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PREFACE

Scope
This manual contains instructions for installing and operating the Parr 6755 Solution Calorimeter. For ease of use, the manual is divided into 13 chapters.

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

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

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

Additional instructions concerning the installation and operation of various component parts and peripheral items used with the 6755 Solution Calorimeter should be made a part of these instructions. Additional instructions for the optional printer are found in the respective printer package and should be made a part of this book.

<table>
<thead>
<tr>
<th>No.</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>201M</td>
<td>Limited Warranty</td>
</tr>
<tr>
<td>230M</td>
<td>Safety in the Operation of Laboratory and Pressure Vessels</td>
</tr>
</tbody>
</table>

This manual contains detailed instructions related to oxygen bomb calorimetry, standardization of the calorimeter, combustion techniques, and thermochemical corrections.

Note:
The unit of heat used in this manual is the thermochemical calorie, which is equal to 4.1840 absolute joules.
Explanation of Symbols

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>On position</td>
</tr>
<tr>
<td>O</td>
<td>Off position</td>
</tr>
<tr>
<td>~</td>
<td>Alternating Current (AC)</td>
</tr>
</tbody>
</table>

This **CAUTION** symbol may be present on the Product Instrumentation and literature. If present on the product, the user must consult the appropriate part of the accompanying product literature for more information.

**ATTENTION**, Electrostatic Discharge (ESD) hazards. Observe precautions for handling electrostatic sensitive devices.

**Protective Earth (PE) terminal.** Provided for connection of the Protective Earth (green or green/yellow) supply system conductor.

**Chassis Ground.** Identifies a connection to the chassis or frame of the equipment shall be bonded to Protective Earth at the source of supply in accordance with national and local electrical code requirements.

**Earth Ground.** Functional earth connection. NOTE: This connection shall be bonded to Protective earth at the source of supply in accordance with national and local electrical code requirements.

Safety Information

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

**To avoid personal injury:**
1. Do not use in the presence of flammable or combustible materials; fire or explosion may result. This device contains components which may ignite such material.
2. Refer servicing to qualified personnel.
Intended Usage
If the instrument is used in a manner not specified by Parr Instrument Company, the protection provided by the equipment may be impaired.

General Specifications

Electrical Ratings
115VAC, 2.0 Amps, 50/60 Hz
230VAC, 2.0 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
This apparatus is to be used indoors.

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

Provisions for Lifting and Carrying
Before moving the instrument, disconnect all connections from the rear of the apparatus. Lift the instrument by grabbing underneath each corner.

Periodic cleaning may be performed on the exterior surfaces of the instrument with a lightly dampened cloth containing a mild soap solution. All power should be disconnected when cleaning the instrument. There are no user serviceable parts inside the product other than what is specifically called out and discussed in this manual. Advanced troubleshooting instructions beyond the scope of this manual can be obtained by calling Parr Instrument Company in order to determine which part(s) may be replaced or serviced.
Specifications

<table>
<thead>
<tr>
<th>Description</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Probe type</td>
<td>Thermistor</td>
</tr>
<tr>
<td>Thermometer range</td>
<td>10-50 °C</td>
</tr>
<tr>
<td>Resolution</td>
<td>0.0001 °C</td>
</tr>
<tr>
<td>Absolute accuracy</td>
<td></td>
</tr>
<tr>
<td>without calibration</td>
<td>+/- 0.100 °C</td>
</tr>
<tr>
<td>with calibration</td>
<td>+/- 0.0500 °C</td>
</tr>
<tr>
<td>Repeatability, single point</td>
<td>+/- 0.002 °C</td>
</tr>
<tr>
<td>Linearity, 10 °C span</td>
<td>+/- 0.002 °C</td>
</tr>
<tr>
<td>Communications port</td>
<td>Ethernet</td>
</tr>
<tr>
<td>Data logging capacity</td>
<td>1MB (~10000 points)</td>
</tr>
</tbody>
</table>
INSTALLATION

The 6755 Solution Calorimeter requires approximately 4 square feet of work space on a sturdy bench/table in a location free from room drafts or radiant heat sources, (preferably in an air conditioned room providing minimal temperature change), and an electrical outlet.

Other necessary accessories include:
- Chemical balance sensitive to 0.1 mg
- Top loading balance capable of weighing up to 1.5 kg with a sensitivity of 0.1g

Unpack the calorimeter carefully and check the individual parts against the packing list. If shipping damage is discovered, report it immediately to the delivery carrier. Handle the glass cell, Dewar flask and the thermistor probe with care as these parts are fragile and easily broken.

Set the calorimeter on a bench or table in a location that is free from drafts and protected from sources of radiant heat. Temperature changes in the room should be minimal.

Power Connection
Plug the power line into any grounded outlet providing proper voltage that matches the specification on the nameplate of the Calorimetric Thermometer. The calorimeter will draw approximately 100 watts 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 6772 Calorimetric Thermometer Main Menu is displayed.
Motor Installation
Attach the motor cord to the rear of the calorimeter case using the mounting screws provided for safety purposes. Set the cover with its attached stirring shaft onto the stainless steel air can; drop the geared drive belt over the motor and stirrer pulleys. The drive system should run freely. Although the belt may appear to be unusually loose, it is intended to operate under light tension to minimize friction in the stirrer bearing. The gearing on the belt and pulleys will prevent slippage.

Thermistor Probe Installation
Connections for two thermistor probes are located on the back of the 6772 Calorimetric Thermometer. If only one probe is to be used, connect it to the “bucket” connection. If the second probe is used, it should be inserted in the hole on the left, rear of the instrument. Install the thermistor in the cover opening and press the bushing firmly into place to anchor the probe in its proper position. Place the cover on the calorimeter with the orienting pin in the alignment hole.

Communication Connections
There are three RS-232 serial ports at the rear of the calorimeter. These ports are designated Terminal, Printer and Balance. The pin-out of these three ports is identical. The pin-out is illustrated in Table 1.
Table 1
6772 Calorimetric Thermometer Serial Ports Pin-Out

<table>
<thead>
<tr>
<th>9 pin D Connector Pin #</th>
<th>Description</th>
<th>Direction (6300 – External Device)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>Received Data</td>
<td>←</td>
</tr>
<tr>
<td>3</td>
<td>Transmitted Data</td>
<td>→</td>
</tr>
<tr>
<td>4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Signal Ground</td>
<td>← →</td>
</tr>
<tr>
<td>6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Ready to Send (RTS)</td>
<td>→</td>
</tr>
<tr>
<td>8</td>
<td>Clear to Send (CTS)</td>
<td>←</td>
</tr>
</tbody>
</table>

Both the terminal and balance port are female. The printer port uses a male connector.

The 6772 Calorimetric Thermometer is also equipped with an RS-232 port for connection to a 40 or 80 column printer and/or a computer. Before making any of these connections, the data transmission rate of the Calorimetric Thermometer and the printer, balance or computer must be matched. Generally, the baud rates on either device can be changed to achieve this match.

The 6772 will allow the user to specify the IP addresses of one or more Balance Interface devices on the network by selecting the Network Data Device menu in the Communications Controls menu. Balance Interface devices are polled from device 1 to 15 for sample and / or spike weights when the weight entry mode is set to Network.

**Printer Connections**

The printer port settings are on the Communication Controls Menu: Printer Port Communications Menu. The default parameters for the 6772 Calorimetric Thermometer are set up for use with the Parr 1757 Printer. Table 1 identifies and describes the pin-out for the RS-232 port.

**Balance Connections**

The 6772 Calorimetric Thermometer supports input from the multiple balance types. Additionally, a generic input driver is provided for communications with balances that do not conform to the four supported protocols. A new feature supported by all four 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 Calorimetric Thermometer. 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 Calorimetric Thermometer 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 Calorimetric Thermometer.

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

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

\[ \text{[ESC]} \text{ P [CR] [LF]} \]

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

The Calorimetric Thermometer 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.

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

Computer Connections
If the 6772 Calorimetric Thermometer is to be connected to a computer, the Ethernet connection should be used. Calorimetric Thermometer 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 Calorimetric Thermometer. The network DHCP (Dynamic Host Configuration Protocol) server provides this address shortly after the Calorimetric Thermometer is turned on. The address can be seen on the Software & Hardware Info” screen, under Program Info and Control Menu; see the example screenshot. Users who don’t have a network infrastructure can create a simple network by connecting a router with DHCP server capability to the Calorimetric Thermometer using an ordinary CAT 5 network cable. The Calorimetric Thermometer should be connected to LAN side of the router. The PC in turn is also connected to the LAN side of the router using a similar CAT 5 cable. A D-Link 614+ router is recommended for this purpose. A D-Link 614+ router is recommended for this purpose. For this router, operated without a WAN connection, the primary DNS address of the router (WAN setup) must be set to the IP address of the router found on the LAN setup page. Other routers behave differently in the absence of a WAN connection. Providing an active upstream connection to the WAN port of most routers generally minimizes the use of any obscure setup configurations. An FTP enabled web browser can be used to access stored test data. The URL is of the following form:

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

In this case, 192.168.0.125 is the IP address of the Calorimetric Thermometer.
The following screenshot illustrates the contents of the Calorimetric Thermometer data directory as presented by a web browser.

You can drag and drop or copy and paste test data files (with the csv suffix) from the web browser window to any convenient folder or directory on the PC.
The Calorimetric Thermometer offers a web server service. Test reports can be viewed with a web browser using a URL of the following form.

http://192.168.0.125

Where 192.168.0.125 is the IP address of the Calorimetric Thermometer. The following screenshot illustrates the Calorimetric Thermometer home page.

Web server services coming soon!

Features to include:

- Viewing test reports.
- Editing test reports.
- Printing test reports.
- Remote instrument diagnostics.
Clicking on the Sample Data tab displays a list of reports currently in the instrument memory.

- RunDataTemplate.csv
- TEST1.det.finl.csv
- TEST10.det.finl.csv
- TEST11.det.finl.csv
- TEST12.det.finl.csv
- TEST13.det.finl.csv
- TEST14.det.finl.csv
- TEST15.det.finl.csv
- TEST16.det.finl.csv
- TEST17.det.finl.csv
- TEST18.det.finl.csv
- TEST19.det.finl.csv
- TEST2.det.finl.csv
- TEST20.det.finl.csv
- TEST3.det.finl.csv
- TEST4.det.finl.csv
- TEST5.det.finl.csv
- TEST6.det.finl.csv
- TEST7.det.finl.csv
- TEST8.det.finl.csv
- TEST9.det.finl.csv
Clicking on any given report will provide a display similar to the following:

![6772 Calorimeter Report](image)

### Sample ID: TEST1
- **Mode:** Determination
- **Date/Time:** 12/04/04 12:33:00
- **Method:** Dynamic
- **Bomb ID:** 1

### Values
- **Fusel:** 50.0000
- **EE Value:** 803.7698
- **Acid:** 10.0000
- **Sulfur:** 2.0000
- **Jacket Temperature:** 30.009
- **Initial Temp.:** 30.0688
- **Temperature Rise:** 7.6466

### Results
- **Gross Heat:** 10955.0479 Btu/lb

---

**Run List**  **Home**
QUICK START

Before starting to use the calorimeter for the first time, it is recommended that the user perform a dry run with the calorimeter completely assembled, but with no liquid in the Dewar and no sample in the rotating cell. This will give the user an opportunity to become familiar with the individual parts of the calorimeter and the manner in which they must be handled. The calorimeter must be standardized prior to analyzing a sample.

1. Allow at least 20 minutes for the calorimeter to warm up.

2. Turn on the stirrer motor switch on the 6755 calorimeter.

3. Prepare and weigh the sample to 0.0001g or 1 mL in the PTFE dish.

4. Fill the Dewar volumetrically or by weight.

5. Install the thermistor probe in the cover opening and press the bushing firmly into place to anchor the probe in its proper position.

6. Lower the cover assembly with the cell and thermistor probe into the Dewar and set the cover in place on the air can, then drop the drive belt over the pulleys, start the motor and press the start key.

7. The pre-period will now start. When the reactants come to thermal equilibrium, the thermometer will beep. Initiate the reaction by pressing downward on the push rod to drop the sample out of the rotating cell.

8. During the reaction period, the enthalpy change will occur.

9. The calorimeter will again come to equilibrium during the post period and at the conclusion of the test, the calorimeter will signal the user and produce a report.

10. Stop the calorimeter motor, raise the cover carefully and wipe any excess liquid from the parts that were immersed in the Dewar. Remove the thermistor probe from the cover and remove the sample dish from the end of the push rod; then remove the rod and release the glass cell from the drive shaft.

11. Lift the Dewar out of the air can and empty it. Wash and dry all wetted parts carefully.

12. At the end of the testing period, turn OFF the thermometer at the power switch.
OPERATION

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

Note:
Keys with a “double box” in the upper left hand corner lead to sub-menus.

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

1. **Toggles.** These data fields contain ON/OFF or YES/NO choices. Simply touching the key on the screen toggles the choice to the other option. The current setting is displayed in the lower right corner of the key.

2. **Option Selection.** These data fields contain a list of options. Touching the key on the screen steps the user through the available choices. The current setting is displayed in the lower right corner of the key.

3. **Value Entry Fields.** These data fields are used to enter data into the Calorimetric Thermometer. Touching the key on the screen brings up a sub-menu with a key pad or similar screen for entering the required value. Some keys lead to multiple choices. Always clear the current value before entering a new value. Once entered the screen will return to the previous menu and the new value will be displayed in the lower right corner of the key.

4. **Data Displays.** Most of these keys display values that have been calculated by the Calorimetric Thermometer and are informational only. Certain ones can be overridden by the user entering a desired value through a sub-menu. The value is displayed in the lower right corner of the key.

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

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

1. **Escape.** This key is used to go up one level in the menu structure.
2. Main Menu. This key is used to return to the main menu touch screen from anywhere in the menu structure.

3. Start. This key is used to start a Calorimetric Thermometer test.

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

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

Programming
The program in the 6772 Calorimetric Thermometer can be extensively modified to tailor the unit to a wide variety of operating conditions, reporting units, laboratory techniques, available accessories and communication modes. In addition, the calculations, thermochemical corrections and reporting modes can be modified to conform to a number of standard test methods and procedures. Numerous provisions are included to permit the use of other reagent concentrations, techniques, combustion aids and short cuts appropriate for the user’s work.

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

Default Settings
The 6772 Calorimetric Thermometer is preprogrammed with default settings for use with the 1341 Plain Jacket Calorimeter. On the operating controls page of the 6772 Thermometer is the Method of Operation key. This key toggles the thermometer between solution and combustion calorimetry. Make sure that the calorimeter is set to solution calorimetry. This will force the calorimeter to restart and bring up the appropriate set of menus and eliminate all of the keys dedicated to combustion calorimetry.

The default values of the 6772 are designed to operate with the 1341 Plain Jacket calorimeter. Therefore, the following parameters must be changed in the Calorimetry Parameters menu found in the Diagnostics Menu.

<table>
<thead>
<tr>
<th>Correction (K) Parameters:</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>K1</td>
<td>0.5</td>
</tr>
<tr>
<td>K2</td>
<td>0.00096</td>
</tr>
<tr>
<td>K3</td>
<td>1.0</td>
</tr>
<tr>
<td>K4</td>
<td>0.0</td>
</tr>
<tr>
<td>K5</td>
<td>0.0</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Blackout (B) Parameters:</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Misfire Blackout (B2)</td>
<td>72</td>
</tr>
<tr>
<td>Derivative Blackout (B3)</td>
<td>0.5</td>
</tr>
<tr>
<td>Dynamic Blackout (B4)</td>
<td>6</td>
</tr>
<tr>
<td>Equilibrium Blackout (B5)</td>
<td>18</td>
</tr>
<tr>
<td>Dynamic Derivative Blackout (B6)</td>
<td>0.02</td>
</tr>
<tr>
<td>Dynamic Time Blackout (B7)</td>
<td>20</td>
</tr>
</tbody>
</table>
Note:
To perform an exothermic run, set the Tolerance Parameter (L2) to -1.

See Table 1 for a listing of the factory default settings. A more in-depth explanation of these parameters is found on the corresponding parameter group help pages. These default settings remain in effect until changed by the user. Should the user ever wish to return to the factory default settings, go to the Program Info and Control Menu, User/Factory Settings, touch Reload Factory Default Settings and YES. Non-volatile memory is provided to retain any and all operator initiated program changes; even if power is interrupted or the unit is turned off. If the unit experiences an intentional or unintentional “Cold Restart”, the controller will return to the last known settings.

The default parameters of the 6772 Calorimetric Thermometer can be changed to guarantee that the thermometer, when cold restarted, will always be in the desired configuration before beginning a series of tests. Users who wish to permanently revise their default settings may do so using the following procedure:

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

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

Performing an analysis
Tests can be run in a strictly manual fashion or automatically where the thermometer sequences the calorimeter through the pre and post periods. The manual sequencing approach is useful for applications where raw data is logged and subsequently analyzed, off-line. In the automatic mode, the thermometer fully sequences the test and applies real time corrections to the calorimeter temperature rise in order to correct for all systemic heat leak effects. In either case, the operator must determine the appropriate temperature source for the jacket.

- **Probe** – This method uses a thermistor probe attached to the jacket wall to measure the actual temperature of the surroundings (at the chosen point) and the heat leak correction are based upon the actual differences between the bucket and this external jacket temperature.

- **Calculated** – During the initial equilibrium period this method analyzes the actual heat leak rate and calculates the apparent temperature of the surroundings which would generate this rate and applies this calculated jacket temperature for the determination.

- **Fixed** – In this method the operator determines what his jacket temperature will be and enters it into the thermometer. All heat leak corrections are then based upon this fixed jacket temperature.
For most applications the calculated method is recommended.

**Sample size**
The rotating sample cell will hold up to 20 ml of liquid sample or a solid sample weighing up to one gram. More than one gram of solid may be used in some cases, but smaller samples are preferred so that the heat capacity and ionic strength of the system will not change significantly when the reactants are mixed. The Dewar must be filled with not less than 90 ml and not more than 120 ml of liquid to properly cover the rotating cell.

**Filling the Dewar**
It is best to lift the Dewar out of the air can during the filling operation. The liquid to be placed in it can be measured volumetrically, or the Dewar can be placed on a solution or trip balance and filled by weight. After filling the Dewar, set it in the air can and gently push the spacer ring down as far as it will go.

**Loading a solid sample**
Solid samples should be suitably ground so that they will dissolve quickly or mix uniformly with the liquid in the Dewar. Place the 126C PTFE Dish on an analytical balance and weigh the sample directly into the dish. Be careful not to drop any of the sample into the push rod socket. After the final weighing, set the dish on a flat surface and carefully press the glass bell over the dish to assemble the cell. Do not grasp or press the thin-walled glass stem during this operation; it is fragile and will break easily. Instead, grasp the bell and press it firmly onto the dish. Then lift the cover from the calorimeter and attach the cell to the stirring shaft by sliding the plastic coupling onto the shaft as far as it will go and turning the thumbscrew finger tight. If the thumbscrew is not tight against the shaft, the contents will not be released. If necessary, use a 9/64 Allen wrench to tighten further. Hold the cover in a horizontal position and lower it carefully until the bottom of the rotating cell rests on a firm, flat surface; then insert the push rod through the pulley hub and press the end of the rod into the socket in the 126C Sample Dish.

**Loading a liquid sample**
Liquid samples can be measured into the rotating cell either by volume or by weight. Best precision is obtained by weighing, but filling from a volumetric pipette may be adequate in some cases. Set the 126C PTFE Dish on a flat surface and press the glass bell over the dish, handling the glass carefully as described above. If the sample is to be weighed, tare the empty cell on a laboratory balance; insert a pipette through the glass stem and add the liquid, then reweigh the cell. Attach the cell to the stirring shaft and insert the push rod.

**Installing the loaded cover assembly**
Install the thermistor probe in the cover opening and press the bushing firmly into place to anchor the probe in its proper position. Lower the cover assembly with the cell and thermistor probe into the Dewar and set the cover in place on the air can, then drop the drive belt over the pulleys and start the motor as required.
Combining the reactants
Each test in a solution calorimeter can be divided into three distinct time periods:

1. A pre-period during which the reactants are allowed to come to an initial thermal equilibrium. The thermometer will beep to inform the operator that it has established the initial equilibrium and that it is now time to initiate the reaction.

2. A reaction period during which the reactants are combined and an enthalpy change occurs in the system.

3. A post-period during which the calorimeter again comes to equilibrium. The thermometer will produce a report when the final equilibrium has been achieved and that the test is complete.

At the end of the pre-period, start the reaction by pressing the push rod downward to drop the sample out of the rotating cell. This should be done quickly without interrupting the rotation of the rod without undue friction from the finger. Push the rod down as far as it will go; after which it should continue to rotate the pulley. Let the stirrer continue to run during the reaction and the calorimeter reports its results.

Emptying the calorimeter
Stop the calorimeter motor, raise the cover carefully and wipe any excess liquid from the parts that were immersed in the Dewar. Remove the thermistor probe from the cover and remove the sample dish from the end of the push rod; then remove the rod and release the glass cell from the drive shaft. Lift the Dewar out of the air can and empty it; then wash and dry all wetted parts carefully.

The two operating modes, (manual or automatic) are outlined below:

**Manual Test Sequencing**
Some users may wish to construct their own thermogram and apply the classic graphical corrections developed by Dickenson and others. In this case, the actual temperatures can be logged to the memory of the thermometer and then analyzed at the end of the test. These logged temperatures can be recalled to display on the thermometer, printed on an attached printer or transferred to a computer using either the Ethernet Connection or a Compact Flash Card. The Ethernet Connection can also be used to transfer temperatures to a computer for plotting.

First, select the appropriate jacket temperature source as described previously. Then fill the Dewar. Next, prepare and load the reaction. After the calorimeter is fully assembled, turn on the motor, and then turn on the stirrer by pressing the stirrer key on the calorimeter operation menu screen. Turn on the data logger (accessed via the Diagnostics page) in order to periodically record the bucket or calorimeter temperature. The bucket temperature is updated every 12 seconds. Turn on the calorimetric pre-period. The pre-period should last for 6-7 minutes. After the 6-7 minute pre-period test phase, start the reaction by pressing the push rod downward to drop the sample out of
the rotating cell. This begins the reaction and subsequent post-period. The calorimeter temperature should begin to significantly change at this point, indicating sample reaction. The calorimetric post-period should last for an additional 6-7 minutes from sample introduction. At the conclusion of the post-period, turn the stirrer off by pressing the stirrer key once again. The motor switch may be left in the “on” position for subsequent tests. Empty and clean the calorimeter.

If the data log destination is a log file, the log file is located at /flash/datalog.csv and may be retrieved via FTP. The log file is easily imported into a spreadsheet program where the calorimeter temperature can be plotted in order to realize a thermal curve. Instructions for working with or analyzing thermal curves are found in the calculations section.

**Automatic Test Sequencing**

The solution calorimeter will perform all calculations for the user. To do this, first select the appropriate jacket temperature source. For most applications, the calculated jacket approach works well. First, select the appropriate jacket temperature source as described previously. Then fill the Dewar. Next, prepare and load the reaction. After the calorimeter is fully assembled, turn on the motor, and then press the START key located on the right hand side of the screen. This will activate the stirrer that gently circulates the water that surrounds the glass cell. The thermometer will prompt for the sample ID number and the mass of the sample in grams. This begins the calorimetric pre-period. After the thermometer determines that adequate temperature equilibrium is realized, the thermometer will prompt the user to start the reaction by pressing the push rod downward to drop the sample out of the rotating cell. This starts the calorimetric post-period. The calorimeter temperature should begin to significantly rise at this point, indicating sample combustion. The calorimetric post-period will last for an additional 6-7 minutes until the calorimeter temperature drift rate sufficiently stabilizes. At the end of the post-period the calorimeter will signal the end of the test and generate a report.

Stop the calorimeter motor, raise the cover carefully and wipe any excess liquid from the parts that were immersed in the Dewar. Remove the thermistor probe from the cover and remove the sample dish from the end of the push rod; then remove the rod and release the glass cell from the drive shaft. Lift the Dewar out of the air can and empty it; then wash and dry all wetted parts carefully.
**MENU DESCRIPTIONS**

**Note:**

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

**Main Menu**

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

**Start Key:**

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

**Report:**

Press the Report key to begin the reporting process.

**Help:**

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

**Calorimeter Operation Menu**

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

**Temperature Graph:**

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

**Stirrer:**

On / Off This key provides a convenient way to manually start and stop the calorimeter stirrer motor. The motor must be physically turned on for this function to be active.
Temperature vs. Time Plot
Press the Setup key to access the Temperature Plot Setup Menu, which has many keys that permit the user to fully customize both the x (time) axis and the scaling of the y axis.

Temperature Plot Setup Menu

Bucket Plot Symbol: Toggles between:
- No Point
- Small Dot
- Round
- Square
- Up Triangle
- Down Triangle
- Diamond

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

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

Jacket Plot Symbol: 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 run.

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

Operating Controls Menu

Method of Operation:
Offers an operating mode of either combustion or solution. In the solution calorimetry mode, the instrument menu structure is streamlined in order to remove items that are only relevant to combustion calorimetry. The text on the key displays the current instrument operating mode. Pressing the key will prompt the user to restart the calorimeter, loading the appropriate menu structure. Help screens are context sensitive with respect to the operating mode.

Jacket Temp. Source:
Press this key to display a menu that allows the user to select the source for the jacket temperature used for tests.

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

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

LCD Contrast:
This key accesses a sub-menu with a slide control which adjusts the contrast on the LCD display for optimum viewing.
Print Error Messages:
When turned ON, all error messages will be printed on the printer as well as displayed on the screen. When turned OFF, messages will only display on the screen.

Language:
Steps the Calorimetric Thermometer through the installed operating languages.

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

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

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

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

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

User Setup ID
Used to enter a unique identifier for recalling user settings.

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

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

Save User Default Settings:
Used to record the setup to the memory once the user has configured the instrument to their operating requirements.
**Feature Key:**
This key access a screen which allows the user to input a code to access special calorimeter features such as the bar code capabilities or remote calorimeter access.

**Bomb Type Select:**
This key is not a valid function in the 6755 Solution Calorimeter at this time.

**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.

### Data Entry Controls Menu

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

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

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

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

**Reporting Controls Menu**

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

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

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

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

**Communication Controls Menu**
Accesses sub-menus which set the communications protocols for the printer and balances.

**Printer Port (RS-232):**
Accesses sub-menu, Printer Port Communications. Sets the communication parameters for the RS-232 ports used for the printer port. Standard options for data bits, parity, stop bits, handshaking, baud rate and balance type are provided to match any devices that might be connected to these ports.

- Number of Data Bits. Standard options for data bits. Toggles between 7 and 8.
- Parity. Standard options for parity. Choose from None, Odd or Even.

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*Figure 5-6: Reporting Controls Menu*

*Figure 5-7: Printer Port Communications Menu*
- Number of Stop Bits. Standard options for stop bits. Toggles between 1 and 2.
- Handshaking. Standard options for handshaking. Choose from Xon/Xoff, RTS/CTS and None.
- Baud Rate. Standard options for baud rate. Choose from 19.2K, 9600, 4800, 2400, 1800, 1200, 600, 300, 150, 134.5, 110, and 75.
- Printer Type. Toggles between a Parr 1757 and a generic printer. When set for the 1757 Printer, all of the features of this printer, such as bold printing, will be activated.
- Printer Port Loop Back Test. Used for factory testing of the printer port.

**Balance Port (RS-232):**
Accesses sub-menu, Balance Port Communications.

**Balance Type**
Toggles through the available balance templates.

**Customize Balance Settings**
Sets the communication parameters for the RS-232 port used for the balance port. Standard options for data bits, parity, stop bits, handshaking, baud rate and balance type are provided to match any devices that might be connected to these ports.

- Number of Data Bits. Standard options for data bits. Toggles between 7 and 8.
- Parity. Standard options for parity. Choose from None, Odd or Even.
- Number of Stop Bits. Standard options for stop bits. Toggles between 1 and 2.
- Handshaking. Standard options for handshaking. Choose from Xon / Xoff, RTS/CTS and None
- Baud Rate. Standard options for baud rate. Choose from 19.2K, 9600, 4800, 2400, 1800, 1200, 600, 300, 150, 134.5, 110, and 75.
- Data Characters from Balance. This setting is only used when the generic balance format is selected. This value determines the number of numeric data characters (0-9, +, -) to accept. Any additional characters after this value and before the string terminating <CR> are discarded.
• Data Precision. This key allows the user to establish the number of digits to the right of the decimal point that are passed from the balance handler.
• Transfer Timeout (seconds). This value determines how long the interface will wait before giving up on a weight transfer. The value is entered in seconds.
• Balance Handler Strings. This key leads to a submenu that allows balance template to be customized for unique balances or needs.

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

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

Parr offers the following communication cables:

<table>
<thead>
<tr>
<th>25 pin D (male)</th>
<th>9-pin DP</th>
<th>25-pin DP S-T</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1837E</td>
<td>9-pin DP</td>
<td>25 pin DP-Null</td>
</tr>
<tr>
<td>9 pin D (male)</td>
<td>9-pin DP</td>
<td>9-pin DP S-T</td>
</tr>
<tr>
<td>A1892E</td>
<td>9-pin DP</td>
<td>9-pin DP Null</td>
</tr>
</tbody>
</table>

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

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

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

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

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

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

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

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

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

Desel All:
This key deselects all files previously selected.

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

Delete:
This key deletes all selected files.

Convert Type:
This key allows one or more selected tests to be converted from determinations to standardizations and vice versa. This function is only available in combustion mode, since the standardization / determination report concept is not supported in the solution calorimeter mode.
Diagnostics Menu
Provides the user with the means to test many of the components and subsystems of the Calorimetric Thermometer. These capabilities should be used in conjunction with the Maintenance Instructions in order to obtain the maximum benefits from these capabilities.

Calorimetry Parameters:
This key accesses its sub-menu which allows access to the calorimetry parameters used to automatically sequence a calorimetric test. These parameters are organized according to their use. The three types are:

- Tolerance (L) Parameters. These parameters are used primarily to establish appropriate criteria for transitioning from pre-period to post-period as well as ending a test.
- Correction (K) Parameters. These parameters are used for calorimeter temperature drift corrections.
- Blackout (B) Parameters. These parameters establish suitable blackout criteria for the post-period of the testing sequence.

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

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

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

Data Log Interval:
This key displays the interval of which the selected data is logged. The interval in seconds is defined in the Select Data Items sub-menu (normally 12 seconds). This roughly matches the update interval for the bucket temperature.
Data Log Destination:
Options are logfile, printer or both. When the logfile option is selected, the logfile
is located at /flash/datalog.csv. The maximum allowed size for this file is roughly
one megabyte. If the file reaches this size, logging is halted.

Select Data Log Items:
Press this key to access the Data Log Items sub-menu, which provides keys for
fifteen items that can be individually selected for logging. By default, both the
bucket and jacket temperatures are logged. All records are date and time
stamped. The most commonly helpful items to log are:
D0 - Corrected calorimeter drift rate.
Tsum - Accumulated temperature rise.
T1 - Extrapolated temperature rise.
C0 - Temperature conversion counter

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

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

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

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

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

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

Instrument Monitor:
This key accesses its sub-menu screen which provides a summary of most of the important
instrument parameters. This screen is used to detail the course of a test or to observe the
heating / cooling performance of the calorimeter.
User Defined Functions:
This key leads to a sub menu that offers five special purpose/ user/ factory definable function keys.

View System Info:
Press this key to display a screen with current operating system information / statistics such as:
- Processes and their associated PID$s
- Memory
- Mass Storage
- Network
Press the Print key to print this information.

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

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

Standardization
A sample of tris (hydroxymethyl) aminomethane, commonly called TRIS, is furnished with the 6755 Calorimeter to provide a reliable standardizing reagent. TRIS is furnished as a dry powder which can be used directly from the bottle as supplied without further preparation, but undue exposure to air and moisture should be avoided in order to preserve the integrity of the standard.

For standardizing the 6755 Solution Calorimeter, solid TRIS can be dissolved in dilute hydrochloric acid in a controlled reaction for which the amount of heat evolved is well established. In the recommended standardization procedure described below, 0.5 gram of TRIS is dissolved in 100 ml of 0.1 N HCl to evolve 58.738 calories per gram of TRIS at 25°C.

1. Tare the Dewar on a solution or trip balance and add exactly 100.00 + 0.05 grams of 0.100 N HCl.

2. Weigh 0.50 + 0.01 gram of TRIS into the 126C Teflon Dish on an analytical balance to an accuracy of +0.0001 g.

3. Assemble the rotating cell; place it in the calorimeter and start the motor.

4. Let the calorimeter come to equilibrium; then initiate the reaction by depressing the push rod.

5. Analyze the thermogram to determine the net corrected temperature rise, $\Delta T_c$. At the conclusion of the test the instrument will report a net corrected temperature rise, $\Delta T_c$.

6. Calculate the known energy input by substituting in the equation:

$$QE = m [58.738 + 0.3433(25 - T(0.63R))]$$

where:

- $QE$ = the energy input in calories
- $m$ = weight of TRIS in grams
- $T(0.63R)$ = temperature at point 0.63R on the thermogram

Note:

The term, $0.3433(25 - T(0.63R))$, adjusts the heat of reaction to any temperature above or below the 25°C reference temperature.
Calculate the energy equivalent of the calorimeter and its contents by substituting in the equation:

\[ e = \frac{QE}{\Delta T_C} \]

where:
- \( e \) is expressed in calories per °C.

Determine the energy equivalent of the empty calorimeter by subtracting the heat capacity of the 100 g of 0.1N HCl from \( e \), as follows:

\[ e' = e - (100.00)(0.99894) \]

where:
- \( e' \) = energy equivalent of the empty calorimeter in calories per °C.
- 100.00 = mass of 0.100N HCl in grams
- 0.99894 = specific heat of 0.1N HCl at 25°C

Example:
A standardization reaction involving 0.5017 grams of TRIS, and 100.00 grams of 0.100N HCl producing a net corrected temperature rise of \( \Delta T_C = 0.244°C \) with 0.63 rise, \( T(0.63R) \), at 24.301°C.

In this reaction the known energy input is:

\[ QE = 0.5017 \left[ 58.738 + 0.3433 (25 - 24.301) \right] \]
\[ = 29.589 \text{ calories} \]

The energy equivalent, \( e \), of the calorimeter and its contents is then computed:

\[ e = \frac{29.589}{0.244} \]
\[ = 121.27 \frac{\text{cal}}{°C} \]

The energy equivalent, \( e' \), of the empty calorimeter is then computed:

\[ e' = 121.27 - (100)(0.99894) \]
\[ = 21.38 \frac{\text{cal}}{°C} \]

Calculating the Energy Change
The energy change, \( Q \), measured in this calorimeter is calculated by multiplying the net corrected temperature change, \( \Delta T_C \), by the energy equivalent, \( e \), of the calorimeter and its contents.

\[ Q = (\Delta T_C)(e) \]

If \( \Delta T_C \) is measured in °C and \( e \) is expressed in calories per °C, \( Q \) will be reported in calories. (The energy equivalent, \( e \), is determined by a standardization procedure).
The change in enthalpy, $\Delta H$, at the mean reaction temperature is equal to $-Q$ divided by the amount of sample used in the experiment, expressed either in moles or grams.

$$\Delta H_T = \frac{-Q}{m}$$

where $T$ is the temperature at the 0.63R point on the thermogram.

Enthalpy values are usually expressed in kilocalories per mole.

 Procedures for converting enthalpy changes, $\Delta H$, to thermodynamic standard conditions and for using $\Delta H$ in other computations can be obtained from thermodynamics or thermochemistry textbooks, or from literature references.

**Reading the Thermogram**

![Thermogram Diagram](image)

In order to determine the net temperature change produced by the reaction, it is necessary to interpolate a point on the thermogram at which the temperature reached 63 percent of its total rise. This can be done easily by following Figure 2, although other variations of this method can be used as well.

1. Place a straight edge over the preperiod drift line and extend this line well past the point at which the reaction was initiated.

2. Move the straight edge to the postperiod drift line and extrapolate this line backward to the firing time. If there are fluctuations in the drift lines due to noise
or other variations in the signal, use the best average when drawing these extrapolations.

3. Using a centimeter scale, measure the vertical distance, \( R \), between the two extrapolated lines at a point near the middle of the reaction period.

4. Multiply the distance, \( R \), by 0.63.

5. Set the zero end of the centimeter scale on the extrapolated preperiod drift line and move the scale along this line to locate a vertical intercept with the thermogram which is exactly 0.63\( R \) above the preperiod drift line. Draw a vertical line through this point to intercept both drift lines.

6. Read the initial temperature, and the final temperature, at the points of intersection with the drift lines and subtract to determine the corrected temperature rise, \( \Delta T \) (see Figure 2)

\[
\Delta T_c = T_f - T_i
\]
Example - Exothermic Reaction
Problem: Determine the change in enthalpy for solid sodium sulfate, Na$_2$SO$_4$, when dissolved in a 5 gram/liter aqueous solution of barium chloride, BaCl$_2$·2H$_2$O.

Na$_2$SO$_4$ m = 0.1458 grams  
Ba$^{++}$ solution = 100.00 grams

Corrected temperature rise
\[ \Delta T_C = 0.042^\circ C \text{ (from Figure 3)} \]
\[ T(0.63R) = 24.885^\circ C \]

Energy equivalent
\[ e = 121.46 \text{ cal}^\circ C \]

Energy evolved
\[ Q = (\Delta T_C) (e) \]
\[ = (0.042) (121.46) \]
\[ = 5.1013 \text{ calories} \]

Enthalpy change
\[ \Delta H_T = \frac{-Q}{m} \]
\[ = \frac{-5.1013}{0.1458} \]
\[ = -34.99 \text{ cal/g @ 24.885}^\circ C \]

Or, multiplying by 142.04 (the molecular weight of Na$_2$SO$_4$)
\[ \Delta H_T = (-34.99) (142.04) \]
\[ = -4.970 \text{ Kcal/mole @ 24.885}^\circ C \]

Figure 3
Exothermic Reaction

0.1458 g solid Na$_2$SO$_4$ dissolved in 100.00 g of 5 g/l aqueous BaCl$_2$·2H$_2$O

Full scale = 10 mv = 0.1°C  
Baseline = 24.852°C

T$_f$ = 0494 + 24.852  
T$_i$ = .0074 + 24.852  
\[ \Delta T_C = .0420^\circ C \]

T(0.63R) = .0333 + 24.852  
\[ = 24.885^\circ C \]
Example - Endothermic Reaction

Problem: Determine the heat of solution of solid potassium nitrate, KNO₃, when dissolved in water.

\[
\begin{align*}
\text{KNO}_3 \text{ m} & = 0.7180 \text{ gram} \\
\text{Distilled water} & = 100.0 \text{ grams}
\end{align*}
\]

Corrected temperature rise

\[
\begin{align*}
\Delta T_c & = -0.508^\circ \text{C} \text{ (from Figure 3)} \\
T(0.63R) & = 25.400^\circ \text{C}
\end{align*}
\]

Energy equivalent, \(e\) = 121.46 cal/°C

Energy evolved \(Q\) = \((\Delta T_c)(e)\)

\[
= (-0.508)(121.46)
= -61.70 \text{ calories}
\]

Enthalpy change \(\Delta H_T\) = \(-\frac{Q}{m}\)

\[
= -\frac{-61.70}{0.7180}
= 85.94 \text{ cal/g @ 25.400°C}
\]

Or, multiplying by the molecular weight of KNO₃

\[
\Delta H_T = (85.94)(101.10)
= 8.96 \text{ Kcal/mole @ 25.400°C}
\]

Figure 4

Endothermic Reaction

0.7180g solid KNO₃ dissolved in 100.00g of distilled water

\[
\begin{align*}
\text{Full scale} & = 100 \text{ mv} = 1.0^\circ \text{C} \\
\text{Baseline} & = 25.000^\circ \text{C} \\
T_f & = 0.190 + 25.000 \\
T_i & = 0.698 + 25.000 \\
\Delta T_c & = -508^\circ \text{C} \\
T(0.63R) & = 0.400 + 25.000 \\
& = 25.400^\circ \text{C}
\end{align*}
\]
REPORTS

The 6772 Calorimetric Thermometer can transmit its stored test data in either of two ways. The Auto Report Destination key on the Reporting Controls Menu toggles the report destination between the display and an optional printer connected to the RS232 printer port of the Calorimetric Thermometer. 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 Status  This key enables the operator to display five report options:
- only preliminary and final reports
- only final reports
- only preliminary reports
- only pre-weighed sample reports
- all stored reports.

The displayed files can be sorted by filename (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 report to the display, press the DISPLAY key. To send the reports to the printer press the PRINT key.
MEMORY MANAGEMENT

The 6772 Calorimetric Thermometer will hold data for 1000 tests in its memory. These tests may be pre-weights, preliminary or final reports for either calibration or determination runs. Once the memory of the controller is filled, the controller will not start a new analysis until the user clears some of the memory.

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

The controller of the 6772 Calorimetric Thermometer can accept compact flash memory cards. These cards can be used to:

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

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

Examine the rotating cell periodically for leaks by closing the cell and submerging the empty bell in a beaker of water for a period of time comparable to a complete run in the calorimeter. If water migrates into the sealing area between the PTFE dish and the glass bell, the seal is deteriorating and the 126C PTFE dish should be replaced.

After one hundred hours of operation, or once each year, apply one drop of instrument oil or household oil to the motor bearing, and one drop of oil to the bronze stirrer shaft bearing. Be sure to remove any excess oil that may appear at the bottom of the shaft.

There is no routine maintenance recommended for the 6772 Calorimetric Thermometer.

Fuses

The replacement of protective fuses for the 6722 Calorimetric Thermometer should be performed by qualified personnel.

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

Main line protective fuses. Rated Fast-Act 15 Amp, 250VAC (Parr number 139E23)
Fuse F1– Mains stirrer output fuse. Rated Fast-Act 1 Amp, 250VAC, time lag, 5 x 20mm (Parr number 1641E)

This fuse is located on the A1794E I/O board located inside the instrument. To gain access to the inside, first disconnect the power cord from the rear of the unit. Remove the two screws at the upper rear of the unit that secure the front cover. Pry up on the back lip of the cover and carefully hinge it forward. Make sure that the cables attached to the front portion of the cover do not become stretched or disconnected. Follow this procedure in reverse order to re-secure the front cover.

Conversion from 6755 to 6725

All parts needed for this conversion are provided in a 6729 Conversion Set. Installation should proceed step-wise, as follows:

Remove the A1195E2 Thermistor, A130C Cell and A137C Pulley Assembly from the calorimeter cover.

Remove the 123C Dewar and 122C Ring from the air can. Attach the A137C Pulley Assembly with 3/8" I.D. washer from the solution calorimeter cover to the bearing tower on the A120C3 Cover furnished with the conversion set. Attach the A72C2 Impeller Assembly to the drive shaft. Then place the cover assembly on the air can so that the cover is centered in the air can and the alignment slot straddles the alignment pin.

Replace the drive belt, attaching it first to the drive pulley and then onto the driven pulley.

The calorimeter is now ready to operate as a Semi-micro Oxygen Bomb Calorimeter, using instructions furnished with the 6729 Conversion Set.
TROUBLESHOOTING

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

A Misfire Condition Has Been Detected. This error will be generated in the event the total temperature change fails to exceed 0.5°C after the first minute of the post-period.

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

- A cell leak.
- Poor bucket stirring.
- Lid not tight.
- Initial Dewar temperature outside the acceptable range.

The Current Run Has Aborted Due To Timeout. The calorimeter has failed to establish an acceptable final temperature within the time allowed. Possible causes for this error are listed below:

- A cell leak.
- Poor bucket stirring.

There Is A Problem With The Bucket Thermistor. Possible electrical open or short. This error will result if the temperature probe response is not within the expected range. Probe substitution can be useful in determining the cause of the problem (probe or electronics). The valid working range of the probe resistance is 1000 to 5000 ohms.

- Check connection.
- Replace probe.

There Is A Problem With The Jacket Thermistor. Possible electrical open or short. This errors will result if the temperature probe response is not within the expected range. Probe substitution can be useful in determining the cause of the problem (probe or electronics). The valid working range of the probe resistance is 1000 to 5000 ohms.

- Check connection.
- Replace probe.

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.
TECHNICAL SERVICE

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

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

Any correspondence must include the following basic information:

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

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

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

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

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

Ship repair to:

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

Principal Assemblies in 6772 Calorimetric Thermometer

<table>
<thead>
<tr>
<th>Part Number</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1940E</td>
<td>Power Supply</td>
</tr>
<tr>
<td>A1792E</td>
<td>Display Transition Board</td>
</tr>
<tr>
<td>A1993E4</td>
<td>CPU Board</td>
</tr>
<tr>
<td>A1794E</td>
<td>I/O Board</td>
</tr>
<tr>
<td>1803E</td>
<td>Backlight Inverter</td>
</tr>
<tr>
<td>A1808E2</td>
<td>Ribbon Cable</td>
</tr>
<tr>
<td>A1809E2</td>
<td>Ribbon Cable</td>
</tr>
<tr>
<td>A1876E</td>
<td>Touch screen LCD with Cable</td>
</tr>
<tr>
<td>A719E</td>
<td>Cord set, 115V</td>
</tr>
<tr>
<td>1200EEE</td>
<td>Corset, 220V, EU Plug</td>
</tr>
<tr>
<td>1889E</td>
<td>Film, Display Protection</td>
</tr>
<tr>
<td>139E23</td>
<td>Fuse Fast/ Act 15 Amp 250V</td>
</tr>
<tr>
<td>1641E</td>
<td>Fuse Fast/ Act 1.0 Amp 250V, 5 x 20 mm</td>
</tr>
</tbody>
</table>

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

Parts for the 6755 Solution Calorimeter

<table>
<thead>
<tr>
<th>Part Number</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>A338CEB</td>
<td>Stirrer Drive Motor Assembly 115V</td>
</tr>
<tr>
<td>A338CEE</td>
<td>Stirrer Drive Assembly 230V</td>
</tr>
<tr>
<td>A120C</td>
<td>Calorimeter Cover with Stirrer Hub &amp; Bearing Assembly</td>
</tr>
<tr>
<td>122C</td>
<td>Locating Ring (6755)</td>
</tr>
<tr>
<td>123C</td>
<td>Dewar Flask 150 mL</td>
</tr>
<tr>
<td>126C</td>
<td>Sample Dish, PTFE</td>
</tr>
<tr>
<td>128C</td>
<td>Hollow Shaft</td>
</tr>
<tr>
<td>A129C</td>
<td>Push Rod Assembly</td>
</tr>
<tr>
<td>A130C</td>
<td>Sample Cell with Coupling, less 126C Dish</td>
</tr>
<tr>
<td>132C</td>
<td>Bushing for 133C Stirrer Pulley</td>
</tr>
<tr>
<td>133C</td>
<td>Stirrer Pulley</td>
</tr>
<tr>
<td>135C</td>
<td>Drive Belt, Geared</td>
</tr>
<tr>
<td>139C</td>
<td>O-ring, Buna N 2-11/16 ID x 3/32 CS</td>
</tr>
<tr>
<td>752HC2</td>
<td>Air Can, Stainless Steel</td>
</tr>
<tr>
<td>A1195E2</td>
<td>Thermistor Probe Assembly</td>
</tr>
<tr>
<td>3421</td>
<td>Tris (Hydroxymethyl) Aminomethane, Parr Standard for Solution Calorimetry, 100 Gram Bottle</td>
</tr>
<tr>
<td>A137C</td>
<td>Pulley Assembly</td>
</tr>
</tbody>
</table>
DRAWINGS

6772 Schematic
Internal Parts Views of 6772 Calorimetric Thermometer
Internal Parts Views of 6772 Calorimetric Thermometer
Back Panel of 6772 Calorimetric Thermometer

(2) SA1332RD06
6-32 X 3/8 RHMS

327C BACK PANEL REF

SA1332RD06
6-32 X 3/8 RHMS

POWER INPUT

IGNITION

POWER

ETHERNET

BALANCE

TERMINAL

TERMINAL

PRINTER

JACKET BUCKET

ETHERNET

POWER

SA1332RD06
6-32 X 3/8 RHMS

MOTOR
External Parts View of 6755 Solution Calorimeter
Internal Parts View of 6755 Solution Calorimeter
TABLES

Table 1 – Default Settings

**Note:** The following are recommended operation settings for the 1341 Plain Jacket Calorimeter; see the Operation section of this manual for the appropriate parameter changes for use with the 6725 Semi-micro Calorimeter.

**Calorimeter Operation**
- Stirrer: OFF

**Operating Controls**
- Method of Operation: Solution
- LCD Backlight Timeout(s): 1200 S
- LCD Contrast: 30%
- Print Error Messages: ON
- Language: English

**Program Info and Control**
- Settings Protect: OFF

**Data Entry Controls**
- Auto Sample ID Controls: ON

**Auto Sample ID Controls**
- Automatic Sample ID: ON
- Automatic Sample ID Number: 1
- Automatic Sample ID Increment: 1

**Reporting Controls**
- Report Width: 40
- Auto Report Destination: Printer
- Individual Printed Reports: OFF

**Communication Controls**

**Printer Port**
- Number of Data Bits: 8
- Parity: None
- Number of Stop Bits: 1
- Handshaking: None
- Baud Rate: 9600
- Printer Type: Parr 1757

**Balance Port**
- Number of Data Bits: 8
- Parity: None
- Number of Stop Bits: 1
- Handshaking: None
- Baud Rate: 9600
- Data Characters from Balance: 8
- Data Precision: 4
- Transfer Time Out: 10

**Diagnostics**

**Calorimetry Parameters**

**Tolerance (L) Parameters**
- Preperiod Tolerance (L1): 0.002
- Misfire Tolerance (L2): 0.05
- Dynamic Post period Tolerance (L3): 0.002
- Eqlbrm Post period Tolerance (L4): 0.002
- Preperiod Stability (L5): 2
- Preperiod Blackout (L6): 36
- Derivative Wghtng Cutoff (L7): 0.02
- Preperiod Timeout (L8): 42
- Postperiod Timeout (L9): 60
- Preperiod Tolerance Jck Calc’d (L10): 0.5

**Correction (K) Parameters**
- K1: 0.5
- K2: 0.00036
- K3: 1.0
- K4: 0.0
- K5: 0.0

**Blackout (B) Parameters**
- Misfire Blackout (B2): 6
- Derivative Blackout (B3): 0.5
- Dynamic Blackout (B4): 6
- Equilibrium Blackout (B5): 48
- Dynamic Derivative Blackout (B6): 0.02
- Dynamic Time Blackout (B7): 20

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