XRY-1A+

Oxygen Bomb Calorimeter

Operation Manual

Content

This instrument is high precise instrument. Please read the operation manual carefully before using it.

I. Main characteristics and scope

The instrument is designed and made according to GB/T213-2008 Method for the determination of the calorific value of coal, GB/T384-1988 Method for the determination of the heat value of petroleum products and Calibration and Inspection Regulation of People's Republic of China JJG672-2001 Oxygen Bomb Calorimeter, as well as the Shanghai Enterprise Standard Q/YXYY 10 XRY-1 Oxygen Bomb Calorimeter.

The instrument adopts high precision temperature sensor and high-power A/D converter, and uses single chip microprocessor to form an intelligent temperature data collection and transfer system. It uses microcomputer to form an automatic, convenient, rapid, and intelligent instrument. If the user operates the instrument carefully and input all kinds of parameters correctly, the desired measurement data will be getted. The instrument can be widely used to determine the calorific value of materials such as coal, oil, coke, paraffin and other combustible materials etc. It is an ideal option of the thermoelectricity, metallurgical, cement, chemical industries, scientific research institutes, college and universities.

II. Main technical specification and parameters

1、Power supply：AC（220±10%）V 50Hz；

2、Power consumption：≤150W；

3、Heat capacity：(14000～15000)J/K；

4、Temperature resolution: 0.001K；

5、Calorific value：≤60J/g；

6、Temperature controlling range(10～35)℃；

7、Repeatability error (RSD)：≤0.2 %（C grade）；

8、Pressure resistance by oxygen bomb:20MPa；

9、Ambient temperature： (15～28)℃, the temperature fluctuation should not be greater than 1℃ during one determination procedure；

10、Humidity：≤85%；

11. Dimension: 600 ㎜×480 ㎜×460 ㎜

III. Instrument structure and functions

1. Instrument structure:

The instrument adopts integrated structure. Its main parts are shown as Pic 1:

(1) Main unit of calorimeter: It consists of outer bucket, inner bucket, stirring motor and oxygen bomb.

(2) Cover of the water bucket: Double-layer moist cover on both inner and outer bucket. Equipped with stirring motor and temperature sensor.

(3) Stirring motor: Stirring motor for water bath in inner bucket.

(4) Temperature sensor: Precise temperature sensor in inner bucket.

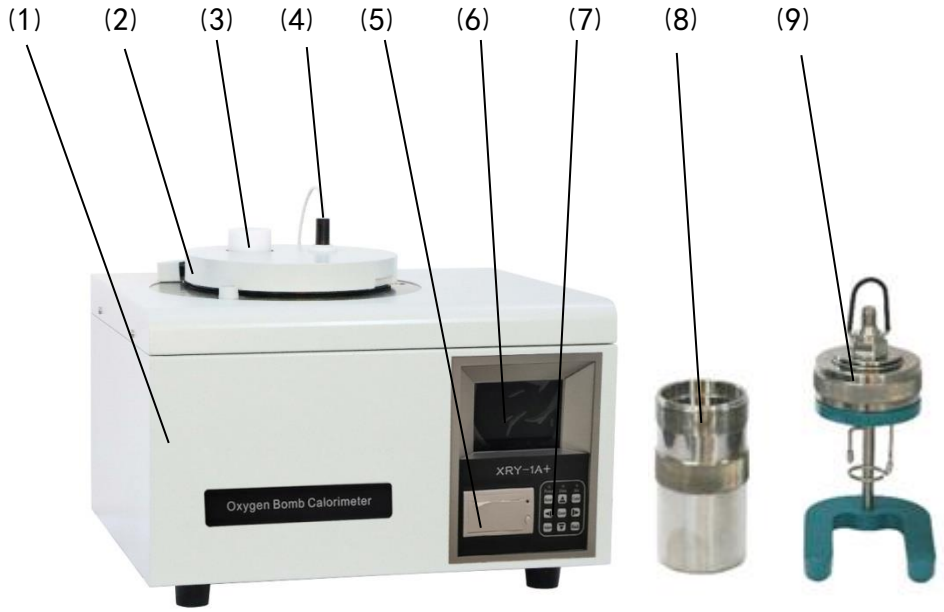
(5) Printer: Micro-needle printer, 40 bytes per row (DBC case).

(6) Screen: Colored LED screen, shows different kinds of parameters.

(7) Operation keyboard: Light-touch type. Details refer to below.

(8) Oxygen bomb cylinder: Stainless steel and high pressure resistance. The sample is tested in the cylinder.

(9) Oxygen bomb head holder



Pic.1

2. Functions introduction:

(1) The main unit of calorimeter is the important part of the calorimeter. It is mainly used to form a constant temperature system, and to ensure it can accurately show the temperature rising curve of the sample under the constant temperature condition during the procedure of ignition and stirring, so that to calculate the heat value of sample.

(2) Bomb head holder, oxygen filling device, and discharging valve: be used to prepare the sample, install the ignition wire, fill oxygen into the bomb and exhaust oxygen

after determination.

(3) Microcomputer(single-chip): one of the most important parts of the instrument. It can automatically control the determination procedure, record and save the test data.

(4) Printer: print out the test data.

IV. Operation procedures

1. Instrument installation

(1) Main unit installation: Place it on a flat table. Keep from other substance more than 10cm around to ease the wire connection and to avoid any interfere to the constant temperature system. Don't place heating or refrigeration device around the main unit.

(2) Wire connection: There are stirring motor wire, ignition connection wire and temperature sensor wire in the main unit. The plugs of these wires are different to avoid operation mistakes.

(3) Ignition and stirring test: start the controlling software, and press the "ctrl"＋ "↑" button (at the same time) or the "ctrl"＋ "→" button (at the same time), it will switch on the ignition circuit or run the stirrer.

2. Determination preparation

(1) Fill water (about 18Kg) into the outer bucket until the outer bucket is full of water. But it should not overflow during manually stirring. In order to let the testing temperature uniform quickly, it is better to leave the water in the room for more than half a day. And the operator should stir the water in the bucket for ten more times after filling it into the outer bucket (the red handle in outer bucket is the manual stirring rod)

(2) Weigh the sample: weigh a certain amount of sample (nearest to 0.0002g) and then place it into crucible.

Note: Weigh 1 g benzoic acid (about two pieces), nearest to 0.0002 g for calibration.

(3) Install the ignition wire: place the bomb lid on the bomb head holder. Get an ignition wire, the length of which is about 9cm (Ni-Cr wire). The ignition wire should just touch the sample, and two ends of ignition wire should be hung on two conductive poles with slot (one of them is the crucible bracket.). Then lock two ends using sleeves. **Note:** keep the ignition wire away from the crucible or metal parts of the oxygen bomb to avoid ignition failure. There is a circular shield above the crucible to avoid burning the

airproof ring.

(4) Fill oxygen: Pour 10 ml distilled water into bomb and screw the bomb lid. Then connect the oxygen filling device to the oxygen flask, and connect the oxygen pipe to the oxygen bomb. Then open the gas valve to fill the oxygen into the bomb slowly. The filling pressure should be limited within 2.5－3.0Mpa. The filling time should not be less than 30s. Put the bomb filled with oxygen into water to check whether it is leaking. If there is no bubble, it indicates that it is not leaking.

Warning: The oxygen bomb is a high pressure-resistant container, please fill oxygen at the correct pressure. Filling oxygen at a higher pressure is not allowed.

(5) Fill water into inner bucket: put the oxygen bomb on the bomb seat in the inner bucket, and then fill distilled water (about 3000g, the water surface is near 2/3 of the oxygen filling valve nut) into the inner bucket; the mass of water for each determination should be the same (error must be ≤±1g). The temperature of the inner bucket should be 0.2K－0.5K lower than that of outer bucket, so that the temperature curve will decrease obviously when the temperature of inner bucket is higher than that of outer bucket at end of the determination. Then put the inner bucket on the outer bucket's insulating seat. The position of the bracket has been regulated before leaving factory, to ensure the same location for each test.

(6) Close the cover of the outer bucket well.

(7) Insert the temperature sensor well

(8) Switch on the red power supply at the back of the instrument, and then the indication light of power on the control panel will be on.

3. Microcomputer control

(1) Switch on the instrument to run the controlling software. The instrument is English interface, so it is convenient and easy to operate.

(2) Select "Bunte determination", "R-P determination", "Bunte calibrate" or "R-P calibrate" (Different item corresponds to different formulas).

(3) Input the mass of the benzoic acid or sample, heat capacity of the instrument, and the additional heat value.

(4) Select the Start item and press the Enter button, then the instrument begins to make calibration or measurement automatically.

(5) Select to calculate the gross or net calorific value when the whole determination

is over (It is not necessary for instrument calibration).

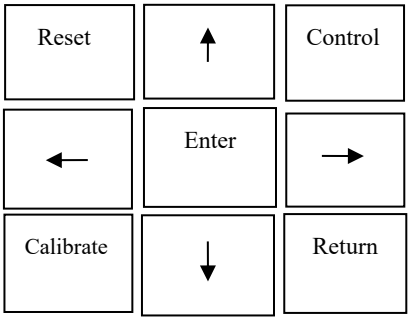
(6) Then select to print at once or later. **Note:** Although the instrument is switch off (power off), the data is still kept in the instrument. When the instrument is restarted, these data can be used until the next determination begins. So operator can also print out the data before next determination.

V. Operation methods of software

The controlling software is the combination of "English menu operation" + "button confirm", so its operation is simple and intuitional.

1. Buttons

There are nine buttons on the instrument panel, such as Reset, Control, Enter, Calibrate, Return, Left "←", Up "↑", Right "→" and Down "↓". They are shown as follows, they'll be introduced one by one as followings:



Pic.2

(1) The Reset button: if the controlling software is interfered by something wrong and locked, then operator can press the Reset button to resume it. This button is seldom to be used.

(2) The Return button: Quit of certain submenu or certain states.

(3) The Control button: it is used with some buttons together, as the function button; By now it only has two functions "control" + "↑" = "ignition manually", and "controlling" + "→"= "stirring manually". And they are used only when debugging the instrument or pre-checking before determination. During the determination, it is not necessary to control the instrument manually and the instrument will run automatically. **Note:** If the operator have used these two functions, he should turn off the power supply

and restart determination.

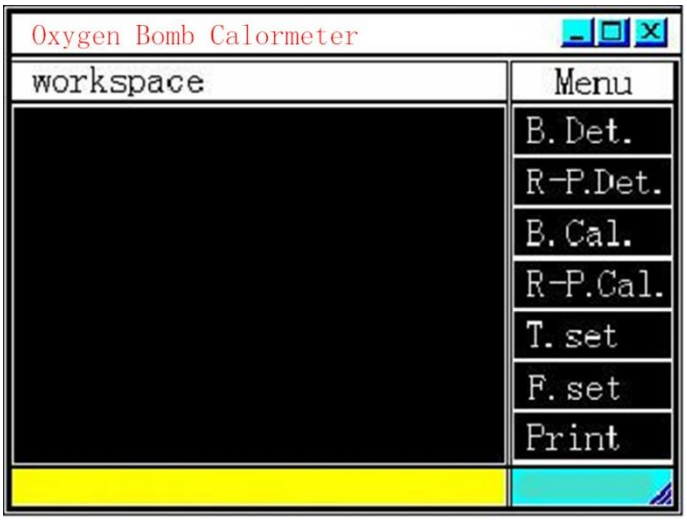
4) The Enter button: it is used to confirm the choice or operation.

5) The buttons of "←","↑","↓"and"→": They are used to move the highlight cursor. Also form the combination function button with Control button too.

6) The Calibrate button: Reserved for the manufacturer, not used by operator.

2. Operation interface

It is shown as Pic.3:



Pic.3

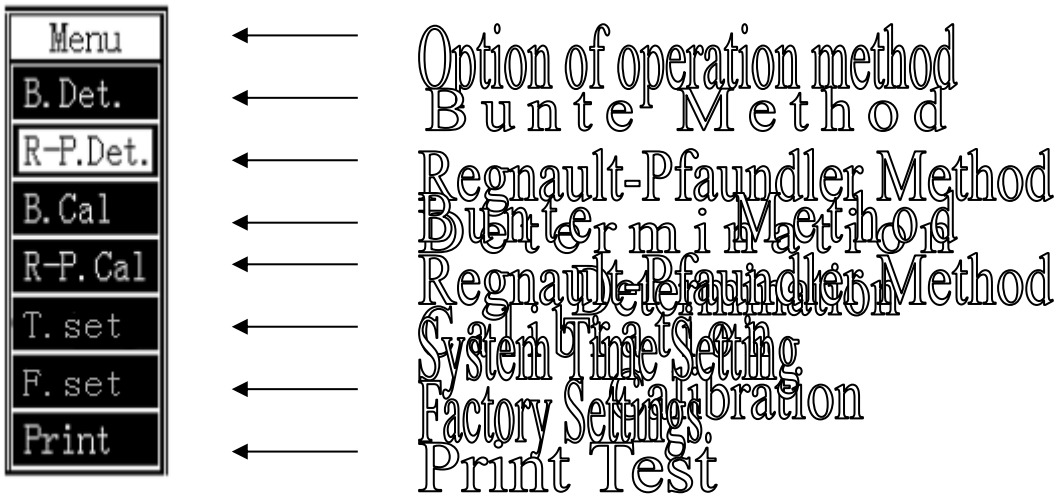
3. Menu:

It is shown as Pic.4:

(1) Bunte method: It adopts the Bunte method to make determination and calculation, and its submenu is similar to the Regnault-Pfaundler method.

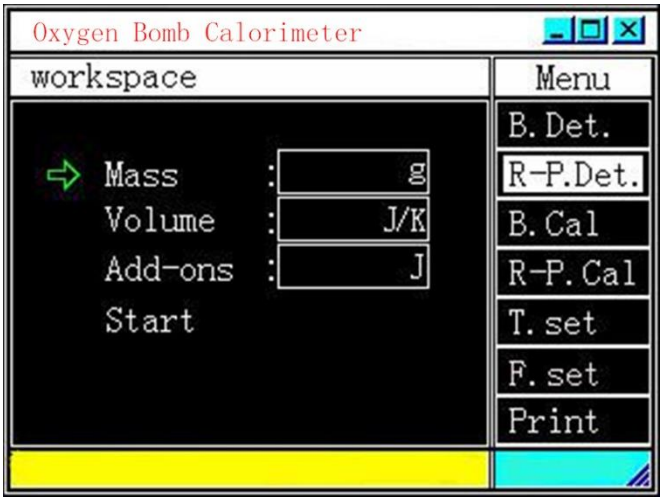
(2) R—P method: It adopts the Regnault-Pfaundler method to make determination and calculation, and its submenu is shown as Pic.5.

Note: Bunte method is suitable for determination of National Standard of People's Republic of China GB/T384 Method for the determination of the heat value of petroleum products. R-P method is suitable for determination of National Standard of People's Republic of China GB/T213 "Method for the determination of the calorific value of coal".



Pic.4

●After choosing the R—P method, it is necessary to modify the instrument parameters: Press the Enter button, and then the "_" cursor appears. Then use the buttons of "←" , "↑" , "↓"and"→" to decrease or increase the number value of each digit. Press the Enter button after input.



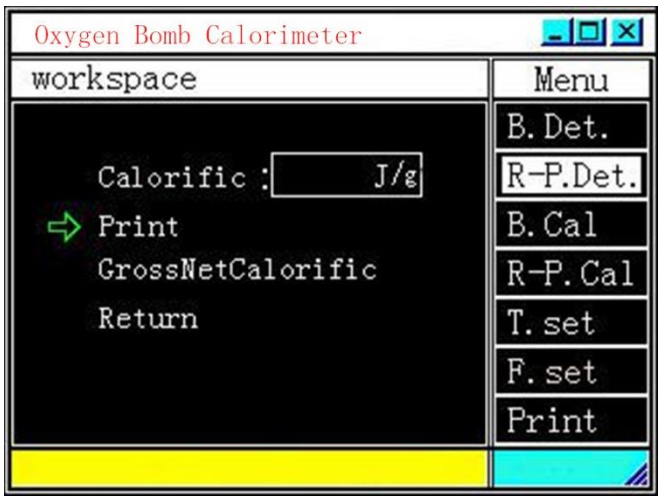
Pic.5

●After inputting the sample mass (Mass), heat capacity of the instrument (Volume), and the additional heat (Add-ons), move the cursor to the Start item, and press the Enter button. (The indication lights of determination and stirring should be off before measurement. If not, the operator should turn off the power supply to restart the determination.)

●The indication light of stirring should be on after it starts measurement. But indication light of determination is still off until ignition. And it is shown as Pic.6:

Pic.6

●After initial period (record temperature for 6—10 times), ignition, main period (about 20 times) and final period (10 times), the following interface will appear(Pic.7).



Pic.7

The operator can select the Gross or Net Calorific Value item (determine, calculate), or the Print item (automatically exit after printing and doesn't calculate the gross or net calorific value).

●If the operator selects the Gross or Net Calorific Value item, the interface is shown as Pic.8:

1)After determining the sulfur content, hydrogen content, total moisture(Mt), and air-dried moisture (Mad), press the Next item to print test data.

2)When the determination is over, the stirrer will stop automatically. The indication lights of determination and stirring will be off.

3) The operation interfaces of Bunter calibration and R—P calibration are the same

as the interfaces described above, so we do not describe it repeatedly.

Pic.8

4. Time set

(1) The instrument can show the "year/month/day" and "hour: minute: second" automatically. After selecting the T.set menu, the operator can modify them. The time has been set before leaving factory. If the operator change the battery or repair the instrument, please reset the time.

(2) The method to input time is similar to the method to input instrument parameters described above, so we do not describe it again.

5. Factory settings

Warning: Select the F.set item to make the parameters back to factory settings. This item is especially reserved for the manufacturer. User should not select it casually in order to avoid disordering the system.

6. Print test data

(1) The operator can print test data by the prompt menu, or print it once more using the print menu.

(2) About the usage of the printer, please see the part of Appendix 3.

VI. Instrument calibration and sample determination

1. Calibrate the instrument

Though the instrument has been calibrated and checked before leaving factory, the operator should re-calibrate the instrument according to the factual circumstance. It is because the operation surroundings are different.

Calibration is used to determine the heat capacity of the calorimeter (instrument coefficient). It is related to the constant temperature system, season, lab circumstance, so the instrument must be calibrated at least once before use. Determine standard benzoic acid sample, the calorific value of which is known, for 5 times for each calibration. If the difference between any two determinations is less than 60 J/K, then record the average value of these determinations as the heat capacity of the instrument. If the difference is more than 60 J/K, then determine one more time, and eliminate the error value at the same time, until it can meet the requirement that the difference is less than 60 J/K.

The valid period of the heat capacity calibration value is 3 months, and the instrument should be re-calibrated after 3 months.

Heat capacity of the instrument will be influenced when some important parts are changed (such as bomb, bomb lid and so on). For this reason, the instrument also should be re-calibrated.

2. Sample determination

Sample determination is the main goal to use the calorimeter; the operation procedures are similar to that of instrument calibration.

The sample only need to be determined once, and the operator can select the determination items as requirements (such as the calorific value of sample, gross or net calorific value and so on.).

3. Determine the gross or net calorific value

If it is necessary to calculate the gross or net calorific value after sample determination, please use proper method to determinate the hydrogen contents, sulfur contents, total moisture (Mt) and air-dried moisture (Mad), and input them into the corresponding text box. Then the controlling software will calculate the gross or net calorific value automatically (Please refer to the methods to determine these parameters mentioned above in the professional book).

VII. Attention and maintenance

1. Preparation before determination

(1) Please read the Operation Manual thoroughly before use.

(2) Please read the GB/T213-2008 Method for the determination of the calorific value of coal, or GB/T384-1988 Method for the determination of the heat value of petroleum products in the national standard, as well as the JJG 672-2001 Oxygen Bomb

Calorimeter in the Calibration and Inspection Regulation of People's Republic of China to acquaint and familiar with test regulations, test methods, test procedures and requirements.

(3) Check work environment of the instrument, and ensure it is in accordance with the working environment and working condition stipulated in the Operation Manual.

(4) Check the instrument shell to ensure it is well grounded.

2. Attention during determination

(1) The instrument should be placed in a single room without any sunshine. The workbench is flat and the ambient temperature meets operation requirements of the instrument. In order to ensure the determination accuracy, the temperature change in the room should not be more than 1.0K during each determination. Ensure there is no heat source and air convection.

(2) Select the grade two or higher benzoic acid, which has been calibrated by the calibration department and marked with calorific value, as the standard material

(3) Use the industrial oxygen, the purity of which is more than 99.5%. It is forbidden to use electrolysis oxygen.

(4) The instrument has been equipped with the Ni-Cr ignition wire before leaving factory. If the operator uses other materials as ignition material, please input its calorific value in the text box of additional heat.

(5) Keep the surface of the instrument clean and dry. Do not let water flow into the instrument to avoid any damage to the instrument's circuit board. Especially do not fill too much water into the outer bucket to avoid any damage to the circuit board caused by the overflowing water.

3. Daily maintenance

(1) Check the oxygen bomb using 20Mpa hydraulic pressure regularly, at least once a year.

(2) Clean the oil dirty on the oxygen decompression device to avoid explosion during filling oxygen. And the oxygen decompression device should be checked regularly, at least once a year.

(3) The airproof ring should be checked before use. If the airproof ring is damaged, please change it at once to avoid any leaking.

(4) The instrument should be kept clean and dry after use to prevent it from rust. If it

won't be used for a long time, please exhaust water in the instrument, clean and dry the instrument, and keep it in a dry area.

(5) Add lubricating oil to the rotation shaft of stirring motor every year or every half a year (it is due to the use frequency).

Note: If the ambient temperature is lower than 0 ℃, please drain off water all in the water bath to avoid any damage to the instrument caused by freezing!

VIII. Common problems and solutions

No.	Problem	Cause	Solution
1	Indication light is off.	1.No power supply. 2.The fuse is damaged.	1.Connect the power supply 2.Replace the fuse.
2	Can not be ignited.	1.The ignition electric pole is poor connected with oxygen bomb. 2.The ignition wire is short circuited.	1.Make sure the electric pole and oxygen bomb well connected. 2.Install the ignition wire again.
3	Stirrer doesn't work	1.The stirring motor is blocked. 2.The stirring motor wire is disconnected.	1.Get rid of the blocks. 2.Connect the wire again.
4	Temperature doesn't decrease	1.Water temperature in outer bucket is much lower than ambient temperature. 2.The sample mass is too less.	1.Make determination again after regulating the outer bucket temperature. 2.Use more sample to rise the temperature 2 K~3K.
5	Clock stops	1. the clock has no power	1.Replace battery 2.Manually input time to continue determination.

Warning: If there is any breakdown, please cut off the power supply immediately. The operator can use the instrument again only after it be repaired by professional to avoid accidents!

IX. Printing format

1. Printing report for sample determination

Note: Bunte method report is similar to the R—P method report.

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————R-P method determination————

2010-04-13 15:27

Sample No.: _____

Sample mass	1.0127g
Heat capacity	14474J/K
Add-on heat	40J/K
Sulfur contents (Sbad)	0.20%
Hydrogen contents (Had)	0.20%
Total moisture(Mt)	0.40%
Air-dried moisture (Mad)	0.30%
Calorific value of sample:	26526 J/g
Gross calorific value:	26456 J/g
Net calorific value:	26388 J/g

Initial period temperature

23.846℃	23.848℃
23.851℃	23.853 ℃
23.856℃	23.858℃

Main period temperature

23.980℃	24.530℃
24.966℃	25.229℃
25.384℃	25.484℃
25.549℃	25.594℃
25.626℃	25.649℃
25.666℃	25.679℃
25.689℃	25.696℃
25.701℃	25.705℃
25.709℃	25.711℃
25.712℃	25.714℃
25.715℃	25.715℃
25.716℃	25.716℃
25.715℃	

Final period temperature

25.715℃	25.715℃
25.715℃	25.714℃
25.714℃	25.714℃
25.714℃	25.713℃
25.713℃	25.713℃

Operator: _____

2. Calibration report:

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————R—P method calibrate————

2010-04-15 13：02

Sample No.: _____

Benzoic acid mass 1.0191g

Benzoic acid heat value 26466J/g

Heat capacity 14469J/K

Initial period temperature

22.476℃	22.476℃
22.477℃	22.479℃
22.479℃	22.480℃

Main period temperature

22.548℃	22.999℃
23.430℃	23.737℃
23.926℃	24.047℃
24.127℃	24.184℃
24.224℃	24.253℃
24.275℃	24.291℃
24.302℃	24.311℃
24.318℃	24.323℃
24.327℃	24.329℃
24.331℃	24.332℃
24.333℃	24.333℃
24.333℃	24.333℃
24.333℃	24.333℃
24.332℃	

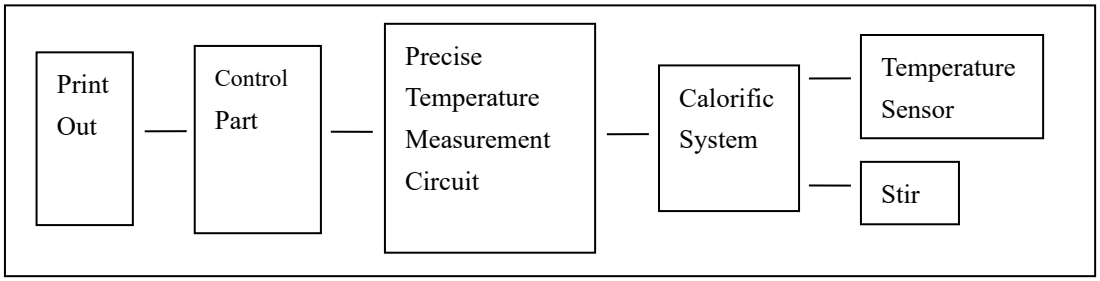
Final period temperature

24.331℃	24.331℃
24.330℃	24.330℃
24.329℃	24.328℃
24.327℃	24.326℃
24.326℃	24.325℃

Operator:_____

3. Principle diagram

Main unit of calorimeter



X. Optional spare parts

1. Benzoic acid 2. Standard coal 3. Oxygen filling device

4. Ignition wire 5. Pellet press machine

XI. Full set and technical documents

1. Full set

No.	Name	Unit	Quantity
1	XYR-1A+ Main unit (including single chip, oxygen bomb, water jacket, water bucket, stirrer)	set	1
2	Oxygen bomb head holder	set	1
3	Oxygen bomb seat	set	1
4	Temperature sensor	piece	1
5	Thermometer（0-50℃）	piece	1
6	Stopper for thermometer (silica gel stopper 2 #)	piece	1
7	Ignition wire (Φ0.1 Nickel-Chromium wire)	meter	10

8	Crucible	piece	2
9	Discharging valve	piece	1
10	Oxygen decompression device	piece	1
11	Oxygen pipe	pair	1
12	O type seal ring（φ20×2.4mm）	piece	5
13	O type seal ring（φ8×1.9mm）	piece	5
14	O type seal ring（φ6×1.9mm）	piece	5
15	Airproof ring (largest O type)	piece	2
16	Benzoic acid	piece	10
17	Water aspirator	piece	1
18	Printing paper	roll	1
19	Stopping ring for thermometer (1#)	piece	4
20	Silicon stopper (7#)	piece	1

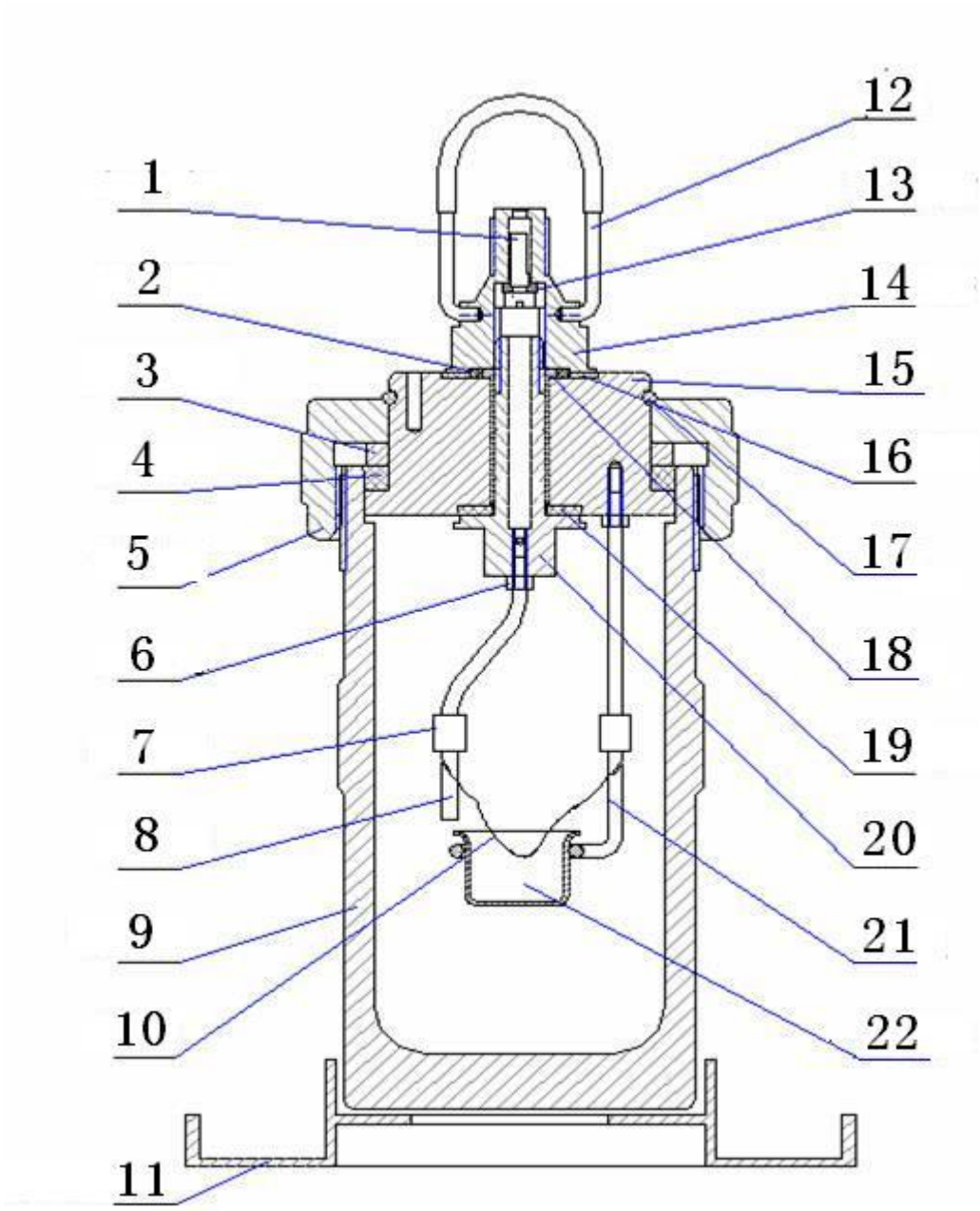
2. Technical Documents

(1) Operation Manual 1 piece

(2) Repair guarantee 1 piece

(3) Quality Certificate 1 piece

Appendix 1: Oxygen Bomb Diagram



1. Valve handle 2. O type airproof ring 3. Fixing ring for airproof ring
4. Airproof ring 5. Bomb cylinder nut 6. Hexagon nut
7. Conductive sleeve 8. Conductive pole 9. Bomb cylinder
10. Ignition wire 11. Oxygen bomb seat 12. Handle ring
13. O type airproof ring 14. Bomb top nut 15. Bomb lid
16. Large insulation gasket 17. Clamp spring 18. Insulation pipe
19. Small insulation gasket 20. Gas valve 21. Crucible holder

22. Crucible

Appendix 2: Concept of Absolute Temperature

Thermodynamic temperature is also called as Kelvin temperature or absolute temperature, and its symbol is K. The temperature at absolute zero is defined as 0 K. The three-phase point of water, which is expressed as the temperature when the water is in the liquid, solid, and gas state at the same time, is defined as 273.16 K.

The temperature when the water freezes at standard atmospheric pressure is at Celsius temperature of 0 ℃, or Fahrenheit temperature of 32 ^{O}F. The temperature is equal to thermodynamic temperature of 273.16 K.

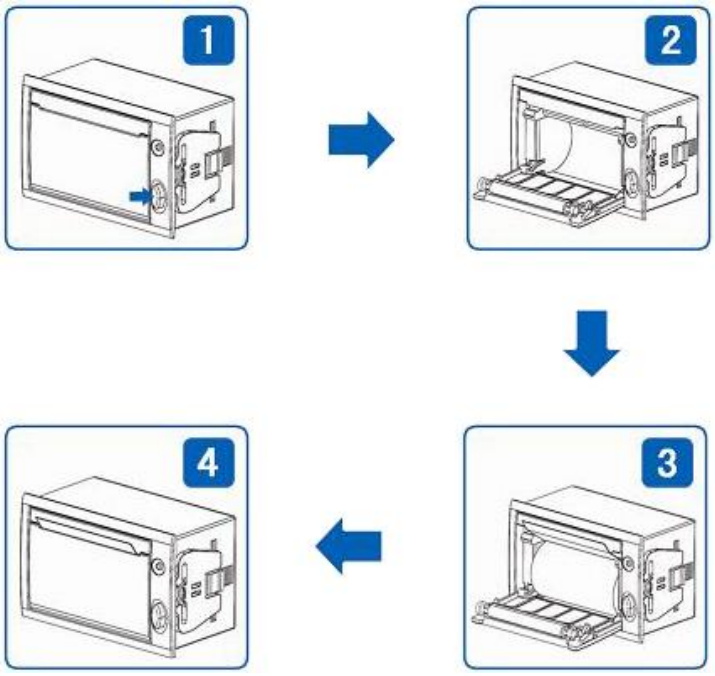
The relationship between absolute temperature and Celsius temperature:

Absolute temperature T≈ Celsius temperature t +273 degree;

Absolute temperature 0 degree≈ Celsius temperature of 273 degree;

Appendix 3: Instruction of Printer

1.Installation of printing paper



(1) As it shows in Pic.1 above, lightly push down the part showed by arrowhead, the

cover of paper storing box will open.

⑵ Put the printing paper in and pull out a little length（Over a little tearing teeth). Pay attention to put the paper in order. The side of the paper with liquid drug(glossy surface) should be put upward. See Pic.3.

(3) Close the cover of paper storing box. After use the print head paper-going axis to press printing paper orderly, press the print head paper-going axis back to the print head.

⑷ Switch on the power of the printer, then press the LF button to make the head rolling. If the paper goes deviously, press the LF button again.

2.Maintenance of the printer

（1） After a long time use of the thermal printer, some dirts will left on the thermosensitive bar and rolling axis. The usage and lifetime will be affected if the operator do not clean it in time. So the Irregular clean is suggested.

⑵ How to clean: Open the paper storing box. Moisten the cotton swab with a little alcohol liquid. Clean the thermosensitive head and rolling axis on the paper storing box lightly.