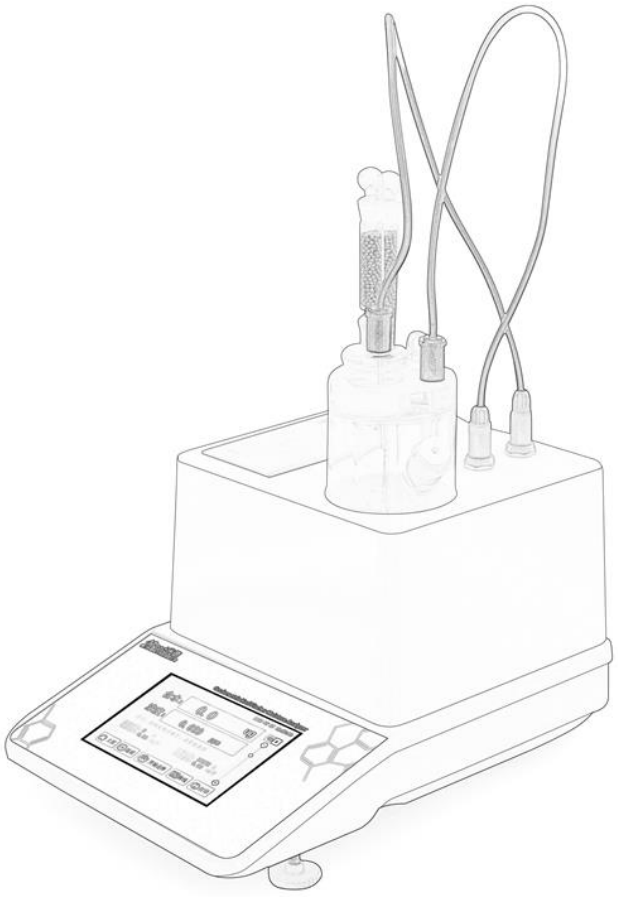
Karl Fischer Moisture Analyzer Manual



Catalog

Chapter 1、Overview

Karl Fischer moisture analyzer is a new generation of trace moisture measuring instrument newly developed by our company. The instrument is designed based on the principle of coulomb titration and using a circuit topology. It has the advantages of high precision, fast electrolysis speed, short equilibrium time, accurate blank deduction, and accurate and reliable analysis results.

1.1 Product Parameters

Principle method: Karl Fischer Coulomb method;
Electrolysis current: ≤500mA;
Measuring range: 3μg～100mg;
Moisture weight resolution: 0.1μg;
Moisture content resolution: 0.001%/ppm;
Accuracy: 3μg (10μg～100μg H_2O),
≤0.3% (more than 100μg H_2O) (excluding sampling error);
Sensitivity: 0.1μg H_2O;
Operation: pure touch operation;
Storage data: 496 groups;
Printing: thermal printing;
Display: 1024×600, 5-inch WVGA (800×480);
Power supply: AC220V, 50Hz, about 30W;
Ambient temperature: 5℃～40℃;
Environmental humidity: ≤55%;
Overall dimensions: 320*220*200mm;

Chapter 2、Appearance and Working Principle

2.1 Appearance

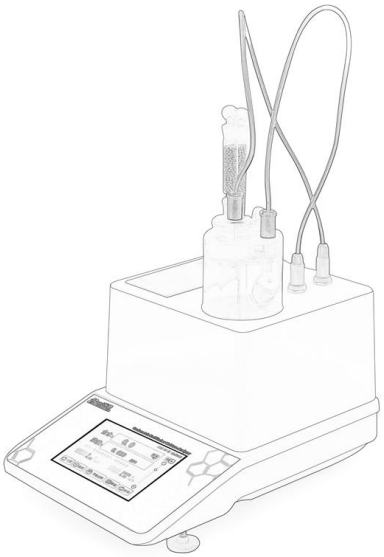


Figure 2-1

2.2 Working Principle

The chemical equation for the reaction between Karl Fischer reagent and water is:

$$I_2+SO_2+3C_5H_5N+H_2O \rightarrow 2C_5H_5N \cdot HI+C_5H_5N \cdot SO_3$$

$$C_5H_5N \cdot SO_3+CH_3OH \rightarrow C_5H_5N \cdot HSO_4CH_3$$

The reagent used is a mixture of a certain concentration of elemental iodine and methanol with dissolved sulfur dioxide. The basis for measurement is that a certain concentration of elemental iodine will undergo the following reaction on the electrolysis electrode after electrolysis:

$$2I^- - 2e \rightarrow I_2$$

$$I_2 + 2e \rightarrow 2I^-$$

When the concentration of elemental iodine changes within a certain range, it will lead to effective changes in the conductivity of the solution. The higher the concentration of elemental iodine, the greater the change in the conductivity of the solution is taken as the effective range of measurement. The electrical conductivity of the solution is reflected by measuring the current value generated at both ends of the platinum electrode. Inject the sample into the electrolyte, and the water in the sample will participate in the reaction. The change of iodine during the reaction can be measured by the instrument. The consumption of iodine can be calculated based on the amount of electricity used to electrolyze the same amount of iodine. The water in the sample will consume the same amount of iodine, so the weight of the water can be calculated directly.

Chapter 3、Acceptance and Installation

3.1 Acceptance

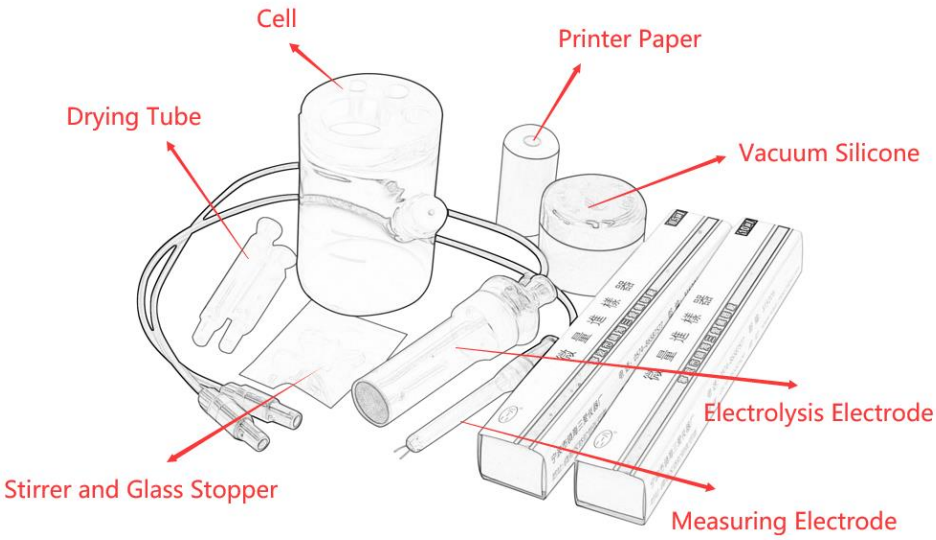
After the instrument has been shipped. First, the appearance of the packaging box should be inspected to see if it has been damaged due to overturning, collision, or falling during transportation. When unpacking, you should carefully check the instrument and accessories, and check whether the accessories are complete according to the product list. If there are any errors, please notify the manufacturer in time.

3.2 Installation environment

The instrument should be placed in a dust-free laboratory. Indoor temperature conditions are between 5℃ ~ 40℃, and the maximum relative humidity is not greater than 55%. The power supply required for the instrument is: AC220±5V, frequency 50±0.5Hz, and current not less than 2A. The neutral wire of the laboratory power distribution system must be well grounded. The instrument should be placed on a level table and a certain distance from the wall. The instrument cannot be placed in direct sunlight! The instrument must not be installed in a room with corrosive gases. Corrosive gases will corrode the instrument circuit and shorten the life of the instrument. The instrument must not be installed in places with high humidity.

3.3 Installation

3.3.1 Place all accessories on the table.

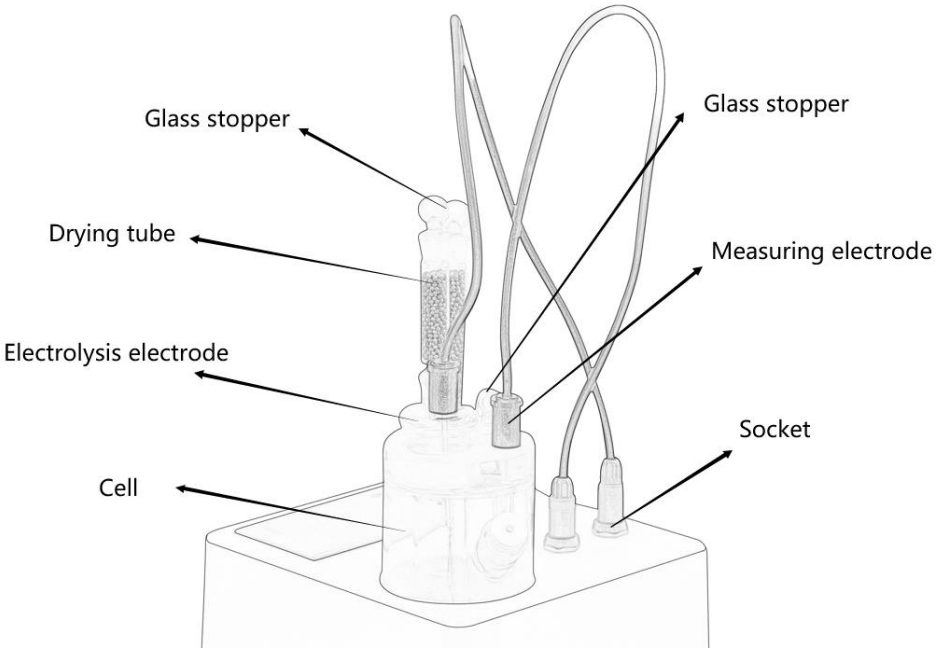


3.3.2 Place the electrolytic cell on the card holder of the Karl Fischer moisture analyzer. Place the stirring bar into the electrolytic cell.

3.3.3 Remove the probe protective cover of the measuring electrode. Insert the measuring electrode into the socket on the electrolytic cell and insert the plug into the corresponding socket. Note: There are corresponding electrodes written next to the socket. In addition, the color of the socket also corresponds to the color of the electrode plug.

3.3.4 Put blue desiccant into the two drying tubes and block the tube openings with glass stoppers. Insert the remaining larger glass stopper and the long drying tube into the jack of the electrolytic cell.

3.3.5 Insert the electrolytic electrode into the socket of the electrolytic cell and insert the plug into the corresponding socket. Then insert the short drying tube into the jack of the electrolytic electrode. The schematic diagram of the assembled instrument is as follows:



3.3.6 Pick up the electrolytic electrode and pour the reagent into the electrolytic cell through the jack of the electrolytic electrode. Pour it between the two horizontal lines on the electrolytic cell bottle, and then insert the electrolytic electrode back into the jack.

3.3.7 Press the button on the printer, open the cover, put the printing paper roll in, and close the cover. Note that there must be some distance between the printing paper.

Notice:
1、Vacuum silicone grease should be applied to the grinding edges of the electrolytic electrode, measuring electrode and glass plug that are in contact with the electrolytic cell. It has two purposes: one is to ensure that water vapor from the external environment will not invade the electrolytic cell, causing rapid failure of the reagents. The second is to prevent the electrolytic electrode, measuring electrode and glass plug from being embedded in the electrolytic cell jack under the action of external air pressure, making it impossible to pull them out.
2、Rotate the electrolytic electrode, measuring electrode and glass plug once a week to prevent the vacuum silicone grease from solidifying.
3、Set a fixed time every month to clean the electrolytic electrode and measuring electrode. Do not use water. You can use organic solvents such as methanol for cleaning. After cleaning, place the electrolytic electrode and measuring electrode in a drying box and dry them at 80 ℃ for 12 hours.
4、When installing the measuring electrode, pay attention to whether the two probes on the measuring electrode are in contact with each other. Make sure that the two probes are separated.
5、Pay attention to the color change of the desiccant. If it changes from blue to light or colorless, you need to replace it with a new one.
6、After the measurement is completed, the instrument will automatically print. If there is no writing on the paper, it means that the printing paper is installed backwards. Adjust the direction of the printing paper.

Chapter 4、Operation and Use

4.1 General operating instructions

The instrument is equipped with a high-sensitivity touch screen. Click any valid key area on the screen, and the instrument will automatically switch to the corresponding interface for corresponding operations.

When you want to input parameters, click the position of the corresponding parameter, and the instrument will pop up a numeric keyboard for parameter input, as shown in Figure 4-1. The function description of each button is as follows:

The numeric keys "0-9" and the decimal point key "." are used to directly enter the corresponding numbers;

The backspace key "←" is used to delete incorrect numbers during input;

The "Cancel" key is used to cancel this input operation;

The "Confirm" key is used to confirm that the data entered this time is valid. If the data is within the allowed range, the newly entered data will be automatically displayed in the corresponding position of the corresponding parameter after pressing the confirmation key; if the data is invalid, it will not be displayed and needs to be re-entered.

Figure 4-1

4.2 Power on

After the electrolytic cell is assembled, connect to the 220V power supply. Press the power button and the instrument will automatically enter the test interface. Click "Home" in the lower left corner of the test interface to enter the main interface. The main interface is shown in Figure 4-2. In the main interface, click the function button, and the instrument can switch to other corresponding functions. The specific instructions are as follows:

"Settings" function: used to set test options, date and time, sound and display equipment, language, communication and other parameters;

"Test" function: used to measure the moisture content of samples;

"Method" function is used to select the concentration calculation formula and enter the corresponding parameters;

"Data" function, used to query historical data;

"Help" function is used to view information, common faults and precautions related to the instrument.

Figure 4-2

4.3 Test Option Settings

Before conducting a test, you should ensure that the options or parameters related to the test process are set correctly.

Click the "Test" button in the main menu interface to enter the test interface, and then click "Options" to enter the test options interface, as shown in Figure 4-3.

In this interface, you can set various parameters related to the test process, such as test method, injection delay time, maximum electrolysis current and other options. After the settings are completed and confirmed, click the "OK" button to save the set parameters. You can also click "Return" to return directly to

the test interface. At this time, the new setting parameters will not be saved.

Current test method: used to set the currently used test method. The instrument has six test methods, and the specific parameter settings of each method can be changed. When selecting the current test method, make sure that the parameters of the selected test method are set correctly. Please refer to 4.4 Test Method Settings.

Sample Injection delay: used to set the delay time from pressing the "Start Injection" key to completing the injection, ranging from 0-99 seconds. During the actual measurement process, in order to ensure that the measurement results can be obtained correctly, the injection must be completed within the delay time after pressing the "Start Injection" key.

Max electrolysis current: used to set the maximum electrolysis current value during the measurement process, ranging from 1 to 500mA.

Max electrolysis time: the maximum time for testing. At the specified time, even if the measurement has not been completed, the instrument will directly stop measuring and calculate the measured moisture content.

Auto print results: When this option is turned on, the instrument will automatically print the test results after the measurement is completed.

Auto save results: When this option is turned on, the instrument automatically saves the test results after the measurement is completed.

Sample No: The number of the sample to be tested. After entering this item, the instrument will print this item when printing the results so that it can be used to distinguish the test results.

Background rejecion: Used to deduct blank current.

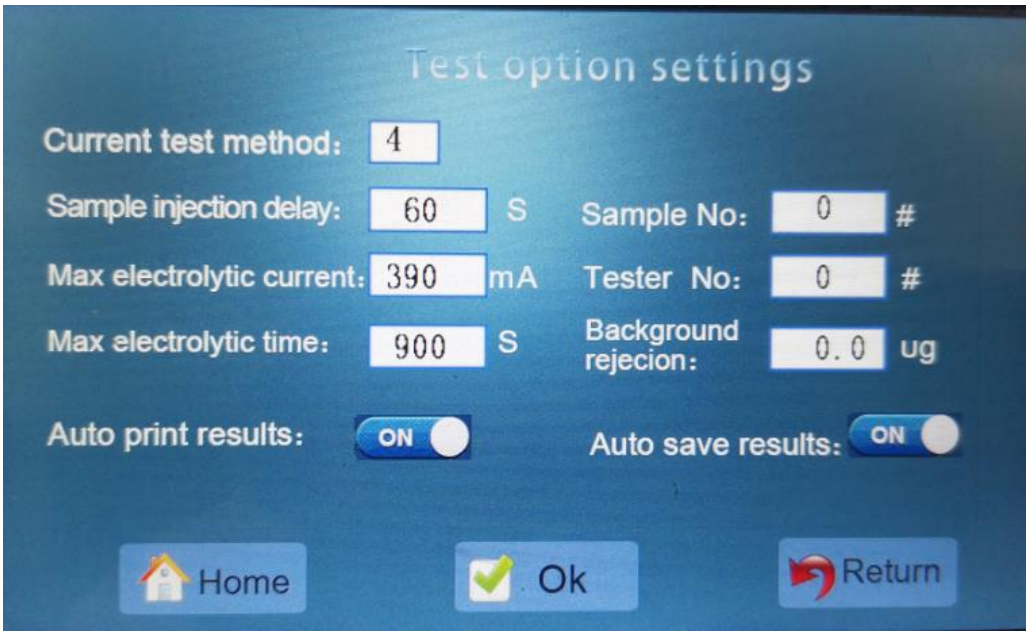


Figure 4-3

4.4 Test Method Settings

Click the "Method" button in the main interface to enter the setting method menu interface, as shown in Figure 4-4. You can select any one of eight concentration formulas as the measured concentration formula. Click the drop-down box after "Concentration Formula:" and a list of concentration formulas will pop up, as shown in Figure 4-5. Select a concentration formula and enter the parameters related to the formula. After the input is completed, press the "Save" key to store the parameter settings related to this method, and press the "Apply" key to use this method as the currently valid test method.

Figure 4-4

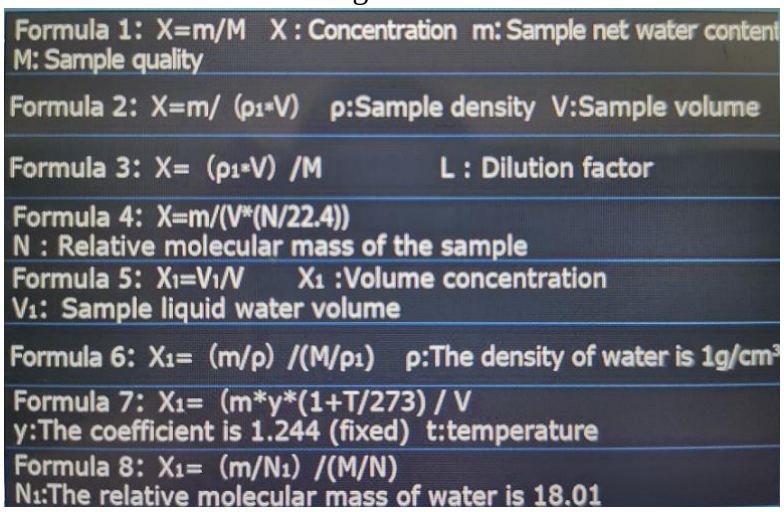


Figure 4-5

4.5 Blank current deduction

The amount of blank current has a great influence on the measurement accuracy. The instrument has the functions of measuring blank current and subtracting blank current.

After the interface displays "It is read. Please inject the sample", press the "Sample Injection" button, wait for the measurement to end, the buzzer will sound, and the water content value will be displayed on the test interface. Repeat the above operation several times and calculate the average value. Click "Options" on the test interface, find “background rejection”, enter the calculated average value in the corresponding column, then click "OK" to save, and then click "Return" to return to the test interface.

4.6 Calibration of the instrument

When the instrument is ready, pure water can be used to check the measurement accuracy. The inspection method is as follows:

(1) Use a 0.5μl microsampler to extract 0.2μl of pure water and wipe the needle clean.

(2) Press the "Sample Injection" button to start the delay.

(3) Inject pure water into the electrolytic cell through the injection port before the delay ends. The needle tip must be inserted into the electrolyte and avoid contact with the inner wall of the titration cell or the electrode. The titration will start automatically after the delay.

(4) The buzzer sounds and the instrument reaches the end point. The displayed result should be 200μg±13μg water (including injection error). Repeat 2 to 3 times. If the number is within the error range, it means that the measurement accuracy of the instrument is accurate.

4.7 Measurement Operations

Please refer to the table below for sample sampling volume:

Moisture Content	Injection Volume
100%	2mg～1mg
50%	5mg～2mg
10%	20mg～5mg
1%	200mg～20mg
0.10%	2g～200mg
0.01%	2g 以上

Turn on the power of the instrument, enter the test interface, and press the "+" and "-" keys to adjust the stirring speed of the stirrer, usually to the middle.

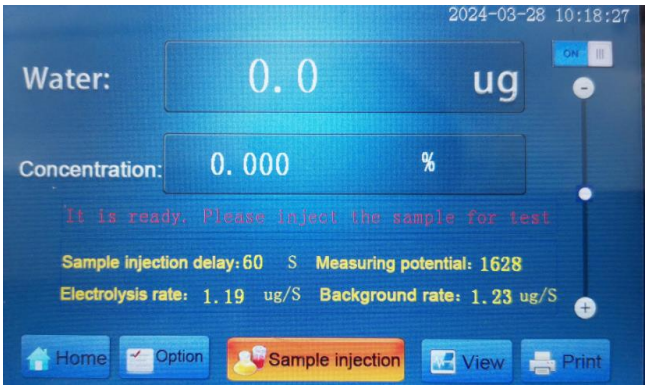


Figure 4-6

The measurement steps are as follows:

4.7.1 The coulometric reagent used for the first time contains excess elemental iodine, the color is dark red, and "iodine status" will be displayed in the middle of the screen. It is necessary to use a 100ul microsyringe to extract 10~20ul of pure water and inject it into the electrolytic cell from the injection port. Repeat the operation until the liquid in the reaction cup becomes lighter in color. At this time, "The instrument is stabilizing, please wait" is displayed in the middle of the instrument.

Note: If too much pure water is added, it will take a long time for the instrument to stabilize.

4.7.2 If the color of the added Karl Fischer reagent is light yellow and "The instrument is stabilizing, please wait" is displayed in the middle of the screen, there is no need to add water and just wait for the instrument to stabilize.

4.7.3 When the instrument is stable, the middle position of the interface will display “It is ready.Please inject the sample for test”, and the instrument can be used normally.

4.7.4 It is recommended to set the "Sample Injection Delay" to 60 seconds. Set “Max electrolysis time” to 900 seconds. If the moisture content of the sample cannot be measured within 15 minutes, reduce the sample amount and re-measure.

4.7.5 Prepare the 0.0001g balance.

4.7.6 Take a sample with a liquid sampler and wipe the needle clean. Place the liquid sampler on the 0.0001g balance and press the tare key to return the weight to zero. After the sample is injected, wipe the needle clean and weigh it on the 0.0001g balance. The value displayed at this time is the weight of the sample injected into the electrolytic cell. The value needs to be accurate to 0.0001g. (You can also record the weight of the injector before and after injection to calculate the injection weight)

4.7.7 After "It is ready. please inject sample for test" is displayed in the screen, click "Sample Injection". After confirming that the injection delay has started counting down, insert the liquid sampler from the injection port and insert the needle into the liquid surface. Be careful to hold the needle tube properly while inserting the needle. After injecting the sample, pull out the injector.

4.7.8 After injecting the sample, the Karl Fischer moisture analyzer starts measuring. Click "Home", "Method" in turn; find "Sample quality" and enter the injected sample weight in the corresponding column, accurate to 0.0001g. Then click "Concentration Unit" and select the desired unit. Finally, click "Apply" to save, click "Return" to return to the test interface, and wait for the measurement to end.

4.7.9 The beep sounds and the measurement ends. The screen will display the values of "water" and "concentration". Among them, "water" refers to the weight of water contained in the sample; "concentration" refers to the moisture content of the sample.

4.7.10 After the instrument is stabilized again, the next test can be started.

Notice

- If you are not sure how much sample should be added, it is recommended to test it once with 0.5g sample, and then adjust the amount of sample that should be added based on the test results. It is generally recommended that the “water” of the measurement results should be no less than 200ug and no more than 1000ug, preferably around 500ug.
- Prepare extra liquid samplers and long needles. A liquid sampler can only be used to measure one type of sample, and the same sample can be sampled repeatedly, but multiple samples cannot be sampled.
- Under normal circumstances, the electrolysis rate at the end of the measurement will be relatively close to the background rate, generally about 0.2ug/S higher than the background rate. If the electrolysis rate at the end of the measurement is much higher than the background rate, there is something wrong with this measurement and the repeatability of the measurement data may be poor.
- Pay attention to the color change of the desiccant in the drying tube. If it changes from blue to light, it needs to be replaced.
- If the liquid level in the electrolytic cell is lower than the lowest white horizontal line on the bottle, reagents need to be replenished.
- If the instrument suddenly terminates the measurement during measurement. It may be that the two platinum wires of the measuring electrode are touching each other. Just separate them.

4.8 Historical data query

In the main menu interface, click the "Data" button to enter the historical data query interface, as shown in Figure 4-7. In the data query interface, you can view the results of each set of the last 500 experiments. Each set of test results has a number. The smaller the number, the closer the time of the set of tests is to now. You can click the "Previous Page" or "Next Page" button to turn pages of measured historical data. Click the check box in the "Select" area to select this set of test results. Click the "Print" icon to print out the selected

test results for each group.

Figure 4-7

4.9 Help

In the main menu interface, click the "Help" button to enter the help interface, as shown in Figure 4-8. In the help interface, you can view relevant information about the instrument such as its name, model, software version number, etc. Click the "Page Turn" button to view relevant information such as precautions for using the instrument and basic operations. You can also directly click the button on the right to quickly switch content.

Figure 4-8

Chapter 5、Precautions

5.1 Precautions for reagents

5.1.1 During the normal measurement process, each 100ml of reagent can react with no less than 1g of water. If used for too long, the sensitivity of the reagent will decrease and new reagents will need to be replaced.
5.1.2 If strong bubbles are found on the surface of the reagent or the color of the reagent turns light reddish brown during the measurement process, the reagent needs to be replaced.
5.1.3 The instrument has not stabilized for more than half an hour. At this time, you should click the stirring knob button to stop stirring, and observe whether there is obvious brown iodine produced on the platinum mesh at the bottom of the electrolytic electrode. If there is no or very little iodine produced, the reagent should be replaced.
5.1.4 If the electrolysis rate remains above 1.0ug/S after the instrument is stabilized, the reagents need to be

replaced.

5.1.5 Be careful not to inhale or touch the electrolyte with your hands. In case of skin contact, rinse thoroughly with tap water. Maintain good ventilation in the test room.

5.2 Precautions for measurement

The typical measurement range of the Karl Fischer moisture meter is 3 μg ~ 100 mg. In order to obtain accurate measurement results, the injection volume of the sample needs to be controlled according to the moisture content of the sample.

5.3 Safety Precautions

Make sure the power supply you are using has a ground wire!

Please do not work in an environment with explosion risk! The instrument shell is not completely sealed, and there is a possibility of explosion caused by the entry of sparks and gases.

When working with chemicals and solvents, follow the manufacturer's instructions and general laboratory safety practices!

All Karl Fischer reagents are flammable and toxic. If Karl Fischer reagent comes into contact with skin, rinse immediately with plenty of water. If your eyes accidentally come into contact with Karl Fischer reagent, rinse immediately with plenty of water and consult a doctor.

If the instrument fails, do not open the instrument case, please contact after-sales personnel.

Chapter 6、Maintenance

6.1 Maintenance of reagents

Store the reagents in a well-ventilated place where the temperature is less than 35℃ and the relative humidity is no more than 75%.

6.2 Replacement of silicone pad

If the silica gel pad of the inlet is used for too long, the shrinkage performance of the silica gel pad will deteriorate and it will not be able to prevent moisture in the atmosphere from entering the electrolytic cell. At this time, the silica gel pad needs to be replaced.

6.3 Desiccant replacement

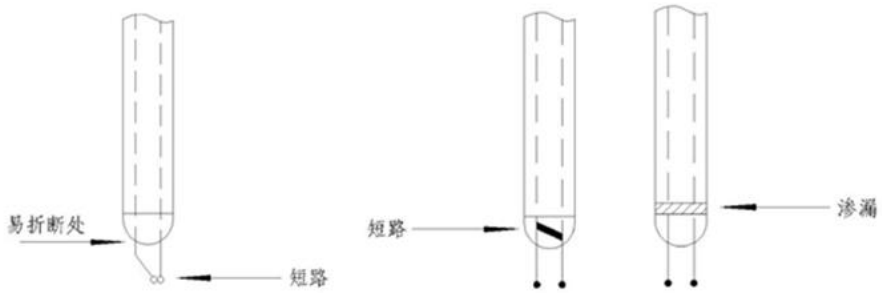
When the desiccant in the drying tube changes from blue to light or colorless, the desiccant should be replaced with a new one.

6.4 Maintenance of electrolytic cell grinding mouth

The electrolytic electrodes, measuring electrodes and glass plugs on the electrolytic cell should be rotated once a week. Once the vacuum silicone grease hardens, the electrolysis electrode cannot be pulled out.

Please drop alcohol on the edge of the grinding mouth and let it soak for a while before trying again. First try to see if you can rotate the electrolytic electrode, measuring electrode and glass plug; as long as they can be rotated, they can be easily pulled out.
(Note: It is not advisable to apply too much vacuum silicone grease, otherwise it may fall into the electrolytic cell and cause measurement errors)

6.5 Maintenance of measuring electrodes



When the magnetic stirrer rotates rapidly, be careful not to let the stirrer beat and damage the measuring electrode.

When putting in or taking out the measuring electrode, turn off the power and be careful not to let the measuring electrode touch the hole wall of the electrolytic cell.

When the instrument prompts a short circuit, take out the measuring electrode and use tweezers to separate the platinum wire at the bottom of the measuring electrode.

When the measuring electrode is contaminated, the measuring electrode can be wiped with acetone. If the dirt on the platinum wire still cannot be removed, please use an alcohol lamp to bake the platinum wire ball end of the measuring electrode. (Note that the ball end of the platinum wire should be slowly brought close to the flame of the alcohol lamp to avoid the electrode glass from exploding due to rapid heating)

6.6 Maintenance of electrolytic electrodes

6.6.1 Disassemble the electrolysis electrode

Because the platinum wire and platinum mesh are stretched out from the cross section of the ground connection part of the electrolytic electrode, when pulling out the electrolytic electrode, be careful not to touch the top and hole wall of the electrolytic cell.

6.6.2 Cleaning of electrolytic electrodes

The following phenomena may occur when the electrolytic electrode is contaminated:

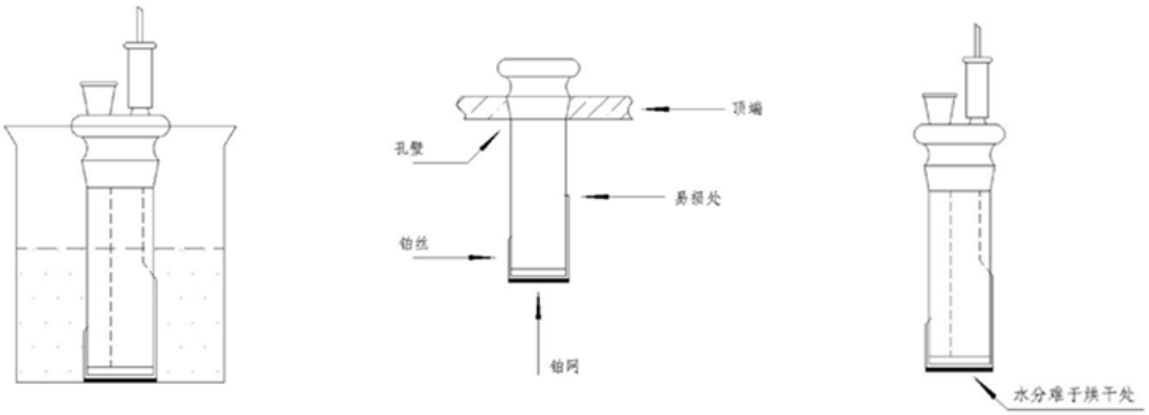
1) The electrolysis efficiency decreases and the measurement time becomes longer.

2) The blank current increases due to the adhesion and absorption of moisture by the contaminated parts.

3) The titration speed is unstable and cannot reach the end point.

If the above situation occurs, you can use alcohol to clean the dirt on the surface of the glass and the platinum mesh (be careful not to damage the platinum wire and platinum mesh). Pour alcohol into the electrolytic electrode, seal the interface of the drying tube with a rubber stopper or similar, and shake it thoroughly to remove internal dirt. Then pour alcohol on the entire outer surface of the glass piece to rinse, but do not rinse the electrode leads. (Be careful not to damage the platinum wire and platinum mesh during

cleaning)



6.6.3 Drying of electrolytic electrodes

Dry the electrolytic electrode with hot air from a hair dryer. The sand core part is a place where moisture is difficult to dry, so it must be dried thoroughly. When there may be residual moisture, the electrolytic electrode can be placed in a vacuum dryer and dried for about 12 hours.

6.7 Maintenance of electrode plugs and sockets

If the plugs and sockets of the measuring electrode and electrolytic electrode are frequently moved, the outside of the plug will gradually loosen. After long-term use, dirt will adhere to the plugs and sockets, which will also cause poor contact, so they need to be trimmed and cleaned.

(1) When the connection between the plug and the socket is loose, use pliers to press the outer metal piece of the plug evenly inward.

(2) Clean the plugs and sockets, wipe the metal parts with alcohol respectively, and wipe off the dirt to ensure good contact.

Chapter 7、Troubleshooting

7.1 Measuring open circuit

When the screen displays "Measuring Open Circuit", perform the following operations:

Check whether the plugs and sockets of the measuring electrode and electrolysis electrode are in good contact;

Check whether the platinum wires at the bottom of the measuring electrode touch together;

Check the leads on the measuring and electrolytic electrodes for breaks.

7.2 Measuring short circuits

When the screen shows a short circuit:

Check whether the measuring electrode plug or socket is short-circuited;

Check whether the measuring electrode is short-circuited;

Check whether the measuring electrode is leaking, causing the instrument to fail to reach the end point. If the measuring electrode leaks, the instrument cannot reach the end point even if the titration time exceeds

half an hour (this is not a reagent problem).

When replacing new reagents, an electrode short circuit may also occur. At this time, you can slowly drip in a small amount of distilled water until the screen displays normally.

7.3 Electrolysis circuit

When the screen displays "Electrolysis Circuit":

Check whether the electrolytic electrode plug and socket are connected;

Check whether the lead on the electrolysis electrode is connected properly or broken;

When measuring oil products, if more samples are injected into the electrolytic cell but are not discharged in time, the oil samples will accumulate on the platinum mesh on the outer layer of the electrolytic electrode, making the electrolytic electrode unable to contact the reagents, resulting in an electrolysis circuit. At this time, the excess oil sample should be extracted in time.

Appendix: Terms of Service

The Karl Fischer moisture analyzer is an analytical instrument product researched and developed by our company. Our company is responsible for the after-sales service of the instrument.

Statement as follows：

- Under normal conditions of use:

From the date you receive the product, if it is installed and used in accordance with the instruction manual, if there is a non-human quality problem with the core components within one year, we will provide free repairs; other accessories have a one-year warranty.

- Damage caused by human factors, force majeure and other factors:

The product will be sent back to the designated location for repair by the user. Qunlong Instruments will charge accessories fees and appropriate maintenance labor fees. The repaired product will be given a 6-month quality guarantee; during this period, if the same fault occurs during normal use, free repair will be provided.

- The warranty only covers the instrument structure, electronic circuits, and non-vulnerable components; it does not include the following parts:

Sampler, electrolytic cell, measuring electrode, electrolytic electrode, drying tube