successful standardization, the 6200 Calorimeter should be ready for testing samples.

Chapter 1
Concept of Operation

Overview

- The 6200 Calorimeter has been designed to provide the user with:
- A **traditional design** calorimeter with removable oxygen combustion vessel and bucket.
- A **moderately priced** calorimeter which uses real time temperature measurements to determine heat leaks using a controlled calorimeter jacket.
- A **high precision** calorimeter capable of exceeding the repeatability and reproducibility requirements of all international standard test methods.
- A **compact** calorimeter requiring minimum laboratory bench space.
- A modern intuitive **graphical user interface** for ease of operation and training.
- A calorimeter with up to date **digital hardware, software** and **communication** capabilities.
- A calorimeter that is **cost effective** and which can incorporate a user's current vessels, buckets, and accessories.

Removable Oxygen Combustion Vessel

The Model 6200 Calorimeter utilizes the Parr 1108P Oxygen Combustion Vessel. More than 20,000 of these reliable 1108 style oxygen combustion vessels have been placed in service on a world wide basis. This vessel features an automatic inlet check valve and an adjustable needle valve for controlled release of residual gasses following combustion. They are intended for samples ranging from 0.6 g to 1.2 g with a maximum energy release of 8 kcal per charge.

The 1108P Oxygen Combustion Vessel is made of high-strength, high nickel stainless steel designed to resist the corrosive acids produced in routine fuel testing. An alternative 1108PCL vessel is available, constructed of an alloy containing additional cobalt and molybdenum to resist the corrosive conditions produced when burning samples containing chlorinated compounds.

The Model 6200 can also be equipped with a variety of special purpose oxygen combustion vessels for unusual samples and/or applications. The 1104 High Strength Oxygen Combustion Vessel is designed for testing explosives and other potentially hazardous materials. The 1109A Semimicro Oxygen Combustion Vessels can be fitted along with its unique bucket to test samples ranging from 25 mg to 200 mg.

Removable Bucket

The A391DD removable bucket has been designed to hold the combustion vessel, stirrer, and thermistor with a minimum volume of water and to provide an effective circulating system which will bring the calorimeter to rapid thermal equilibrium both before and after firing.

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 combustion vessel.
- 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.

Full Microprocessor Based Data Acquisition & 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.
- Complete freedom for reagent concentrations and calculations.
- Unlimited choice of reporting units.
- Automatic vessel 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 6200 Calorimeter is equipped with one USB connection for direct communication with its printer and other peripherals. It is also equipped with an Ethernet network connection for connections to laboratory computers.

Chapter 2

Installation

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

Environmental Conditions

The 6200 Calorimeter is completely assembled and given a thorough test before it is shipped from the factory. If the user follows these instructions, installation of the calorimeter should be completed with little or no difficulty. If the factory settings are not disturbed, only minor adjustments will be needed to adapt the calorimeter to operating conditions in the user's laboratory.

This apparatus is to be used indoors. It requires at least 0.75 m^2 (8 sqft) of workspace on a sturdy bench or table in a well-ventilated area with convenient access to an electric outlet, running water and a drain. The supply voltage must be within ± 10 % of marked nominal voltage on the apparatus. The supply voltage receptacle must have an earth ground connection.

Required Consumables, Utilities & Power Requirements

The 6200 Calorimeter System requires availability of Oxygen, 99.5 % purity, 17 MPa (2500 psig) maximum.

The power requirements for the subassemblies of the 6200 Calorimeter are:

Calorimeter
120VAC, 6.0 Amps. 50/60 Hz
240VAC, 3.0 Amps. 50/60 Hz

1759 Printer
100 to 240 VAC, 1.4 Amps 50/60 Hz

1759 Printer Supplies
334C Printer Paper
381C Printer Ribbon

Filling the Jacket Reservoir

The water reservoir of the calorimeter must be filled with approximately 1.4 L of water (distilled or deionized preferred). This must be done prior to turning on the heater and the pump. The reservoir is filled through the tank fill elbow on the back of the calorimeter. The tank is full once water stands in the horizontal run of the filling elbow.

Power Connection

Plug the power line into any grounded outlet providing proper voltage that matches the specification on the nameplate of the calorimeter. The calorimeter will draw approximately 300 W of power. Grounding is very important not only as a safety measure, but also to ensure satisfactory controller performance. If there is any question about the reliability of the ground connection through the power cord, run a separate earth ground wire to the controller chassis.

Turn the power switch to the on position. After a short time, the Parr logo will appear on the LCD display followed by a running description of the instrument boot sequence. When the boot sequence is complete, the calorimeter Main Menu is displayed. Go to the Calorimeter Operation page and turn the heater and pump on. This begins circulating and heating the calorimeter jacket water. Add water to the filling elbow at the rear of the instrument as required in order to keep it full.

Jacket Cooling Water Connection

It becomes necessary to use the jacket cooling water connection only if the calorimeter operating room temperature exceeds 24 °C (75 °F).

When required, an external water source is used to cool the jacket of the 6200 Calorimeter. This is done in either of the following ways:

1. Tap water is used for cooling and then run to a drain.

2. Cooling water is re-circulated to the calorimeter from a Parr 6510 Water Handling System.

The water that provides the cooling goes through a heat exchanger and does not mix with the water in the jacket and its reservoir. There is a very low cooling load and tap water up to a temperature of 27 °C should be adequate.

Tap Water Cooling

Connect the tap water supply to the cold water inlet on the back of the calorimeter using either 1/4″ copper or nylon tubing (HJ0025TB035). A 196VB metering valve is provided with the calorimeter. This valve should be installed in this inlet line near the calorimeter. This valve is used to adjust the flow of water to the heat exchanger to compensate for differences in tap water temperatures and water line pressures. Once the calorimeter is operating at equilibrium, check the jacket temperature that is displayed on the operating page. If this temperature is cycling significantly, close down on the metering valve to reduce the flow of cooling water. If the jacket rises above its 30 °C set point, open this valve to increase the cooling. A flow rate of 100 mL/min is generally all that is required.

Connect the cooling water outlet on the back of the calorimeter to a drain using either nylon (HJ0025TB035) or copper 1/4″ tubing. A shut off valve in tap water supply line is also a good idea if the calorimeter will not be used for an extended period.

Cooling with the Water Handling System

If the calorimeter is to be operated with a Parr Water Handling System, connect the pump output to the cooling water inlet and connect the cooling water outlet to the return connection on the water handling system. With this installation it is neither necessary nor desirable to install the 196VB metering valve in the inlet line. It is a good idea to keep all water line runs as short as practical to avoid unwanted temperature changes in the water between the source and the calorimeter.

Oxygen Filling Connection

The 6200 Calorimeter is equipped with an automatic oxygen filling system for the combustion vessel. This system consists of an oxygen pressure regulator with a relief valve that mounts on an oxygen tank and a controlled solenoid inside the calorimeter. To install the regulator on the oxygen supply tank, unscrew the protecting cap from the oxygen 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 outlet and draw up the union nut tightly, keeping the gages tilted slightly back from an upright position. Connect the regulator to the oxygen inlet fitting on the back of the calorimeter case. This hose should be routed so that it will not kink or come in contact with any hot surface. Connect the high-pressure nylon hose with the push on connector to the oxygen outlet fitting on the back of the calorimeter. All connections should be checked for leaks. Any leaks detected must be corrected before proceeding. Instructions for operating the filling connection are in ***Chapter 3 - Operation***.

Adjust the pressure regulator to deliver 3.0 MPa (435 psig) of O_2. Assemble the oxygen combustion vessel without a charge and attach the filling hose to the vessel inlet valve. Press the **O2 FILL** key on the Calorimeter Operation page and observe the delivery pressure on the 0 MPa to 4 MPa (0 psi to 600 psi) gage while the oxygen is flowing into the vessel. Adjust the regulator, if needed, to bring the pressure to 3.0 MPa (435 psig). If there is any doubt about the setting, release the gas from the vessel and run a second check.

Printer and Balance Connections

Connect the printer to the calorimeter at this time. The Parr 1759 Printer is configured and furnished with a cord to connect directly to the USB port on the back of the calorimeter.

If a balance is to be attached to the calorimeter it will be necessary to use a USB hub so that multiple devices can be connected. Any standard USB hub can be used.

See ***Chapter 13 - Communication Interfaces*** for more information.

Standardizing the Calorimeter

The calorimeter must be accurately standardized prior to actually performing calorimetric tests on sample materials. Review ***Chapter 6 - 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 standardization mode on the CALORIMETER OPERATION menu, with the **OPERATING MODE** key. If two combustion vessels and buckets 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 menu, using the **BOMB/EE** key. All combustion vessel and bucket combinations 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 temperature of the calorimeter one degree. Repeated standardization with any given combustion vessel and bucket combination should yield an energy equivalent value with a range of 14 cal_{IT}/°C, centered around the mean value for all tests using that vessel and bucket combination. The calorimeter is ready for testing samples after an energy equivalent value has been obtained.

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 4 mm or smaller tube fittings, tighten the Swagelok nut 3/4 turns from finger-tight.

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

1. Insert the tubing with pre-swaged ferrules into the fitting body until the front ferrule seats.

2. Tighten the nut by hand. Rotate the nut to the original position with a wrench. An increase in resistance will be encountered at the original position. Then tighten slightly with a wrench. Smaller tube sizes (up to 3/16" or 4 mm) 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.

Figure 2-1

Swagelok Tube Fittings

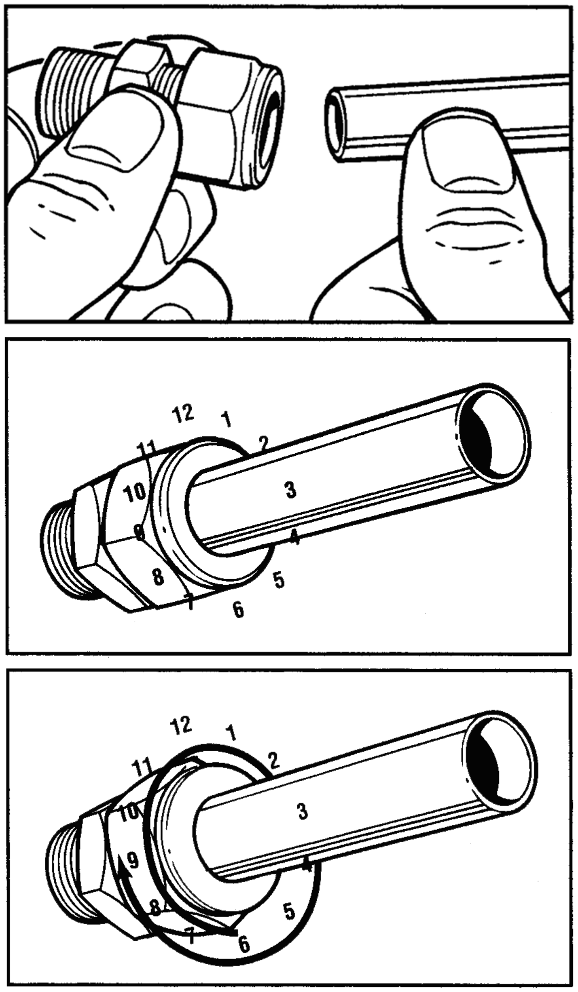


Figure 2-2

6200 Calorimeter Back Panel

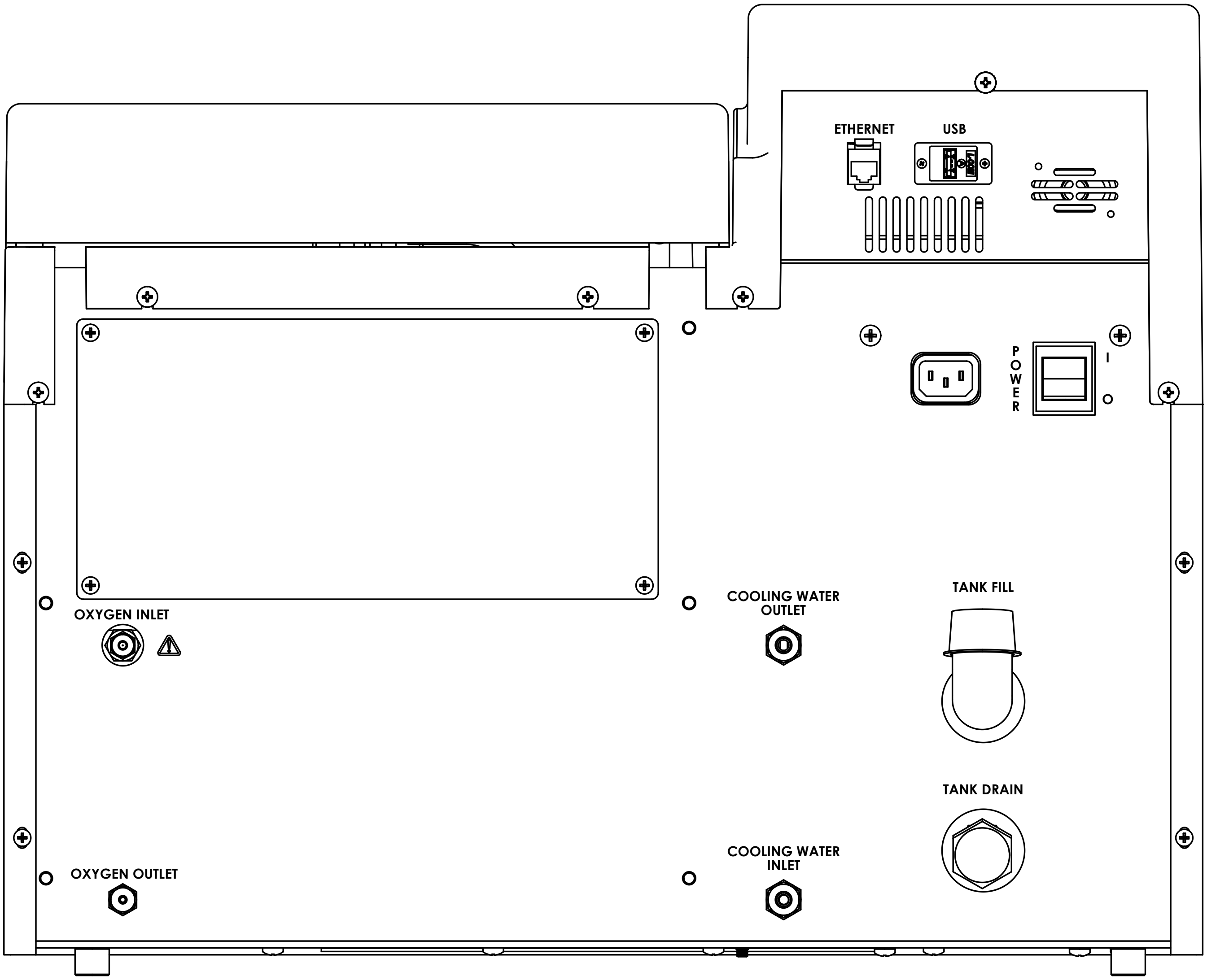


Figure 2-3

Closed Loop Configuration with 6510

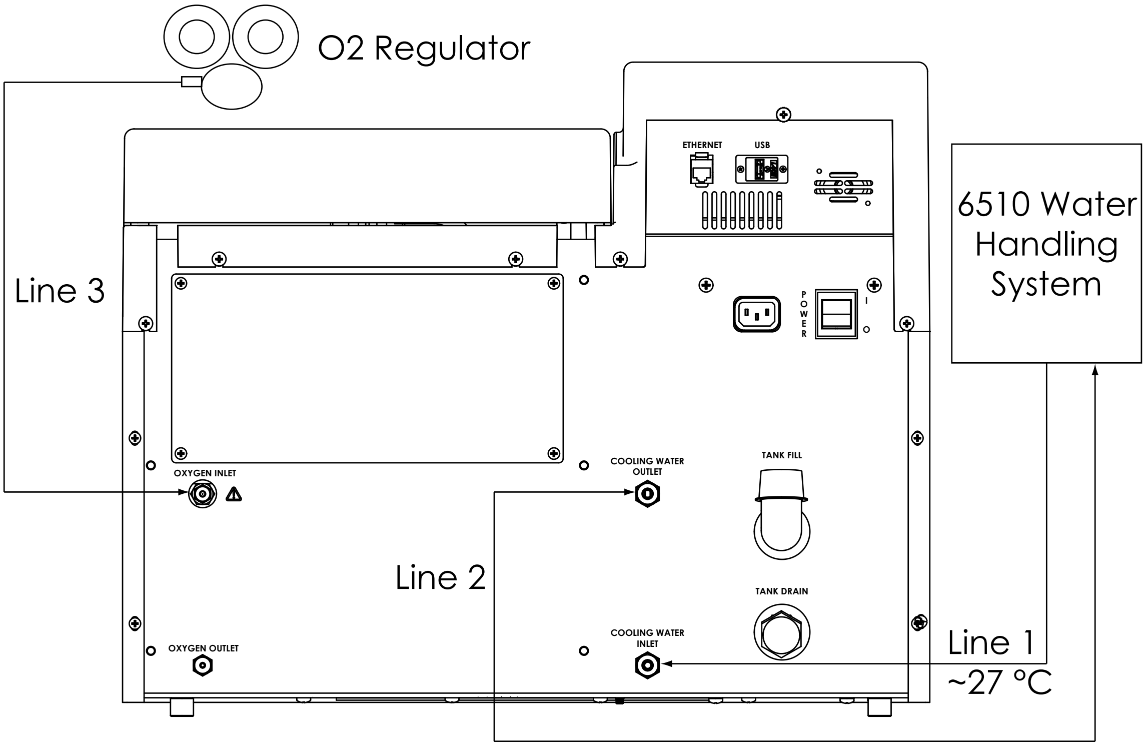
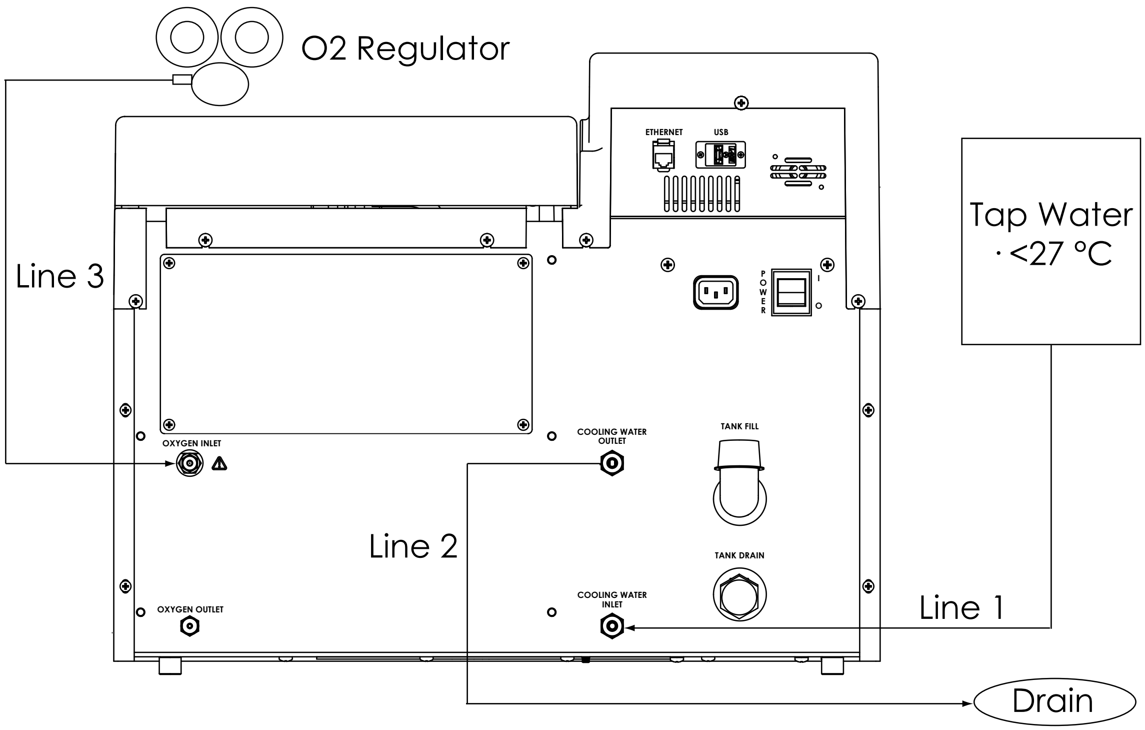


Figure 2-4

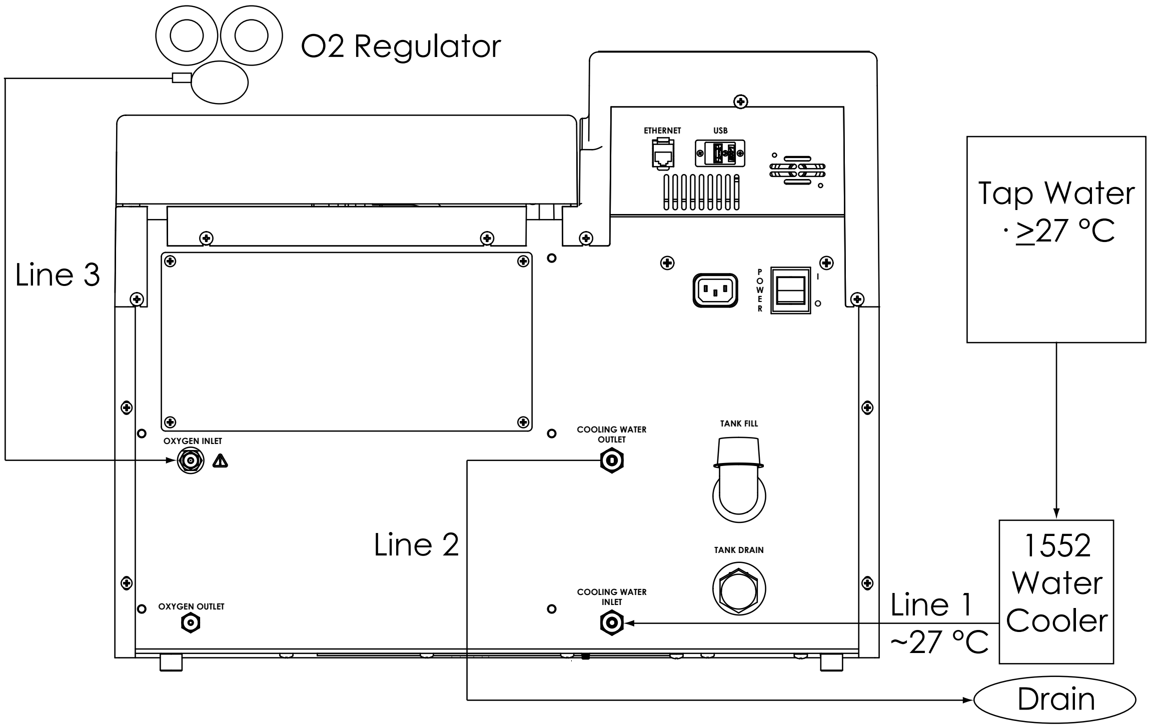
Open Loop Configuration with Tap Water



Line 1 & 2 – Maximum length of 10 ft, 1/4” OD, Polyurethane (Part Number HJ0025TB035)
Line 3 – Maximum length of 25 ft, 1/8” OD, Nylon (Part Number HX0012TB024)

Figure 2-5

Open Loop Configuration with 1552



Line 1 & 2 – Maximum length of 10 ft, 1/4” OD, Polyurethane (Part Number HJ0025TB035)
Line 3 – Maximum length of 25 ft, 1/8” OD, Nylon (Part Number HX0012TB024)

Chapter 3

Program Installation & Control

Software Installation

The program in the 6200 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 ***Chapter 5*** 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. A more in-depth explanation of these parameters is found on the corresponding parameter group help pages within the software. 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 & CONTROL** menu, **USER/FACTORY SETTINGS**, **RELOAD FACTORY DEFAULT SETTINGS** and confirm **YES**.

User Default Settings

The user parameters of the 6200 Calorimeter can be saved to guarantee that the desired calorimeter configurations can always be recalled before beginning a series of tests. Users who wish to permanently save their default settings may do so using the following procedure:

1. Establish the operating parameters to be stored as the user default settings.
2. Go to the **PROGRAM INFO & CONTROL** menu, **USER/ FACTORY SETTINGS**, **USER SETUP ID**, and enter the desired **USER SETUP ID**.
3. Select **SAVE USER DEFAULT SETTINGS**

To re-load the user default settings, go to the **PROGRAM INFO & CONTROL** menu, **USER/FACTORY SETTINGS**, **RELOAD USER DEFAULT SETTINGS**, and confirm **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.

Factory Default Settings

Calorimeter Operations	
Operating Mode	Determination
Bomb Installed/EE	1/2400.0
Heater and Pump	OFF

Operating Controls	
Method of Operation	Dynamic
Reporting Units	BTU/lb
Use Spiking Correction	OFF
"OTHER" Multiplier	4.1868
LCD Backlight Timeout(s)	1200 S
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
Prompt for Spike before Weight	OFF

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	62-1108
Reload Factory Default Settings	
Reload User Default Settings	
Save User Default Settings	

Calibration Data & Controls	
Calibration Run Limit	10
EE Max Std Deviation	0
Heat of Combustion of Standard	6318.4
Bomb Service Interval	500
Control Chart Parameters	
Charted Value	HOC Standard
Process Sigma	0.1
Temp Rise High Warning	3.3
Temp Rise Low Warning	2
Use Bomb	1

Bomb 1 Through 4

EE Value	2400
Protected EE Value	OFF

Thermochemical Corrections	
Standardization Correction	
Fixed Fuse Correction	ON 50
Acid Correction	Fixed HNO3 10.0
Fixed Sulfur Correction	ON 0.0
Determination Correction	
Fixed Fuse Correction	ON 50
Acid Correction	Fixed HNO3 10.0
Fixed Sulfur Correction	OFF 0.0

Net Heat/Dry Factors	
Net Heat/Dry Factors	Net Heat & Dry Disable
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

Calculation Factors	
Nitric Acid Factor	1.58
Acid Multiplier	0.0709
Sulfur Value is Percent	ON
Sulfur Multiplier	0.6238
Fuse Multiplier	1
Use Offset Correction (ISO)	OFF
Offset Value	0

Data Entry Controls	
Prompt for Bomb ID	ON
Weight Entry Modes	Touchscreen
Acid Entry Mode	Touchscreen
Net Heat Entry Modes	Touchscreen
Auto Sample ID Controls	ON
Sample Weight Warning above	2
Spike Weight Entry Mode	Touchscreen
Sulfur Entry Mode	Touchscreen
Moisture Entry Mode	Touchscreen
Auto Preweigh Controls	ON

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

Preweigh Sample IS Controls	
Automatic Preweigh ID	ON
Automatic Preweigh ID Increment	1
Automatic Preweigh ID Number	1

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

Communication Controls	
Printer Type	Parr 1759
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	

Diagnostics

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
Log Trigger	Timebase

Chapter 4

Operation

Operating the 1108P Oxygen Combustion Vessel

Detailed instructions for preparing the sample and charging the 1108P Oxygen Combustion Vessels are given in Operating Instructions No. 205M. Follow these instructions carefully, giving particular attention to the precautions to be observed in charging and handling the vessel.

Operating the Filling Connection

To fill the combustion vessel, connect the hose to the vessel inlet valve and push the **O2 FILL** button on the calorimeter control panel. The calorimeter will then fill the combustion vessel to the preset pressure and release the residual pressure in the connecting hose at the end of the filling cycle. It will take approximately 60 seconds to fill the vessel. During this time a countdown timer on the **O2 FILL** button will display the remaining fill time. Pushing the **O2 FILL** button a second time will stop the flow of oxygen at any time. Once the display returns to its normal reading, the user can disconnect the coupling and proceed with the combustion test.

If the charging cycle should be started inadvertently, it can be stopped immediately by pushing the **O2 FILL** key a second time.

During extended periods of inactivity, overnight or longer, close the tank valve to prevent leakage. When changing oxygen tanks, close the tank valve and push the **O2 FILL** key to exhaust the system. Do not use oil or combustible lubricants on this filling system or on any devices handling oxygen under pressure. Keep all threads, fittings, and gaskets clean and in good condition. Replace the two 394HCJE O-rings in the slip connector if the connector fails to maintain a tight seal on the vessel inlet valve.

The recommended filling pressure is 3.0 MPa (435 psig or 30 bar). This pressure is prescribed by most of the standard bomb calorimetric test methods. Higher or lower filling pressures can be used, but the combustion vessel must never be filled to more than 4.0 MPa (600 psig or 40 bar).

Operating the Calorimeter

All operations required to standardize the 6200 Calorimeter, or test an unknown sample, should proceed step-wise in the following manner:

1. Turn on the calorimeter and activate the pump and heater using Calorimeter Operations. **Allow at least 20 min for the calorimeter to warm up and the jacket temperature to stabilize. Once the jacket temperature comes within 30 °C ± 0.5 °C and stays there for approximately 15 min, the calorimeter is ready to begin testing.** The **START** key will be available at this time. The combustion vessel parts should be wetted and then dried in the manner used at the conclusion of a test. This serves to wet all sealing parts as well as leaving the vessel with the same amount of residual water which will exist in all subsequent testing.

2. Prepare the sample weighing the material to 0.1 mg and charge the oxygen combustion vessel as described in the section entitled **Operating the Filling Connection**. Using an additional combustion vessel and bucket can increase the throughput of the 6200 Calorimeter. With this arrangement, the calorimeter can operate almost continuously since the operator will be able to empty a vessel and recharge it while a run is in progress. A vessel and bucket for the next run will be ready to go into the calorimeter as soon as it is opened. Each vessel and bucket combination will have to be standardized separately and the proper energy equivalent for each set must be used when calculating the heat of combustion.

3. Fill the calorimeter bucket by first taring the dry bucket on a solution or trip balance; then add 2 kg ± 0.5g of water. Distilled water is preferred, but demineralized or tap water containing less than 250 mg/kg (250 ppm) of dissolved solids is satisfactory. The bucket water temperature should be approximately 3 °C to 5 °C below the jacket temperature. It is not necessary to use exactly 2 kg, but the amount selected must be duplicated within ± 0.5 g for each run. Instead of weighing the bucket, it can be filled from an automatic pipet, or from any other volumetric device if the repeatability of the filling system is within ± 0.5 mL.

4. To speed and simplify the bucket filling process, and to conserve water and energy, Parr offers a closed circuit Water Handling System (No. 6510). This provides a water supply, cooled to the starting temperature and held in an automatic pipet ready for delivery in the exact amount needed to fill the bucket. Instructions for this automatic system are given in Operating Instruction No. 454M.

5. Set the bucket in the calorimeter. Attach the lifting handle (421A) to the two holes in the side of the screw cap and partially lower the combustion vessel into the water. Handle the vessel carefully during this operation so that the sample will not be disturbed. Push the two ignition lead wires into the terminal sockets on the combustion vessel head. Orient the wires away from the stirrer shaft so they do not become tangled in the stirring mechanism. Lower the vessel completely into water with its feet spanning the circular boss in the bottom of the bucket. Remove the lifting handle and shake any drops of water back into the bucket and check for gas bubbles.

CAUTION! If bubbles continue to rise from the combustion vessel after the air in the screw cap has escaped the test must be stopped and the vessel not fired until the leak has been corrected.

6. Close the calorimeter cover. This lowers the stirrer and thermistor probe into the bucket. Make sure that the bucket thermistor does not touch the bucket or vessel when the lid is lowered.

7. Select determination or standardization as appropriate on the **CALORIMETER OPERATION** menu by toggling the **OPERATING MODE** key. After pressing the **START** key, the calorimeter will now prompt the operator for Bomb ID number, sample ID number, sample mass and spike mass in accordance with the instructions set into the operating controls page.

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

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

10. Open the cover and remove the combustion vessel and bucket. Remove the combustion vessel from the bucket and open the knurled valve knob on the vessel head to release the residual gas pressure before attempting to remove the cap. This release should proceed slowly over a period of not less than one minute to avoid entrainment losses. After all pressure has been released, unscrew the cap, lift the head out of the cylinder and examine the interior of the vessel for soot or other evidence of incomplete combustion. If such evidence is found, the test will have to be discarded. Otherwise, wash all interior surfaces of the vessel, including the head, with a jet of distilled water and collect the washings in a beaker.

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

12. Analyze the vessel washings to determine the sulfur content of the sample if it exceeds 0.1 %. Methods for determining sulfur are discussed in Operating Instructions No. 207M.

13. At the end of the testing period, turn OFF the calorimeter using the **MAIN MENU**, ⏻ key.

Samples and Sample Holders

Particle Size and Moisture Content. Solid samples burn best in an oxygen combustion vessel when reduced to 250 μm (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 in giving controlled burn rates, the pelletizing of samples keeps the sample in the fuel capsule during combustion.

CAUTION! 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 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.

CAUTION! It must be remembered, that a combustion aid adds to the total energy released in the vessel 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 combustion vessel which might result from rapid combustion of the loose powder.

Oxygen Charging Pressure

The 6200 Calorimeter has been designed to operate with an oxygen filling pressure of 3.0 MPa (435 psig or 30 bar). Significant changes from this value are not recommended.

Combustion Capsules

Non-volatile samples to be tested in Parr oxygen combustion vessels are weighed and burned in shallow capsules measuring approximately 1" dia. and 7/16" deep. These are available in stainless steel, fused silica, fused quartz, and platinum alloyed with 3.5 % 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/quartz 43A3 capsule is required. When superior corrosion resistance is needed, the platinum rhodium 43A5 capsule is required.

The stainless steel capsules will acquire a dull gray finish after repeated use in an oxygen combustion vessel 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 0.7 mm (0.025").

New capsules are heated in a muffle furnace at 500 °C for 24 h by Parr 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 furnace 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 a combustion aid such as mineral oil to retard the burn rate and avoid development of high pressures.

Partial drying may be necessary if the moisture content is too high to obtain ignition, but if the sample is heat sensitive and cannot be dried, a water soluble combustion aid such as ethylene glycol can be added to promote ignition.

Coarse Samples

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

Corrosive Samples

The 1108P Oxygen Combustion Vessel is made of a corrosion resistant alloy designed to withstand the corrosive mixture of sulfuric and nitric acids produced in normal fuel testing operations. Samples containing chlorine and particularly samples containing more than 20 mg of chlorine samples with high sulfur contents will greatly accelerate corrosion of the vessel. An alternate 1108PCL is available constructed of an alloy selected to specifically resist the corrosive effects of samples with high chlorine or chloride.

While no material will offer complete corrosion resistance to these samples, the 1108PCL offers significantly enhanced corrosion resistance for this service.

Explosives and High Energy Fuels

The 1108P and 1108PCL used in the 6200 Calorimeter have been designed to provide highly automated testing of routine samples. Materials which release large volumes of gas, which detonate with explosive force, or burn with unusually high energy levels should not be tested with these combustion vessels. Rather, they should be tested in a model 1104 High Pressure Oxygen Combustion Vessel 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 stainless steel 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.

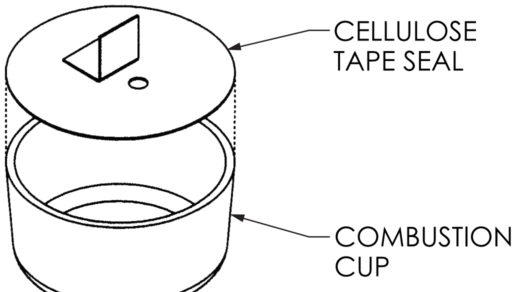
The weight of the tape disc must be determined separately and a correction applied for any elements in the tape which might interfere with the determination. The approximate Heat of Combustion of the tape is 26.5 kJ/g (6.3 kcal/g). An actual amount should be determined by running a blank test with tape alone using a sample weighing 1.0 g. 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.

Note: Heat of Combustion of Spike must be entered in cal_{IT}/g.

Figure 4-1

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 combustion vessel with the usual oxygen charging pressure.
- The calorimeter will fire the combustion vessel and complete the test in the usual manner.

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 combustion vessel, 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:

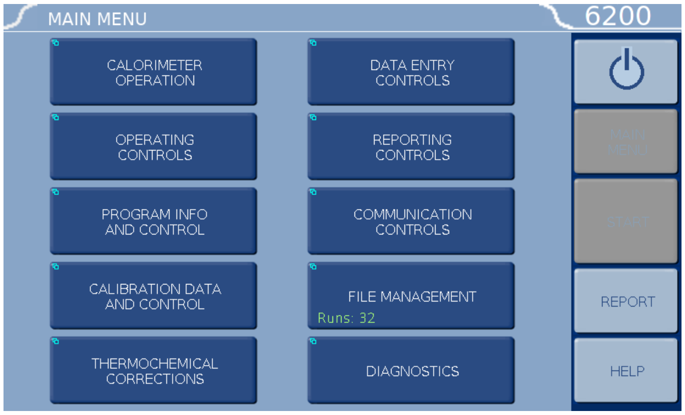
- Some part of the sample must be heated to its ignition temperature to start the combustion and, in burning, it must liberate sufficient heat to support its own combustion regardless of the chilling effect of the adjacent metal parts.
- The combustion must produce sufficient turbulence within the vessel 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 250 μm (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 combustion vessel. The bottom of the cup should always be at least one-half inch above the bottom of the vessel or above the liquid level in the vessel 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.

Chapter 5

Menu Descriptions

Menu Structure

All calorimeter configurations and operations are handled by a menu-driven system operated from the bright touchscreen display. The settings and controls are organized into ten 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 Touchscreen from anywhere in the menu structure.

3. **Start.** This key is used to start a calorimeter test.

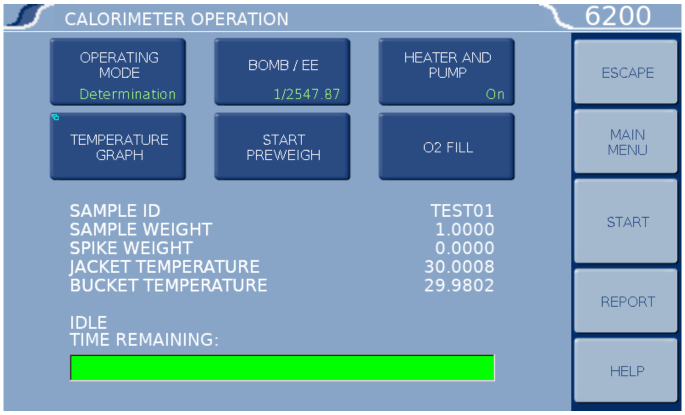
4. **Report.** This key is used to access the test results stored in the calorimeter, to enter thermochemical corrections and to initiate report on the display, printer or attached computer.

5. **Help.** This key is used to access help screens related to the menu currently displayed on the Touchscreen.

6. **Abort.** This key appears in the start key location while the test is running. Pressing this key will abort the test in progress.

7. ⏻ This key appears on the main menu only and is used to prepare the calorimeter for turning off the power.

CALORIMETER OPERATION



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

OPERATING MODE: Sets the operating mode by toggling between standardization and determination.

BOMB/EE: Used to identify the bomb presently installed in the calorimeter and its EE value.

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 jacket to the correct starting temperature before testing can commence.

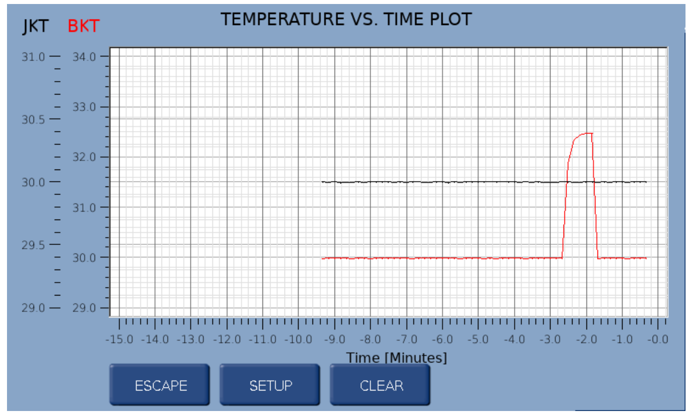
START PREWEIGH: This key is used to start the sample preweigh process. The user is presented with or prompted for a sample ID. Next, the user is asked to key in the associated sample mass or alternatively the mass can be retrieved from a connected balance.

O2 FILL: This key is used to activate the oxygen filling system used to fill the combustion vessel. Pressing this same key while the vessel is filling will abort the process.

TEMPERATURE GRAPH: Press this key to view the Temperature vs. Time Plot Screen.

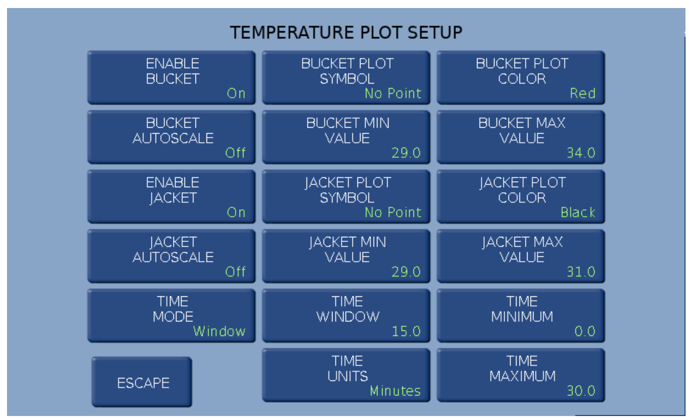
CALORIMETER OPERATION (CONTINUED)

TEMPERATURE VS. TIME PLOT SCREEN



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



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

CALORIMETER OPERATION (CONTINUED)

TEMPERATURE PLOT SETUP (CONTINUED)

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 Symbol, above).

JACKET MIN VALUE: Press this key to access its numeric dialog box to set a minimum jacket value.

TIME WINDOW: Sets the time scale for the X-axis.

TIME UNITS: Toggles between minutes and seconds.

BUCKET PLOT COLOR: Toggles between:

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

BUCKET MAX VALUE: Press this key to access its numeric dialog box to set a maximum bucket value.

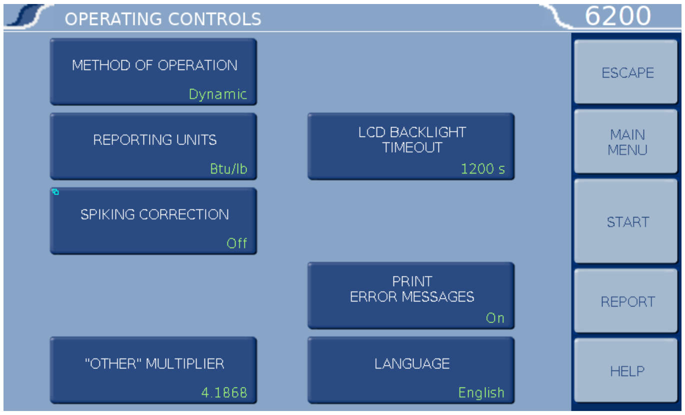
JACKET PLOT COLOR: Toggles between: (Same as Bucket Plot Color, above.)

JACKET MAX VALUE: Press this key to access its numeric dialog box to set a maximum jacket value.

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

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

OPERATING CONTROLS

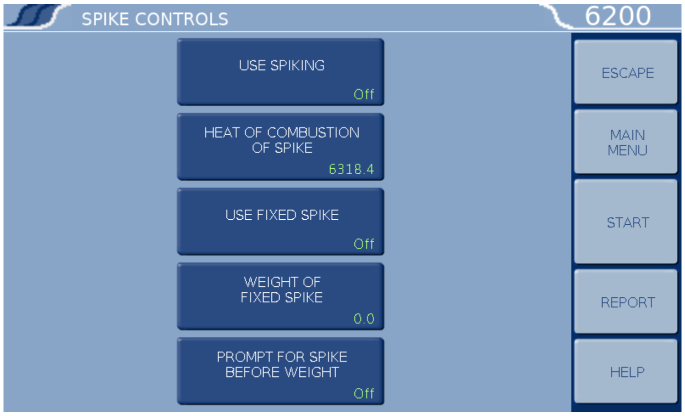


METHOD OF OPERATION: Offers an operating mode of either dynamic or equilibrium. In most cases, the dynamic mode with its curve matching capability will save approximately 3-4 minutes per test and will produce the same operating precision as the slower equilibrium mode.

REPORTING UNITS: Toggles between BTU/lb, cal/g, J/kg, other, and MJ/kg. A user selected set of reporting units may be programmed by selecting "Other".

SPIKING CORRECTION: Accesses sub-menu, Spike Controls. Spiking is the material addition, such as benzoic acid or mineral oil, to samples which are difficult to burn in order to drive the combustion to completion.

SPIKING CONTROLS



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.

OPERATING CONTROLS (CONTINUED)

SPIKING CONTROLS (CONTINUED)

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 a sub-menu keyboard.

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.

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

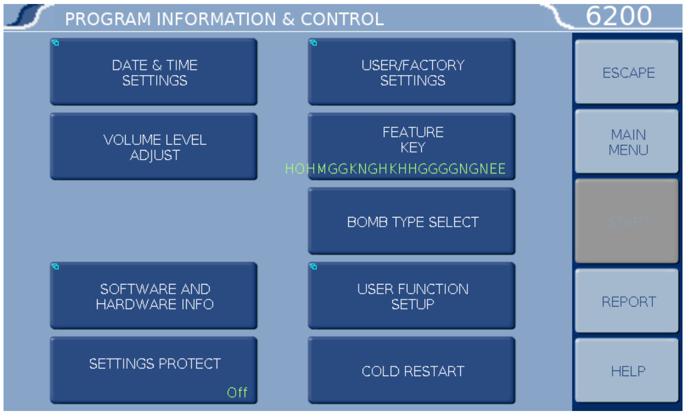
"OTHER" MULTIPLIER: This button allows the user to enter a final multiplier that is used when the reporting units are set as "Other".

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

PRINT ERROR MESSAGES: When turned ON, all error messages will be printed on the printer as well as displayed on the screen.

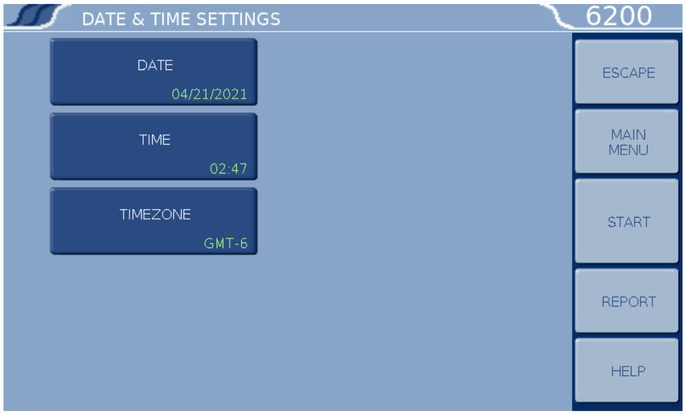
LANGUAGE: Steps the calorimeter through the installed operating languages.

PROGRAM INFORMATION & CONTROL



DATE & TIME SETTINGS: Access the sub-menu on which Date & Time are set.

DATE & TIME SETTINGS



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

TIME: Display current time and accesses the sub-menu on which time is set in (HH:MM) format.

TIME ZONE: Allows the user to select the local time zone. Pressing the button will toggle through the time zones.

PROGRAM INFORMATION & CONTROL (CONT.)

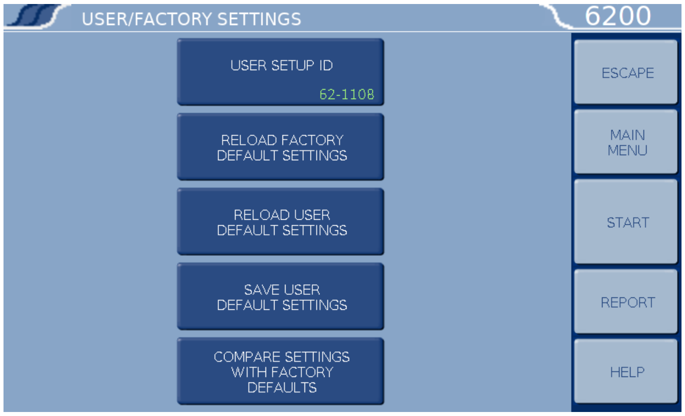
VOLUME LEVEL ADJUST: Opens a window with a slide adjustmenr to set the volume of the key clicks and alarms of the calorimeter. Default is 85 %.

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.

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/FACTORY SETTINGS



USER SETUP ID: Used to enter a unique identifier for recalling user settings. After assigning a User Setup ID the Save User Default Settings must be used in order to save the settings.

RELOAD FACTORY DEFAULT SETTINGS: Used to erase all of the settings and restore the factory default settings.

PROGRAM INFORMATION & CONTROL (CONT.)

USER/FACTORY SETTINGS (CONTINUED)

RELOAD USER DEFAULT SETTINGS: Used to load/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.

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.

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.

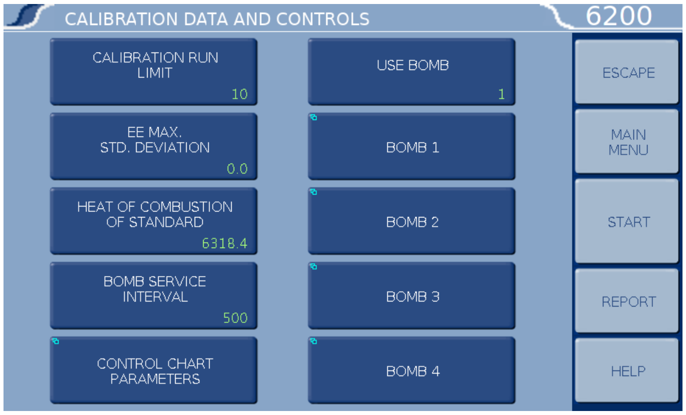
FEATURE KEY: Unique Feature Keys obtained from Parr allow the user to access capabilities on the instrument such as bar code interfacing or remote operation of the calorimeter.

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.

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

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

CALIBRATION DATA AND CONTROLS



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 view or printing.

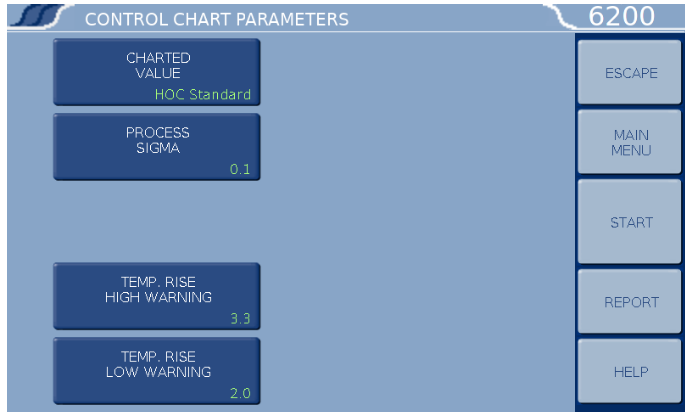
EE MAX STD DEVIATION: Displays the maximum relative standard deviation in percent that will be permitted for any EE value calculated by the calorimeter and accesses the sub-menu on which this limit is set. If this value is exceeded, the user will be warned to take corrective action before proceeding with testing. This calorimeter is capable of achieving a value of 0.17 % or better for 10 tests. 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 cal_{IT}/g.

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. This interval may be more frequent 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.

CONTROL CHART PARAMETERS



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.

Note: The 6200 is a 0.1 Process Sigma calorimeter.

TEMP. RISE HIGH WARNING: Sets the high warning limit on the bucket temperature rise. A temperature rise higher than this value will generate a warning message.

TEMP. RISE LOW WARNING: Sets the low warning limit on the bucket temperature rise. A temperature rise lower than this value will generate a warning message.

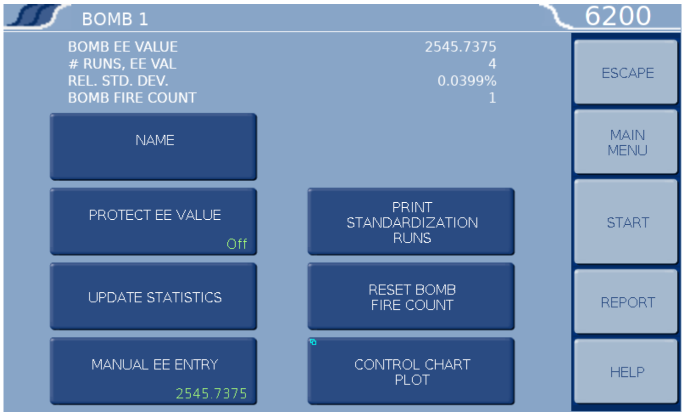
USE BOMB: Displays the bomb number of the bomb currently installed in the calorimeter and toggles through the four possible bomb numbers. The left and right arrow keys are used to toggle through the bomb identification numbers available for each bomb.

CALIBRATION DATA AND CONTROLS (CONT.)

BOMB 1 - BOMB 4: Leads to sub-menus for Bomb 1 - Bomb 4. 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 throughput. 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 two bombs. Each bomb should be assigned a bomb number. Set prompt for bomb ID to "ON".

BOMB 1



- BOMB EE VALUE. Displays the calculated EE value for the corresponding Bomb 1.
- # OF RUNS, EE VALUE. Displays how many runs have been used to determine the EE value.
- REL. STD. DEV. Displays the relative standard deviation for the series of tests used to determine the current EE value in percent of the EE value.
- BOMB FIRE COUNT. Displays the current bomb firing count or the number of times the bomb has been fired since it was last serviced. When this count matches the limit set by Bomb Service Interval, the user will be informed that the bomb is ready to be serviced.

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: When set to ON, protects the EE value if the user does not wish to have the calorimeter automatically update its own EE value.

CALIBRATION DATA AND CONTROLS (CONT.)

BOMB 1 (CONTINUED)

UPDATE STATISTICS: This key will cause the EE value for this bomb ID to be updated using the most recent standardization runs; if the EE value is not protected. (The number of standardization runs used is equal to the value entered into the Calibration Data and Controls Menu under Calibration Run Limit. If less runs are available than the number specified, all runs will be used.)

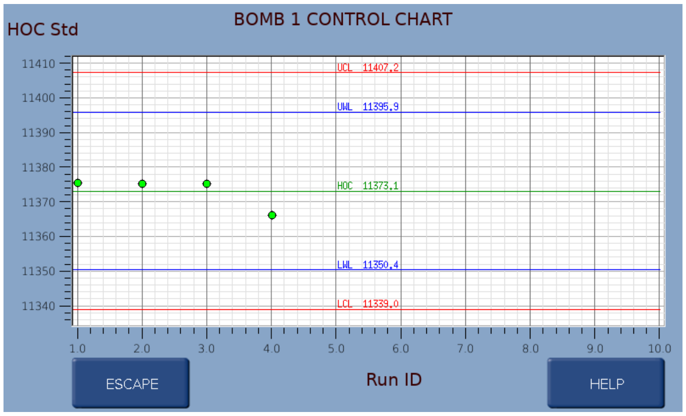
MANUAL EE ENTRY: This key allows the user to manually enter an EE or calibration factor for a given calorimeter ID or bomb head. If an EE value is manually entered, it is necessary to turn the Protect EE Value ON in order to prevent this value from being overwritten by an automatic update.

PRINT STANDARDIZATION RUNS: 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.

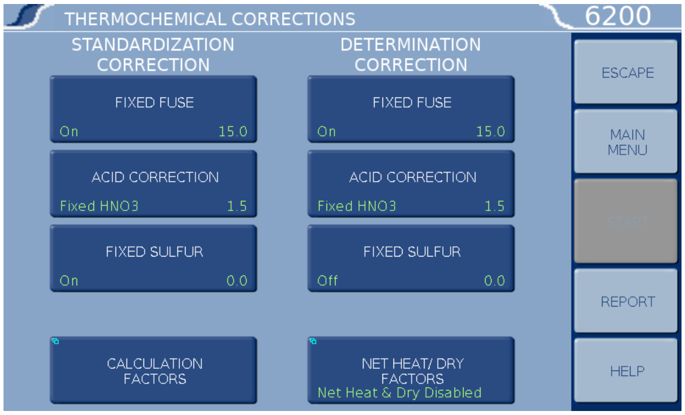
RESET BOMB FIRE COUNT: After bomb service, press this button to reset the fire count to zero.

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.



THERMOCHEMICAL CALCULATIONS



STANDARDIZATION CORRECTION

FIXED FUSE CORRECTION: Displays both the ON/OFF of the fixed fuse corrections for standardization runs and the value of the correction. This key toggles the correction ON/OFF and accesses a sub-menu on which the value is set. An appropriate fixed fuse value is 50 calories for the 1108P style oxygen combustion vessel.

ACID CORRECTION: Press this key on the LEFT side to toggle between Fixed HNO_3, Calculated HNO_3, Entered Total, Entered HNO_3, and Fixed Total for the acid correction for determination runs. Press it on the RIGHT side to access the Acid Correction numeric dialog box on which the value can be set.

Options for the Acid Correction:

- Fixed HNO_3
- Calculated HNO_3
- Entered Total
- Entered HNO_3
- Fixed Total

These options are discussed further in ***Chapter 8 - Calculations***.

FIXED SULFUR CORRECTION: Displays both the ON/OFF of the fixed sulfur corrections for standardization runs and the value of the correction. This key toggles the correction ON/OFF and accesses a sub-menu on which the value is set. When benzoic acid is used as the calibrant, a fixed sulfur value of zero should be used.

THERMOCHEMICAL CALCULATIONS (CONT.)

DETERMINATION CORRECTION

FIXED FUSE: Displays both the ON/OFF of the fixed fuse corrections for determination runs and the value of the correction. This key toggles the correction ON/OFF and accesses a sub-menu on which the value is set.

ACID CORRECTION: Press this key on the LEFT side to toggle between Fixed HNO_3, Calculated HNO_3, Entered Total, Entered HNO_3, and Fixed Total for the acid correction for determination runs. Press it on the RIGHT side to access the Acid Correction numeric dialog box on which the value can be set.

Options for the Acid Correction:

- Fixed HNO_3
- Calculated HNO_3
- Entered Total
- Entered HNO_3
- Fixed Total

These options are discussed further in ***Chapter 8 - Calculations***.

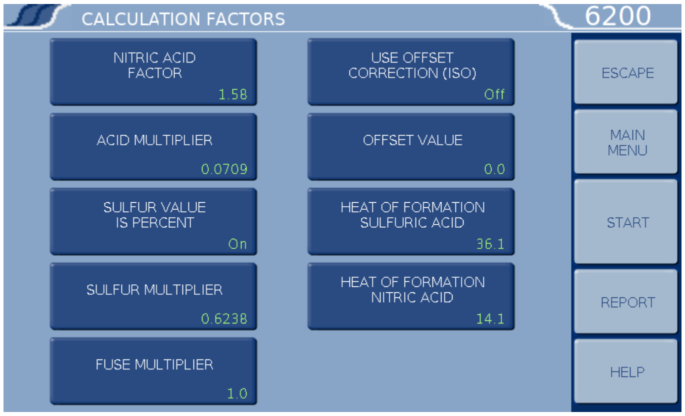
FIXED SULFUR: Displays both the ON/OFF of the fixed sulfur corrections for determination runs and the value of the correction. This key toggles the correction ON/OFF and accesses a sub-menu on which the value is set.

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, a preliminary report will not print, rather only a final report will be generated. If values for these corrections are entered into these lines, and the toggle is set to OFF, then the fixed value will be used in the preliminary report, but not in the final report.

THERMOCHEMICAL CALCULATIONS (CONTINUED)

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

CALCULATION FACTORS



NITRIC ACID FACTOR: The default is 1.58 cal per 1 kcal of released energy.

ACID MULTIPLIER: The multiplier is the normality of the sodium carbonate used to titrate for the acid correction. The default value of 0.0709 allows for direct entry of the acid correction in calories. If the bomb rinsings are titrated in order to determine the acid correction, this multiplier is the concentration of the base (equivalents/L) or normality used for titration. In this case, the acid correction is entered as milliliters of base used to titrate the bomb rinsings.

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 thread used to ignite the sample. If another measurement is used, the correction factor must be entered here.

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.

THERMOCHEMICAL CALCULATIONS (CONTINUED)

CALCULATION FACTORS (CONTINUED)

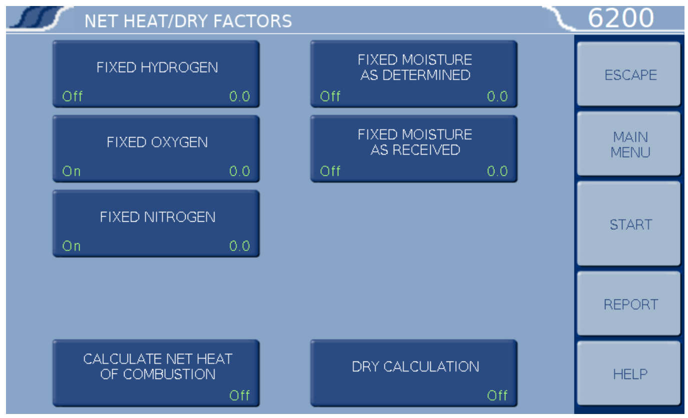
To use these calculations, set to ON and enter the appropriate value as the offset value.

OFFSET VALUE: Entry for the value when use offset correction is turned ON.

HEAT OF FORMATION, SULFURIC ACID: The heat of sulfuric acid formation used in the e_2 correction calculation (cal/mEq) (where mEq is milliequivalent).

HEAT OF FORMATION, NITRIC ACID: The heat of nitric acid formation used in the e_1 correction calculation (cal/mEq).

NET HEAT/DRY FACTORS



FIXED HYDROGEN: ON/OFF and value entry.

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

PROMPT FOR BOMB ID: 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 Touchscreen, network or through the balance (USB) port.

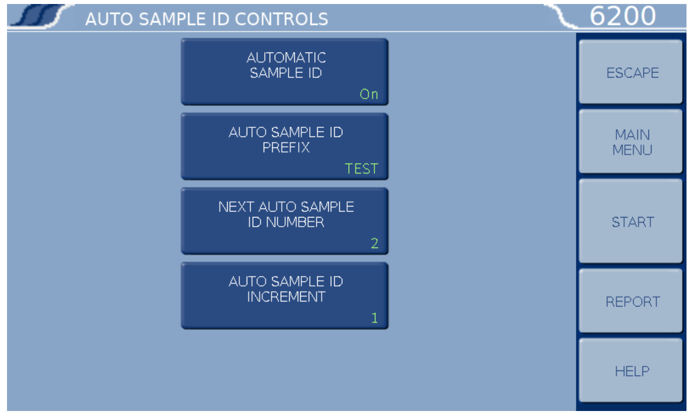
ACID ENTRY MODE: This key steps through the options for entering acid correction value either manually through the Touchscreen or automatically through the balance (USB) port.

NET HEAT ENTRY MODES: This key accesses a menu listing options for entering hydrogen, oxygen, and nitrogen content for calculating the net heat of combustion either manually through the Touchscreen or automatically through the balance (USB) port.

AUTOMATIC SAMPLE ID CONTROLS: Accesses sub-menu for controlling the automatic assignment of sample identification numbers.

DATA ENTRY CONTROLS (CONTINUED)

AUTOMATIC SAMPLE ID CONTROLS



AUTOMATIC SAMPLE ID: When set to ON it will automatically assign sample identification numbers in accordance with instructions set in the other two keys on this menu.

AUTO SAMPLE ID PREFIX: An entry here will be used as a prefix for all sample IDs.

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.

AUTO SAMPLE ID INCREMENT: Establishes the increment between sample numbers.

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 Touchscreen, automatically through the balance (USB) port or through a network.

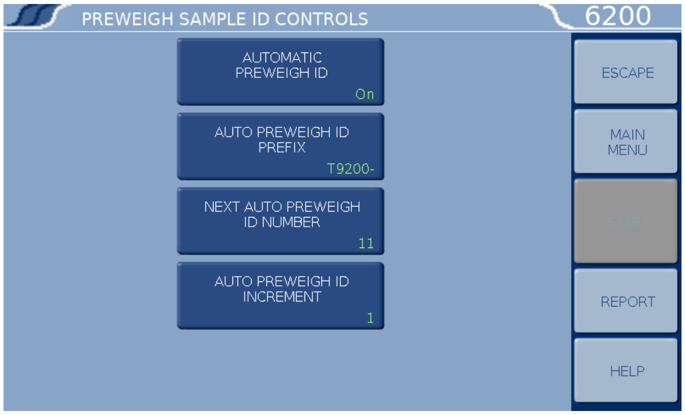
DATA ENTRY CONTROLS (CONTINUED)

SULFUR ENTRY MODE: This key toggles steps through the options for entering sulfur correction value either manually through the Touchscreen or through the balance (USB) port.

MOISTURE ENTRY MODE: This key steps through the options for entering the moisture percentage whether manually through the Touchscreen or automatically through the balance (USB) port.

AUTO PREWEIGH ID CONTROLS: Accesses sub-menu, used to automatically assign sample identification numbers when a series of samples are pre weighed ahead of the time they are actually tested.

PREWEIGH SAMPLE ID CONTROLS



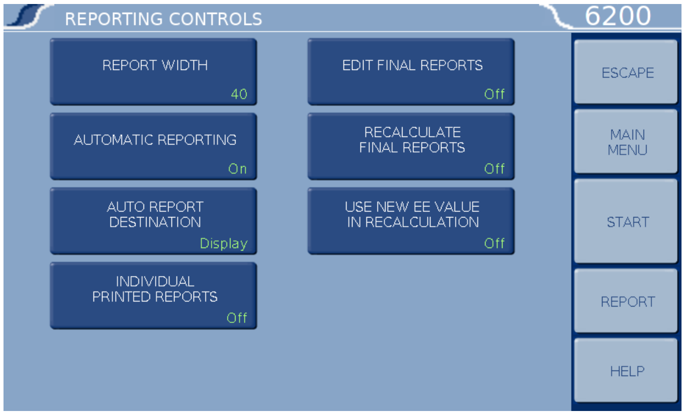
AUTOMATIC PREWEIGH ID: ON/OFF toggle for this feature.

AUTOMATIC PREWEIGH ID PREFIX: An entry here will be used as a prefix for all pre-weigh sample identification numbers.

NEXT AUTOMATIC PREWEIGH ID NUMBER: Shows the next sample identification number which will be assigned and is used to enter the beginning Sample ID of any series.

AUTOMATIC PREWEIGH ID INCREMENT: Establishes the increment between samples.

REPORTING CONTROLS



REPORT WIDTH: The column width of the printer being used can be set to 40 or 80 columns. Select 40 when the 1759 Printer is used.

AUTOMATIC REPORTING: 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 this automatic reporting feature is turned ON. When this is turned OFF, reports will only be generated through the reporting controls.

AUTOMATIC REPORT DESTINATION: Directs the reports to the printer port or the display.

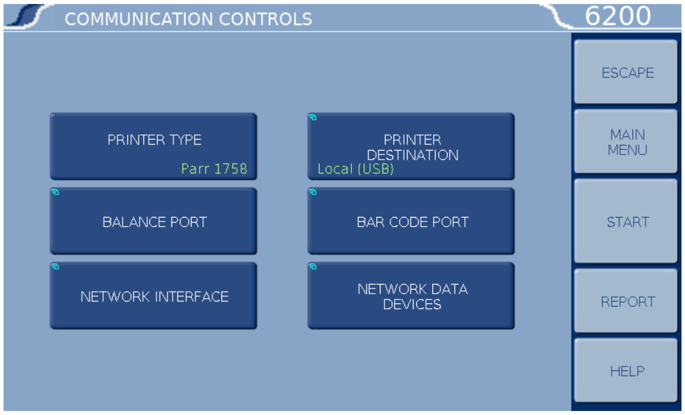
INDIVIDUAL PRINTED REPORTS: When set to ON, will generate extra spacing for each report printed. In the OFF position, only one header will be printed for a series of tests.

EDIT FINAL REPORTS: When set to ON, enables the user to revise sample weight and thermochemical corrections.

RECALCULATE FINAL REPORTS: When set to ON, causes a recalculation of stored final reports using calibration data and menu settings currently in the calorimeter.

USE NEW EE VALUE IN RECALCULATION: When set to ON, any recalculation made will use the most recent EE value in the calculations. In the OFF position, all calculations will be made using the EE value which was effective when the test was originally run.

COMMUNICATION CONTROLS

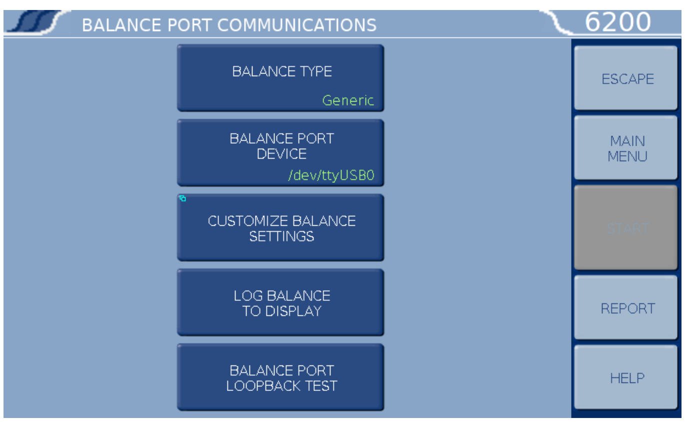


COMMUNICATION CONTROLS: Accesses sub-menus which set the communications protocols for the printer and balances.

PRINTER TYPE: Toggles between a Parr 1759 and a generic printer. When set for the 1759 Printer, all of the features of this printer, such as bold printing, will be activated.

BALANCE PORT: Accesses sub-menu, Balance Port Communications.

BALANCE PORT COMMUNICATIONS



BALANCE TYPE: This key 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. Balances which emulate the serial port over the USB connection may register as a /dev/ttyACM0 instead of /dev/ttyUSB0.

COMMUNICATION CONTROLS (CONTINUED)

CUSTOMIZE BALANCE SETTINGS: Accesses sub-menu that sets the communication parameters for the balance port. Standard options for data bits, parity, stop bits, handshaking, baud rate and balance type are provided to match the settings of an attached balance for proper communication.

- » 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, 2000, 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 the balance template to be customized for unique balances or needs.

COMMUNICATION CONTROLS (CONTINUED)

BALANCE PORT COMMUNICATIONS (CONTINUED)

LOG BALANCE TO DISPLAY: This button will direct 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: This key initiates a loopback test on the port. A special loopback plug is required in order to perform this test.

NETWORK INTERFACE: Accesses the Network Communications submenu for entering details needed to communicate over a network. Options include using a DHCP server or static IP address.

PRINTER DESTINATION: Accesses a submenu for choosing whether to print to an attached printer or to a network printer. If a network printer is to be used the IP address of the printer will also be entered here.

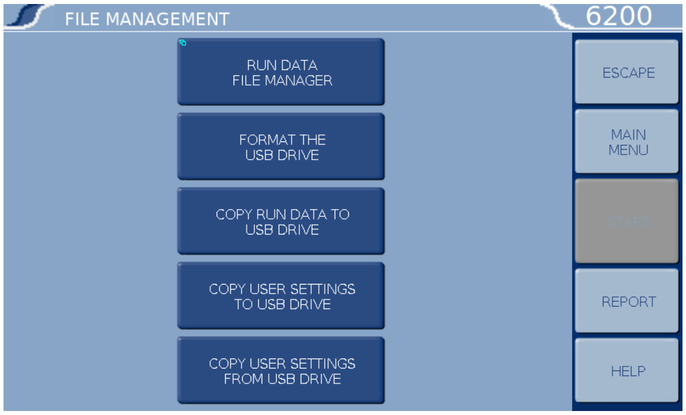
BAR CODE PORT: Accesses a submenu to set up a Bar Code Scanner for use with the calorimeter. Note: Feature key required to utilize this function.

NETWORK DATA DEVICES: Accesses a submenu to input the IP addresses of one or more networked devices such as balances and proximate analyzers. Devices are polled from device 1 to 15 for sample and/or spike weights when the weight entry mode is set to "Network".

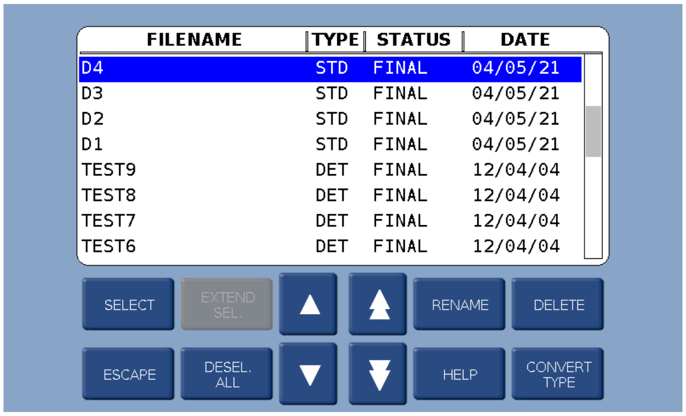
NETWORK DATA DEVICES: This key allows the user to specify the IP addresses of one or more Balance Interface devices on the network. Balance Interface devices are polled from device 1 to 15 for sample and/or spike weights when the weight entry mode is set to Network.

Further information on establishing communications for the Printer, Balance, Network Interface, Bar Code and other Network Data Devices can be found in *Chapter 13 - 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.



Note: See the ***Report Generating*** section in Chapter 10.

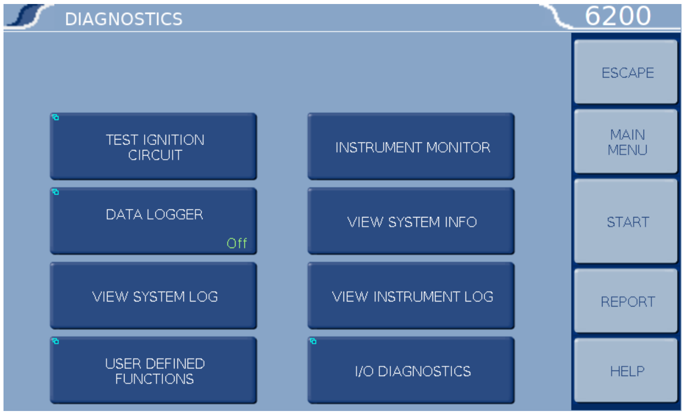
FORMAT THE USB DRIVE: This key provides access to a function that will format the user installed USB flash drive in a manner that is compatible with the CPU Boot loader. Formatting the flash drive this way is recommended prior to installing any program update files on the USB drive.

COPY RUN DATA TO USB DRIVE: This key copies all test data to a USB drive 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.

COPY USER SETTINGS TO USB DRIVE: This key copies all previously saved user setups to the drive.

COPY USER SETTINGS FROM USB DRIVE: This key copies all user setups previously saved to the drive back to the calorimeter controller memory. This feature can be used to configure multiple calorimeters in an identical manner.

DIAGNOSTICS

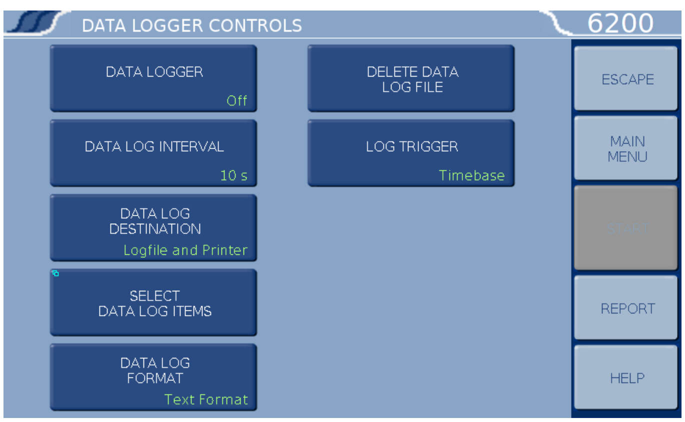


Allows the user to test many of the components and subsystems of the calorimeter. These capabilities must be used in conjunction with the Maintenance Instructions to obtain the maximum benefits from these capabilities.

TEST IGNITION CIRCUIT: The key activates the ignition circuit. A volt meter can be placed across the ignition leads to ensure that the actual firing charge is reaching these contacts.

DATA LOGGER: This key displays and leads to sub-menus which control the data logging function of the calorimeter.

DATA LOGGER CONTROLS



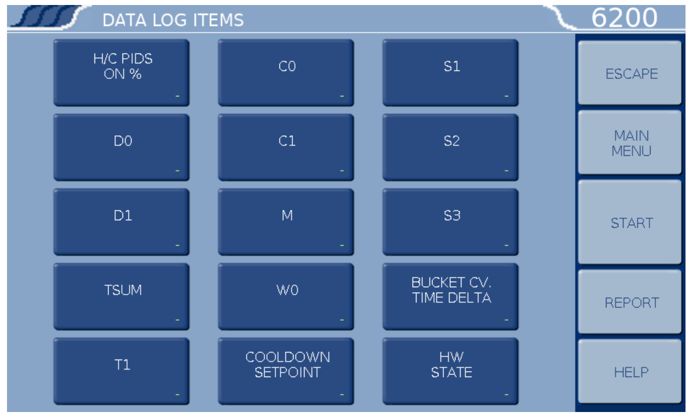
DATA LOGGER: This key toggles the data logging function ON/OFF.

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.

DIAGNOSTICS (CONTINUED)

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.

DATA LOG ITEMS



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

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

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

DELETE DATA LOG FILE: When this key is pressed the contents of the data log file are deleted.

DIAGNOSTICS (CONTINUED)

DATA LOG ITEMS (CONTINUED)

LOG TRIGGER: Toggles between Timebase and Bucket Conversion.

- » TIMEBASE. The data will be recorded in the log file at regular intervals as defined by Data Log Interval. Note that the bucket temperature is updated approximately every 10 s so setting the Data Log Interval to less than 10 s is not recommended.
- » BUCKET CONVERSION. The data will be recorded in the log file when the bucket temperature is updated.

VIEW SYSTEM LOG: This key is used to display the contents of /flash/log/messages. This file is used primarily to log application program debug messages.

USER DEFINED FUNCTIONS: This key leads to a sub-menu that offers five special purpose user/factory definable function keys.

INSTRUMENT MONITOR: This screen provides a summary of important instrument parameters. The monitor is used to detail the course of a test or to observe the heating/cooling performance of the calorimeter.

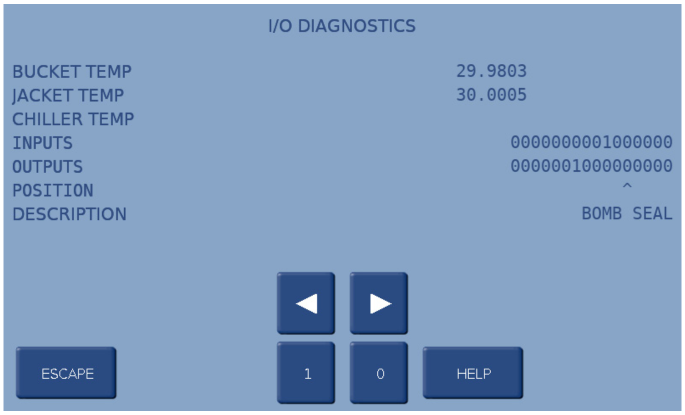
VIEW SYSTEM INFO: This key accesses current program information and settings such as: Processes and their associated PIDs (proportional (P), the integral (I), and the derivative (D) controls), memory, mass storage, network.

VIEW INSTRUMENT LOG: This screen displays the contents of tmp/instlog. This file, among other things, is the logfile destination for the data logger.

I/O DIAGNOSTICS: This key accesses a sub-menu which allows the user to manipulate digital outputs for troubleshooting.

DIAGNOSTICS (CONTINUED)

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.

Note: Not all functions are implemented on the 6200 Isoperibol Calorimeter.

CHAPTER 6
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 g nor more than 1.1 g. 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 cal/°C.
- H = Heat of combustion of the standard benzoic acid sample in cal/g.
- m = Mass of the sample.
- T = Temperature rise in °C.
- e_1 = Correction for heat of formation of nitric acid in calories.
- e_2 = Correction for sulfur which is usually 0.
- e_3 = Correction for heating wire and combustion of cotton thread.

Standard Materials

A bottle of 100 (1 g) 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 and is certified to ISO 17034. Additional benzoic acid pellets can be obtained from Parr. Alternatively, 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 vessel 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 6200 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 6200 Calorimeter automatically determines and uses (by default) 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 AND CONTROLS menu under the EE Value for each bomb.

The user can choose 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 menu using **PROTECT EE VALUE.**

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 11). A list of all tests associated with any Cal ID can be printed from the CALIBRATION DATA AND CONTROL menu 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 AND CONTROLS menu - **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.

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

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

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 % RSD.
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

Chapter 7

Standardization (Precision Method)

Achieving a Higher Precision Class, < 0.1 %

The Parr 6200 calorimeter is a versatile instrument that can be adjusted for many purposes and uses. It also can be operated with significantly better precision than the published specifications using our defaults. It has a proven history not only for precision applications like ASTM D4809, but also for research applications with a precision class down to 0.01 %. In order to achieve this certain factors should be better controlled, as discussed below. Please keep in mind that this is a general suggestion, and not a mandatory requirement, and in practice the selection of factors and how rigorous to control them depends on the specific need. A user should refer to the relevant standard for more specific guidance for their application.

Test Room

For better precision and long term stability the room temperature should be held constant or controlled within a reasonably narrow temperature band, not only during the day but between the seasons of the year as well. Rapid room temperature oscillation should not be present. The calorimeter shall not be exposed to direct sun rays, direct air flow from any vents, or located near any equipment that can change the surrounding temperature near the calorimeter.

Starting Temperature

The starting temperature of the bucket water should be controlled and be in a close range in all experiments. The best way to achieve that is to use the Parr 6510 Water Handling System. Alternatively a big jar of water thermally equilibrated with the room temperature can serve as a water source.

Bucket Water

Bucket water mass constancy is one of the major factors. For a general 0.1 % precision class a mass of 2 kg ± 0.5 g is sufficient. However it should be more tightly controlled for better precision. For example a precision method ASTM D4809 mandates weighing 2 kg ± 0.05 g of water.

Another thing that is often overlooked, is the time between the end of weighing the water and placing the loaded bucket in the calorimeter. This time should be minimized to reduce water evaporation and should also be reasonably constant in all experiments. This will result in minimizing the variance from the evaporation.

Energy Range of the Experiments

A calorimeter is normally standardized for a very specific energy. When a 1 g benzoic acid tablet is used this is 26.5 kJ, which yields approx. 2.5 °C temperature rise in the Parr 6200 calorimeter with 1108 style vessel. The energy range where this standardization is valid is normally defined as ± 25 % to ± 30 % depending on the standard. Thus the sample mass should be adjusted for the experiment temperature rise to be consistent and within those limits. For better precision a smaller interval can be considered, e.g. precision method ASTM D4809 requires to control the temperature rise within ± 0.3 °C for all determinations.

Sample Cup

A platinum sample cup with low thermal mass, although not necessarily required, is beneficial for high precision measurement. Its ability to better heat through leads to better sample combustion which can be critical for samples with combustibility issues.

Sample Mass

Generally weighing the sample to ± 0.1 mg is sufficient for the majority of applications, however precision method ASTM D4809 mandates the sample masses to be recorded to ± 0.01 mg. Semi-micro samples can also benefit from the use of more sensitive balances.

Fuse Mass

It may be necessary to weigh the fuse and enter fuse correction instead of using the fixed correction for high-precision applications. The variance in the cotton thread length, or in burned metal fuse wire is small, but it becomes detectable when high-precision measurements are performed.

Calorimeter Control Limit Values in J/g

When Benzoic Acid is Used as a Test Sample (Precision Method)

Accepted heat of combustion taken as 26454 J/g.
Instrument precision 0.05 %.
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			39.7
2	48.8	0.131 %	28.1
3	57.7	0.114 %	22.9
4	62.2	0.105 %	19.9
5	65.1	0.098 %	17.8
6	67.2	0.094 %	16.2
7	68.8	0.091 %	15.0
8	70.2	0.088 %	14.1
9	71.4	0.086 %	13.3
10	72.4	0.084 %	12.6
11	73.2	0.082 %	12.0
12	74.0	0.081 %	11.5
13	74.7	0.080 %	11.0
14	75.4	0.078 %	10.6
15	75.9	0.077 %	10.3
16	76.5	0.077 %	9.9
17	77.0	0.076 %	9.6
18	77.5	0.075 %	9.4
19	77.9	0.074 %	9.1
20	78.4	0.074 %	8.9
21	78.7	0.073 %	8.7
22	79.1	0.073 %	8.5
23	79.5	0.072 %	8.3
24	79.8	0.072 %	8.1
25	80.1	0.071 %	8.0

CHAPTER 8

Calculations

Calculating the Heat of Combustion

The 6200 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 combustion vessel 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 cal/g and degrees Celsius and then converted to other units if required.

Temperature Rise

The 6200 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 ***Chapter 6 - 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 combustion vessel, bucket or amount of water) is changed.

Thermochemical Corrections

Nitric Acid Correction.

In the high pressure oxygen environment within the oxygen combustion vessel, nitrogen that was present as part of the air trapped in the vessel 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 vessel, 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 applied by the calorimeter is calculated as:

e_3 = (fuse value) (fuse multiplier from calculation factors 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 cal is applied to all tests. Total errors of more than 5 cal will seldom occur when using a fixed fuse correction and the fuse wire supplied by Parr.

When using the 1108P Oxygen Combustion Vessel, there are two components to the fuse correction:

- 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 be calculated. In the 6200 Calorimeter this is a fixed correction of 10 cal per test.

Cotton has a heat of combustion of 4 kcal/g. 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 g which would release 12 cal as it burns. Heavier threads weigh up to 0.010 g per 10 cm and increase this correction to 40 cal 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 combustion vessel 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 cal from electrical heating plus 12 cal from the burning thread for a total of 22 cal per test. The thread supplied by Parr has a mass of approximately 1 mg/cm. This results in a total fuse correction of 50 cal.

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 vessel washings to determine the total acid present. This is assumed to be all nitric acid with a heat of combustion of -14.1 kcal/mol. 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/mol or -36.1 cal/mEq [where mEq is 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 6200 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 HNO_3. A correction of 10 calories is a good number for the fixed nitric acid value. 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 6200 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 6200 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.

Acid and Sulfur Corrections

- **Total acid** is the amount of base required to titrate the vessel washings (mL).
- **Nitric acid** is that portion of the total acid in the vessel washings that result when the nitrogen in the air that is trapped in the combustion vessel 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 cal/mEq.
- **Heat of formation of sulfuric acid** (from SO_2) is 36.1 cal/mEq.
- **Sample mass** is the mass of sample burned in the combustion vessel (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 vessel washings.

Sulfur Correction:

e_2 = (percent sulfur)(sample mass)(sulfur multiplier)(heat of formation of H_2SO_4).

Acid Correction:

In the 6200 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 = (nitric acid value)(acid multiplier)(heat of formation of nitric acid)

For an 1108P style oxygen combustion vessel, the default nitric acid value is 10 and acid multiplier is 0.0709. The heat of formation of nitric acid is 14.1 cal/mEq so the calculation is:

$e_1 = (10)(.0709)(14.1)$ or $e_1 = 9.9969$ (rounds to 10)

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.

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

The calculation is:

e_1 = [((total acid)(acid multiplier)) – (% sulfur)(sample mass)(sulfur multiplier)](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/mEq a 1 g sample with 25 mL of washings and 2 % sulfur would result in the following calculation:

$e_1 = [((25)(.0709)) – (2)(1)(.6238)]\ 14.1$
$e_1 = [(1.7725) – (1.2476)]\ 14.1$
$e_1 = [.5249]\ 14.1$
$e_1 = 7.40$

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 vessel 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 HNO_3: 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 vessel, the oxygen filling pressure, and the quantity of energy released.

For the calculated nitric acid method:

e_1 = (nitric acid factor/1000)(Energy Equivalent)(corrected temperature rise)

Example: For a test run with energy equivalent of 2425.07 and a corrected temperature rise of 2.6348 would result:

$e_1 = (1.58/1000)(2425.07)(2.6348)$
$e_1 = 10.10$ cal

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.

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 HNO_3. A correction of 10 cal 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.

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 oxygen combustion vessel conditions, which is -72.2 kcal/mol or -36.1 cal/mEq. But remember, a correction of 14.1 cal/mEq 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 cal/mEq (44.0 kcal/mol). For convenience, this is expressed, in the ASTM e_2 formula, as 13.7 cal (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 6200 Controller for dealing with these different procedures.

The analysis of vessel washings in these methods call for a titration, first using 0.1N barium hydroxide (V_2) followed by filtering, and a second titration using 0.1N HCL(V_1) 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, V_1 and V_2, to be entered into the controller directly for the calculation of the total acid correction. V_1 should be entered at the prompt for acid and V_2 is entered at the prompt for sulfur.

The settings in the ***Settings for ISO & BSI Methods*** table below 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:

> V_1(Acid Multiplier)(Heat of Formation of Nitric Acid) + V_2(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 cal.

Settings for ISO & BSI Methods

Page	Line	Setting	Value
Thermochemical Corrections	Acid Correction (STD)	Entered HNO3	13
	Fixed Sulfur STD	Off	7
	Acid Correction (DET)	Entered HNO3	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

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 6200 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} = The spiking material (cal/g)

M_s = Mass of spiking material

This factor is added to the calculations through the OPERATING CONTROLS menu by selecting the **SPIKING CORRECTION** key to access **SPIKE CONTROLS**, when **USE SPIKING** is set to **ON**. **HEAT OF COMBUSTION OF SPIKE** is entered as calories per gram (cal/g). 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**.

CAUTION! When using a spike it is very important to be aware of the total energy being released by the sample and the spike. Do NOT exceed 8000 calories total energy released.

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 oxygen combustion vessel. 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. Parr calorimeter performs the automatic calculations of net heat of combustion in accordance with ASTM Method D5865. Please refer to it for further details. Alternative calculation schemes exist (e.g. for liquid fuels), refer to the correspondent standards.

NOTE: the calorimeter expects H N O S values to be entered on the as-received moisture free basis. For conversion from/to other basis please refer to ASTM Method D3180.

Chapter 9

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

Chapter 3 - Program Installation and Control, provides the default settings used to setup the method preferred by the user.

Refer to the ***Chapter 10 - Reporting Instructions***, 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 ***Chapter 8 - Calculations***.

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** menu, then the user must manually enter the values at the prompt.

If values for these corrections are not available, the operator can use the **SKIP** key to bypass any of the corrections. The report will remain at preliminary status 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 ***Chapter 8 - Calculations***, Thermochemical Calculations section.

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

Chapter 10
Reporting Instructions

Report Options

The 6200 Calorimeter can transmit its stored test data in either of two ways. The **REPORT DESTINATION** key in the **REPORTING CONTROLS** menu 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.

Reports can also be downloaded to a PC via the Ethernet port or copied using an USB stick.

Report Generation

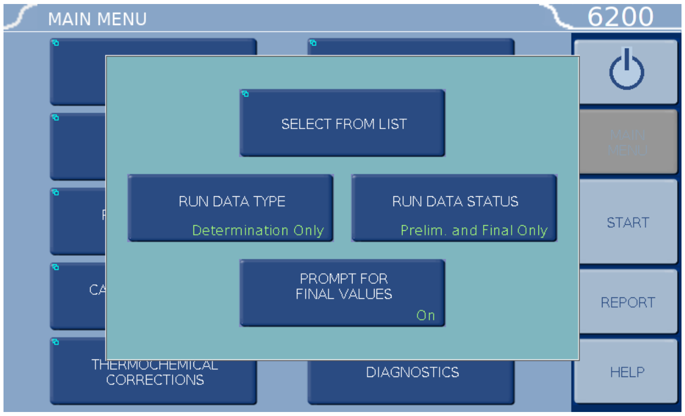
There are two kinds of calorimeter reports: Preliminary and Final.

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.



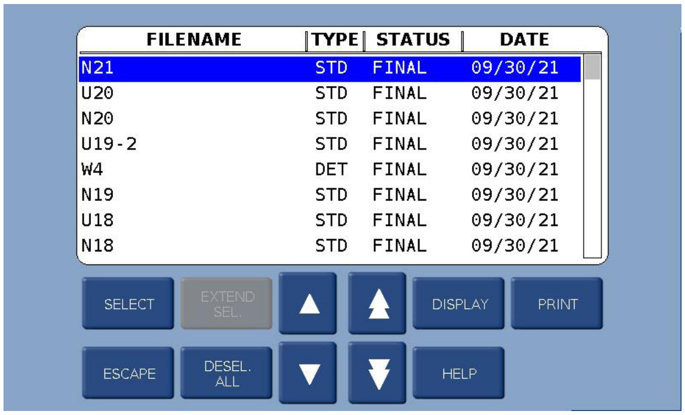
Select From List: This key displays the stored results specified with the following two keys:

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 sample 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.

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.

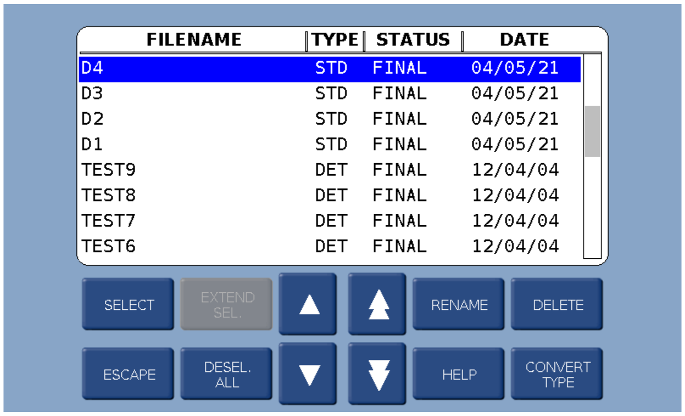
Net Heat of Combustion

To have the Net Heat of Combustion print as part of preliminary and final reports, go to the **NET HEAT/DRY FACTORS** in the **THERMOCHEMICAL CORRECTIONS** menu and turn on **CALCULATE NET HEAT OF COMBUSTION**. During the reporting process, the controller will prompt for the hydrogen (H) value.

Chapter 11

File Management

The 6200 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.



Clearing Memory

The **FILE MANAGEMENT** key on the main menu leads to the FILE MANAGEMENT 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.
- Once a file is deleted there is no way to recover the data.

USB Flash Drives

The controller of the 6200 Calorimeter can accept USB flash drives. These drives 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.
- Restore or update the controller's operating system.

USB flash drives are inserted into USB ports on the back of the control section of the calorimeter. Keys are provided on the FILE MANAGEMENT menu to initiate each of the above actions with the exception of restoring or updating the controller's operating system.

NOTE: If the calorimeter cannot read the USB drive, use F**ORMAT THE USB** function.

Chapter 12

Maintenance & Troubleshooting

Oxygen Combustion Vessel

Under normal usage the 1108P Parr Oxygen Combustion Vessel will give long service if handled with reasonable care. However, the user must remember these vessels are continually subjected to high temperatures and pressures that apply heavy stresses to the sealing mechanism. The mechanical condition of the vessel must therefore be watched carefully and any signs of weakness or deterioration should be replaced before they fail. It is recommended the 1108P Oxygen Combustion Vessel have O-rings and valve seats replaced after 6 months, 500 firings or at more frequent intervals if the vessel has been subject to heavy usage or if it shows any evidence of damage. Detailed information can be found in Manual 205M supplied as a part of this manual. This 1108P Oxygen Combustion Vessel is the only part of the calorimeter system that requires routine maintenance. All other problems will require diagnosis and parts replacement.

Jacket Temperature Troubleshooting

The jacket temperature is monitored with the use of a thermistor installed in the temperature control assembly. This assembly is heated by a heater cartridge, A1459DD. In the **DIAGNOSTICS** menu, select **INSTRUMENT MONITOR**. If the heater PID is ON and reading 100 %, yet the jacket is at ambient temperature, check the following possible causes.

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

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

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

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

If there is no voltage present, and the reset button on the thermostat is not tripped, refer to the error code “There Is A Problem With The Jacket Thermistor” for further troubleshooting. There may also be a problem with the calorimeter controller, A1279DD3, and Parr Customer Service should be contacted.

Fuses

The replacement of protective fuses for the 6200 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. Please contact Parr Customer Service for instructions on accessing the I/O board.

Note: Check the labels on the instrument for correct fuse rating.

Part No.	Description	Type	Ratings
139E23	Lines Protective Fuses	Fast-Acting	15 Amps, 250Vac
1641E6	Heater Fuse (F2)	Slo-Blo	2.5 Amps, 250Vac
1641E7	Pump Fuse (F1)	Slo-Blo	1 Amp, 250VAC

6200 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 Button Dim:
The **START** button will be dim (not lit) when the calorimeter is not ready to begin a test. 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 ± 0.5 °C it will be another 15 min before the **START** button lights 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 0.05 °C after the first minute of the post-period.

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. When the heater and pump are initially turned on the heater will be at 100 % power until the jacket temperature approaches 29.5 °C. Once it gets close the power being applied to the heater will be cut back to avoid overshooting the target of 30 °C.

The calorimeter will turn off the heater and pump if the heater is at 100 % power for more than 10 minutes. If the jacket water is approximately 21 °C or less when the heater and pump is first turned on it is not unusual to get this error. In this case clear the error and restart the heater and pump. If the error occurs again then there could be a problem.

- Check the 2040E Thermostat reset
- Check the water level in the calorimeter

A Preperiod Timeout Has Occurred.
The calorimeter has failed to establish an acceptable initial temperature, prior to firing the combustion vessel, within the time allowed. Possible causes for this error are listed below:

- A vessel leak
- Poor bucket stirring
- Metal to metal contact between the bucket and the jacket
- Lid not tight
- Foam seal has deteriorated
- Bucket temperature outside the acceptable range (3 °C to 5 °C below the jacket setpoint)
- Jacket requires water

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 vessel leak
- Poor bucket stirring

There Is A Problem With The Bucket Thermistor.
Possible electrical open.

- Check connection to board
- Replace probe
- Room temperature is below 10 °C (50 °F)

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 1 kΩ to 5 kΩ.

- Check connection to board
- Room temperature is below 10 °C (50 °F)

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 combustion vessel has been fired. When this count exceeds a preset limit (usually 500) this message will be issued each time the vessel is used for a test. Perform vessel maintenance and reset the vessel fire count on the **CALIBRATION DATA AND CONTROL** page for the 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. See ***Chapter 11 - File Management***.

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 g.

Chapter 13

Communications Interfaces

Printer Port

The 6200 Calorimeter is equipped with a USB port for connection to either a 40 or 80 column printer and/or a balance.

The default parameters for the 6200 Calorimeter are set up for use with the Parr 1759 Printer.

Balance & Port Input Driver Specifications

The 6200 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

The ID field must contain "S_" to indicate a stable mass. The data field contains the current mass, right justified, with a decimal point. The balance should be configured to send continuously.

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

Sartorius Balance Interface

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

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

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

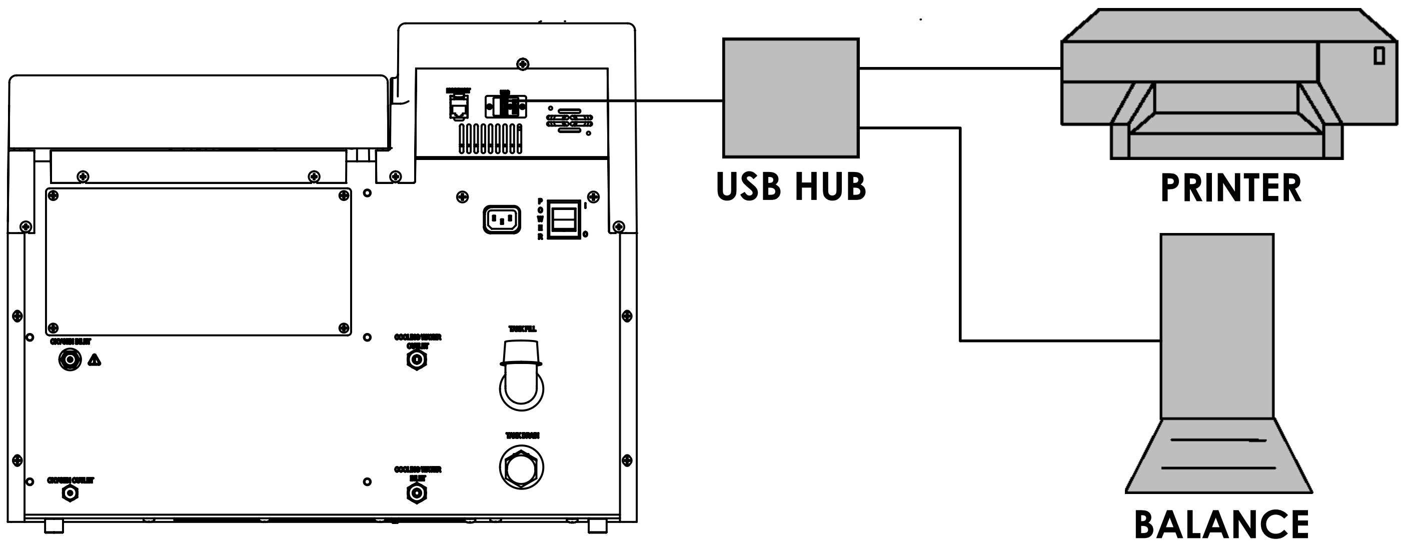
The calorimeter will send this command string once every few seconds after the **ENTER** key has been pressed during a mass entry sequence. The **ENTER** key should only be pressed when the mass reading is stable. However, unstable readings will be rejected and a warning will be issued. Acknowledging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

Generic Interface

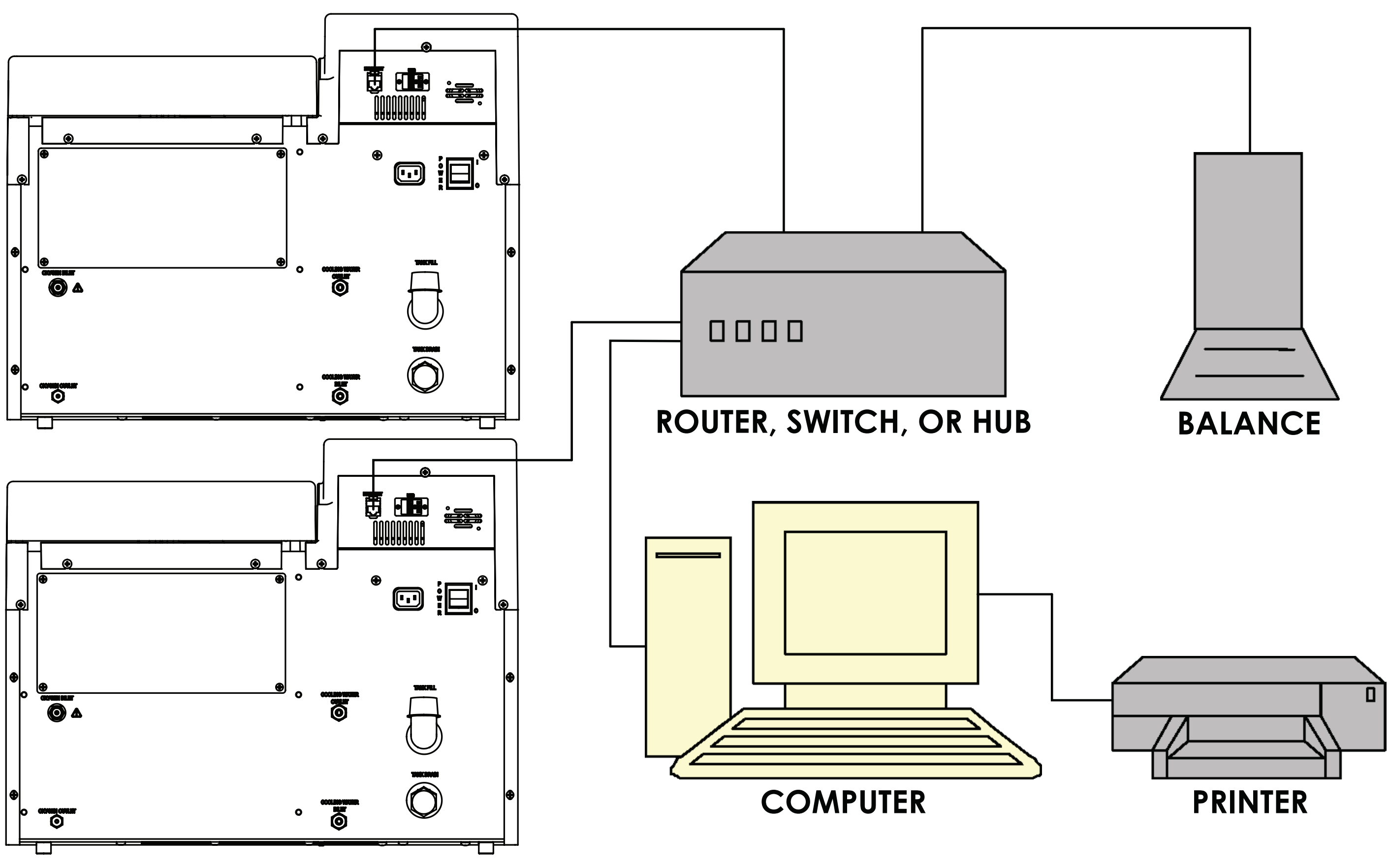
The data field should consist of 9 numeric characters (0 through 9, +, - and space) terminated with a carriage return (CR). Leading zeros may be blanked as spaces and are counted. Non-numeric characters are ignored and will reset the input buffer if the data field has not been filled. Any characters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

Field	Length
data	8
CR	1

6200 Calorimeter Peripherals

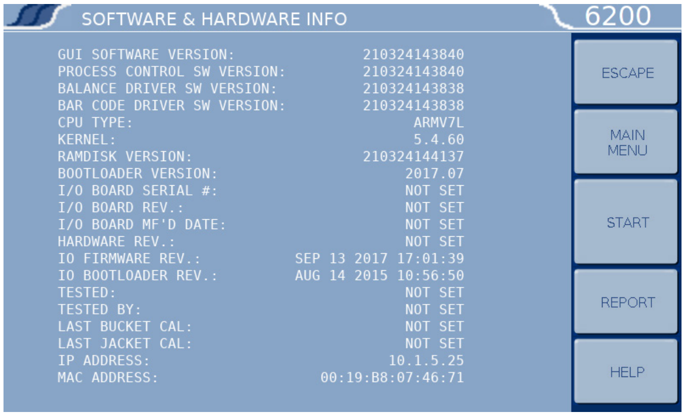


6200 Multiple Alternate Configurations



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 & CONTROL** menu. See the example screenshot.



Users who don't have a network infrastructure can create a simple network by connecting a router with DHCP server capability to the calorimeter using an ordinary CAT 5 network cable. The calorimeter should be connected to LAN side of the router. The PC in turn is also connected to the LAN side of the router using a similar CAT 5 cable. 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.

Alternatively, a PC can be connected directly to the calorimeter via an Ethernet Cable. Both PC and 6200 Calorimeter must be assigned static IP addresses from the same subnet (e.g. 192.168.1.125 and 192.168.1.110 with net mask 255.255.255.0).

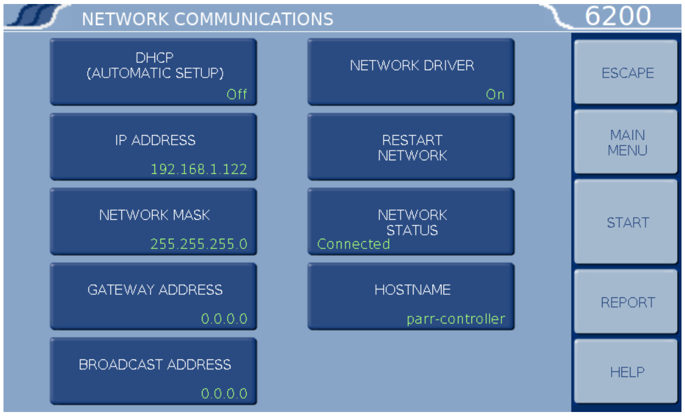
An FTP enabled web browser can be used to access stored test data and datalog file at:

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

Data Log:
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.

Advanced Network Options



Advanced network options can be changed by navigating to the **COMMUNICATION CONTROLS** menu and selecting **NETWORK INTERFACE** key to access the **NETWORK COMMUNICATION** submenu.

A ZeroConf implementation is present in the firmware of Parr Calorimeters, thus the user does not need to know the calorimeter's dynamic IP address. ZeroConf implementation includes avahi-daemon to do a multicast DNS broadcast of services provided by the calorimeter. These services include VNC (with a valid feature key), SSH, FTP and HTTP. In order for this scheme to work in a Windows environment, Bonjour Print Services for Windows (from Apple) must be installed on the client PC. The Bonjour for Windows package provides Bonjour zero-configuration networking services for use by Windows applications. Bonjour enables computers and devices to automatically discover each other using industry standard IP protocols without the need to manually enter IP addresses or configure DNS servers. Avahi broadcasts the services provided using mDNS. This allows the user to use the text address parr-controller.local to connect to the calorimeter. For instance, a SSH session would look like: *ssh parr-controller.local* or in your web browser: *http://parr-controller.local*

The HOSTNAME button on the network configuration screen allows the user to give a meaningful name to the calorimeter, such as parr6200-lab. The default HOSTNAME is parr-controller. The suffix .local must be appended to the hostname when accessing calorimeter services.

Test Data Files

6200 Data File Naming Convention

Test files are stored in the calorimeter with a unique file name. See the following table for details.

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

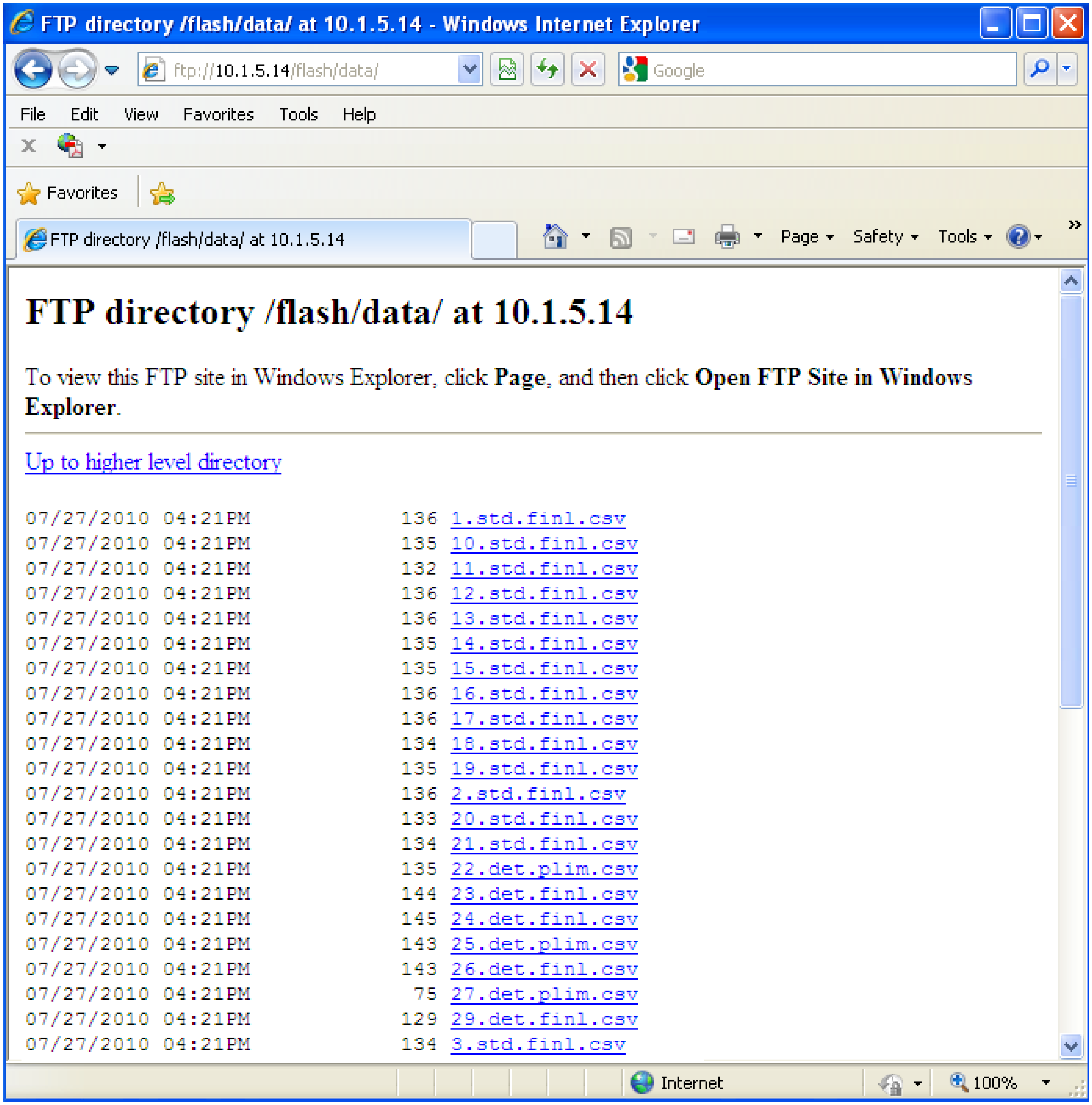
6200 Calorimeter Run Data Template

Each test file contains information separated by commas. See the following table for a list of the data contained within the file. In the data directory on the calorimeter there is also a RunDataTemplate.csv file that contains column headings for the test file data and a RunDataTemplate.txt file that is a duplicate of the table below.

Field	Description
SampleID	char[16]
Timestamp	MM/DD/YY HH:mm:ss
Mode	0 = undetermined (preweigh), 1 = determination, 2 = standardization
Method	0 = undetermined (preweigh), 1 = equilibrium, 2 = 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 mult in effect at time of report, if Units = 4 (other)
BombID	[1,4]
BombEE	bomb energy equivalent

Field	Description
SampleWt	sample weight
SpikeWt	spike weight
Fuse	fuse value
FuseFinal	fuse value is final (0 if not final, 1 if 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)
MADFinal	moisture as determined value is final (dry calc option)
JacketTemp	jacket temperature
InitTemp	initial temperature
DeltaT	temperature rise
HOC	gross heat of combustion
NetHOC	net HOC (if net calc option 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)
OxygenFinal	oxygen value is final (net calc option)
Nitrogen	nitrogen value (net calc option)
NitrogenFinal	nitrogen value is final (net calc option)
MAR	moisture as received value (dry calc option)
MARFinal	moisture as received value final (dry calc option)
DryNetHOC_AR	net HOC as received (if all dry and net values provided)
BombName	user friendly name for bomb

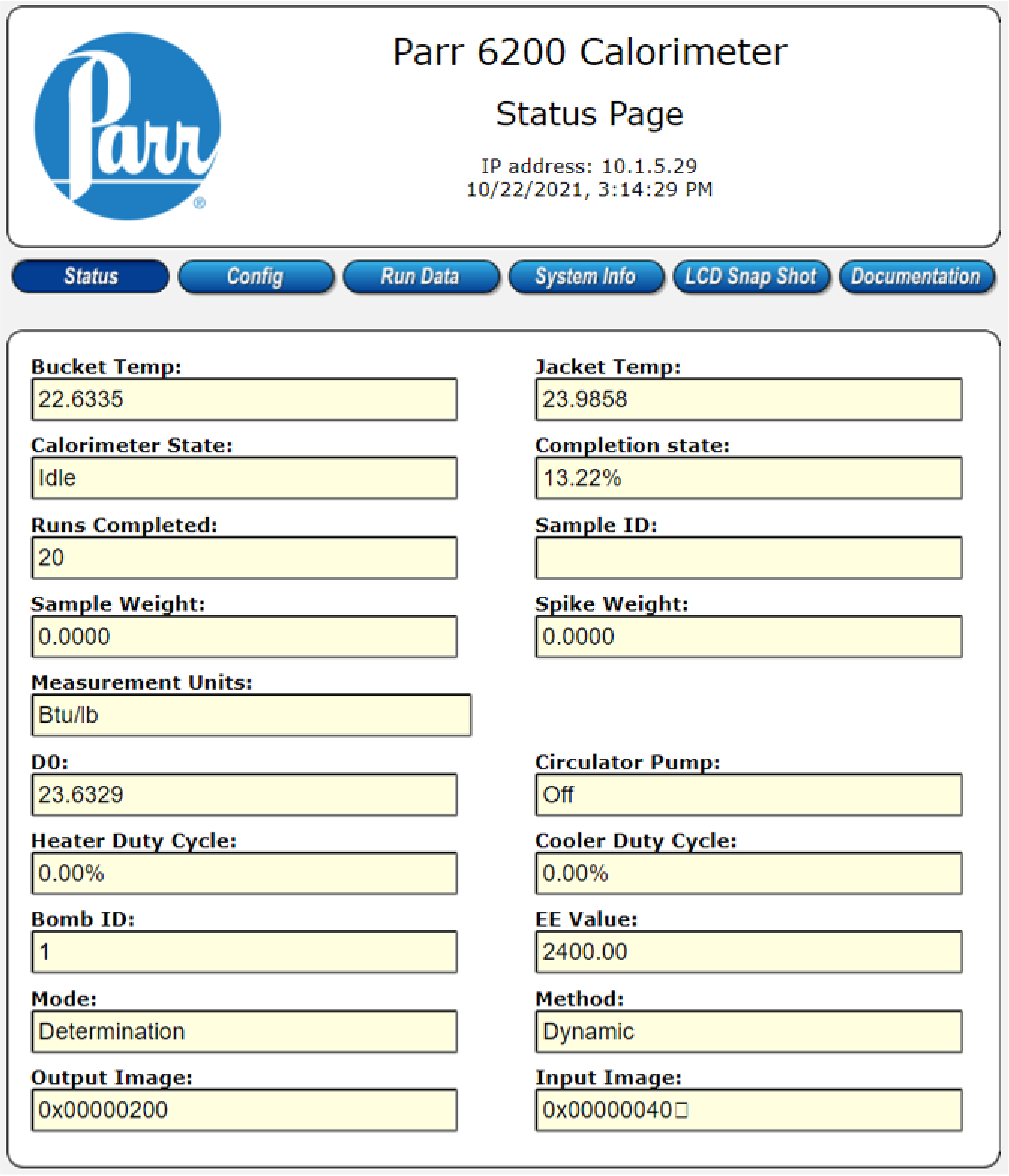
The following screenshot illustrates the contents of the calorimeter data directory as presented by a web browser.



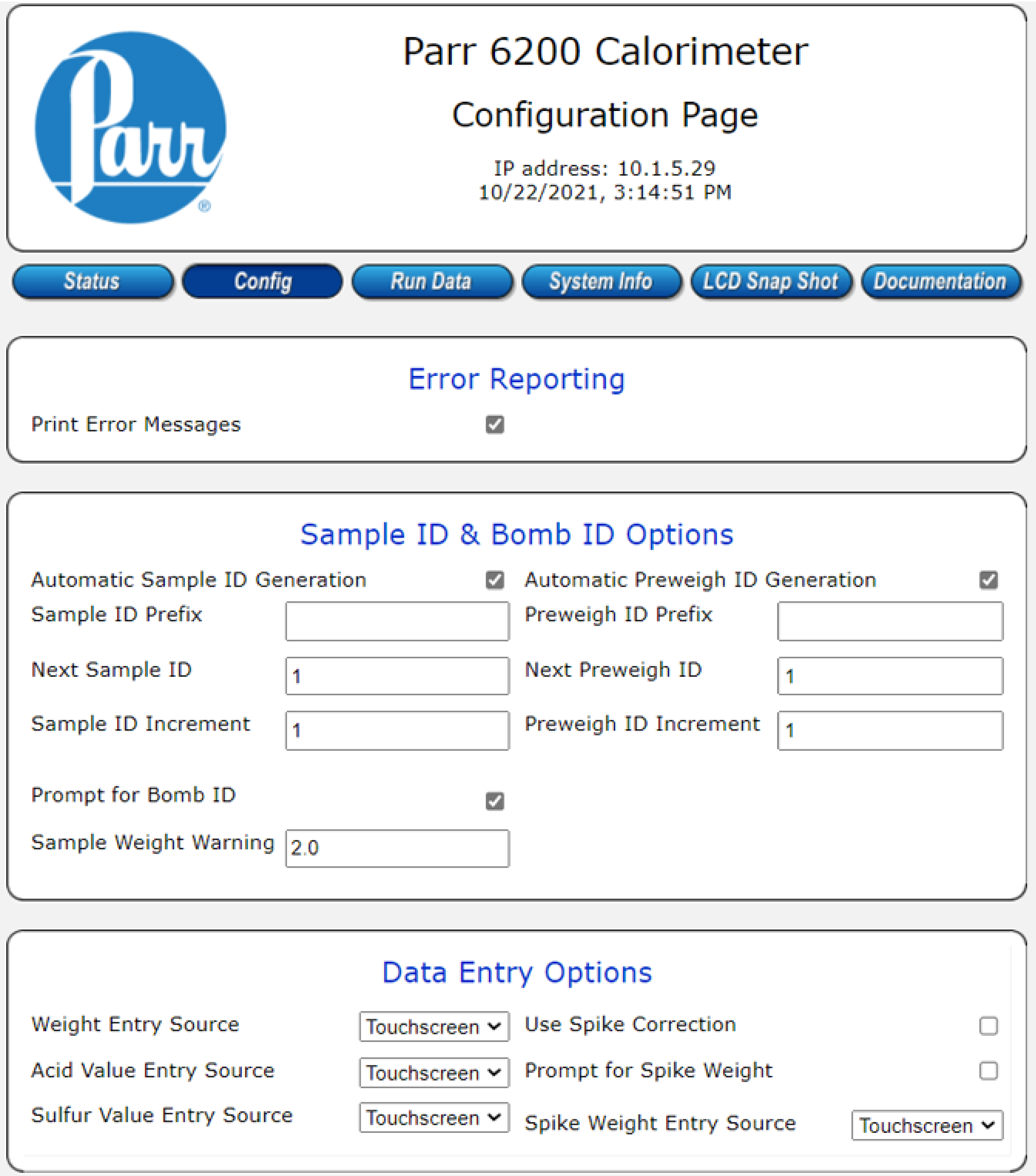
The calorimeter offers a web server service. Test reports can be viewed with a web browser using a URL of the following form.

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.

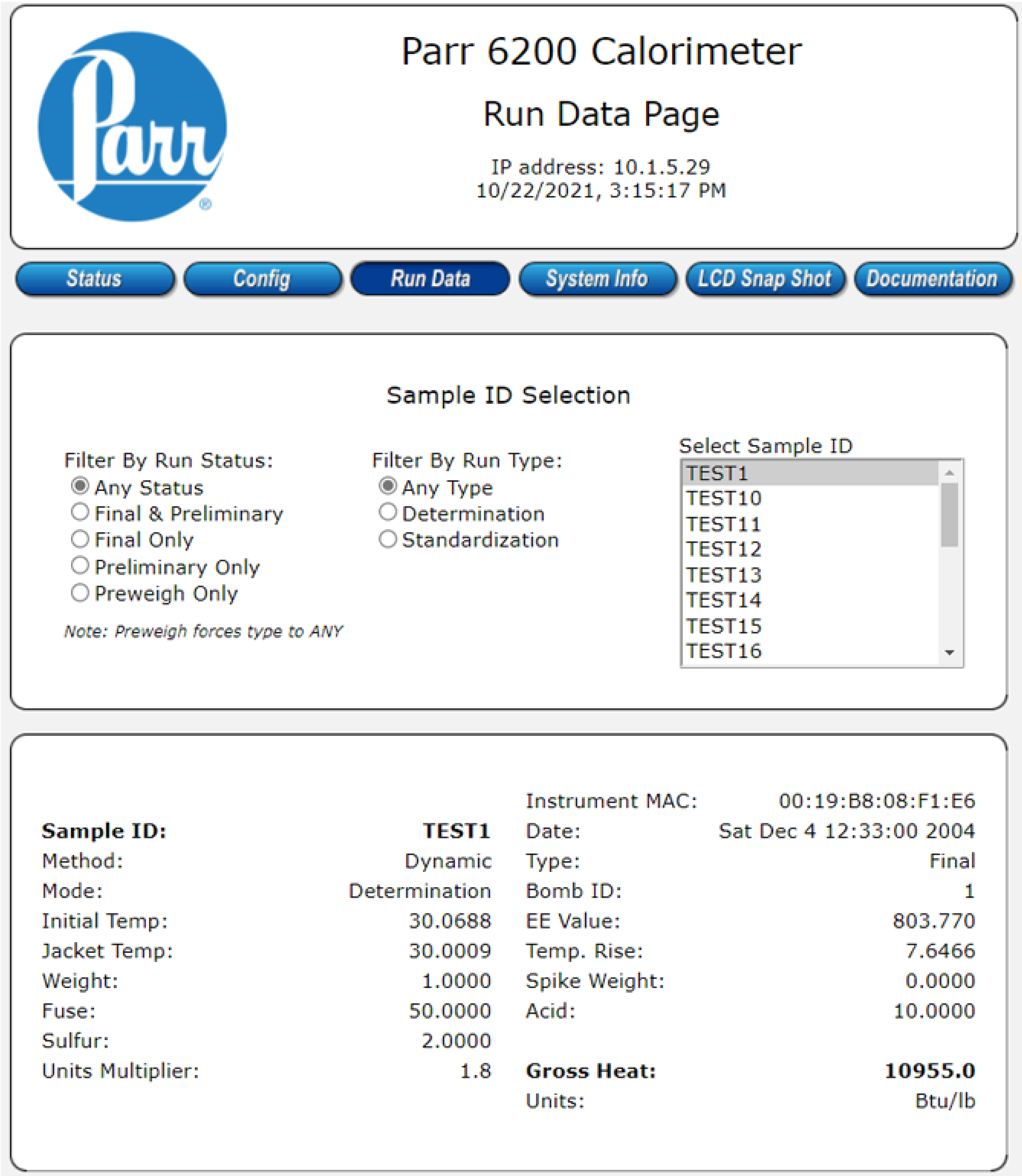


Clicking on the Config button will display the screen below. Changes made on this screen will change the settings in the calorimeter.

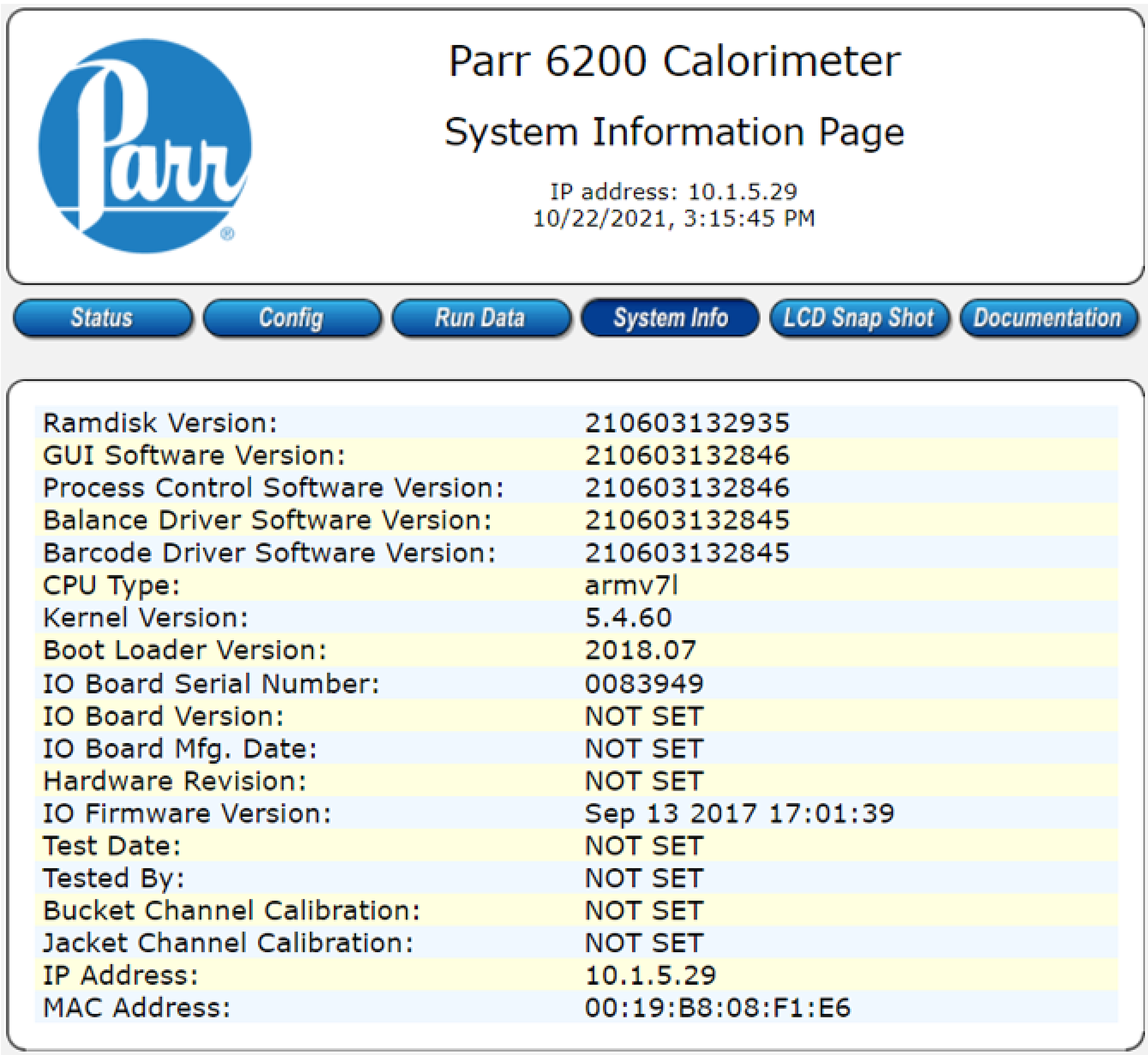


Clicking on the Run Data button displays a list of reports currently in the instrument memory.

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



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



Ramdisk Version:	210603132935
GUI Software Version:	210603132846
Process Control Software Version:	210603132846
Balance Driver Software Version:	210603132845
Barcode Driver Software Version:	210603132845
CPU Type:	armv7l
Kernel Version:	5.4.60
Boot Loader Version:	2018.07
IO Board Serial Number:	0083949
IO Board Version:	NOT SET
IO Board Mfg. Date:	NOT SET
Hardware Revision:	NOT SET
IO Firmware Version:	Sep 13 2017 17:01:39
Test Date:	NOT SET
Tested By:	NOT SET
Bucket Channel Calibration:	NOT SET
Jacket Channel Calibration:	NOT SET
IP Address:	10.1.5.29
MAC Address:	00:19:B8:08:F1:E6

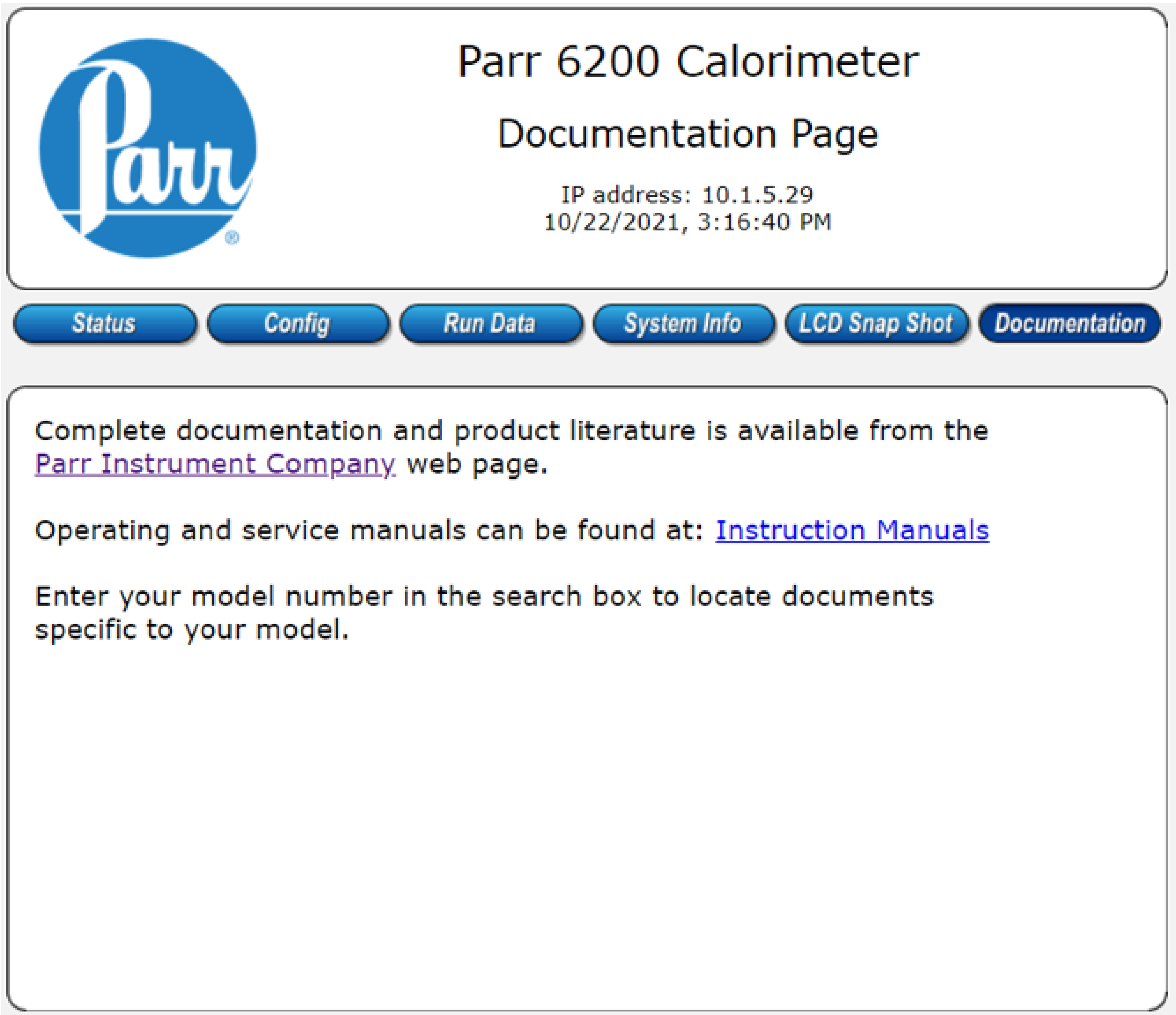
Clicking on the LCD Snap Shot button will display the current menu screen displayed by the calorimeter. If the backlight is not on, this screen will display a blank blue square.

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.



Optional Feature Keys

When purchasing a Remote Feature Key, Samba Server Feature Key, or Bar Code Feature Key, the user must supply Parr with the MAC address of the calorimeter (found in the **SOFTWARE & HARDWARE INFO** screen). This allows Parr to activate the feature key. In order to enable the calorimeter to use the purchased feature, the feature key needs to be entered into the instrument. Select the PROGRAM INFORMATION & CONTROL page 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.

Remote Operation

The 6200 can be operated remotely via PC over a network with the purchase of the 1965E Remote Feature Key.

Bar Code Port

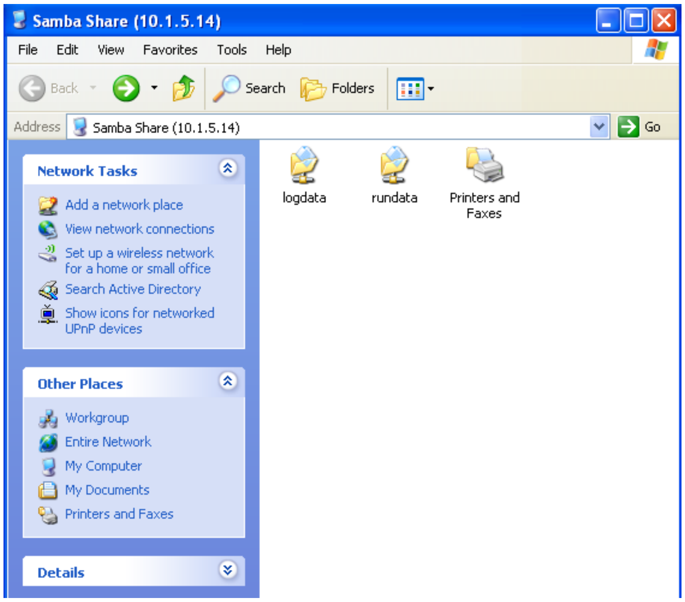
The use of barcodes in the laboratory has become a highly accurate, rapid and inexpensive way to identify samples. The only scanner currently supported is Parr part no. 1952E2.

Samba Server Feature

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 6400 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.

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



CHAPTER 14

Parts Lists & Drawings

Parts Lists

Principal Assemblies in Calorimeter

Item	Description
1108P	Oxygen Combustion Vessel
A391DD	Oval Bucket
A570DD	Regulator Assembly, Oxygen
A1279DD3	Controller Assembly
A1311DDEB	Pump Assembly, Circulating, 115V
A1311DDEE	Pump Assembly, Circulating, 230V
A1268DD	Motor Assembly, Pump, 12V
A1276DD	Cold Water Solenoid
A1284DD2	Stirrer Hub Assembly
A297E	Lead Wire
A1278DD	Oxygen Solenoid, w/o fittings
1940E	Power Supply
897E	Capacitor, 40V, 81000 uF
1317DD	Lid Seal
1417E2	Thermistor Bucket
538VB	Male Connector 1/8 NPTM-T-BT Nylon
549DD	Gas Spring
139E23	Fuse Fast/ Act 15 Amp 250V
1641E	Pump Fuse (F1), Fast-Act, 1 Amp, 250VAC
1641E2	Heater Fuse (F2), Fast-Act, 2.5 Amps, 250VAC

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

A1279DD3 Controller Assembly

Item	Description
2395E3	CPU Board 6200
A2140E	I/O Board
2400E	LCD Touchscreen, 5”
A2402E	Speaker Assembly with Cable
A2403E	Power Cable Assembly
A2166E	Cable, I/O to CPU USB
A2167E	Cable, USB Peripheral
1783E	Modular coupler, feed thru RJ45
2396E	Cable, HDMI Male Extension 12”
2397E	Cable, Micro USB / OTG Male 8”
2401E	Splitter Cable
2406E	CAT6 Cable, RJ45 12”
SA1332RP04	RHMS, 6-32 x 1/4 PH 18-8
SA1140RP06	RHMS, 4-40 x 3/8 PH 18-8
SA1140RP04	RHMS, 4-40 x 1/4 PH 18-8
SN1140HLHJ	Nut, 4-40 Hex Lock Nylon Ins

Temperature Control Assembly

Item	Description
1281DD	Manifold, Temperature Control
1417E	Thermistor, Jacket
538VB	Male Connector, 1/8 NPTM-T-BT Nylon
252HWHJ	Elbow, Hose Barb, 1/2 x 3/8 M
280HWHJ	Elbow, Hose Barb, 1/4T X3/8 M
A1459DDEB	Cartridge, Heater Assembly 120V
A1459DDEE	Cartridge, Heater Assembly 240V
535VB	Male Connector, 1/4T x 3/8 NPTM
A92HWBB	Male Connector, 1/4T x 1/8 NPTM
283VB	Adapter, Male 1/4T x 1/8 NPTM
A1276DD	Cold Water Solenoid Assembly w/ connector
117HW3	Elbow, Male, 1/4T x 1/8 NPTM
2040E	Thermostat, Manual Reset

A1284DD2 Stirrer Hub Assembly

Item	Description
1282DD	Hub, Stirrer
1283DD2	Shaft, Stirrer
1242DD3	Pulley, Timing
682DD	Snap Ring, Internal .50
683DD	Wave Spring, .50 OD
684DD	Ball Bearing
A540DD	Stirrer Assembly
1288DD	Coupler, Stirrer Shaft
SA1140RD04	Set Screw, Pulley

Water Tank Assembly

Item	Description
1301DD	Water Tank, 6200
386VB	Nipple, 1/2 NPT, Nylon
413VB	Cap, 1/2 NPT, Nylon
387VB	Elbow, 1/2” NPT, Plastic
1020DD	Plug Cap
271HWHJ	Hose Barb, Male, 1/2T – 1/2NPTM

Cooling Water Supply

Item	Description
328VB	Union, Bulkhead 1/4 Tube
196VB	Valve, Brass, 1/4 Tube
343VB	Port Connector, 1/4 Tube, Brass
A1276DD	Cold Water Solenoid Assembly w/connectors

Oxygen Filling System

Item	Description
244VB	Union Bulkhead, 1/8 Tube
A476A3	Slip Connector w / 1/8 NPT
438VB	Elbow, 45 °, 1/8 NPT x 1/8 Tube
HX0012TB024	High Pressure Tube, 1/8, Nylon
180VB	Male Elbow 1/8 T x 1/8 NPTM
527VB	Restrictor 0.012 – 1/8 NPT
A1278DD	Oxygen Solenoid Assembly
697HC2	Filter Sintered Bronze
243VB2	Male Connector, 1/8 T x 1/8 NPT
394HCJE	O-Ring EP 3/8 ID X 1/16 CS

6200 Stirrer Motor and Drive

Item	Description
1593DD	Mount, Motor, 6200
1241DD2	Belt Timing, 6200
1242DD	Pulley Timing, 6200
A1268DD	Motor, Stirrer, 6200

Spare and Installation Parts

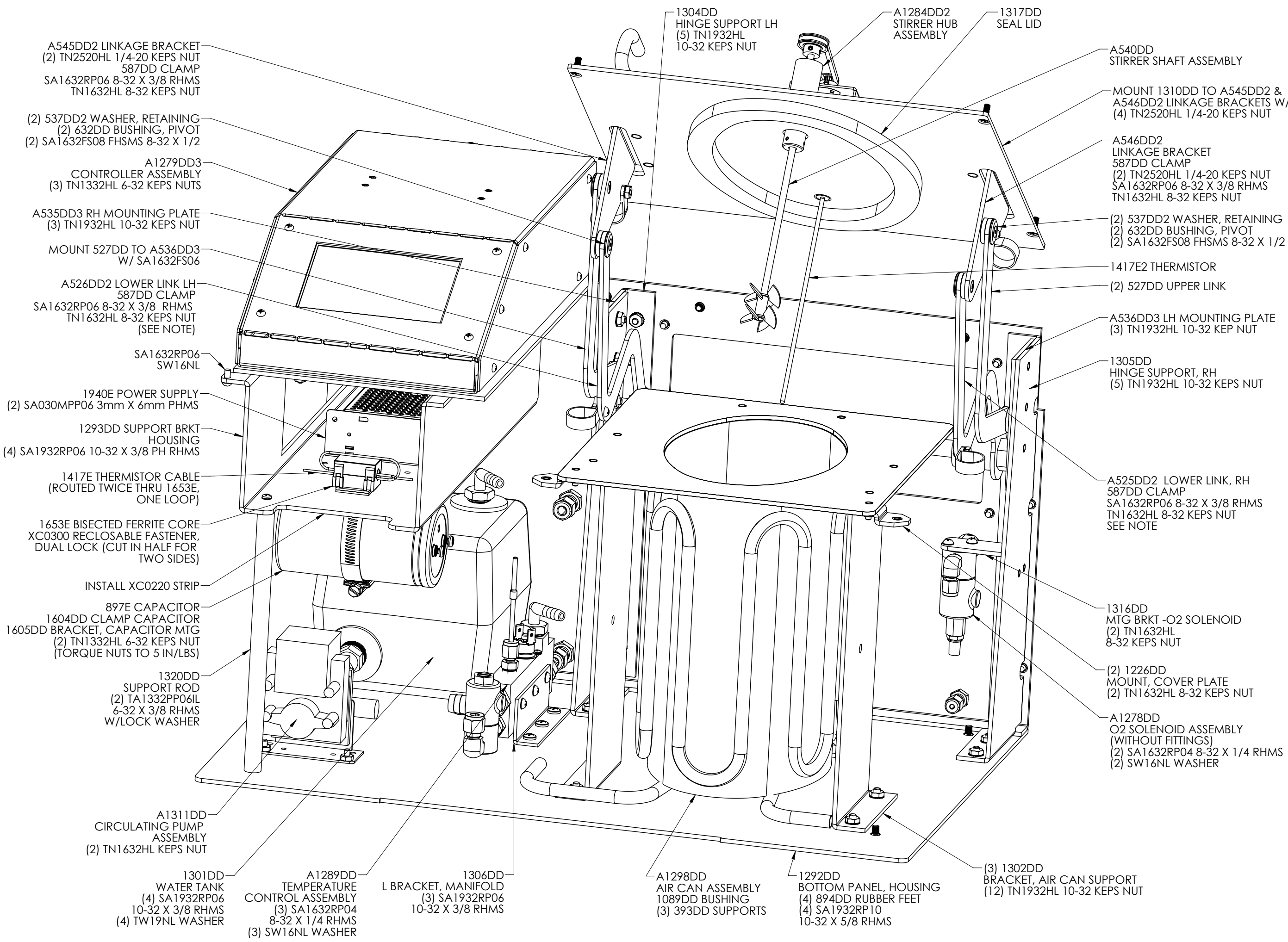
Item	Description
20VB	Valve Seat
230A	O-Ring, Vessel Head, 2-3/8 ID
238A	O-Ring, 3/16 ID
394HCJE	O-Ring, 3/8 ID
415A	O-Ring, 7/16 ID
3415	Benzoic Acid Pellets, 1 g, 100 pcs
421A	Vessel Lifter
43AS	Sample Capsule, SS
475A	Service Clamp Head
A719E	Cordset 115V
A719EEE	Cordset 230V
HJ0025TB035	Tube, Nylon 1/4 OD
A570DD	Oxygen Regulator
143AC	Insulator, Delrin
388A	Spacer
401A	Sleeve Insulator
96AC	Electrode Insulator
378A	Packing Cap
PA1332PP04	6-32 x 1/4 PHMS T316
A38A	Head Support and Stand
840DD2	Heat Wire
845DD2	Ignition Thread
A391DD	Oval Bucket
1889E	Display Protection Film
1610DD	LCD Stylus

NOTE: Parts may change depending on which oxygen combustion vessel was ordered with the calorimeter.

Drawings

Figure 14-1

6200 Isoperibol Calorimeter Cutaway Front

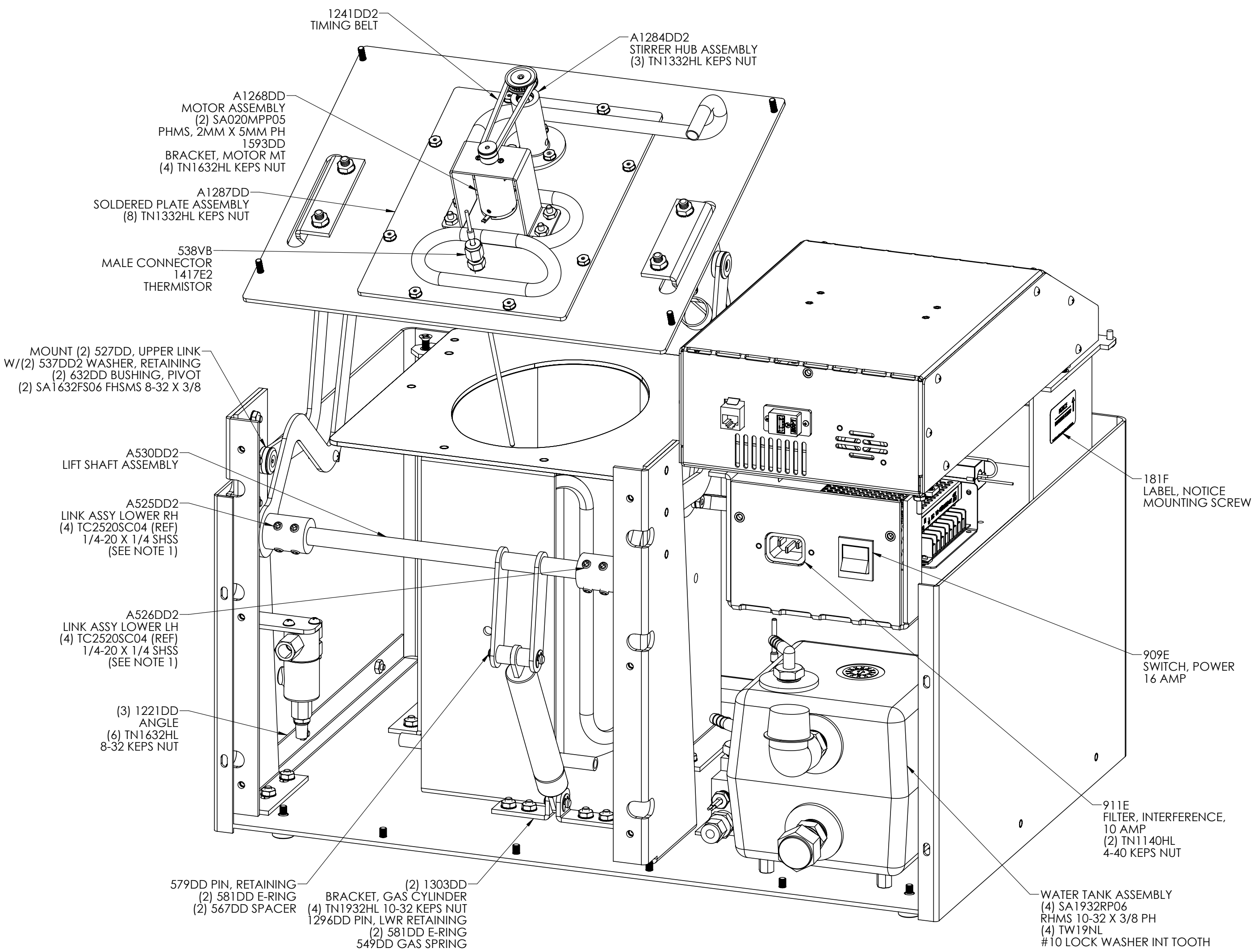


NOTE: INSTALL SET SCREWS INTO TWO SETS OF HOLES 90 DEGREES APART

NOT SHOWN:
1295DD COVER, CONTROLLER
1297DD COVER, LID
1294DD ENCLOSURE, FRONT HOUSING

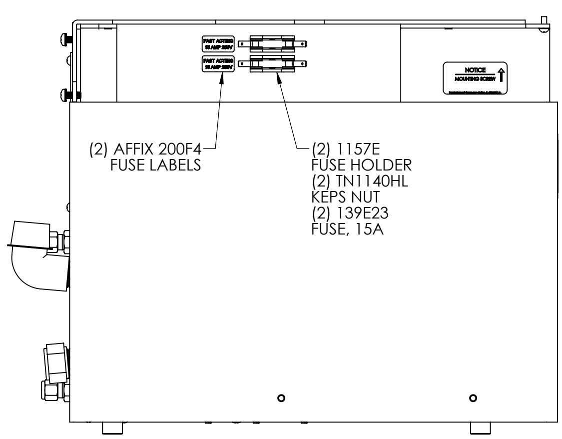
Figure 14-2

6200 Isoperibol Calorimeter Cutaway Rear



NOT SHOWN:
1295DD COVER, CONTROLLER
1297DD COVER, LID
1291DD BACK PANEL
1339DD PANEL, REAR ACCESS

NOTES:
1) TORQUE (8) TC2520SC04 SCREWS OF A525DD2 AND A526DD2 LINK ASSEMBLIES ONTO A530DD2 LIFT SHAFT ASSEMBLY TO 50 IN/LBS AT ASSEMBLY.



LEFT SIDE VIEW

Figure 14-3

A1279DD3 Control Schematic

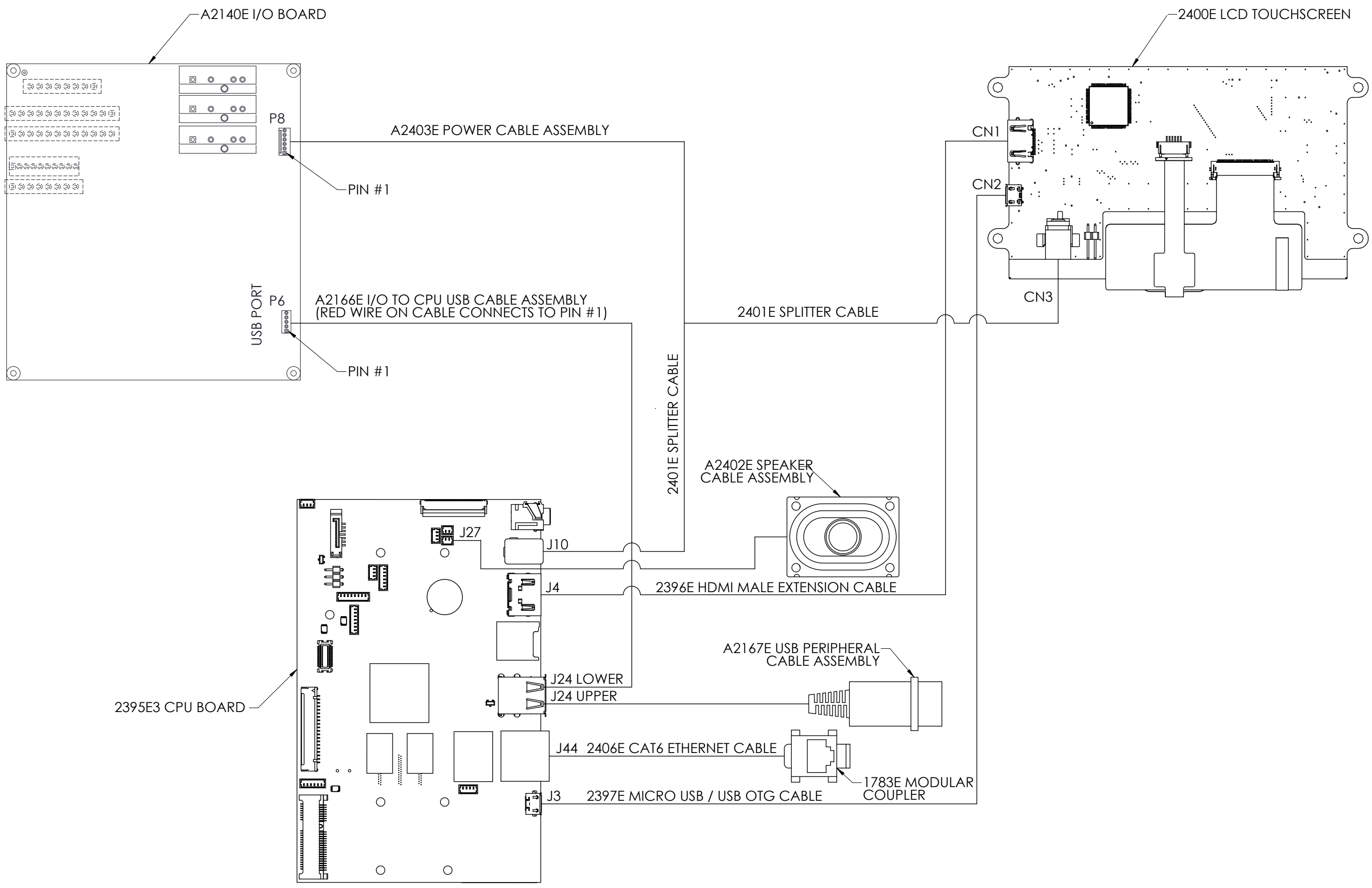


Figure 14-4

Oxygen Solenoid Assembly & Fittings

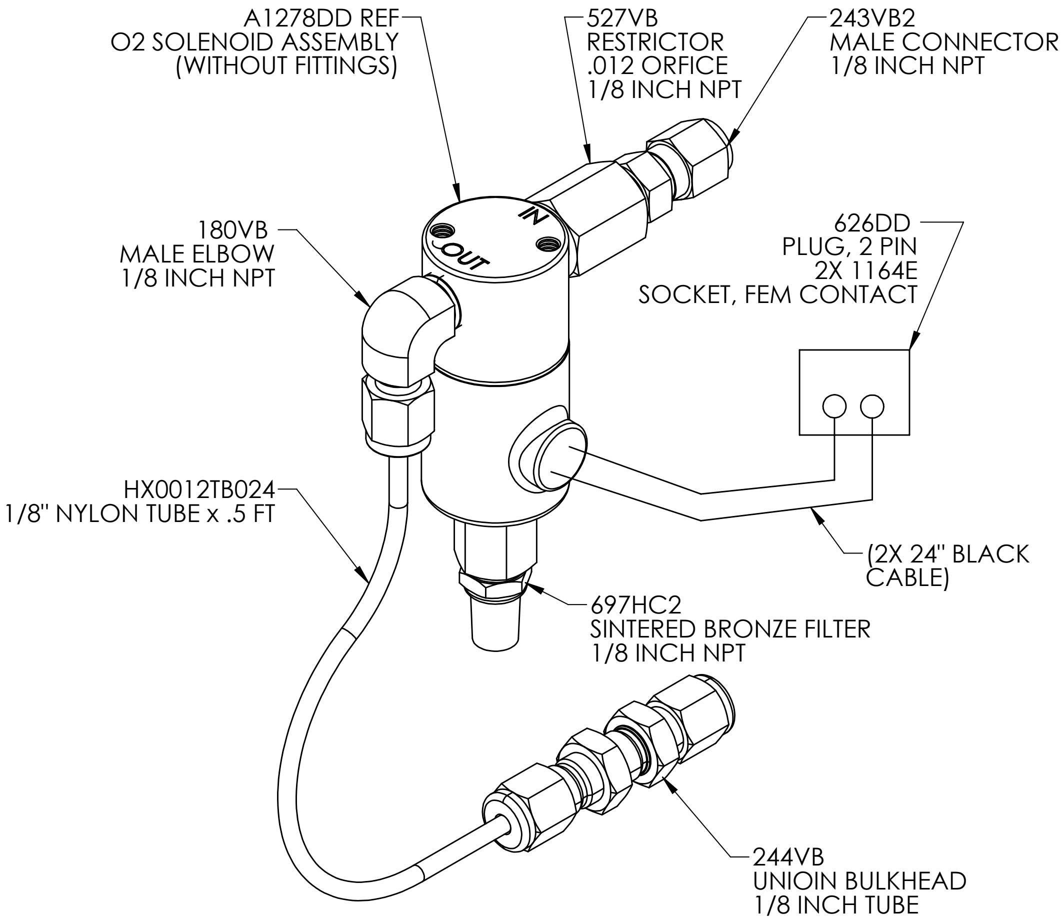


Figure 14-5

Water Tank Assembly

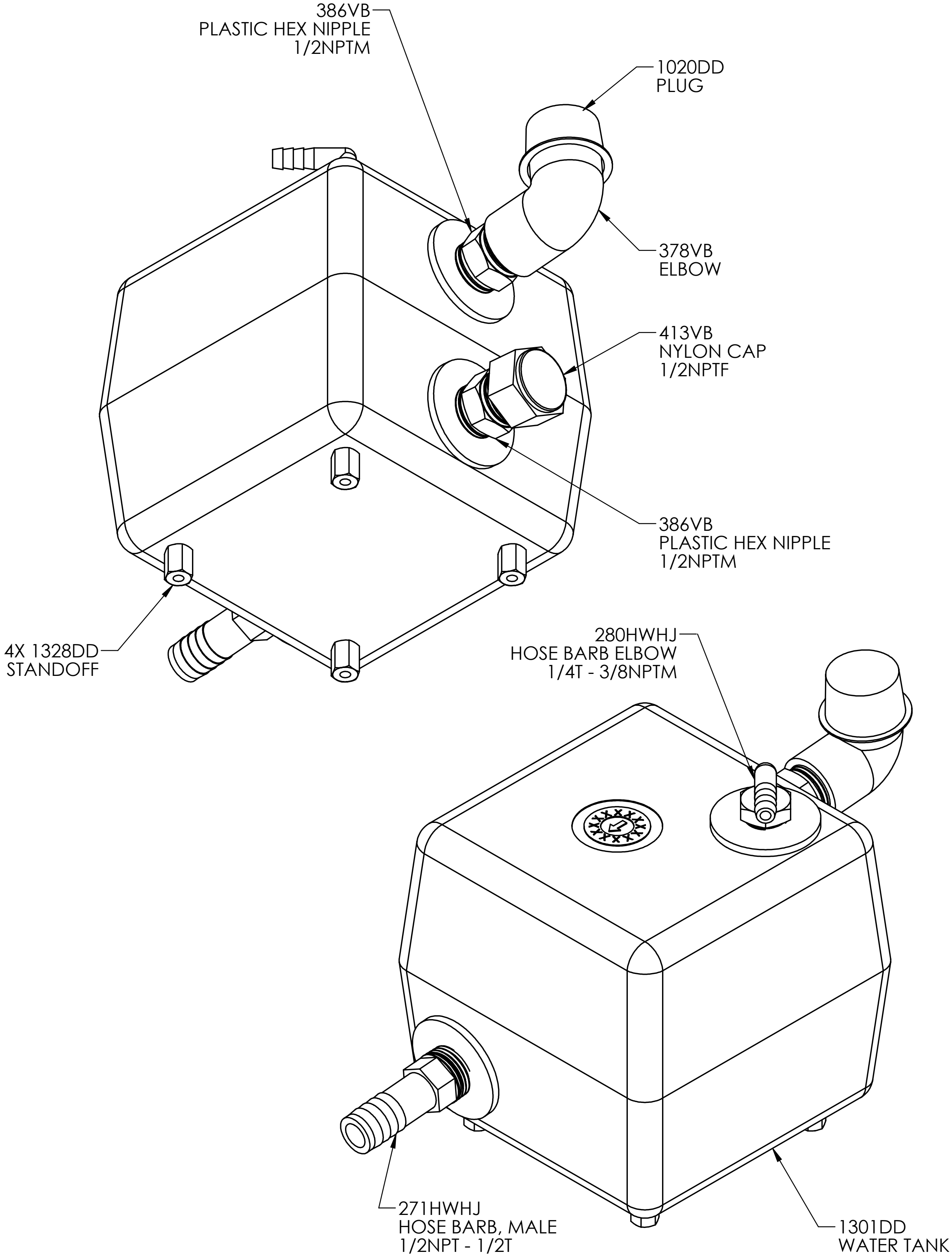


Figure 14-6

A1311DD Circulating Pump Assembly

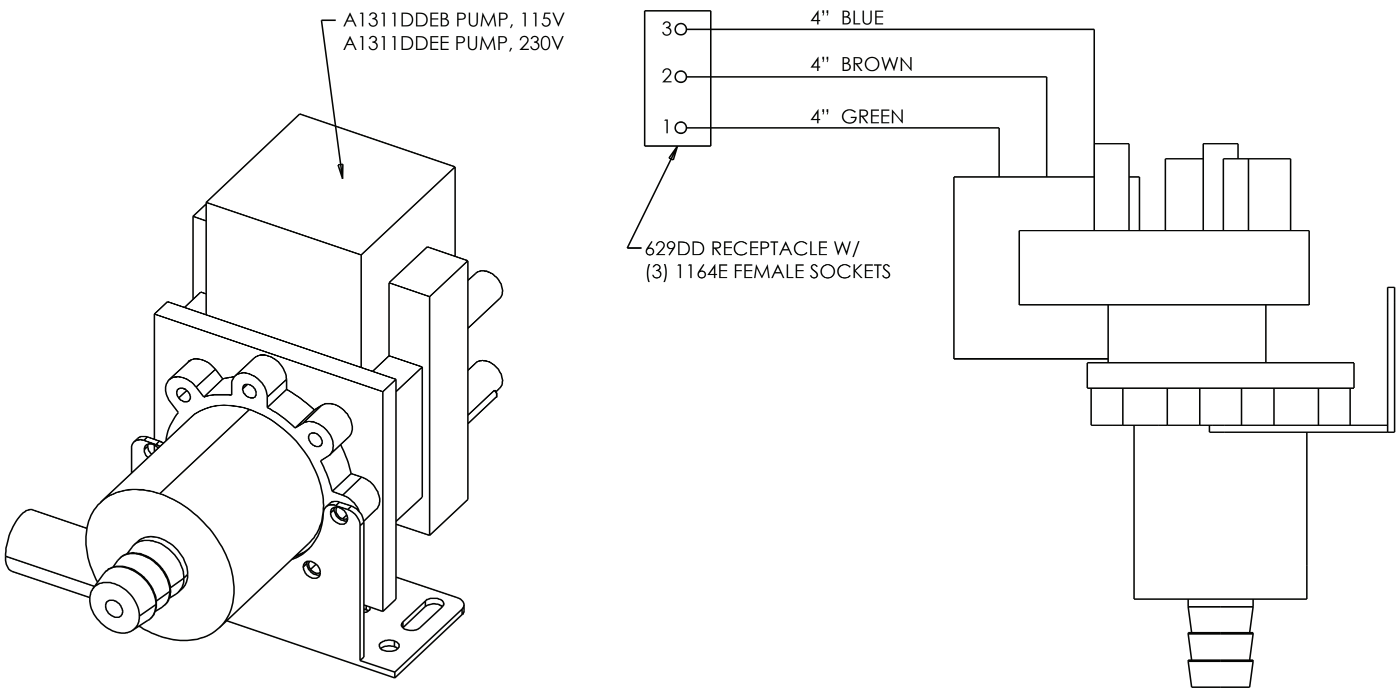


Figure 14-7

Temperature Control Assembly with Fittings

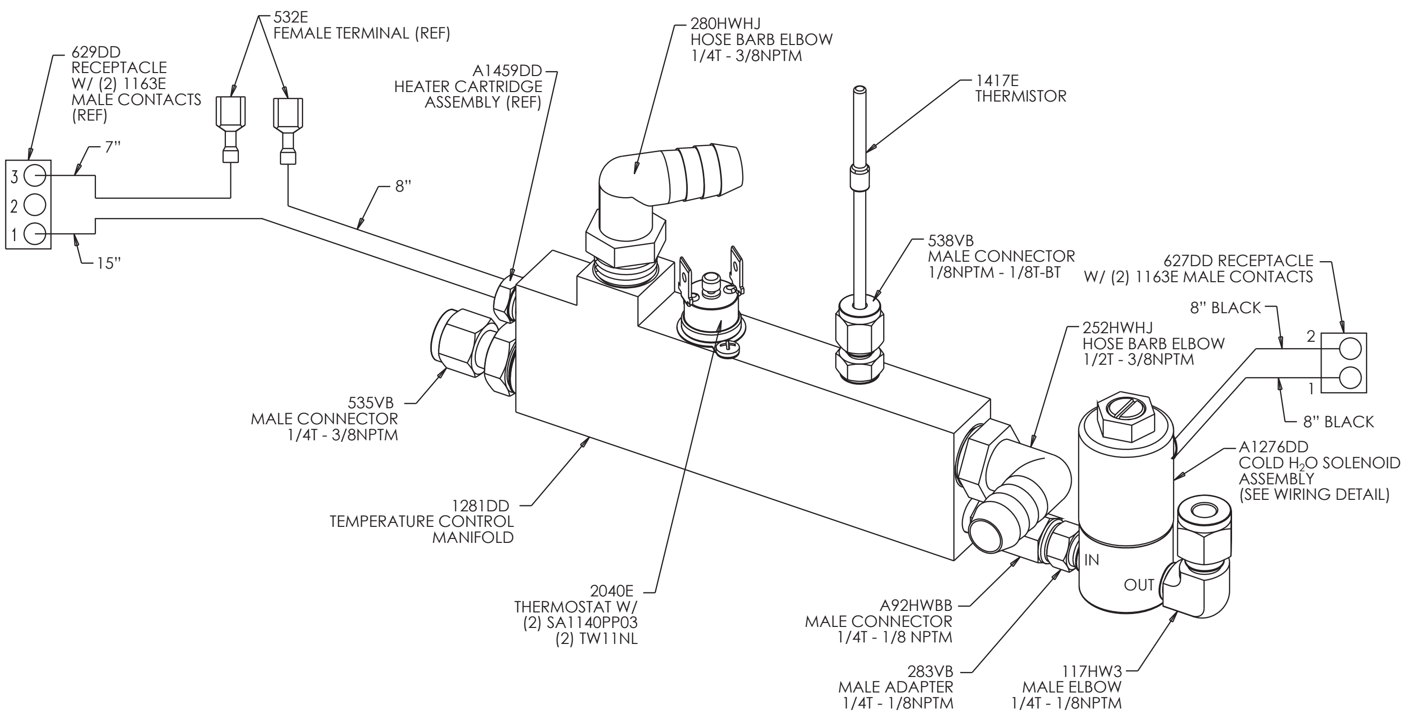
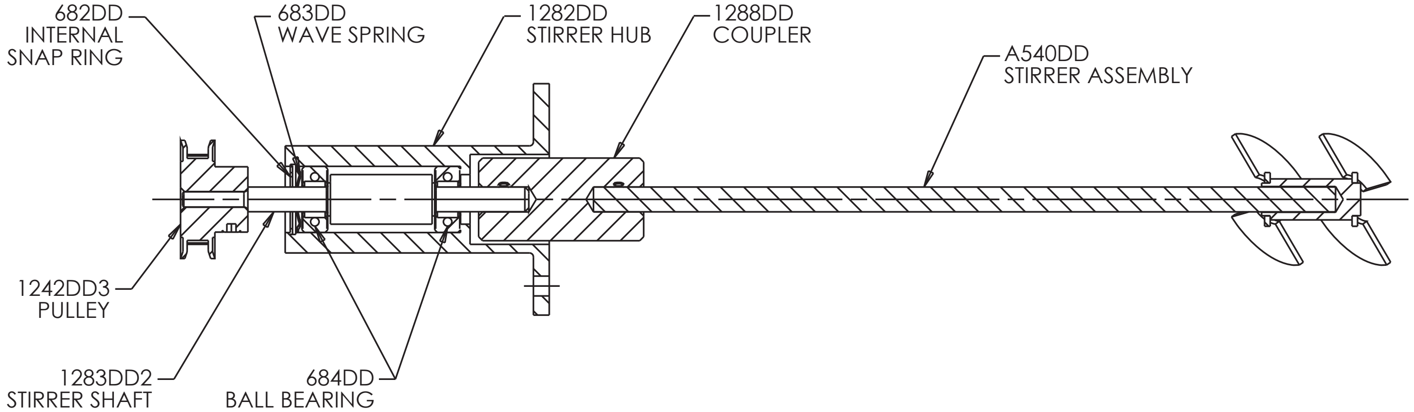


Figure 14-8

A1284DD2 Stirrer Hub Assembly



NOTE: APPLY THREAD SEALANT (LOCTITE OR EQUIVALENT) TO SET SCREW IN 1242DD2 PULLEY BEFORE INSTALLING.

Figure 14-9

Stirrer Motor Assembly

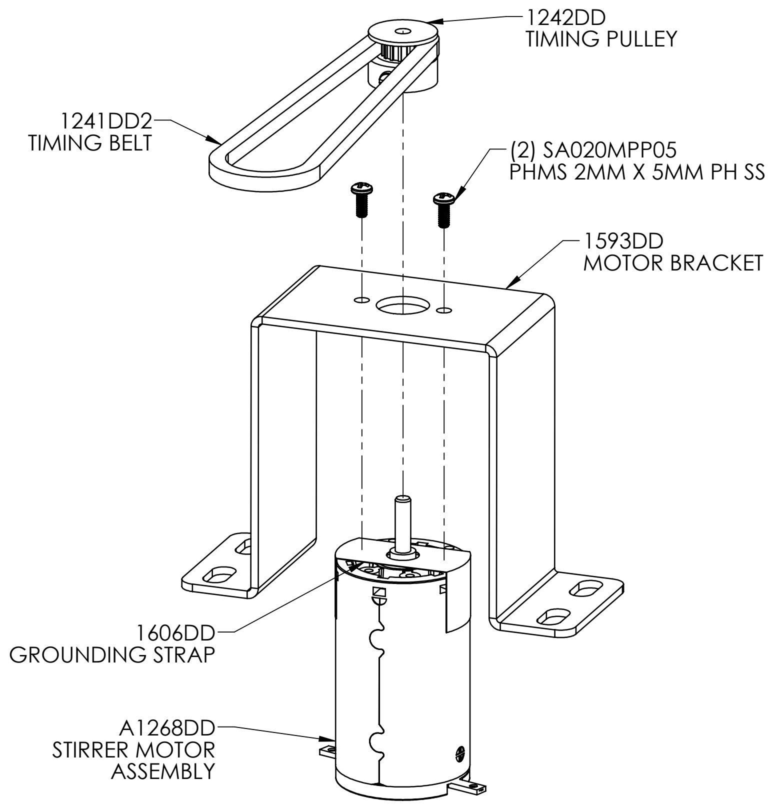
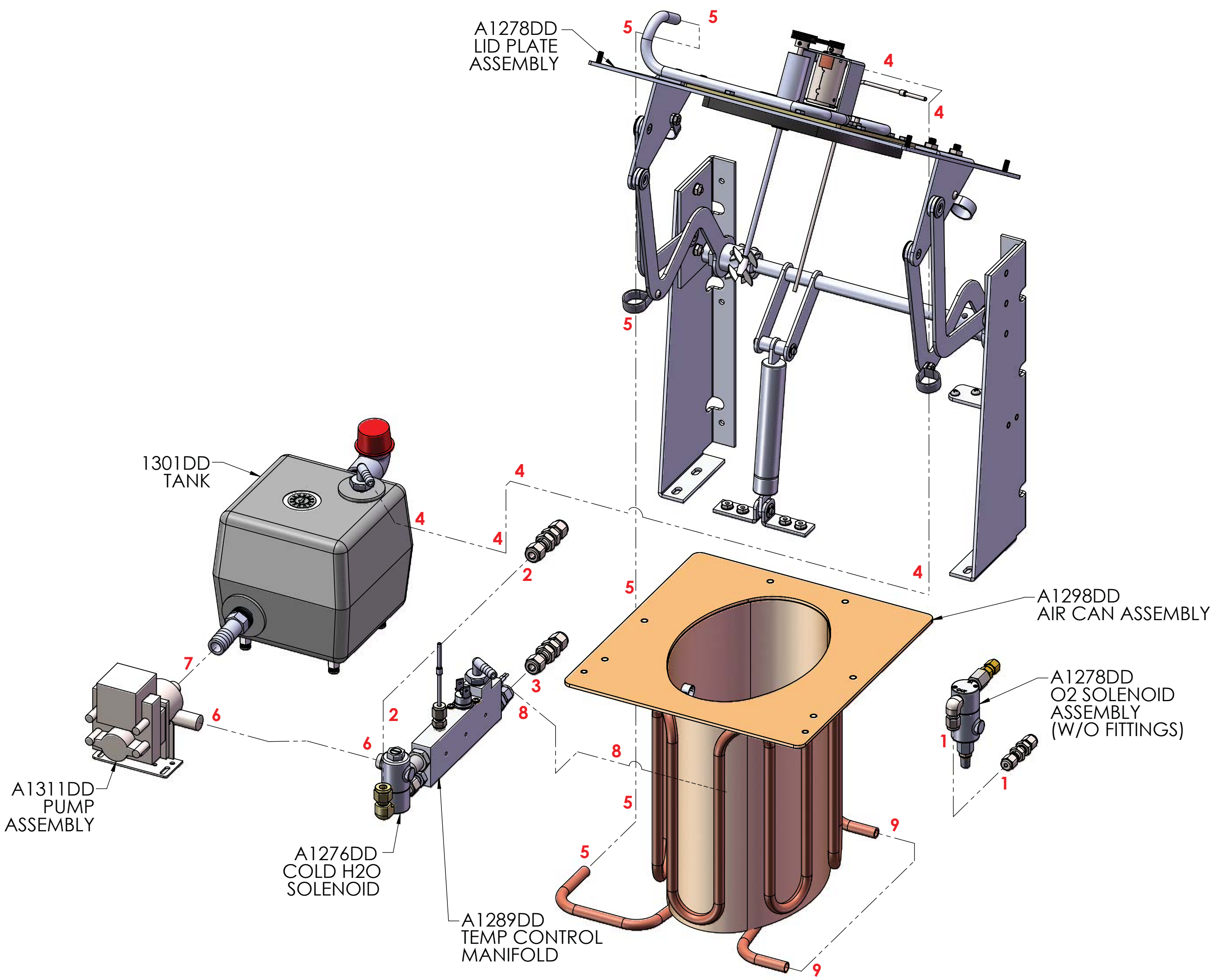


Figure 14-10

6200 Internal Plumbing Diagram



No.	Tubing Part No.	Length	End No. 1	Loc No. 1	End No. 2	Loc No. 2
1	HX0012TB024	6"	243VB2	A1278DD	244VB	1291DD
2	HJ0025TB035	13.5"	328VB	1291DD	117HW3	A1289DD
3	HJ0025TB035	1.5"	328VB	1291DD	535VB	A1289DD
4	JU0038TB062A	40"	BA0037TB032	A1287DD	279HWHJ	1301DD
5	JU0038TB062A	30"	BA0037TB032	A1287DD	BA0037TB032	A1298DD
6	JT0056TB062A	4"	252HWHJ	A1289DD	H2O OUTLET	A1311DD
7	JT0062TB062	2.5"	271HWHJ	1301DD	H2O INLET	A1311DD
8	JT0038TB062A	6.5"	280HWHJ	A1289DD	BA0037TB032	A1298DD
9	JT0038TB062A	12"	BA0037TB032	A1298DD	BA0037TB032	A1298DD

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