



Laboratory Equipment Manufacturer
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Operation Manual for Bomb Calorimeter **CALO-11/11A/13/15**



PLEASE READ THIS MANUAL CAREFULLY BEFORE OPERATION

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MRC.VER.01-2.12

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Chapter 1 Instrument Performance and Characteristics

1. 1 Scope of Application

This instrument is suitable for application to such industries or sectors as electric power, coal, paper-making, petrochemical industry, cement, agriculture & husbandry, pharmaceuticals, scientific research and teaching for measuring of calorific values of combustible of coals, coke, petroleum and cement raw materials.

1. 2 Performance Index

Temperature-measuring range	5-44°C
Resolution	0.0001°C
Heat capacity stability	≤0.15%

1.3.1 High automation and High efficiency

1. Automatic water temperature adjustment and water weighing as well as automatic measuring of total water volume.
2. External balance weighing connected to the calorimeter, automatic send and save and pronounce by voice the sample weight, the operation is simple and reliable.
3. The jacket water and the testing water is separated and the jacket water temperature is always higher than the bucket water temperature and the difference is relatively constant, thus to isolate the influence of the environment to the bucket.
4. Easier operation and convenient maintenance;
5. Double-head oxygen bomb. Firing wire is tied by sleeve-pressure method which is convenient, safe and reliable.
6. Automatic identification of the heat capacity corresponding to 100 oxygen bombs.
7. Test time per sample of CALO-11 is less than 11 mins, CALO-13 less than 13 mins, CALO-15 less than 15 mins, which is fast, of strong adaptability to the environment and high efficiency.

1.3.2 High Precision and Accuracy

The system is of high precision and accuracy based on standard operation.

1.3.3 Easy operation

1. Light and aesthetic.
2. The instrument is controlled by only one switch.
3. It prompts in English characters in the complete process, test completion just by prompt-based operations.
4. Software featuring good error tolerance. No worry about wrong operations.
5. Convenient calculation and printing of gross/net calorific values.
6. Capable of multi-control and will not be interfered by each other. Automatically identify the parallel samples and calculate the average and error value. Capable of multi-copy printing.
7. Rich inquiry functions, supportive to ambiguous inquiry.

1.3.4 Diagnosis system

1. During tests, the system has self-diagnosis function, which can accurately judge each

functional part of the system. It also prompts the user clearly so as to facilitate the maintenance and ensure normal operation of system.

2. Watch-dog and mechanical protection devices are capable of providing whole-process protection to effectively avoid abnormal incidents.

1.3.5 Asynchronous multi-control

Test software of ECNQ/11 calorimeter is acceptable to multi-control. Each principal machine of multi-control calorimeter is completely independent from one another without mutual interference, which can reach the performance of single -control calorimeter.

1.3.6 Operating Platform

The test-control program operates on Windows XP.

Chapter 2 Instrument Composition and Working Principle

2. 1 Instrument Composition

The ECNQ calorimeter is mainly composed of ECNQ calorimeter principal machine, ECNQ-YD oxygen bomb, ECNQ-CYQ micro-type oxygen filler, ECNQ test-control interface, ECNQ test-central software, computer and printer (refer to Diagram.2-1). In addition, users can have such optional devices as electronic balance, balance interface, ECNQ balance weighing software and relevant loginories.

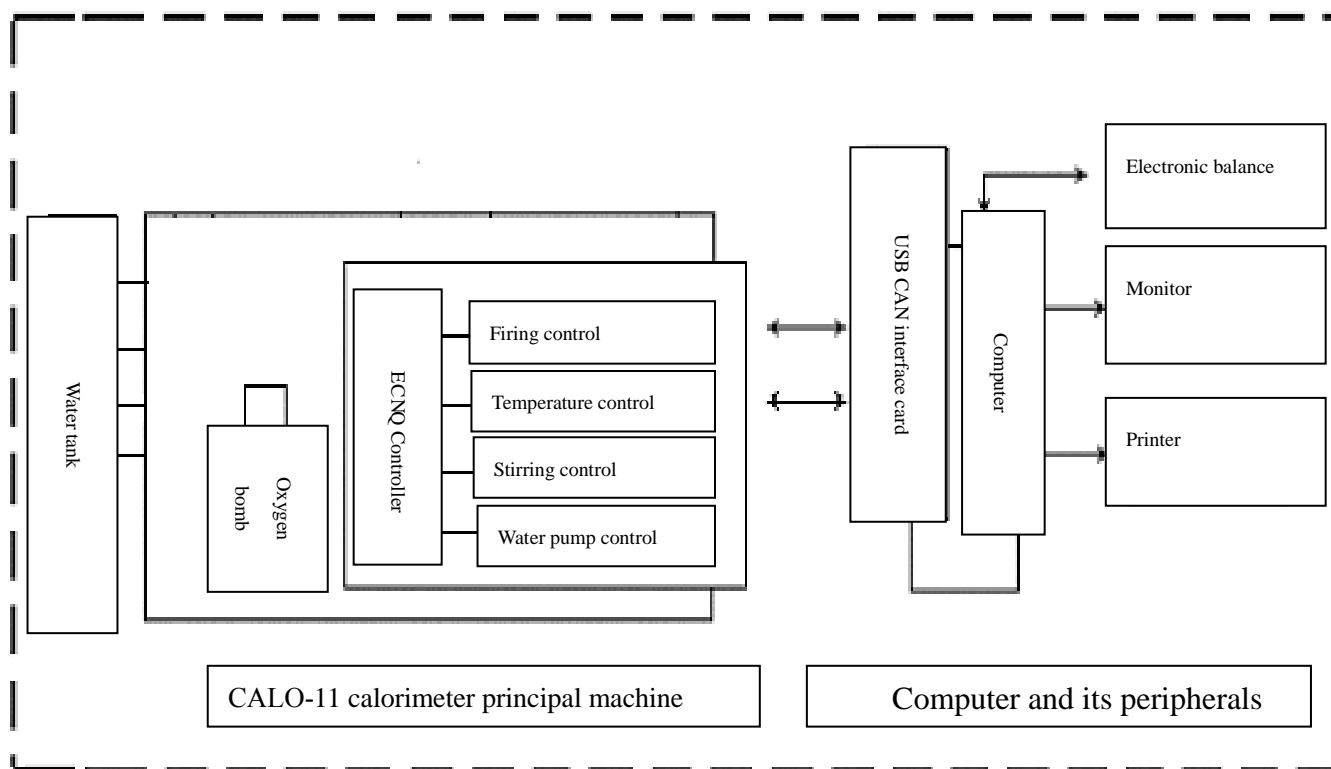


Diagram 2-1

2.1.1 CALO calorimeter principal machine

CALO calorimeter principal machine is composed of bucket, jacket, constant volume tank, water tank, driving pump, heating parts, temperature testing parts and other controlling and protecting parts;

1. Structure diagram(refer to Diagram 2-2a、 2-2b).
2. Brief introduction of the inner parts:
 - Bucket: Fix the oxygen bomb and circulate water around the bomb and replace the testing water.
 - Jacket: Provide a stable environment for the bucket, to avoid the influence of the ambient temperature. The spray stirring way makes sure the outer jacket water temperature controllable and uniform.
 - Constant volume tank: To measuring the volume of the bucket water.
 - Water tank: Be able to contain 22KGS water, equipped with heat dissipation and water judgement devices.
 - Heating parts: External heating mode, to heat up the jacket water; the constant volume

tank heating, to adjust the temperature of the testing water.

- Temperature testing parts: To test the temperature of the bucket, jacket, constant volume tank, with controlling and protecting parts.

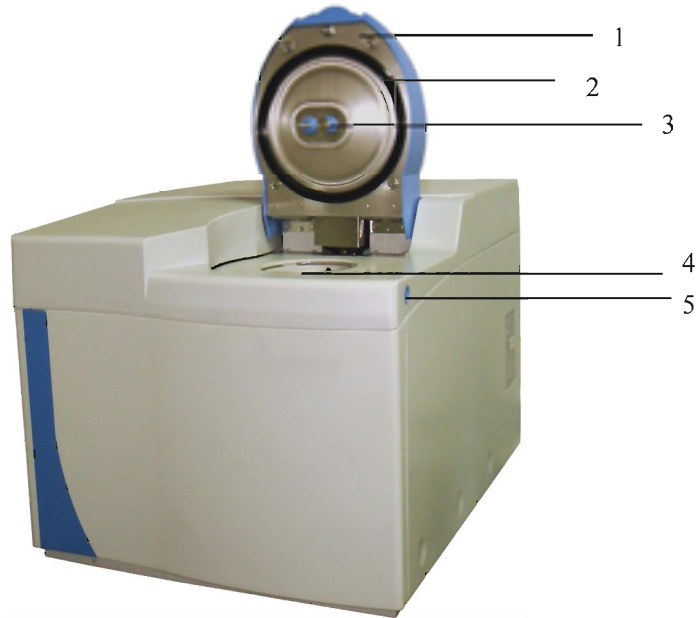


Diagram 2—2 Diagram of CALO-11/CALO-13 Calorimeter Principal Machine

- 1 — bucket cover 2 — sealing ring 3 electrode rod
4 — bucket 5 — fixing bolt of bucket cover

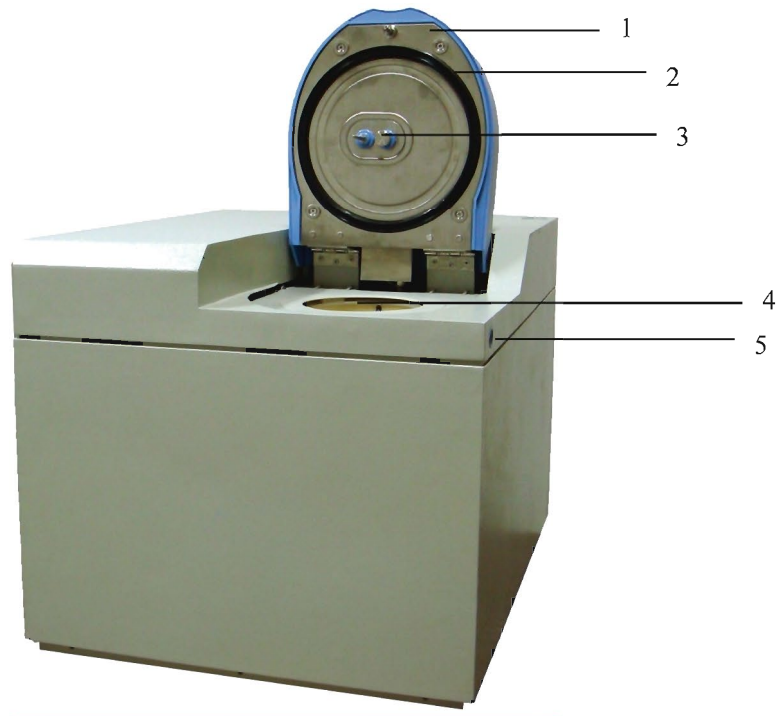


Diagram 2—2b Diagram of CALO-15 Calorimeter Principal Machine

- 1 — bucket cover 2 — sealing ring 3 electrode rod
4 — bucket 5 — fixing bolt of bucket cover

2.1.2 ECNQ—CYQ Micro-type Oxygen Filler

1. **Structure diagram**(refer to Diagram 2-3)
2. **Characteristics:** Simple structure and easy operation. Gas passage is well sealed and rubber sealing ring can be changed easily.

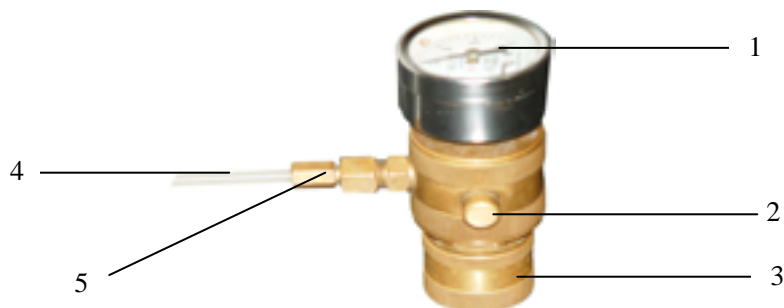


Diagram 2—3 Micro-type Oxygen Filler
 1 —— pressure gauge 2 —— switch 3 —— pressure sleeve
 4 —— oxygen tube 5 —— lock nut

2.1.3 ECNQ-YD oxygen bomb

1. **Structure Diagram** (refer to Diagram 2-4, 2-5)
2. **Characteristics:** Diaphanous appearance, small size, light weight. Firing wire is tied by sleeve-pressure method, which is convenient, safe and reliable.

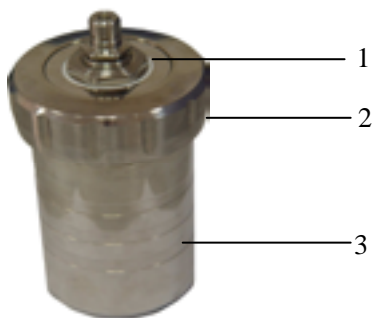


Diagram 2-4a ECNQ/11/ECNQ/13 Oxygen bomb appearance
 1 —— oxygen bomb core 2 —— oxygen bomb cover
 3 —— oxygen bomb cylinder



Diagram 2-4b ECNQ/15 Oxygen bomb appearance

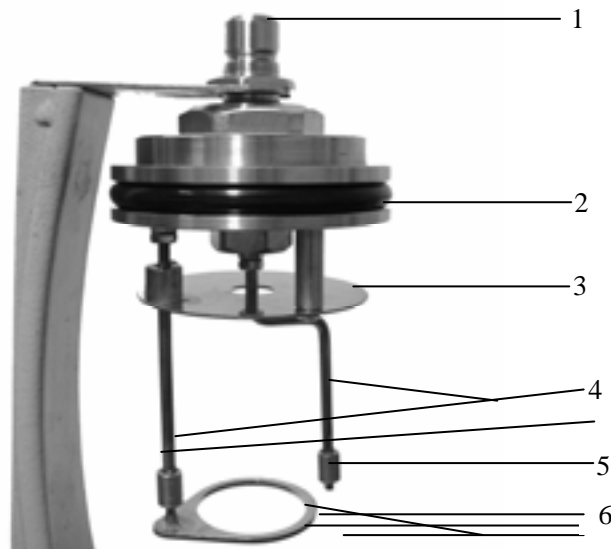


Diagram 2-5 Inner structure of oxygen bomb (oxygen bomb core)

- | | | |
|----------------------|-------------------------------|-----------------|
| 1 — air nozzle | 2 — sealing ring | 3 — fire baffle |
| 4 — electrode rode | 5 — firing wire pressing ring | |
| 6 — crucible support | | |

(Note: 3、4、5、6 can be called crucible support components)

2. 2 Working Principle

The isothermal bomb calorimeter features the jacket water temperature keeping constant, and judge the finishing of the test by the bucket temperature changes. Then correct the heat exchange between the bucket and jacket by the cooling correction formula. It can be used to test the calorific value of solid or liquid combustibles such as coal, coke, biomass, petroleum and so on. The main principle is: weighing some weight of the sample, place it into the bomb, and fill sufficient oxygen into the bomb, and place the bomb into a bucket with some volume of water, burn the sample completely in the bomb and the heat released by the sample are absorbed by the water around the bomb. Calculate the raised water temperature. When the jacket water temperature is constant, the heat capacity of the absorbing water and other parts in the water is constant. The calorific value can be calculated by the raised water temperature.

2. 3 Working process

After powering on and entering the test-control system, the system will do the necessary self-diagnosis and then to balance the temperature, after that the system will enter into test mode. Weighing some weight of sample in the crucible and put it into the bomb, fix the firing wire and filling the oxygen into the bomb, after(45~60)s of oxygen filling, then put the bomb into the bucket. Input the sample weight, the system will automatically start the test. First, the constant-volume water will be heated up, and bucket water feed and the bucket begins to stir, after a time of balancing,

the system will enter into the initial period of the test, then ignite, primary period, the finish period, calculate the test result, bucket water drain and complete the test process. During the test, the other parts will synchronously or asynchronously control the system.

During test process, if any part of the system works abnormally, prompts message will be displayed on the screen so that the user can make a simple maintenance.

Chapter 3 System Installation and Test-run

3. 1 Installation Description

3.1.1 Software Condition:

1. Operation system: Windows XP (English Version);
2. Basic Configuration:

CPU	over 1.79GHz
Memory	over 256MB
Video card	Standard VGA 1024X768 mode
Hard disc	20 GB required at least
Drive	CD-ROM drive
Other equipment:	mouse, keyboard

3. 2 Installation Process

3.2.1 Pre-installation Preparations

1. Prepare suitable special-purpose laboratory.
2. Prepare oxygen bottle. Oxygen purity and the oxygen pressure inside the bottle should conform to the standard.
3. Prepare about 50 Kg distilled water or deionizer water.
4. Prepare analysis balance with sensitivity no lower-than 0.1 mg, and associated drying utensils.
5. Prepare nippers, scissors, spanners, screw-drivers, pincers, angle spoons and other basic tools.

3.2.2 Layout

1. Typical Layout: Place computer (principal machine, monitor and keyboard) in the middle and with printer and ECNQ/11 calorimeter on their left and right side for convenient operation.
2. While connection between water tank and principal machine of calorimeter, place the calorimeter firstly. Then place the water tank under the table (it is necessary to ensure that water tank is below the principal machine of calorimeter). If the calorimeter is CALO-11A vertical type, it's no need to place the water tank.

3.2.3 The connection of the calorimeter

1. Check the fuse box of the instrument power supply socket to see if the fuse tube has been installed. Check the instrument to see if short-circuiting has occurred among the machine shell, power line and ground line. Check the voltage at instrument installation site and check the instrument case for reliable ground. Connect the three cord power with the calorimeter.
2. Open the shell and cover of the calorimeter and take out the puffs and other fillings out

- between the bucket and the shell.
3. Connection of USBCAN interface card. Connect the USB port and CAN of the calorimeter with the computer and fix it with screws.
 4. Connection of the card reader (bomb identifier): Connect nine-cord interface of the card reader with the COM1 or COM2 interface of the computer, and the round interface with the mouse interface of the computer.
 5. Connection of the purifier: Connect the OUT end of the purifier with the calorimeter OUT end and the IN end with the calorimeter IN end. Pay attention that the length of the tubes should be suitable.

Note: The purifier should be replaced once a year.

6. Connection of the water tank: Connect one end of PU tube with the quick coupling of the I/O port of the calorimeter, the other end with the quick coupling of the water tank. The black water hose is covered outside the PU tube, one end to be connected with the I/O port and fix with the clamp, the other end to be connected with the black port of the cover of the water tank.
7. Connect the water level signal wire with the calorimeter.
8. Fix the stirring filter screen after cleaning.
9. Switch on power supply and start computer to set up test-control software.

3.2.4 The installation and uninstallation of the software

3.2.4.1 The installation of the software

1. Test-control software of CALO calorimeter is stored in a CD.
2. Check if the message que has been installed. (Method: Start→Control Panel→Add/Delete program→Add/Delete components→Select “message que” in the component list→Click Next, and install as per the prompt)
3. Insert the CD marked “Test-control Software of CALO Calorimeter” into the CD-ROM. Use “Resource Manager” to find CD-ROM and open it. Find “Setup” icon under root directory.
4. Click “Setup” icon two times, install the program of CALO Calorimeter and USBCAN service program according to the prompt. After finishing the installation, the system will generate the shortcut of CALO Calorimeter on the desktop.
5. Right click “My computer” →Property→ Hardware→ Equipment manager→ Click “+” under, If you can see “MRC USB CAN”, it shows that USB-CAN card and driving program have been installed. Or else, click “Scan” and reinstall the program according to the prompts.
6. After finishing the above steps, the software has been installed.

3.2.4.2 The uninstallation of the software

Click Windows “Control Panel” by mouse, then click “Add/Delete Program” twice and open this window. Choose “Change or Delete Program” menu, then click “CALO Calorimeter” In program group, then click “Delete” button to unload the program. Program group and shortcut of CALO calorimeter system can be deleted safely and quickly according to the prompts on the screen. However, related parameter files and database files of CALO calorimeter system couldn't be uninstalled and will remain in the operating system.

3.2.4.3 Authorization

After the hardware and software are installed, double click “CALO Calorimeter” icon on the

desktop to enter the test- control program, press “F6” key (or, click “Start” in the taskbar → “All Programs” → “MRC” → “USBCAN” → “MRC USBCAN service program”) to open “MRC USB CAN service program” window, select the bucket number, click “Tools” in the menu bar → “Authorization”, feedback the contents of test boxes “Card Verification Code” and “Local Verification Code” in the “Authorization” window to MRC for authorization, and then input or copy the authorization code given by the company to the text box of “Authorization Code”, click “Add”, the prompt shows it has successfully authorized to a date, indicating a successful authorization.

3.3 Debugging

After the installation of the hardware and software, and successfully authorized, fill sufficient water into the water tank, and then enter into the debugging step. Double click the icon of the “CALO Calorimeter” on the desktop to enter into the test-control program, click: Tools → “System test” to do the following operations:

3.3.1 Jacket water feed

Open the cover, click “water makeup test”, the system will automatically filling water of the jacket and the cover in a spiral circulation way (At the same time please check if the operations of the pumps and valves are correct, and if there is any water leakage at the connection of the tubes.) When the water run out from the warning pipe of the jacket, it shows the jacket is fully fed and stop water feeding by click “stop water makeup” and then click “water drain test” to circulate the jacket water for about 5 minutes and check if the pumps and valves are correctly operated and if there is any water leakage.

3.3.2 Constant volume test、 Bucket water feed、 Bucket water drain

Click “Constant volume test”→“Bucket water feed”→“Bucket water drain”, after repeat it for three times, then click the “Constant volume test”, check if there is any bubbles and if it can be filled full, (The time for fully filled is more than 2 seconds), if the water pressure is normal, if the pumps and valves are correctly operated, if there is any water leakage at the connection of the tubes. If everything is in order, click “Bucket water feed” after the air drain constant volume metering (about 15s), check if there is any bubbles between the pump and the bucket, and after the first bucket water feed if it can be drained completely, if the stirring is in order, if there is any water leakage at the joint. After that, click “Bucket water drain”, Check if the pumps and the valves are correctly operated, if the bucket can be drained completely and if there is any water leakage at the joint.

3.3.3 Add water

There are two methods for the water feeding of CALO calorimeter:

Method 1, directly feed water at water tank cap. For this method, it’s mainly used when debugging, the debugging technician will check if the water level is suitable(the water level is 45mm lower than the cover) after the jacket water feeding and constant volume test, or else water should be added.

Note: The debugging technician should check the water level before the end users operate it.

Method 2, Feed water into the bucket after opening the cover. Enter the test-control software, if the system prompt “water insufficient (in the water tank)”, the progress indication main window will show the water insufficient information, and automatically prompt window for feeding water. The operator needs only to feed water into the bucket slowly. During this, the

system will automatically finish the water feeding to the jacket, constant volume tank, and then bucket drain. If the water level is normal, the water drain valve and pump will stop and give prompts to the operator to stop water feeding. After 5 seconds, the system will automatically start the temperature balance, to provide a reliable environment temperature. After the temperature balance, tests can be done. (When stop water feeding, if there is any water left in the bucket not draining back to the water tank, please don't worry, during the temperature balance, the system will automatically conduct the bucket water drain.

When the water level is normal of daily use, there is no need to add water every day. To open the water tank cover and add water according to the prompts, thus to make sure it is at the most suitable level.

3.3.4 Online test

After overall completion of hardware and software installation, cover the shell and carry out system on-line test. If no abnormal matter happens, it will be regarded as installation completion after testing a group of qualified heat capacity and calorific value test. If not, carry out itemized inspection of hardware installation and software setup according to malfunction-shooting procedure. Carry out correct malfunction-shooting or reinstallation just in case.

3.3.5 Usage of Oxygen Bombs

3.3.5.1 Pre-use Check

Strict quality inspection has been done for oxygen bomb before delivery. It is unnecessary for the user to dismantle it. But quality re-inspection is essential. Following items shall be checked:

1. Check the bomb is clean if there is any scrap irons, greasy dirt or impurity.
2. Check oxygen nozzle and the insulation sleeve of the bomb core to see if it's loosened.
3. Check if the electrode rods are loosened and fire baffles (round stainless steel sheet) are tightly fixed.
4. Check if the crucible support is properly fixed.
5. Check if oxygen inlet hole is smooth passage (bomb core).
6. Check the threads on oxygen bomb cylinder and bomb cover as well as sealing rings are with foreign matters.
7. After oxygen is filled, put the oxygen bomb into water. In case of bubble observed, it means that this oxygen bomb leaks.
8. Check the bomb appearance if there are collision traces

3.3.5.2 Use of Oxygen Bomb

1. Hang the oxygen bomb core onto the bomb support.
2. Place the dried cold crucible on the weigh pan of electronic balance to measure its weight. Tare and reset it to zero.
3. Place the thoroughly mixed sample into already weighed crucible by clean nippers and sample ladle (coal sample shall be uniformly mixed). Record its mass. Place sample-containing crucible onto crucible support of oxygen bomb.
4. Connect both ends of firing wire with two electrode rods of oxygen bomb and press them tightly by gland ring. Firing wire is prohibited to contact crucible and break.
5. Bend the wire to circular arc shape to make firing wire close to or slightly contact sample. Do not insert the firing wire into coal if it's coal sample.
6. After firing wire is installed, stably place the oxygen bomb core into oxygen bomb cylinder

- filled with 10ml of distilled water, then fasten the oxygen bomb cover.
7. Oxygen filling, please refer to Chapter 3.3.6.2.
 8. Stably place the oxygen bomb filled with sufficient oxygen into the bucket of instrument, and then fasten the bucket cover.
 9. After test, open the bucket cover and take out the oxygen bomb, then release residual oxygen from oxygen bomb through release valve and wash it by the distilled water or deionized water, make sure there is no firing wire and dirt left in it. At last, clean the parts of the bomb by the bomb towel.

Caution:

1. Please handle the bomb with care.
2. Fire baffle is forbidden from short-circuiting to two electrode rods.
3. Oxygen bomb must be cleaned and wiped up by a special-purpose towel before and after each test.
4. Before installing firing wire each time, completely clean the firing wire and other foreign matters remaining on electrode rods and inside the gland ring.
5. It is forbidden to fill oxygen at over pressure (normal pressure 2.8~3Mpa). Keep relatively unified filling time (30~45sec).
6. Oxygen bomb cover shall not be over-tightened. After it reaches the position, slightly fasten it.
7. Inspect oxygen bomb for quality at regular interval. Carry out hydraulic test at least once a year (to be presented to Sundry for inspection and testing).
8. An ID card is installed at the bottom of oxygen bomb for identification. It can be removed for cleaning.

3.3.6 Installation and usage of micro-type oxygen filler

3.3.6.1 Installation and inspection

1. Carefully inspect each component for fixing and appearance for damage before installation.
2. Connect pressure-reducing valve, oxygen bottle and oxygen filler as per Diadram.3-1. Then fasten the lock nut.
3. Turn on oxygen bottle main valve, regulate pressure-regulating screw lever on pressure reducer to make low pressure gauge reading be 2.8~3Mpa. Be sure of no leakage throughout the gas circuit, otherwise it shall be re-installed until normal pressure is indicated.
4. Carry out oxygen filling at a trial basis. Be sure of no leakage and easy operation at this stage. Pressure indication on oxygen filler shall be same on the whole as low pressure gauge indication on pressure-reducing valve.

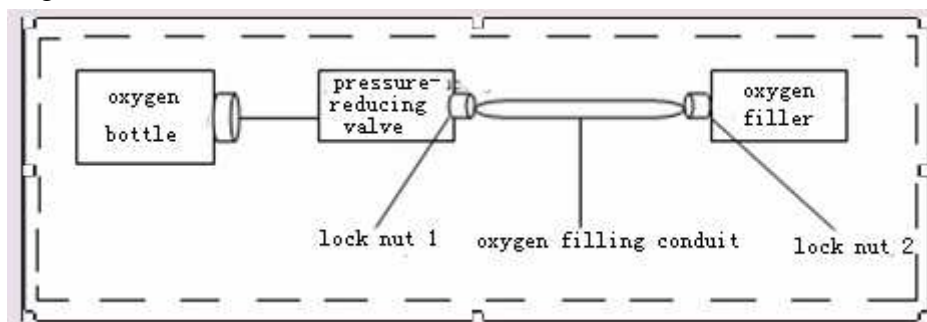


Diagram 3-1

3.3.6.2 Usage

1. Open oxygen bottle valve (according to high pressure gauge indications on pressure-reducing valve, oxygen pressure in bottle must be higher than 4Mpa).
2. Lift the pressure sleeve of oxygen filler to make oxygen filler nozzle point at oxygen bomb head, and then insert it into oxygen bomb head. When oxygen bomb head tightly contacts oxygen filler nozzle, drop the pressure sleeve of oxygen filler, then oxygen filler automatically locks the oxygen bomb.
3. Pressure gauge should show 2.8 ~ 3.0Mpa when opening the switch of micro-type oxygen filler. Fill oxygen for 30 ~ 45seconds, and then close the switch of micro-type oxygen filler.
4. Lifting the oxygen filler cover and take out the oxygen filler, at this time, it should be possible to see that valve core on oxygen bomb is extruding upward, which shows that oxygen bomb has been filled with sufficient oxygen.

Caution:

1. Above procedures shall be strictly observed for oxygen filling and handle with care when using.
2. Only when oxygen filler automatically locks oxygen bomb head, can oxygen filling switch of micro-type oxygen filler be opened.
3. Close oxygen main valve and release residual oxygen from the circuit after test completion each day (Method: close oxygen bottle valve for oxygen-filling through oxygen bomb up to zero indication by oxygen pressure gauge).
4. Fire and smoke are prohibited at oxygen filler and oxygen bottle disposition site.
5. Never bend and twist oxygen filling conduit.
6. Inspect at regular interval the indications on pressure gauge of oxygen filler and on low pressure gauge of pressure-reducing valve to see if both are in conformity.
7. Don't use any oil or grease in nuts at the connections in whole oxygen circuit.

Chapter 4 Instrument Use and Operating Instructions

4. 1 Start-up and exit of Test-control Software

4.1.1 Start-up of Test-control Software

There are two ways to start up the test-control software of CALO-11 calorimeter.

Way 1 Setup of test-control software will enable automatic set-up of a quick way on Windows desktop and menu item in Windows program group. Directly click the quick icon named “CALO-11 calorimeter”, or start up from “Start-up-> Program-> CALO-11 Calorimeter”.

Way 2 Choose “Automatically Program Entry” item from test-control software system setting. It will automatically enter test-control system at next start-up of Windows.

4.1.2 Exit of test-control software

1. Before Windows Exit or computer shutdown, shut down test-control system to ensure no damage to the test data and parameter files.
2. Click “Exit” menu under “System” main menu to Exit CALO calorimeter test-control software and return to the desktop.
3. Click “Shut down Computer” under “System” main menu to Exit CALO calorimeter test-control software and Windows directly, besides it will automatically shut down computer.

4. 2 Main window frame functions

The main window body of CALO calorimeter test-control system is mainly composed of headline column, quick push-button column, window body and status column.

Diagram 4-1 shows the interface of CALO calorimeter test software after temperature balance treatment.

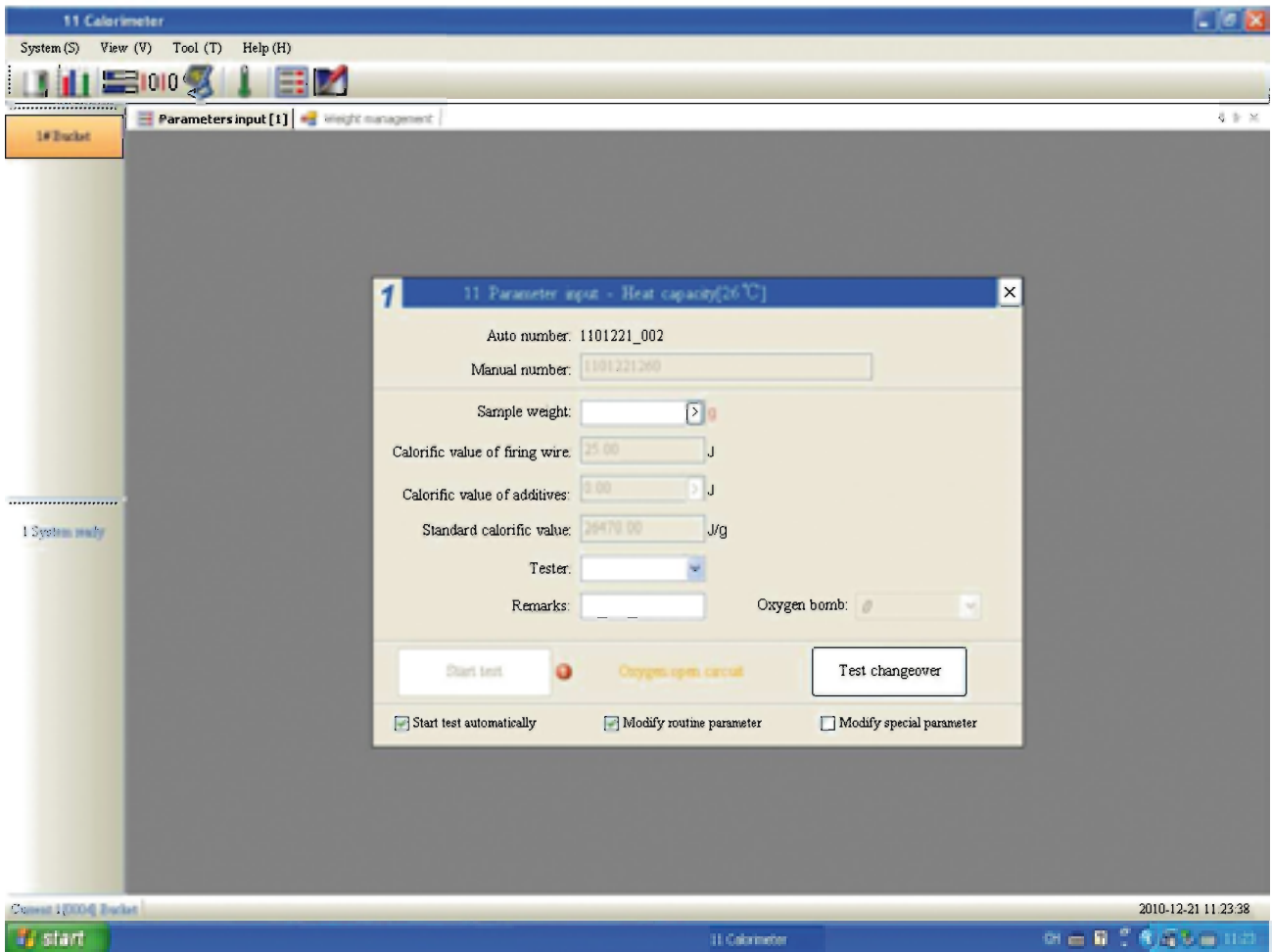


Diagram 4-1

Diagrams 4-2 and 4-3 show the parameter input window.

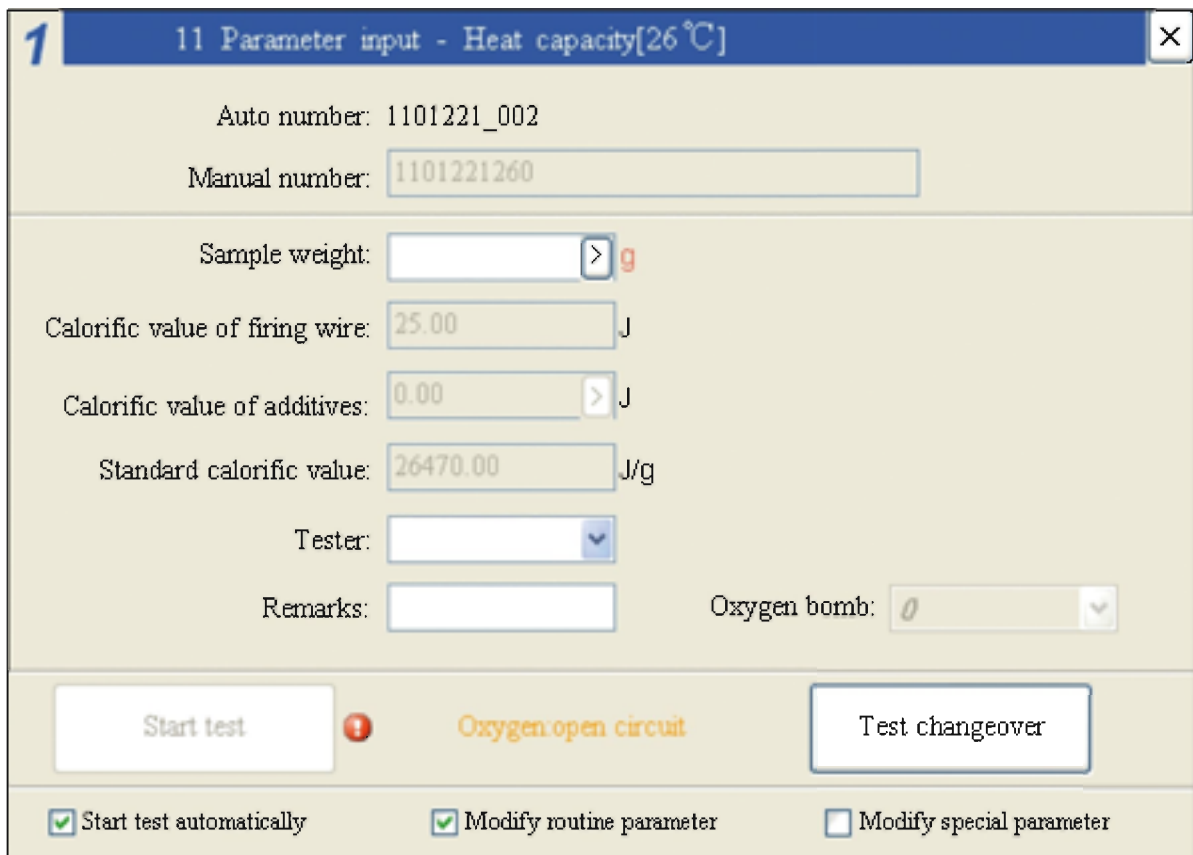


Diagram 4-2 Heat Capacity Input window

Diagram 4-3 Calorific Value Input window

Description of parameter input window:

1. Automatic coding with values as "bucket No. (1-digit) + year (2-digit) + month (2-digit)+date(2-digit)+sample number(3-digit)". During heat capacity test, it is unnecessary to input the manual number, which will be generated by the system itself. Manual number is composed of "bucket" No.(1-digit) + year (2-digit) + month (2-digit) + date (2-digit) + jacket control temperature (2-digit) + oxygen bomb number (1-digit)". Manual number of heat capacity consists of at most 50 characters (one Chinese character or letter is implied as one character).
2. Sample weight: Can be input manually or click ">" to send the sample weight of the "weight management" Window to the sample weight column.
3. Input of Calorific value of Additives: When the test sample contains additives, you can input the calorific value of additives directly, or you can click ">" button in "Calorific value of Additives" input frame. A window as shown in Diagram 4-4 will appear. Input the additives weight, then click "OK". System will automatically convert the additives weight into calorific value of additives and transmit this value to the input window of calorific value of additives. Clicking "Cancel" will return to parameter input window directly and no conversion will be done.

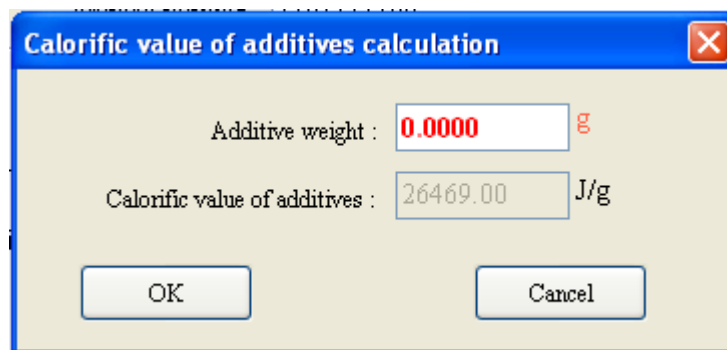


Diagram 4-4

4. Input of Had: You can input Had value directly, or you can click “>” button in “Had” input frame. As shown in Diagram 4-5, you can input Mad、Vat and Aad, then click “OK”. System will automatically calculate Had according to empirical formula and transmit it to Had input window. Clicking “Cancel” will return to parameter input window directly.

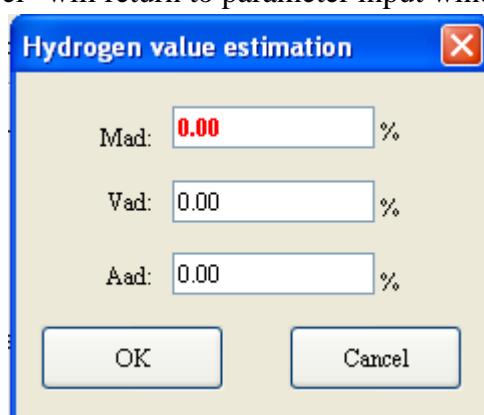


Diagram 4-5

5. Type: This column mainly used to set the limit of the allowed input sample weight range. For example, if the type is biomass, only the sample weight of (0.3~2.5) is allowed.
6. Oxygen Bomb: Default value of oxygen bomb is zero. If oxygen bomb is equipped with ID card, the ID card of this card will be automatically transmitted to oxygen bomb number frame when it is identified by a card reader. When the heat capacity measured by the same ID code is qualified, corresponding heat capacity at the control temperature corresponding to this ID code will be saved, otherwise system will display “This oxygen bomb number doesn’t have the corresponding heat capacity” while calorific value measurement.
7. Test Changeover: Clicking this button before test can shift between calorific value test and heat capacity test. Changeover is prohibited during test.
8. Start Test Automatically: If this function is chosen then system will automatically enter test status when oxygen bomb is placed and oxygen bomb is in "Ready" status. If not then “Start Test” button must be clicked to enter test status.
9. Stop test: If click this button during the test, and confirm to stop, the current test will be stopped.
10. Parameter Modification
 - a) Modify Routine Parameters: You can modify the test sample weight, analyst, remarks, manual number, Mt% Mad% %, Sb, ad% and Had%. This modification is valid before test

result is obtained.

b) Modify Special Parameters: You can modify the oxygen bomb number, calorific value of firing wire, calorific value of additives, reference calorific value and test type. Oxygen bomb numbers are impossible to be modified during test.

c) Setting Modification Authority: Only after correct password is input, can parameter be modified.

d) If a wrong parameter or no parameter is input, the system will display a red mark. Use the mouse to point at this mark to get the related information on allowable input range. After correct parameter is input, the red mark will disappear automatically.

4.2.1 System Main Menu

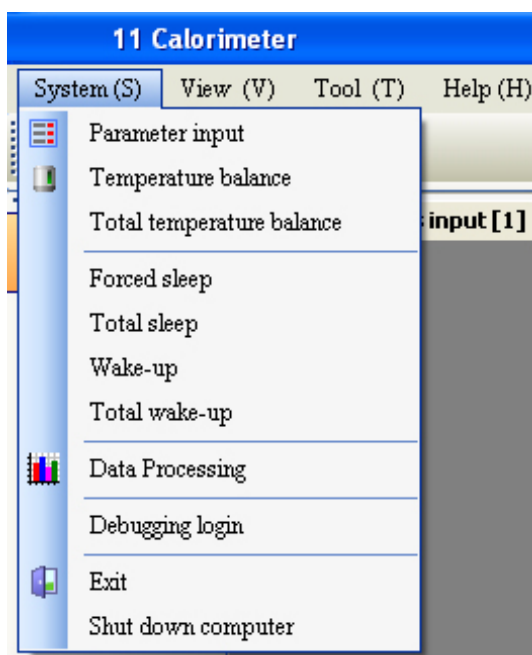


Diagram 4-6 System main menu

System main menu is mainly composed of parameter input, temperature balance, data processing and exit etc.

1. Parameter Input: If you choose this submenu or click "Parameter Input" button in quick button column, system will automatically open the parameter input window of current selected bucket.

2. Temperature Balance: If you choose this submenu or click "Temperature Balance" button in quick button column, system will automatically conduct temperature balance for the current selected bucket.

3. Total Temperature Balance: If you choose this submenu, system will conduct temperature balance for all on-line calorimeter's master machines.

4. Sleep mode and Wake-up: It will automatically enter dormant status after test is finished for some time. At this time, control temperature of jacket will fall for 3°C. After dormancy for some time, system will automatically wake up and jacket temperature will rise up to the control temperature. Inputting the test sample weight or clicking the parameter input headline column can wake up the instrument.

5. Data Processing: This system is designed to manage the test data better and more effectively. It includes the management of heat capacity data and calorific value data. During test, it is allowed to login the database. Choosing this submenu or clicking "Data Management" button in quick button

column can enter the window as shown in Diagram 5-2. Details refer to Chapter 5.

6. Debugging Login: If you click this submenu, an login window will appear. Only after correct password is input, can the system enter debugging status. Only debugging personnel can use this function.

7. Exit: If you chose “Exit” menu or click “Exit” button in quick button column, you can exit the test system.

8. Shut down Computer: If you choose this menu, you can not only exit this test system, but also you can exit windows system and automatically shut down the computer.

4.2.2 View Main Menu, as shown in Diagram4-7

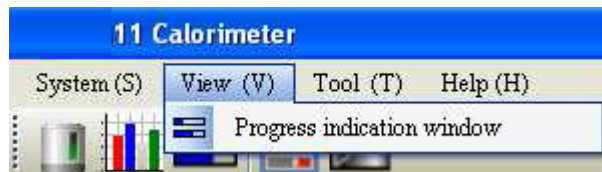


Diagram 4-7 View Main Menu

Progress Indication Window: Clicking this menu once will open this window, which is displayed in main window (Implicit position of progress window is the leftmost row). This window displays all bucket numbers which are on-line with system and their running status of each bucket during test. If you click this menu once again, this window will automatically close.

4.2.3 Too Main Menu

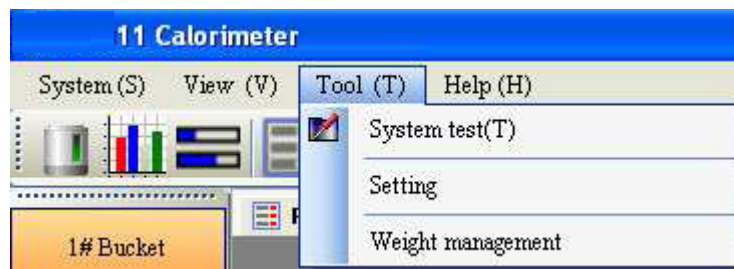


Diagram 4-8 Tool Main Menu

1. System Test

After you choose this submenu or click “system test” button once in quick button column or press “Ctrl+T”, system will enter login window (as shown in Diagram4-8-1). If you input correct password, you can enter system test window (as shown in Diagram4-8-2), where you can test each functional parts of the system.

In order to ensure system safety, system automatically locks some related operations while conducting one operation. After this operation is completed successfully, system will automatically release them.



Diagram 4-8-1

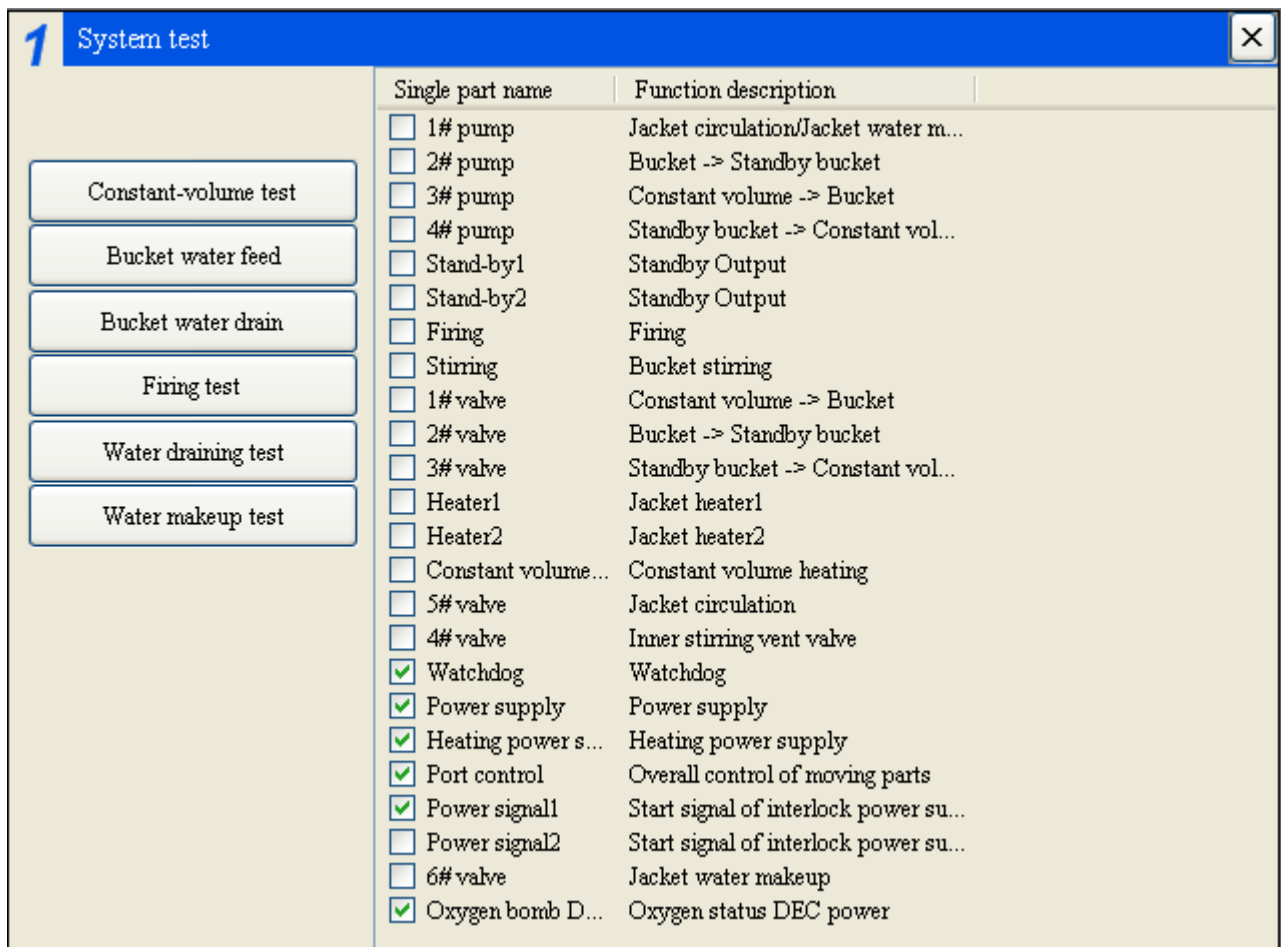


Diagram 4-8-2

Detail Description of System Test:

- Constant-volume Test: If you click this button once, system will pump water from water box into constant-volume vessel and automatically conduct constant volume treatment.
- Bucket Water Feed: If you click this button once, bucket drains water firstly, then it simulates the water feed process as normal test. After bucket water feed is finished, system will automatically start mixing.
- Bucket Water Drain: If you click this button once, system will transfer water from bucket to water box.

- Firing Test: Aim to check firing system's functional status.
- Water Draining Test: It is to drain water from jacket
- Water Makeup Test: System automatically makes up water into jacket.

2. Setting

This menu can be used to setup to font size, background color and test parameters etc. It is divided into general setting option card (Diagram4-8-3) and specific setting option card (Diagram4-8-4)

The screenshot shows a 'Setting' dialog box with a blue title bar and a close button (X) in the top right corner. The dialog has two tabs: 'Common' (selected) and 'Subcontrol'. The 'Common' tab is divided into several sections:

- Balance online:** Contains dropdown menus for 'Serial' (set to 'invalid'), 'Data bit' (set to '7'), 'Baud rate' (set to '1200'), 'Stop bit' (set to '1'), and 'Parity check' (set to 'Odd'). There is also an unchecked checkbox for 'Additive weighing allowable'.
- Check:** Contains five checkboxes: 'Display bucket number at right bottom corner' (unchecked), 'Automatically run program' (unchecked), 'Automatic temperature Balance' (unchecked), 'Toolbar using big icon' (checked), and 'Manual window arrangement allowable' (unchecked).
- Data:** Contains a text input field for 'Calorific value of additives' (set to '26469.00') with 'J/g' as a unit, and a dropdown menu for 'Serial number of card reader' (set to 'COM1').
- Font:** Contains three dropdown menus for 'Menu' (set to 'Example'), 'Progress indication' (set to 'Example'), and 'Status column' (set to 'Example'), each with a '...' button to the right. Below these are three more dropdown menus: 'Progress indication column width' (set to '110'), 'Progress indication row height' (set to '40'), and 'Progress indication position' (set to 'Left'). A 'Default value' button is located at the bottom of this section.

A 'Save' button is located at the bottom right of the dialog.

Diagram 4-8-3

Description of Common Setting:

- Serial Port No. of Card Reader: Only when card reader is properly connected with serial port of computer and the corresponding serial port number is chosen, can card reader work, otherwise code of ID card at the bottom of oxygen bomb can't be transmitted to parameter input window. We suggest card reader shall be connected with 2# serial port. 1# Serial port is implied for updating the computer program.
- Balance online:
 - a) Serial port setting: Serial port of the computer and the balance. Normally there are two serial ports, one is COM1 and the other is COM2; please set COM2 as the serial port for the

communication between the computer and the balance.

- b) Baud rate: For this parameter, please refer to the code of the balance menu, make sure the baud rate of the code can be in conformity with the software (Such as Satorius balance, code 514 refers to Baud rate 1200, code 515 is 2400, code 516 is 4800, code 517 is 9600 and code 518 is 19200).
- c) Parity check: Please refer to the code of the balance menu.
- d) Data bit: Please refer to the code of the balance menu.
- e) Stop bit: Please refer to the code of the balance menu.
- f) Additive weighing allowable: After selecting this, it's allowed to weighing the weight of the additives, and the weight should be less than 0.5g.

- Multiple-choice: Choose the desired items and Exit after saving
 - Display bucket number at right bottom corner: If you choose this option, number of current bucket will be displayed at right bottom of main interface.
 - Automatically run program: After you start computer, it will automatically enter the interface of test-control software.
 - Automatic Temperature Balance: After you start the test-control software, system will check the running status or conditions firstly to see if they are normal or meet the requirement, then it will automatically conduct temperature balance, otherwise automatically temperature balance will not be carried out.
 - Toolbar using big icon: you can change the size of tool column icon.
 - Manual Window Arrangement Allowable: you can drag the window body by hand so as to change the position of window body in main interface.
- Calorific Value of Additives: If you input or modify the calorific value of additives and save it, calorific value of additives in calculation window of calorific value of additives (Diagram4-4) will change correspondingly. If you input the additives weight in this window, system will automatically calculate the calorific value

According to individual requirement, you can click “...” button at the right of character frame to set the character size for menu column, status column and progress indication window. Set the column width, row height and position of progress indication window by up and down buttons. If you click “Default” button, the character, position and width etc will be recovered to the default values set in factory.

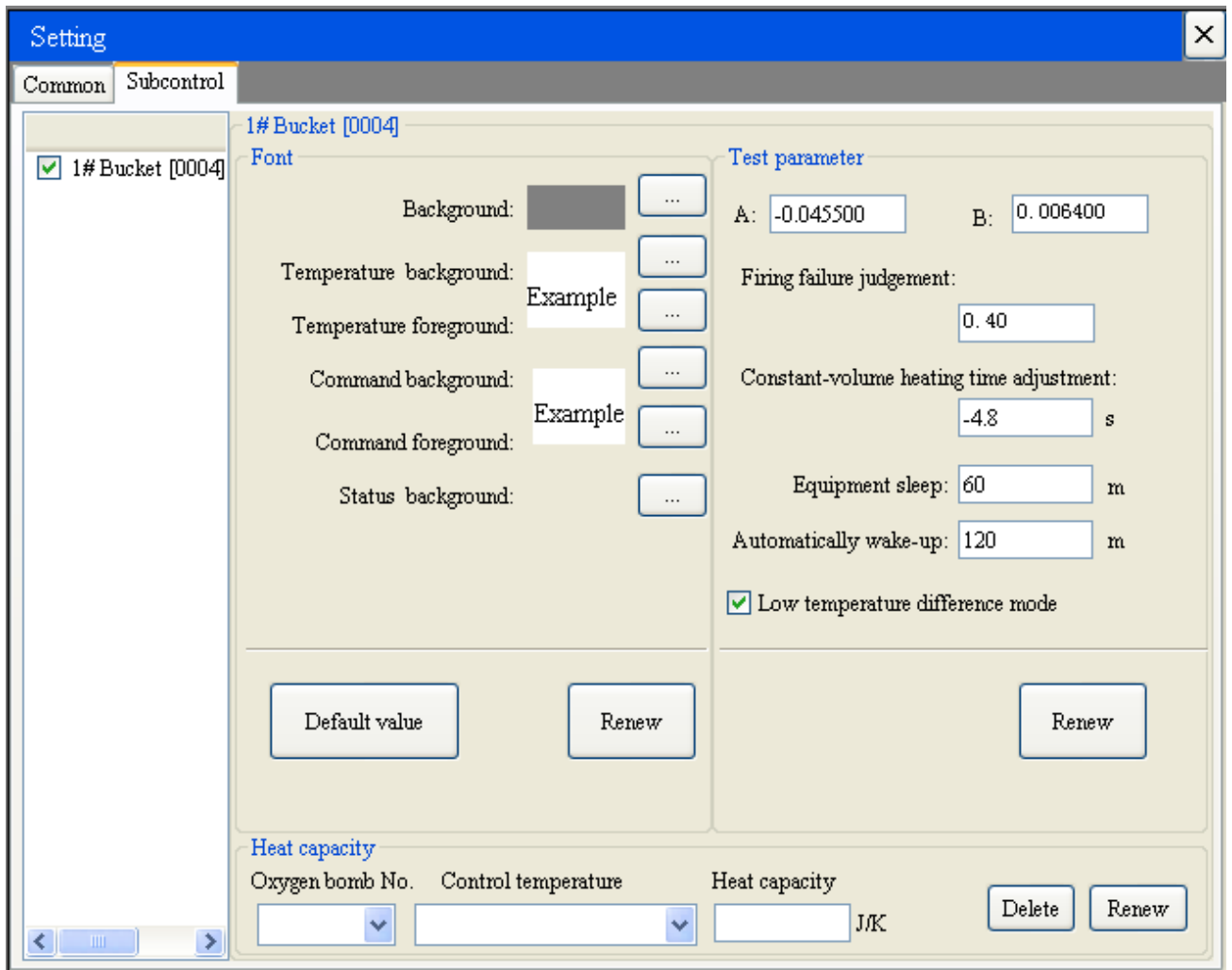


Diagram 4-8-4

In subcontrol setting, you can separately set several buckets. As shown in Diagram 4-8-4, if you choose No.1 bucket multiple-choice frame, you can modify the related test parameters of No.1 bucket. Now, we introduce the subcontrol setting in detail as following:

- Test Parameter

—a) Constant-Volume Heating Time Adjustment: Software can adjust the heating time automatically according to the temperature of constant-volume vessel.

—b) A, B value: After parameter test meets the requirement, system will automatically write in or renew A、B values (While leaving factory, implicit values of A and B are -0.0455 and 0.0064 respectively).

—c) Equipment Sleep: It is to set the time to let equipment dormant after test completion.

—d) Automatically Wake-up: It is to set the time to wake up the equipment after dormancy.

—e) Low temperature difference mode: After selected, the system will control the controlling temperature of the jacket as per low temperature difference mode.

- Color: Click the “...”button at the right of color frame, system will automatically open the color box, then choose the desired color to change the foreground color and background color of temperature window and command window etc. After restarting the test-control software, color will change accordingly. Click “default”, color will be resumed to the status while leaving factory.

When changing the above mentioned parameter setting, “Renew” button should be clicked to put it into effect.

- Heat Capacity
 - a) After a group of heat capacity is qualified at the same jacket control temperature, oxygen bomb number and its corresponding heat capacity will be automatically saved in the individual setting.
 - b) For the oxygen bombs with same serial number, if a group of heat capacity is qualified under different jacket control temperature, system will automatically save the heat capacity at this control temperature in the individual setting.
 - c) Delete: Select the oxygen bomb number, the system will automatically delete the corresponding heat capacity at the corresponding jacket control temperature when not in testing mode; if the system is under testing, it is not allowed to delete the heat capacity.
 - d) Renew: Click this button, system will automatically save the modified heat capacity.
 - e) The left textbox can be used to select the bucket number, the right window shows the relevant parameter of the bucket.

2. Weight management

The calorimeter has the function of external balance weighing and be connected to the calorimeter, automatic send and save and pronounce by voice the sample weight, the window and functions are shown are follows:

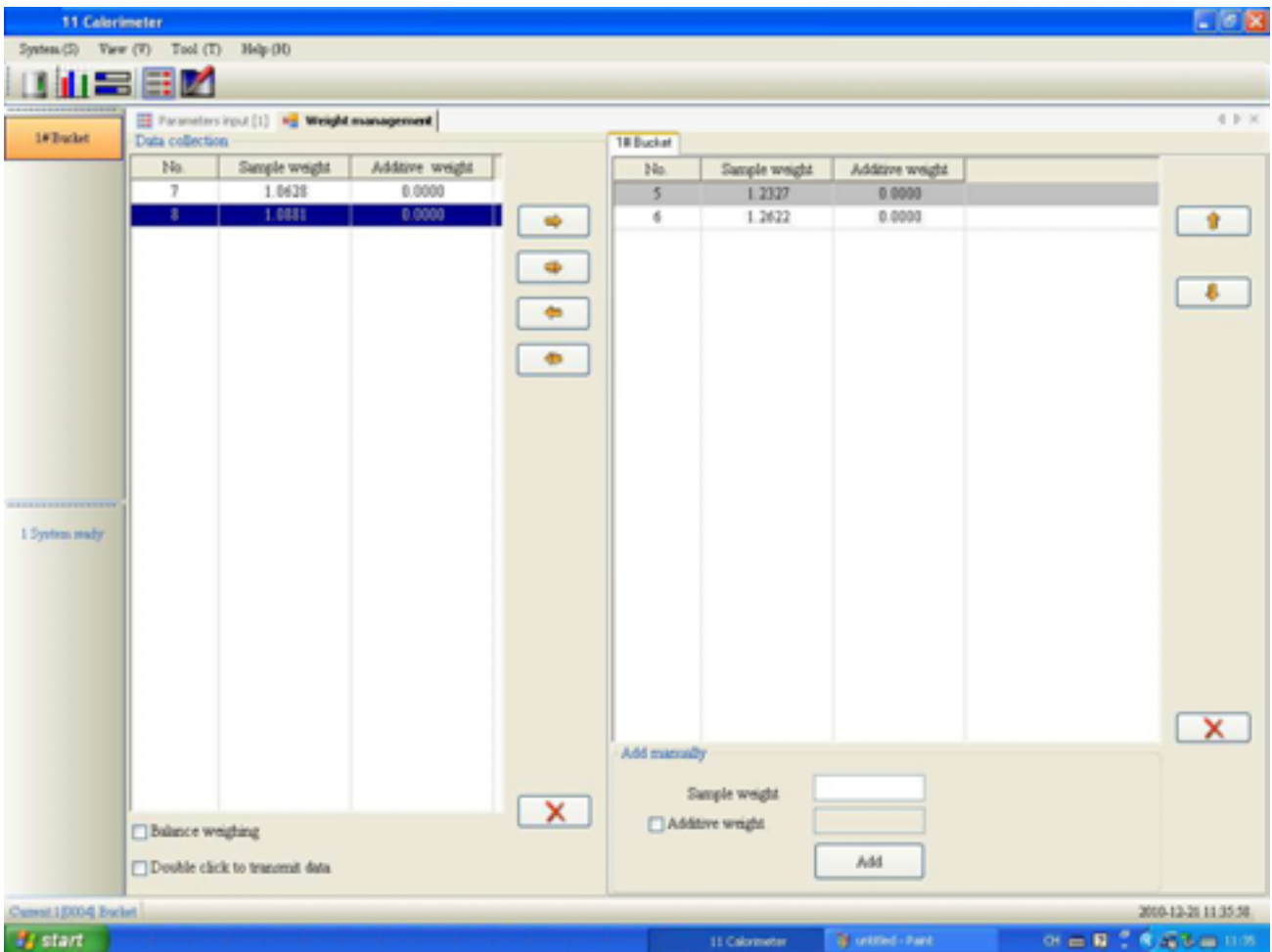


Diagram 4-8-5

Note: If there is sample weight record waiting for test in the window, when close the window, the record will not be lost, and keep in the window. For the already done test, the record will be deleted automatically. If the window is closed, the sample weight record waiting for test will not be sent to the sample weight column of “Parameter input” window. If the window keeps open, the record will be sent to the sample weight column.

4.2.4 Help Main Menu

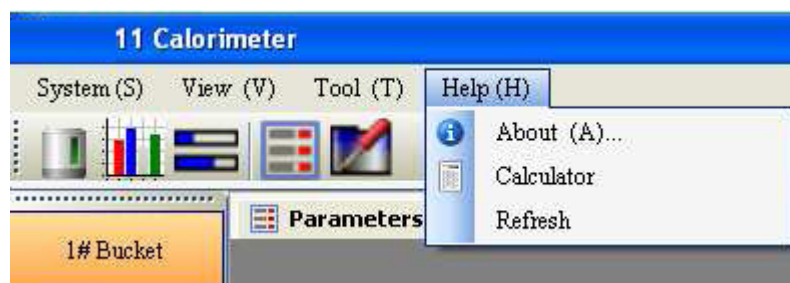


Diagram 4-9 Help Main Menu

Help main menu is mainly composed of about etc.

1. About: Click this or pressing “Ctrl+A”, you can get the information on edition.

4. 3 Test Process

Step One: Switch on the control power supply for the computer and the calorimeter.

Step Two: Start computer and enter the test-control status of ECNQ/11 calorimeter system. Click “ECNQ Calorimeter” icon on the desktop to enter the main program window.

Step Three: Temperature balance. Choose the progress indication window in the main program window, then click “system”-> “temperature balance” or “Total temperature balance”. (Or click “tools” -> “setup” -> “Common” and select “Automatically run program” and “Automatic temperature balance”, When start the computer next time, the program will automatically run and enter into temperature balance.

Step Four: When temperature balance, do the preparation such as weighing the sample, opening the oxygen bottle and check the air tightness of the bomb, check the bucket and filter screen if it's clean, close the cover and set the test method. (Click “Parameter input”, “Test changeover”, then the test method can be changed between calorific value and heat capacity test.). After system temperature balance is completed, progress indication window will display “System Ready”

Step Five: Put oxygen bomb after oxygen filling into the bucket (If there is bomb identifier, please scan it before put it into the bucket.) After bucket cover is closed, input the sample weight and other parameters, such as Mad, additives and so on.

Step Six: click [Start Test] to enter test, if implicit setting is “Start Test Automatically” there is no need to click. After entering the qualified sample weight, the system will automatically start the test. At the same time, the progress indication window will show the progress bar.

Step Seven: During the test, choose “Modify Routine Parameter” or “Modify Special Parameter” to modify the according parameter, or click “Stop test” to stop the test.

Step Eight: After the test is finished, the test data will be saved in the database. Open the bucket cover and take out the bomb from the calorimeter, release the gases remained in the bomb, check the burning status, and clean the bomb. If necessary, please repeat step six to step eight.

Step Nine: After finishing all the tests of the current date, enter into database and select the records, and click Edit->Print, the system will print the selected records.

Step Ten: Clear up the lab, exit the program and shut down the computer.

4. 4 Announcement

Standard operation or not will effect the test result, so please obey the details when operating.

1. Requirement for samples and crucibles: Mix the samples thoroughly, the crucible should be clean and of constant weight. If there is sprinkled sample, it should be treated as waste; it's prohibited to use the samples weighed the day before testing and the samples after failing to ignite.
2. The bomb should be taken carefully, when put samples, please tight up the bomb cap, pay attention the position of the crucible and the firing wire be changed by the any shock.
3. The cleaning of the bomb: The water to clean the bomb should be sufficient, the water temperature should be suitable, it's better if it's close to the water tank temperature. Every time when cleaning the bomb, the cleaning time should be almost the same. The residues of the bomb cylinder should be clean, there should be no firing wire or dirt left in the electrode rod, the fire baffle should be clean too. There should be no short circuit of the firing baffle

and the electrode rod. When doing the first test after starting the calorimeter, please wash the bomb (or place it in the bucket for temperature balance), then use it; Every test, 10ml of distilled water should be added into the bomb, the surface of the bomb should be cleaned by the special towel.

4. Check the air tightness of the bomb: In order to make sure the test result is reliable, every time before doing the test, the bomb should be checked for its air tightness. (Test method: put the bomb with sufficient oxygen into water, the water should be sufficient enough to drown the bomb, after dipping for one minute, check if there is any leakage. The time of dipping the bomb every time should be the same, and the temperature of the water to dip the bomb and the temperature of the water tank should be the same. If there is any leakage, please find out the reason. After solving the problem, the previous sample should be treat as waste; if there is no leakage,
5. After finishing the test, take the bomb out slightly and stably, and release the waste gases through the releasing valve, then to check the burning condition of the sample.
6. Check if the bucket and the filter screen are clean, if there is any dirt, please deal with it and make sure the filter screen is correctly installed.
7. Take the bomb out from bucket, if some water dropped out on to the cover of the bucket shell, please wipe it promptly.
8. Clean the bomb after finishing the current day test and close the oxygen bomb cylinder, release the gas remained in the oxygen tube and make sure the high pressure of the pressure gauge indicates 0Mpa.

Note: When the computer is under use, do not remove the USBCAN card, or it may hurt your computer as well as the relevant cards.

Chapter 5 Data Management

(Take CALO-11 for example, the other types are similar)

5. 1 User login



Diagram 5-1

Description of Database login:

1. Run the test software of CALO calorimeter and click “system” -> “Data management” (Or click “Data management” in the shortcut menu), the system will popup the login window (as showed in Diagram 5-1)
2. You can either input the user name or choose user through the list, after selecting the user, press “Enter” to the password input window. If three times of wronged password, the system will automatically close the login window. There is no password while leaving factory. You can automatically login the “Data Management” while starting the test-control software.
3. Clicking “Exit” button will return to main window. Clicking “Login” will enter the data management main window as shown in Diagram5-2 or 5-3.
4. Enter into the database main window, point the mouse to the column head, press on the left button, drag and adjust the location of different columns in order to convenient the checking.

5. 2 Main window

Data management main window of CALO calorimeter test software is composed of headline column, menu column, quick column and data display column. The menu column in detail as follows.

5.2.1 Item Main Menu

This menu includes the conversion of calorific value and heat capacity. After completion, heat capacity window or calorific value window is shown as Diagram 5-2 or 5-3.

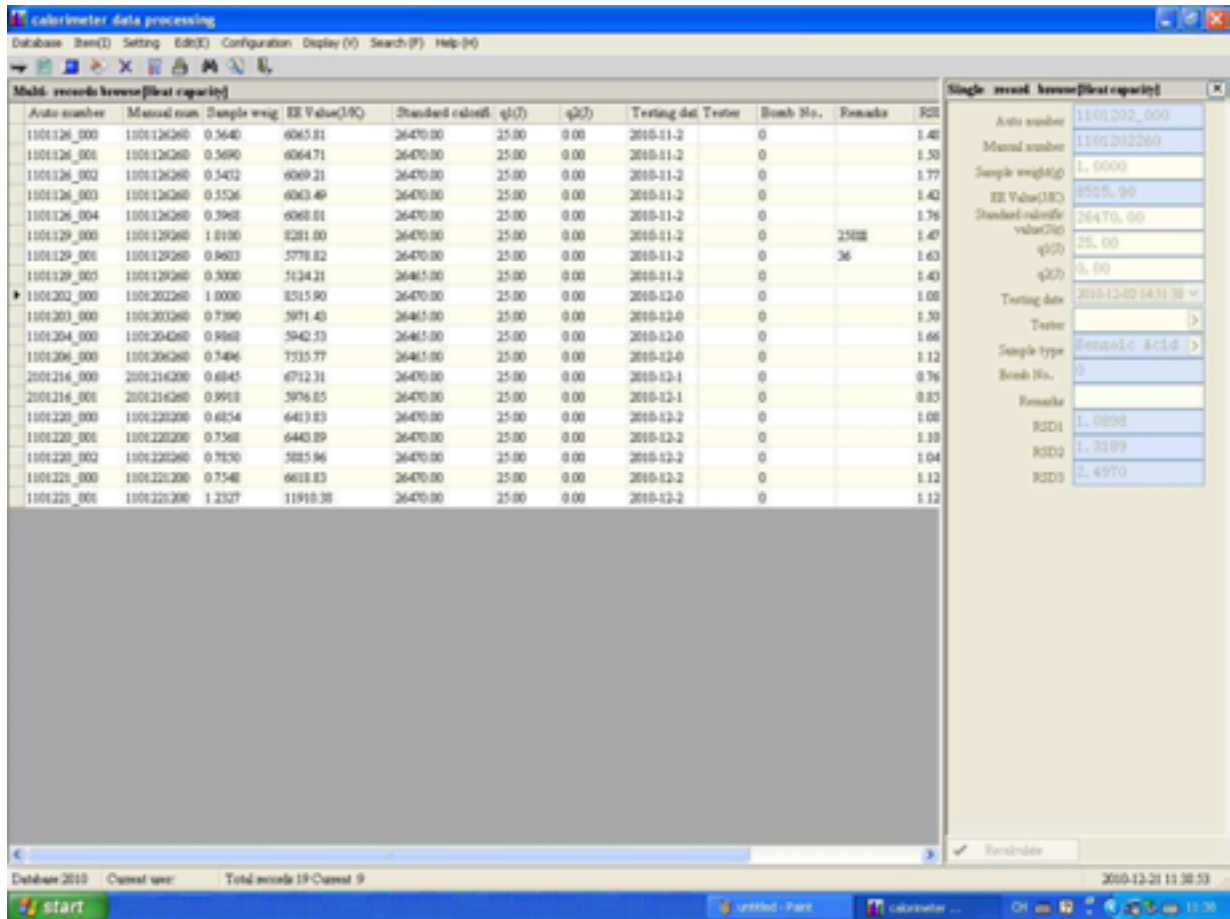


Diagram 5—2

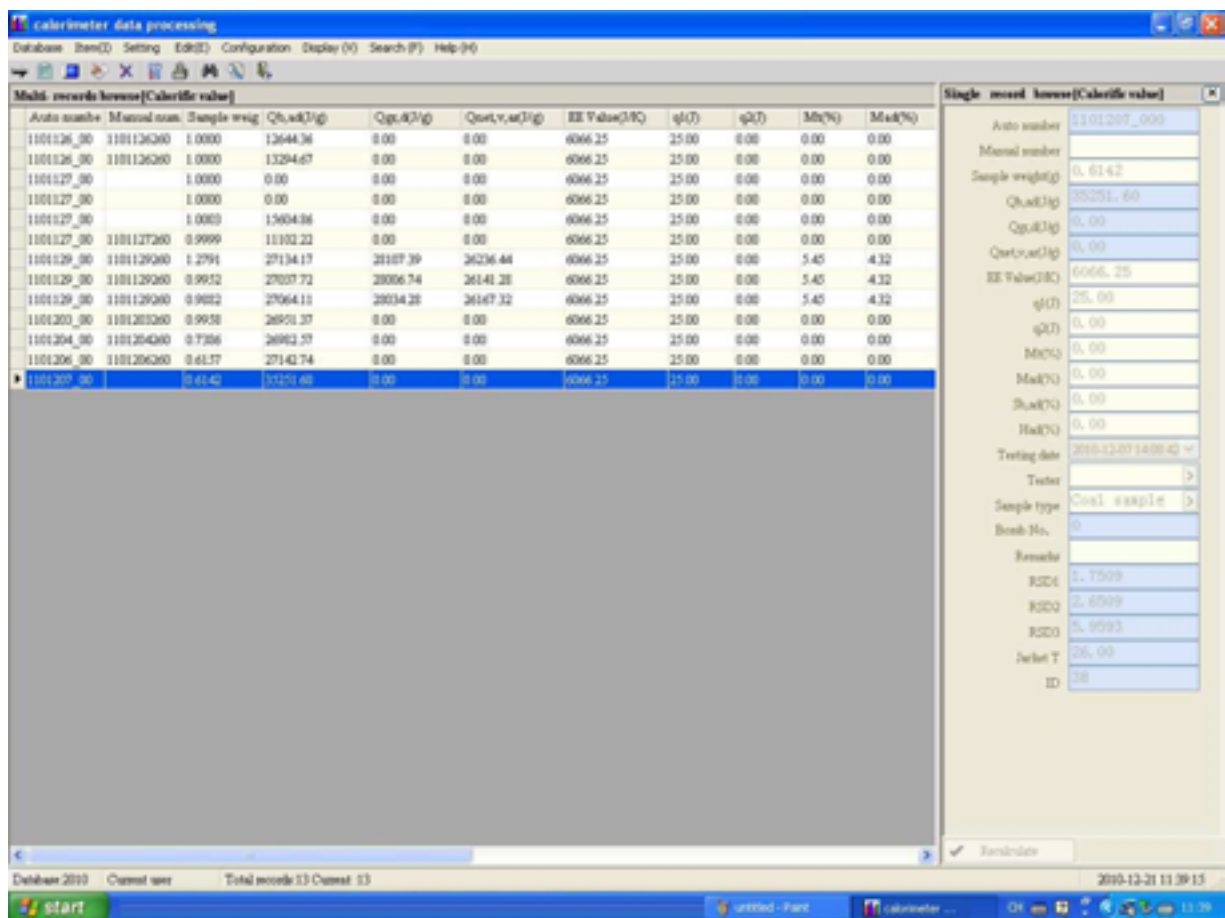


Diagram 5—3

5.2.2 Setting Main Menu (Diagram 5-4)

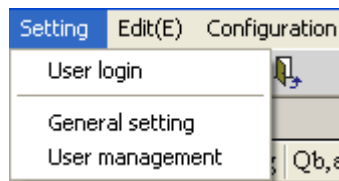


Diagram 5-4 Setting Main Menu

1. User Login: Different user can login according to their authorities.
2. General Setting: Be used for setting the analysis unit information, printing copies, database backup strategy (For-detail, please refer to Diagram 5-5 Basic Setting, Diagram 5-6 Advanced setting and Diagram 5-7 Database Backup Strategy).

● Basic Setting

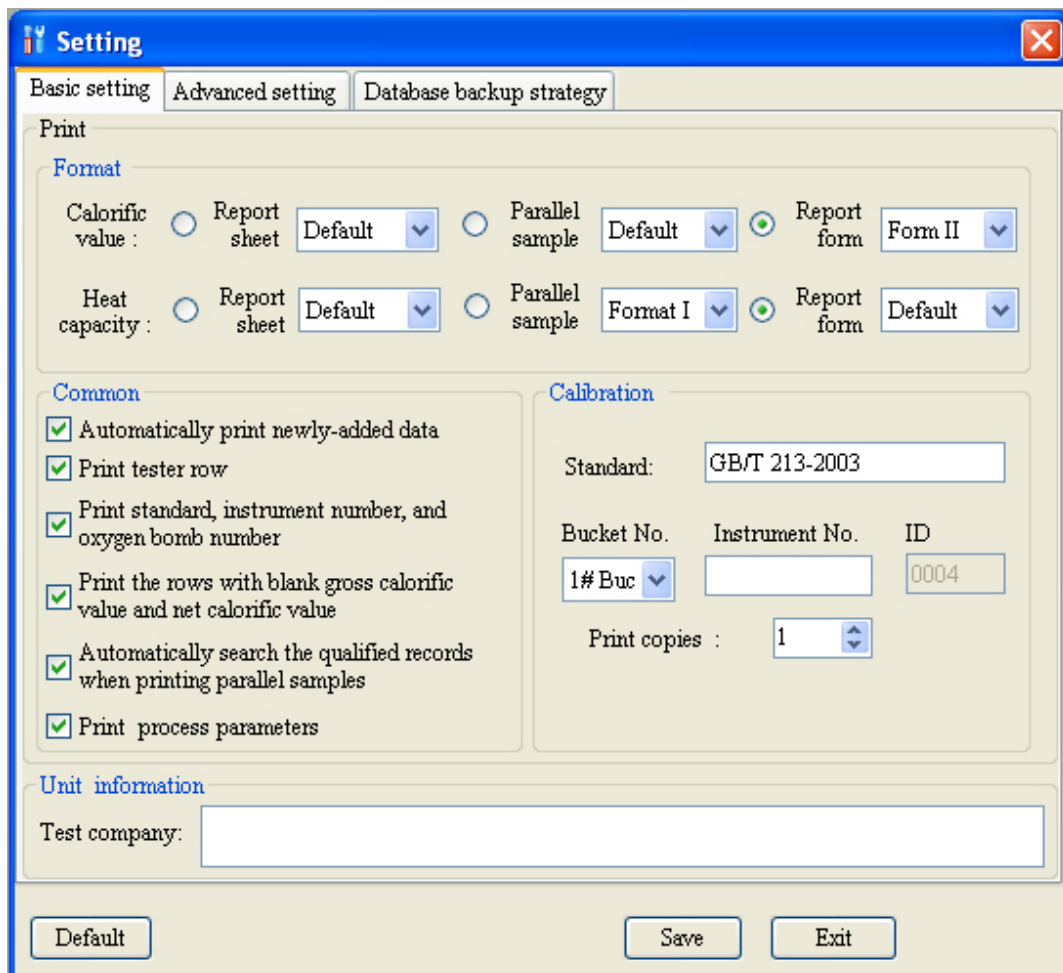


Diagram 5-5

This is to set the printing content and report form etc.

- a) Report Sheet: It is displayed in single-record form during print preview.
- b) Parallel Sample: Parallel sample test shall be conducted according to the manual number of current selected records during print preview. If manual number is not empty and it meets the parallel sample judgment conditions, all the records will be displayed.

c) Report Form: It is displayed according to the report form during print preview. There are three kinds of report form for calorific value. Heat capacity has one kind of report form only.

d) Print Tester row: If you choose this, it will print the analysis unit, analyst, auditor column, otherwise it will not be printed.

e) Print Tester: If you choose this, it will print analyst while print preview and automatic print, otherwise it will not be printed.

f) Print Process Parameters: If you choose this, system test process parameters will be printed while print preview and automatic print.

g) Automatically Print Newly-added Data: When test results of calorific value or heat capacity comes out, it will automatically print out the report sheet and parallel sample. For the printer you can't set the paper size (such as laser printer), we suggest canceling this choice for saving.

h) Print Standard, Instrument Number, and Oxygen Bomb Number: When report sheet of calorific value is set to "Form 2" or parallel sample way is set to "Form 1", it shall print out the standard; system number and oxygen bomb number after you choose this function.

i) Print Copies: It is to set the print copies.

j) Print the rows with blank gross calorific value and net calorific value: If choose this, it will automatically print the blank gross and net calorific value when both preview and print.

k) Automatic search the qualified record when printing parallel samples: If choose this, it will automatically select the qualified record of parallel samples when both preview and print, or it will print all the parallel samples.

- **Advanced setting**

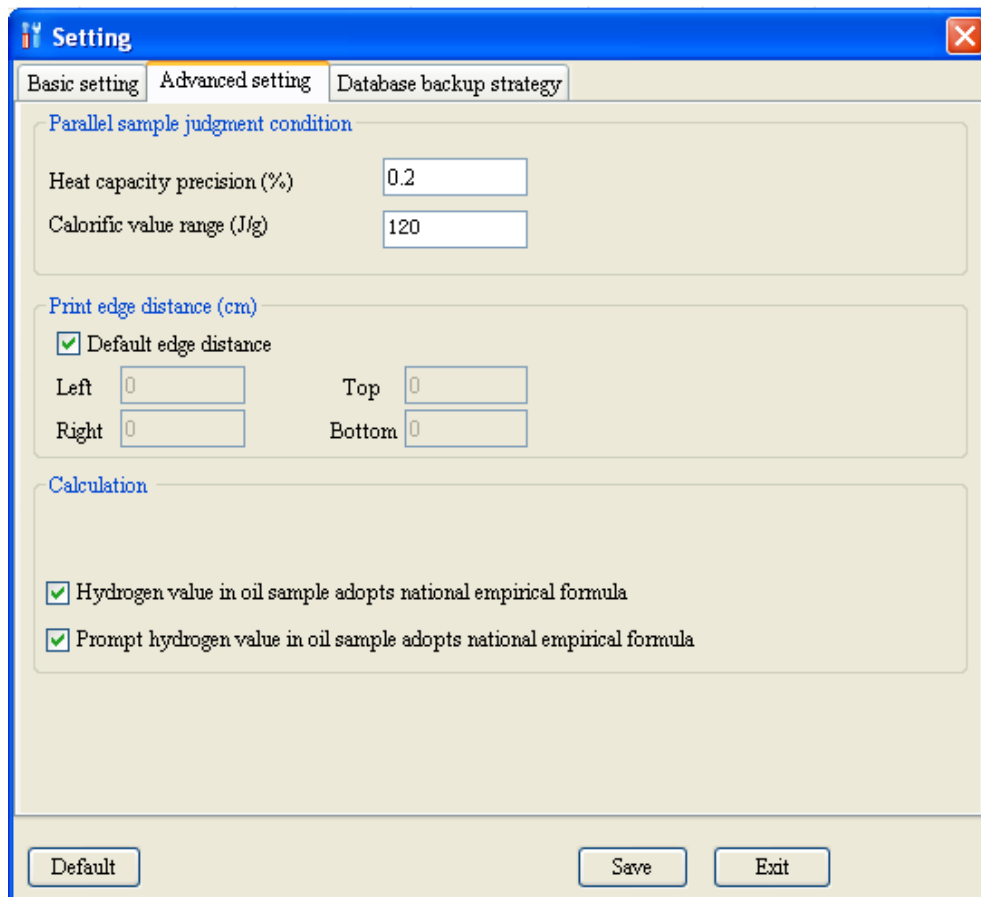


Diagram 5—6

- a) Parallel Sample Judgment Condition: Implicit value is usually used to judge if parallel sample is qualified.
 - b) Print edge distance: Set the print position on the paper.
 - c) Hydrogen value in oil sample adopts national empirical formula: If you choose this option, hydrogen value will be automatically calculated while the test of light oil or heavy oil.
 - d) Prompt Hydrogen value in oil sample adopts national empirical formula: If you choose this option as well as Hydrogen value in oil sample adopts international empirical formula , the system will automatically show prompt message while the test result of light oil or heavy oil are saved in the database or recalculate.
- Database Backup Strategy (as shown in Diagram5-7)
 - a) Backup after program runs n times: If you choose this option, when data management program runs for the setting times, backup shall be carried out while Exiting database program.
 - b) Backup while quitting data management program: If you choose this option, backup will be automatically carried out while exit data management program each time.
 - c) Backup Directory: After you click “Open. . . ” and choose the effective directory, required data shall be duplicated under this directory.
 - d) Backup File List: Choose a record in “Back File List”, and then click “Restore”. After restore, data management program must be restarted; otherwise all the changes to database are invalid.

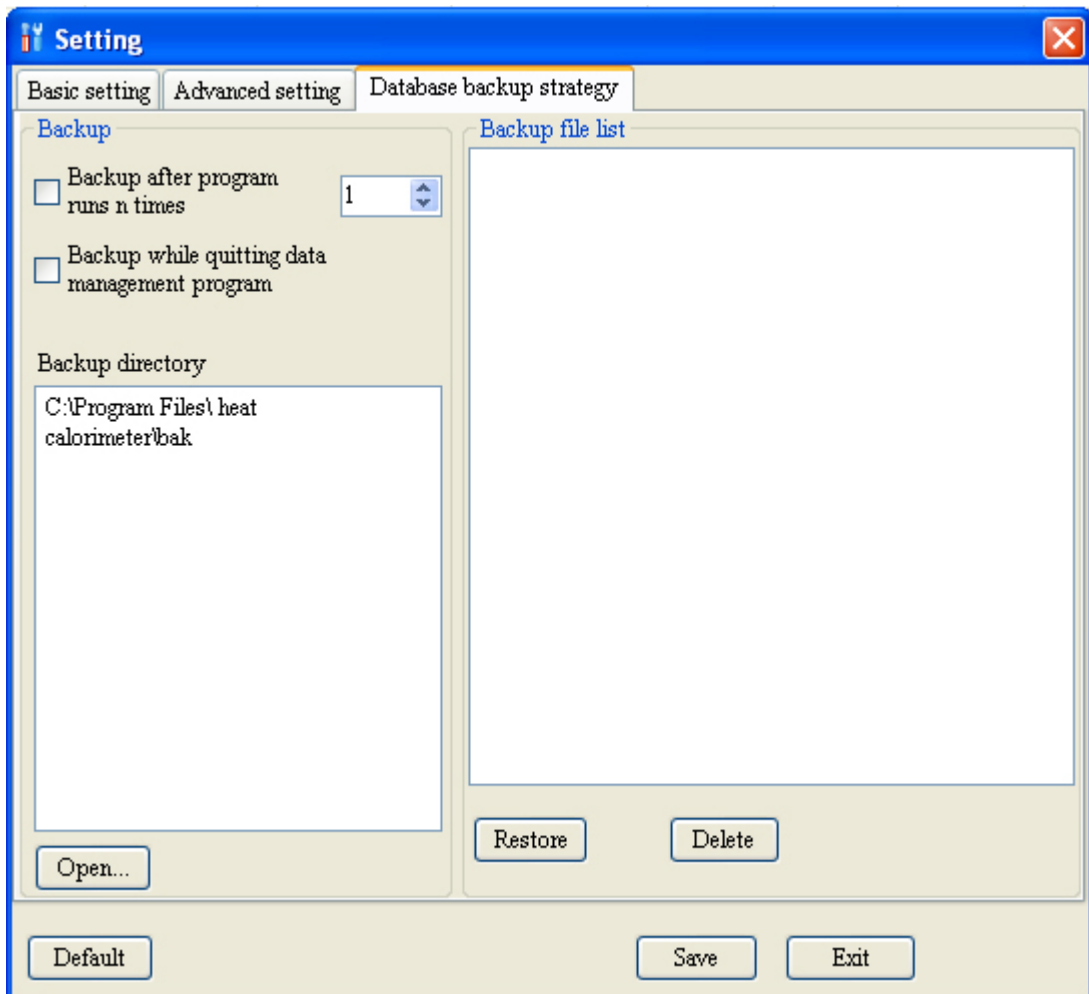


Diagram 5-7

3. User Management: This is used to add, modify and delete user as well as determine the authority level of usage (as shown in Diagram 5-8).

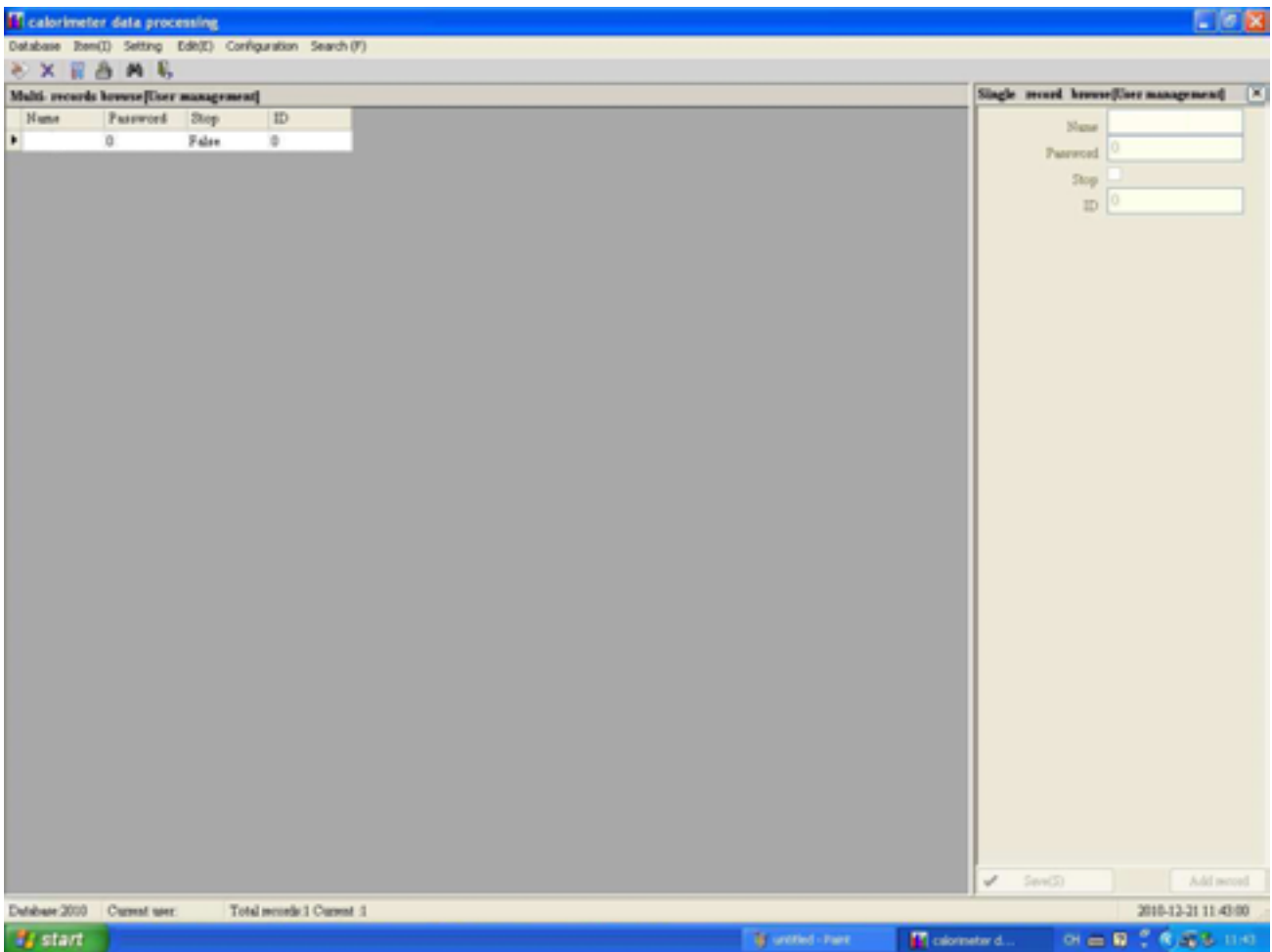


Diagram 5-8

- a) User with ID of zero (0) can't be deleted and changed to out of service status.
- b) Data with same user name can't be added or ID can't be modified.
- c) User who logs currently can't be deleted.
- d) User who has been chosen by main program and are performing test can't be deleted, otherwise data saving will fail.

5.2.3 Edit Main Menu (as shown in Diagram 5-9)

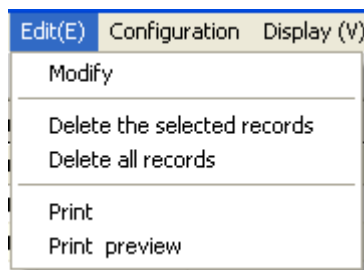


Diagram 5-9 Edit Main Menu

1. Modify: After clicking this option, it will automatically open the single record for review and then enter the editing status.
2. Delete the Selected Records: Delete all records selected in the multi-record view.

3. Delete all records: Delete all records displayed in the multi-record view.
4. Print: Print the records selected in the multi-record view.
5. Print Preview: Preview the records selected in the multi-record view (as shown in Diagram 5-10).

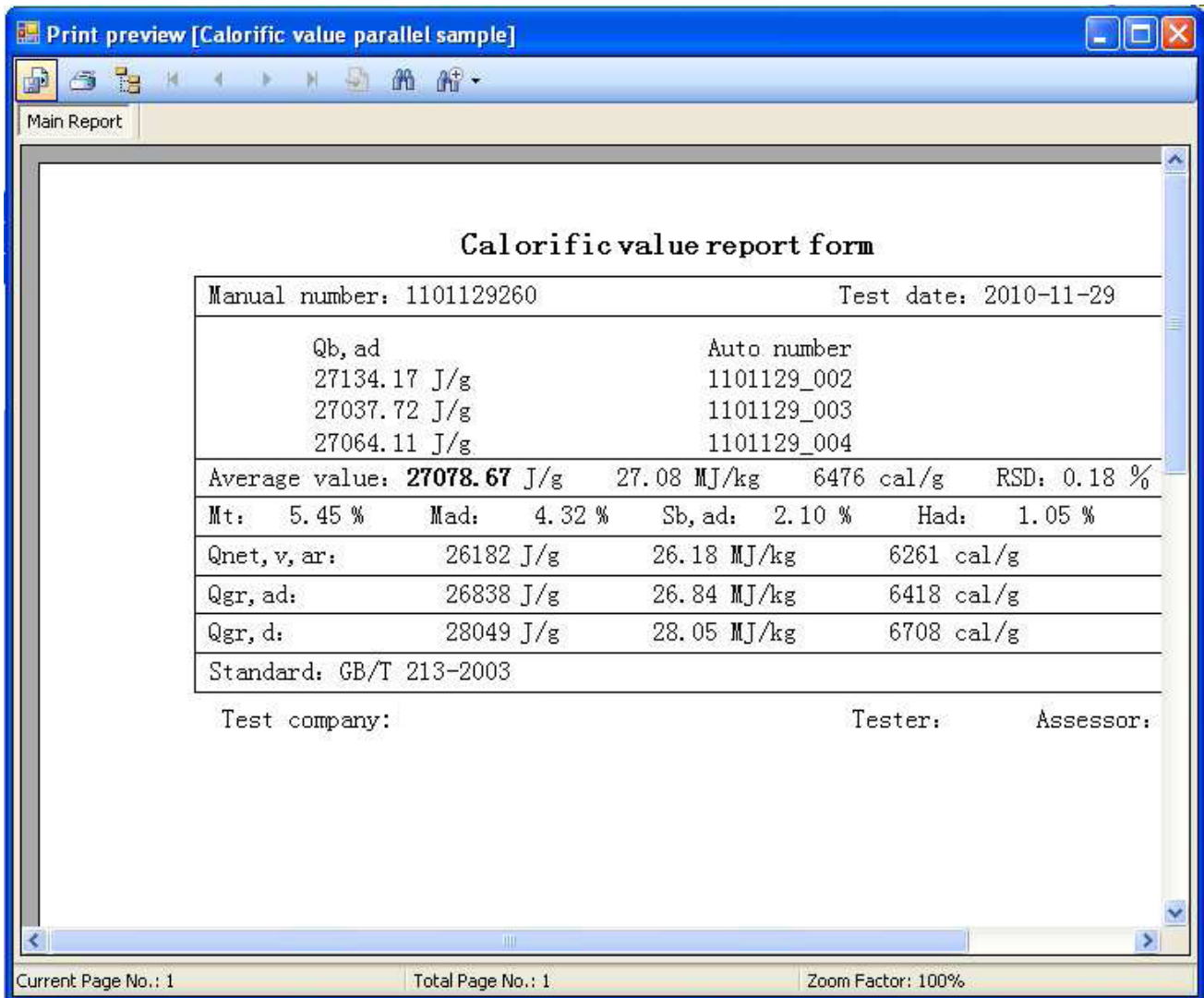


Diagram 5-10

5.2.4 Configuration Main Menu (as shown in Diagram 5-11)

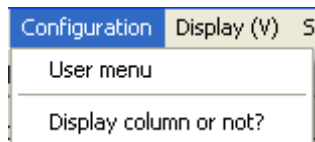


Diagram5-11_Configuration main menu

1. User Menu: This is to configure the authority for the user who is logging currently (as shown in Diagram 5-12).
 - To configure the authority for the user who is logging currently.
 - Directly click or cancel “User Menu” under configuration, then click “Renew Display” to renew this user menu immediately.

- “Setting”, “User Login”, “configuration” and “User Menu” are always visible.
 - If you have chosen “User Menu”, this means that this user can directly configure his (her) or other people’s user password and menu authority. Therefore, authority of “User Menu” is usually assigned to system administrator.
 - If you want to choose “User Menu” under “Configuration” after you cancel “User Menu”, you must verify your identity by use of the authority of system administrator.
 - “—” represents the decollators of menu column, which can be used for display only.
2. 2. Display: This is to configure the rows to be displayed in current multi-record browse window (as shown in Diagram5-13).
- To configure “Multi-record browse” window of current report form and the rows to be displayed in “Single-record browse”.
 - To configure calorific value, heat capacity and follow-up report forms.
 - Not to make row configuration for each user.

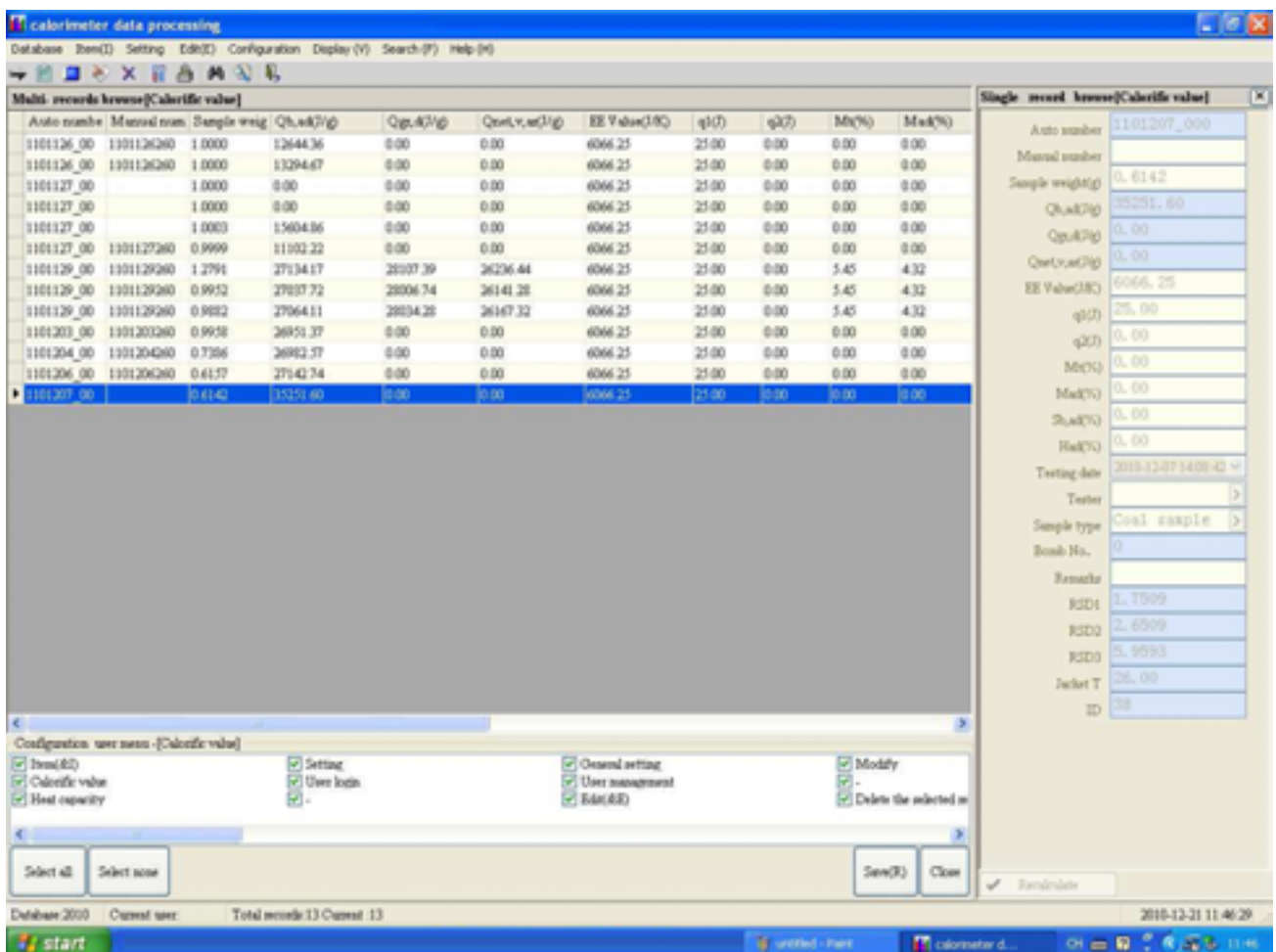


Diagram 5-12

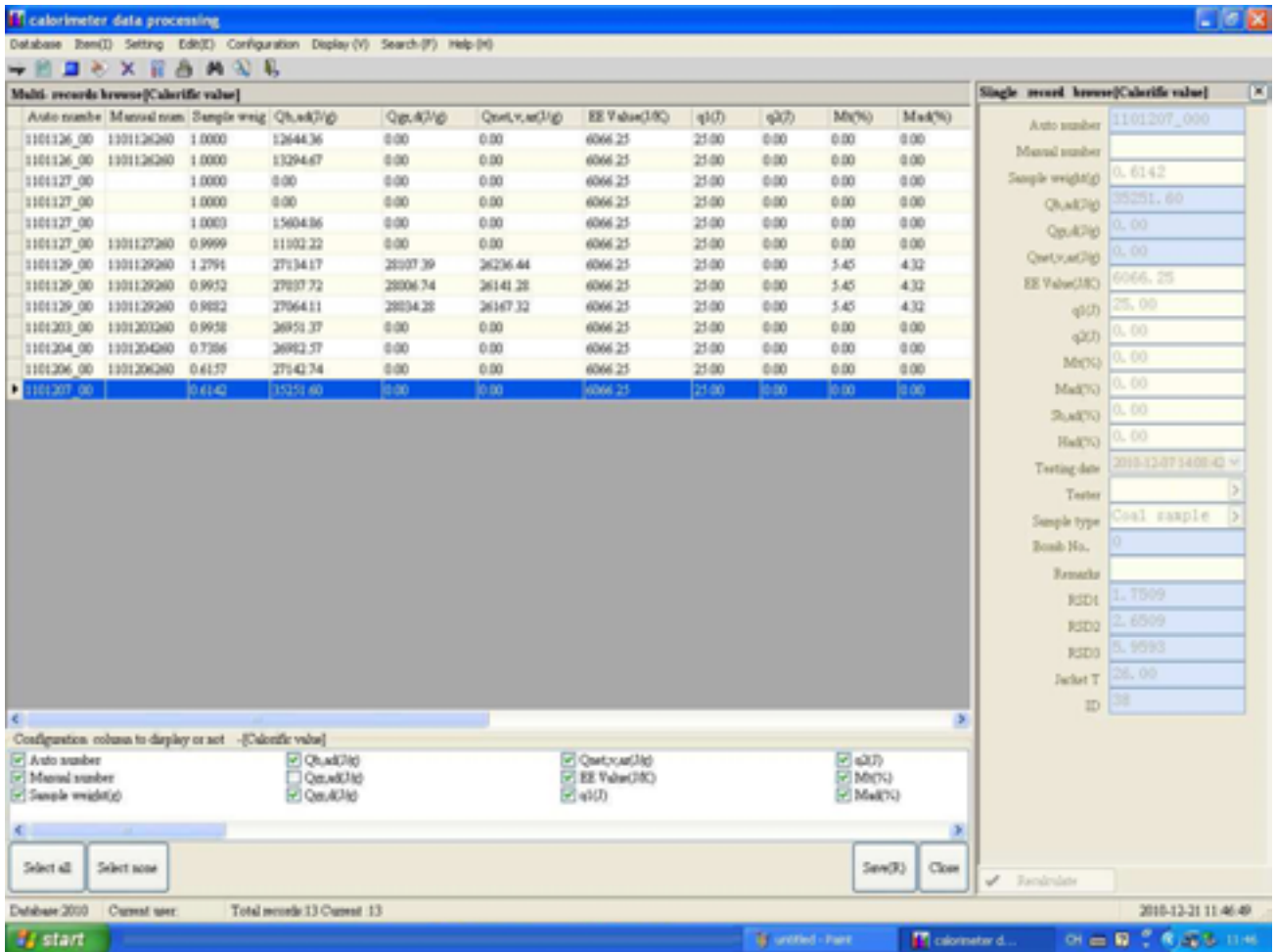


Diagram 5-13

5.2.5 Display Main Menu (as shown in Diagram 5-14)

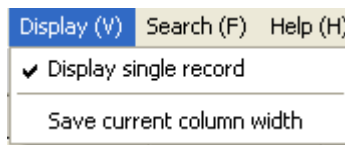


Diagram 5-14_Display Main Menu

1. Display Single-record:

a) Clicking it twice can open/close single-record browse

b) Choosing this menu will open single-record browse window (as above diagram), otherwise single-record view window will be closed.

2. Save Current Column Width: Width of each row in current multi-record browse window after adjustment will be saved.

- To save the width of each column in current report form.
- To configure calorific value, heat capacity, user management and follow-up report forms.
- Not to make width setting for each user.
- To limit special users to use this function by “User Management”.

5.2.6 Search Main Menu (as shown in Diagram 5-15).

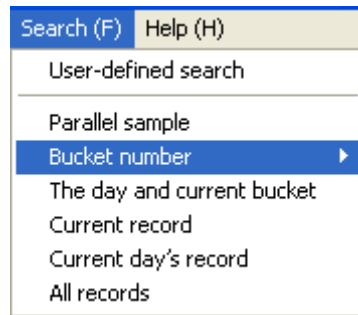


Diagram 5-15 Search Main Menu

1 Parallel Sample: To display all records at current day with same number as manual number and to display the related parallel sample information such as average value, precision etc..

2 Bucket Number: To display all data of a bucket at current day.

3 Current Record: To display the data with the same number as manual number of current selected record and the same test data as above-mentioned record.

4 Current Day's Record: To display all the records with same test date as current selected record.

5 All Records: To display all records in the database.

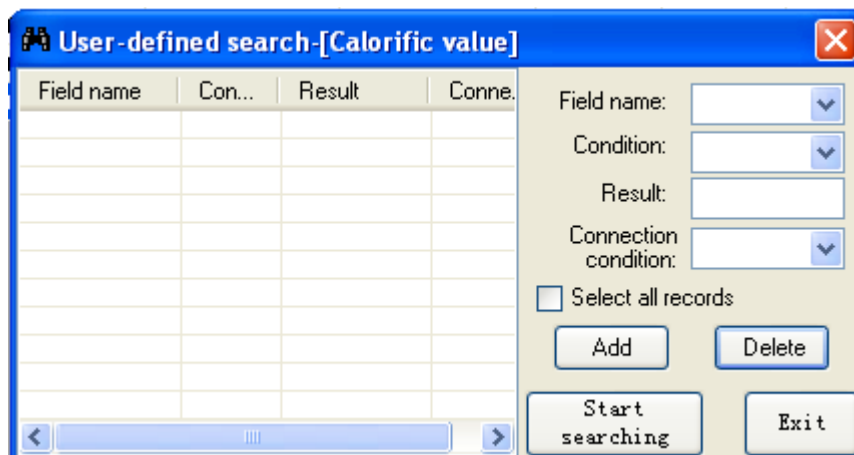
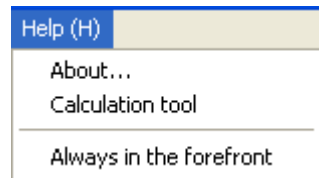


Diagram 5-16

- Field Name: It means the field name of all rows in current report form.
- Condition: “Like” represents similarity.
- Connection Condition: “And” represents that two conditions must be met simultaneously. “Or” represents that only one of two conditions is met.
- Add: Input the content to be searched and click "Add", then it will be transmitted to the condition sheet at the left.
- Delete: Choose a row of condition in the left condition sheet, then click “Delete” to delete it.
- Select All Records: After you choose this option and click “Start Searching”, all records in current multi-record browse list will be displayed.

5.2.7 Help

1. About: To display the information on data edition number etc.
2. Calculation tool: To call out the system calculator for calculation.
3. Always in the Forefront: After you choose this option, this window will always keep at the forefront of the window. If the window size is minimized, this function will be inactive.



Chapter 6 Instrument Maintenance

Calorimeter is a fine measuring instrument, the daily maintenance will directly have influence on the stability and accuracy of the instrument. So the daily maintenance is very important, please not the following points during the daily maintenance.

6.1— The maintenance of the instrument and the water tank

1. The instrument must be reliably ground to avoid any influence of test result not stable caused by the un-ground or not reliably ground.
2. After finishing the test every day, make sure the cover is half opened in order to avoid the rusts on the electrode rod, at the same time there will be dusts and other impurities falling to the bucket, thus to pollute the bucket and the temperature probe.
3. After finishing the test every day, the bomb is prohibited to be placed in the bucket. Take out the bomb from the bucket and clean and wipe it and place it in the proper place.
4. Keep the shell of the instrument clean.
5. Never carry any foreign matter or impurities into bucket so as to avoid pollution of water quality and blockage of pipes.
6. Change water in the water tank at least once half a year
7. Use “System Check” to check each part of system if abnormal incident happens.
8. Wipe the shell of the instrument after cutting off the power supply when it's dirty, use the wet towel with some detergent (soap) to wipe the shell and the bucket slightly, then wipe it by a clean towel to remove the detergent. When operating, make sure the following (Or else it will be all on the user's risk):
 - a) Before wiping, make sure the power supply is cut off in order to avoid the electric shock and damages to the instrument.
 - b) When wiping the bucket, block the inlet hole and the filter hole by the clean towel in order to avoid the detergent to enter into the water pump and water tank thus to effect the test result.
 - c) When wiping, avoid any water into the instrument thus lead to the malfunction.

6.2— Maintenance of Oxygen Bomb

1. Before the test, be sure to fill adequate oxygen into empty oxygen bomb and immerse the filled oxygen bomb in water for several minutes so as to observe its sealing performance to ensure reliable test results.
2. After test completion each time, be sure to flush bomb components with distilled water and wipe them dry with special towel.
3. Change the sealing ring in use by standby ring if oxygen leakage from bomb has been found, otherwise bomb can not be used any longer.
4. Never use oxygen bomb when its screw threads have failed.
5. Inspect oxygen bomb at regular interval. Carry out hydraulic test at least once each year (to

be presented to MRC for such test).

6. Be sure to carefully take out and place oxygen bomb from and into bucket to avoid bucket damage due to collision or drop.
7. Be sure to clean the crucible after use. Better bake it at high temperature of electric oven for 3-5 minutes.

The crucibles and the oxygen bombs are not prohibited to be placed in corrosive

8. environment.

6.3 Maintenance of Micro-type Oxygen Filler

1. Oxygen used for test must satisfy GB213-2003 requirements. It is forbidden to use electrolytic oxygen. Place oxygen bottles at the site in conformity to regulations
2. Place oxygen fillers on stable work bench.
3. It is forbidden to let oxygen fillers (incl. oxygen filling conduit, pressure reducing valve) contact oil and grease.
4. Avoid bending and twisting oxygen filling conduit on oxygen filler.
5. It is forbidden to have open fire around oxygen filler.
6. Change with new oxygen in case of oxygen pressure in oxygen bottle is lower than 5Mpa. Release oxygen from oxygen bomb, reinstall the bomb and refill oxygen in case of filling pressure is higher than 3Mpa.

Chapter 7 FAQ and solutions

In this chapter, we will introduce some knowledge for the maintenance and malfunction solutions, if the user can not solve it, please contact MRC.

7.1— Can not be on-line

If there is no bucket number shown in the progress indication window of the main window, the reasons may be as follows:

1. The power supply was off, check if the power switch and the air switch on the back panel of the calorimeter were on and check if the inverter was in order.
2. USBCAN communication card was not well connected , please connect the USBCAN interface card with the computer and the calorimeter.
3. The communication cable on the AD was loosened.
4. USBCAN service program was not started, please restart the program.
5. The program was damaged by the virus, reinstall the software after running the antivirus.

If the bucket number shown and disappear alternately in the progress indication window of the main window

- ~~1.~~ 1. Some parts of the instruments was poor contacted.
- ~~2.~~ 2. The software edition didn't match.
- ~~3.~~ 3. Some problem with the AD card.

If the malfunction can't be solved still, please contact MRC.

7.2— Can not enter test status

If the calorimeter was online but can not enter the test status, the reasons may be as follows:

1. The jacket temperature was not in the temperature controlling range, please wait for a moment.
2. The sample weight was not input, or the sample weight in not matched or the status of the bomb is not normal.
3. System abnormal prompted.

If the malfunction can't be solved still, please contact MRC.

7.3— No prompt of bomb connected

First, fix the firing wire on the electrode rod, if there is the prompt of bomb connected, then it must be the bomb or the poor contact between the bomb and the electrode rod, deal with it as follows:

1. Check if the bomb and the electrode rod are well contacted, if not, please polish the bomb cover, air nozzle and the electrode rod.
2. Check if the firing wire and the electrode rod are well contacted, if not, please polish the

electrode rod. If there is prompt after polishing, then it is because of the bomb: check the electrode rod if it's broken first, then check the I/O card.

If the malfunction can't be solved still, please contact MRC.

7.4— Prompt “bomb short circuit”

If the prompt was shown before closing the cover, it's because of the calorimeter, please do as follows:

1. Check if the electrode rod is short circuited.
2. Check if the I/O card is in order.

If the prompt was shown after closing the cover, it's because of the bomb or the water, please do as follows:

1. If the effective distance of the firing wire connected in the electrode rod was too short. ~~1.—~~
2. If the fire baffle and the electrode rod was connected together. ~~2.—~~
3. If the firing wire and the crucible support was connected together. ~~3.—~~
4. If the sealing rings and gaskets were aged and led to short circuit. ~~4.—~~

If there is not firing wire in the bomb, the prompt was shown after closing the cover, it's because of the calorimeter itself or the water.

If the malfunction can't be solved still, please contact MRC.

7.5 —No stirring

1. The motor for stirrer was stuck by impurities.
2. The stirring bar was blocked by impurities.
3. The motor for stirrer can not pump water(the stirrer was power on and there was heat emit)

If the malfunction can't be solved still, please contact MRC.

7.6 —Ignition failure

Check if the sample was burned:

The sample was burned:

1. The calorific value of the sample is too low or not flammable: Please add additives to the samples and test again.
2. If the stirrer worked in order.
3. If the temperature probe was damaged.

The sample was not burned:

1. No oxygen or oxygen insufficient.
2. Leakage with the bomb.
3. Forgot to fix the firing wire.
4. Check if the firing wire was fused: If yes, the firing wire was fixed far away from the sample, or the sample was not flammable; If not, the contact resistance between the electrode rod on the cover and the bomb is too large, or the contact resistance between the firing wire and the bomb is too large.

- I/O card or relay expansion card may be damaged.

If the malfunction can't be solved still, please contact MRC.

7.7— Gas leakage with the oxygen bomb

If the bomb was not badly damaged, four places should be check for the leakage:

- Air nozzle leakage or can not be filled or released: Open the air nozzle and clean the valve core in it, and replace the sealing ring on the cut-off valve and clean it, at the same time clean the air nozzle of the bomb.
- The joint of air nozzle and bomb core: twist the air nozzle tightly again.
- The insulation covering of the bomb core: twist it tightly or change the insulation covering.
- The cap of the bomb: change the sealing ring of the bomb core.

If the malfunction can't be solved still, please contact MRC.

7.8 Gas leakage with the oxygen filler

If there is gas leakage with the oxygen filler, five places should be checked:

- The switch of the oxygen filler: Twist down the switch and check the sealing ring, if it's not damaged, please apply some Vaseline, or else please change the three sealing rings.
- The indicator of the oxygen filler: Twist down the indicator and replace the sealing ring and fix back.
- The joint of oxygen conduit and the oxygen filler: Twine with plastic tape and twist tightly or replace it.
- The joint of the oxygen conduit and the pressure reducing valve: Twine with plastic tape and twist tightly or replace it.
- The joint of the pressure reducing valve and the oxygen bottle: Twine with plastic tape and twist tightly or replace it.

If the malfunction can't be solved still, please contact OTE.

7.9 System prompt “water insufficient” or “ water level of the water tank abnormal”

Prompt “water insufficient”:

- The water in the water tank is insufficient, please open the calorimeter cover, and feed water into the bucket according to the bucket until the water level is normal.
- The plug for the water level determination in the I/O card is not well contacted or disconnected.

Prompt “water level of the water tank abnormal”:

- The plug for the water level determination in the I/O card is not well contacted or disconnected.

If the malfunction can't be solved still, please contact MRC.

7.10 Too long time for temperature balance or can not enter into temperature balance

1. The water in the bucket was not enough or no water in it.
2. The heater of the bucket was damaged or the power was not enough.
3. There was no power supply for the heater.

If the malfunction can't be solved still, please contact MRC.

7.11 Test result is not ideal

1. If the operation is as the instructions.
2. If the weighing balance is in order and if the weighing is accurate.
3. If the sample weight is correctly input.
4. The sample particle size and the uniformity is according to the standard.
5. If the sample was deflagrated or if the sample was spilled out from the crucible or the sample was not completed burned.
6. The oxygen in the bomb was not enough or the oxygen filling time was not the same, or if the bomb was leaked.
7. The water of the bucket was not enough or the temperature probing not good.(Please call for service) .
8. If the sealing ring of the calorimeter cover was not tight enough.
9. Stirring efficiency was not enough.
10. Sometimes added 10ml water into the bomb and sometimes not.
11. The calorimeter was not well ground.

If the malfunction can't be solved still, please contact MRC.

7.12 Can not be printed

1. The print copies were set as 0 in the data processing or the paper type was wrongly set.
2. The set print format is not matched with the selected record, such as the selected record is not matched when printing the parallel sample.

Chapter 8 Determination Method for Heat Capacity and Calorific value

When automatically printing parallel samples or set “choose qualified records when printing parallel samples” in database, the determination method for parallel samples heat capacity and calorific value is as follows:

- **Determination Method for Heat Capacity**

If there are more than 7 heat capacity data provided that bucket number at current day, the oxygen bomb number and the controlling temperature are the same, choose 5 data with the least precision from the latest 7 data. That is, when 7 data, arrange them in order firstly. Choose the average value among 1-5, 2-6 and 3-7 provided that precision is less than setting precision. The same way to calculate in case there are 5-6 data.

- **Determination Method for Calorific value**

If the calorific values with same sample number are more than 4, choose the latest 4 data. Arrange them in order firstly. If range of 1-4 range is $\leq 156\text{J/g}$, then choose the average value, otherwise average value between 1-3 and 2-4 shall be chosen provided that range is $\leq 144\text{J/g}$. If it doesn't meet the condition yet, when range of 2-3 is $\leq 120\text{J/g}$, average value shall be chosen. Otherwise qualified average value is not displayed. If there are 3 data, the rest can be done with the same manner.

If there is no setting mentioned above, the determination for parallel samples the heat capacity and the calorific value is as follows:

- **Heat capacity**

The heat capacity data whose bucket number at current day, the oxygen bomb number and the controlling temperature are the same are treated as parallel samples to calculate the average value only, it will not be treated as the heat capacity of the instrument.

- **Calorific value**

All the calorific value data in the data list are treat as the parallel samples to calculate the average value.

Chapter 9 Formula and Description

1. Cooling calibration value $C = nV_0 + (V_n - V_0) / (T_n - T_0) [(T_0 - T_n) / 2 + \text{integral value in principal period} - nT_0]$

Among V_0 ——bucket temperature-fall speed while firing, K/min;
 V_n ——bucket temperature-fall speed at final point, K/min;
 T_0 ——bucket temperature while firing, K;
 T_n ——bucket temperature at final point, K;

integral value in principal period——existing integral value +current temperature* (time lasting for current temperature—time lasting for previous temperature)

2. Formula for heat capacity

instrument heat capacity $E = (1.0015 * Q * m + q_1 + q_2) / (T_n - T_0 + C)$

Q —— calorific value of benzoic acid, J/g;
 m —— weight of benzoic acid, g;
 q_1 ——calorific value of firing wire, J;
 q_2 ——calorific value of additives, J.

3. Formula for calorific value

- calorific value of determination in oxygen bomb

- $Q_{b,ad} = [E (T_n - T_0 + C) - q_1 - q_2] / m$

(If “calorific value of determination in oxygen bomb includes nitric acid formation heat” is cancelled in system setting, $Q_{b,ad} = Q_{b,ad} - a * m * Q_{b,ad}$

- gross calorific value $Q_{gr,ad} = Q_{b,ad} - (94.1 S_{b,ad} + a Q_{b,ad})$
- constant-volume net calorific value $Q_{net,v,ar} = (Q_{gr,ad} - 206 H_{ad}) (100 - M_{ar}) / (100 - M_{ad}) - 23 M_{ar}$
- gross calorific value as dry basis $Q_{gr,d} = 100 Q_{gr,ad} / (100 - M_{ad})$
- sulfur in oxygen bomb $S_{b,ad} = (C * V / m - a Q_{b,ad} / 60) * 1.6$

among C —— mole concentration of sodium hydroxide solution, about 0.1mol/L;

V —— volume of sodium hydroxide solution used by titration, mL;

60 —— formation heat equivalent to 1mmol of nitric acid, J.

$Q_{b,ad}$ ——calorific value of determination in oxygen bomb, MJ/kg

a —— calibration coefficient of nitric acid:

when $Q_b \leq 16.70 \text{ MJ/kg}$, $a = 0.0010$;

When $16.70 \text{ MJ/kg} < Q_b \leq 25.10 \text{ MJ/kg}$, $a = 0.0012$

when $Q_b > 25.10 \text{ MJ/kg}$, $a = 0.0016$;

- conversion of additives weight
heat value of additives = calorific value of additives * additives weight

4. Formula for calorific value of CWM

- The formula for calorific value of CWM Dry sample is the same with the formula for calorific value of air dry basis coal sample.
- Formula for CWM:
 - ◆ calorific value of determination in oxygen bomb
 $Q_{b,cwm} = [E (T_n - T_0 + C) - q_1 - q_2] / m$
(“Calorific value of determination in oxygen bomb includes nitric acid formation heat” is cancelled in system setting,

$$Q_{b,ad} = Q_{b,ad} - a * m * Q_{b,ad} \quad Q_{b,cwm} = Q_{b,cwm} - 0.0015 * m * Q_{b,cwm}$$

- ◆ gross calorific value $Q_{gr,cwm} = Q_{b,cwm} - (94.1 S_{b,cwm} + a Q_{b,cwm})$

The two parameter $S_{b,cwm}$ and a , the determination is the same with air dry basis coal sample.

- ◆ constant-volume net calorific value $Q_{net,v,cwm} = Q_{gr,cwm} - 206 H_{cwm} - 23 M_{cwm}$

In it, H_{cwm} 、 M_{cwm} represents the mass fraction of CWM hydrogen and moisture, %

Note: CWM sample doesn't have the parameter index of Gross calorific value air dry basis.

5. Formula for oil sample

- **light oil**

$$H_{ad} = 1.196 \times Q_{b,d} / 1000 - 41.4$$

$$Q_{gr,ad} = Q_{b,ad} - 95 S_{b,ad} - 50$$

$$Q_{net,ad} = Q_{gr,ad} - 225.8 H_{ad}$$

- **heavy oil**

$$H_{ad} = 1.124 \times Q_{b,d} / 1000 - 37.6$$

$$Q_{gr,ad} = Q_{b,ad} - 95 S_{b,ad} - 42$$

$$Q_{net,ad} = Q_{gr,ad} - 225.8 H_{ad} - 25.1 M_{ad}$$

6. Basis conversion $Q_d = Q_{ad} \times 100 / (100 - M_{ad})$

7. Others:

a) Message on Main Screen (Bucket number 1~x)

Parameter input:

Test information:

Real-time test message:

Time display:

b) System Parameter

- **Digit of Result**

Sample weight four-digit decimal unit (g)

temperature four-digit decimal unit (°C)

C calibration value four-digit decimal unit (K)

Temperature rise four-digit decimal unit (K/min)
Two-digit for others

● **Sample Weight**

Coal:	Weight range (0.3000~1.5000) g
Light oil:	Weight range (0.2000~0.7000) g
Heavy oil:	Weight range (0.2000~0.7000) g
User-defined:	Weight range (0.1000~14.9900) g
Biomass:	Weight range (0.3000~2.5000) g
CWM:	Weight range (1.0000~2.3000) g
CWM(Dry):	Weight range (0.3000~1.5000) g
Heat-capacity sample:	Weight range (0.3000~1.5000) g