Labotronics

Karl Fischer Titrator

LB-10KFT

Index

1. Safety Measures

- Place the electrolytic cell in a dark place. The indoor temperature is between 5°C and 35 °C and the humidity is less than 75%. Avoid sun exposure and disable the reagent.
- It is recommended to place the room with low ambient humidity to extend the life of the reagent and facilitate the instrument to reach the endpoint quickly after starting up.
- If the instrument is not used for a long time, a large amount of air vapor will enter the pool bottle, which will be absorbed by the ceramic filter plate in the electrolytic electrode.
- After the start-up, the moisture in the ceramic filter plate will be slowly released to the reagent of the large pool bottle.
- In the meantime, it is difficult toreach the balance point in the instrument for a long time.
- It is recommended to start the work every day.
- Be careful not to inhale or touch the reagents by hand. If it is in contact with theskin, rinse it off with water promptly.
- The reagent is corrosive, please keep it in a safe place.
- Do not keep the instrument in direct sunlight or a dusty place.

2. Introduction

Karl Fischer titrator LB-10KFT is a volumetric moisture testing unit programmed by Itanium and a double CPU with direct A/D conversion for testing electrode data. It is characterized by auto stirring and analyzing function. The enclosed titration system avoids deviations and improves the quality index.

3. Features

1. Electric current auto-control and compensation by microcontroller unit
2. Karl Fischer reagent for 1000 continuous sample testing
3. Digital display and operation by touch LCD panel
4. Preinstalled with cubature, statistical calculation and elementary arithmetic
5. High sensitivity and quick analyzing speed
6. Cold rolled steel plate exterior with electrostatic anti-corrosive layer
7. Automatic data printing
8. Network management through USB or RS232 interface communication

4. Specifications

Model	LB-10KFT
Testing range	0.01 µg – 200 mg H_2O
Water content range	0.0001 % - 100%
Sensitivity	0.01 µg H_2O
Rate of electrolysis	2.4 mg H_2O
Electrolysis current	Auto control within 430 mA
Accuracy	Water content 2 µg H_2O Deviations ≤± 1 µg
	Water content 2 µg H_2O Deviations ≤± 2.9 µg
	Water content > 1000 µg H_2O, Deviations ≤ ± 2%(Excluding deviations of sample injection)
Ambient temperature	5°C ~ 40°C
Relative humidity	< 85%
Power consumption	< 40 W
Power supply	AC 220 V ± 10%, 50 Hz ± 2.5 Hz
Dimension	390 × 270 × 190 mm
Weight	7 kgs

5. Applications

Used in chemical industries, petroleum and power industries, pharmaceutical industries, pesticides, rigs, natural gas plants, etc.

6. Instrument Introduction

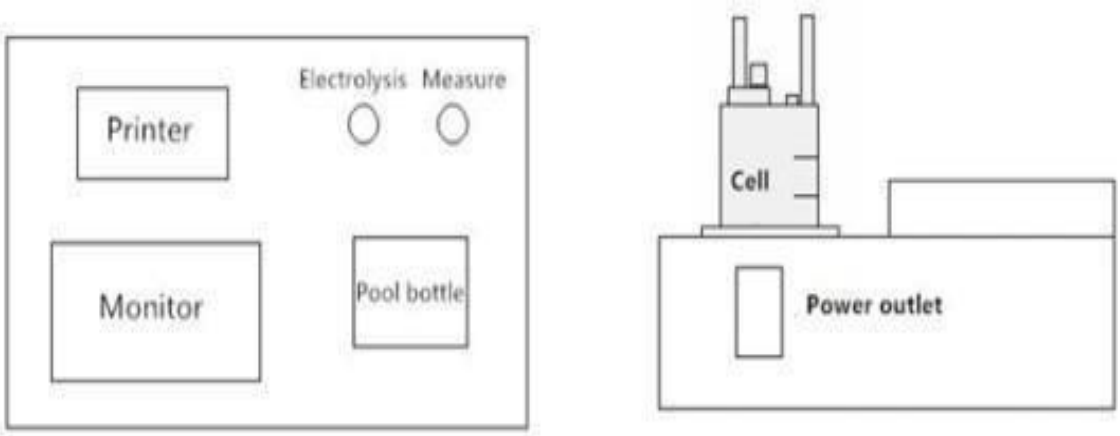

Figure.1

Electrolysis cell structure and assembly

1) Electrolytic cell decomposition

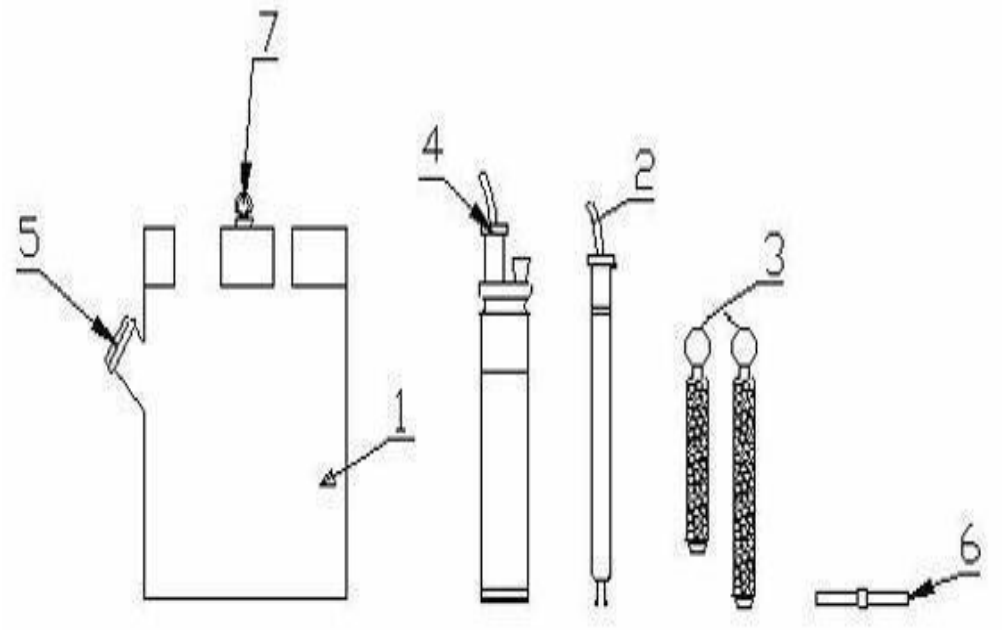

Figure.2

2) After the electrolytic cell bottle is assembled

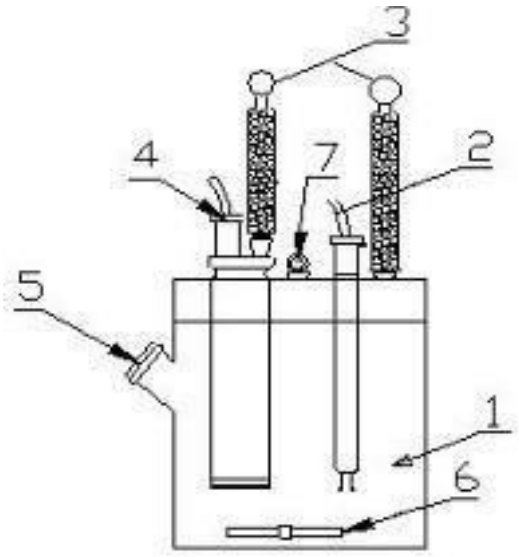

Figure.3

1. Large pool bottle (anode chamber)
2. Measuring electrode
3. Drying tube
4. Electrolytic electrode (cathode chamber)
5. Injection plug
6. Magnetic stir bar
7. Electrolytic cell sealing plug

7. Working Principle

The measuring principle used in this instrument is the Karl-Fischer-Coulomb method. The principle is based on the fact that when water is present, iodine is reduced by sulfur dioxide, and in the presence of pyridine and methanol, pyridine iodine and methyl pyridine thiolate are formed.
The reaction formula is

$I2+SO2+3C5H5N+H20 \rightarrow 2C5H5N \cdot HI+C5H5N \cdot SO3$ ..... (1)
$C5H5N \cdot SO3+CH3OH \rightarrow C5H5N \cdot HSO4CH3$ ..... (2)
During the electrolysis process, the electrode reaction is as follows:
Anode: $2I--2e \rightarrow I2$.....(3)
Cathode: $2H++2e \rightarrow H2\uparrow$..... (4)

The reagent solution used is a mixture of predominant iodine and pyridine- doped pyridine, methanol, etc., and iodine is formed on the anode by electrolysis, and iodine is reacted with water to form hydroiodic acid until all the water has been reacted. The end of the reaction was detected with a platinum electrode.
According to Faraday's law, the formula for calculating the moisture content in the sample is obtained.

$$W = \frac{Q}{10.722} \quad \text{.....(5)}$$

Where: W- moisture content in the sample, in micrograms Q—— Electrolysis power, unit: milli coulomb.
Calculated by the instrument, the moisture content of the tested sample is directly displayed on the display. The instrument uses an electrolysis current automatic control system. The size of the electrolysis current can be determined according to the moisture content of the sample, up to 430 mA.

8. Operations

8.1 Reagent addition

1) Adding reagents in large pool bottles

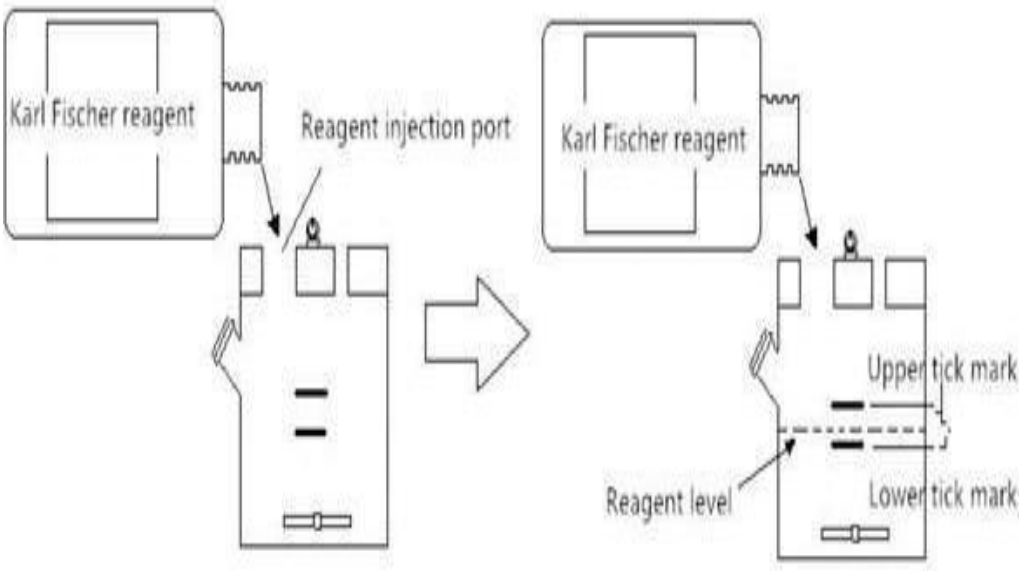

Figure.4

- Inject a new reagent with a dark brown color through the cell mouth.
- Add the middle of the upper and lower tick marks as shown above.

2) Addition of reagent in electrolytic electrode

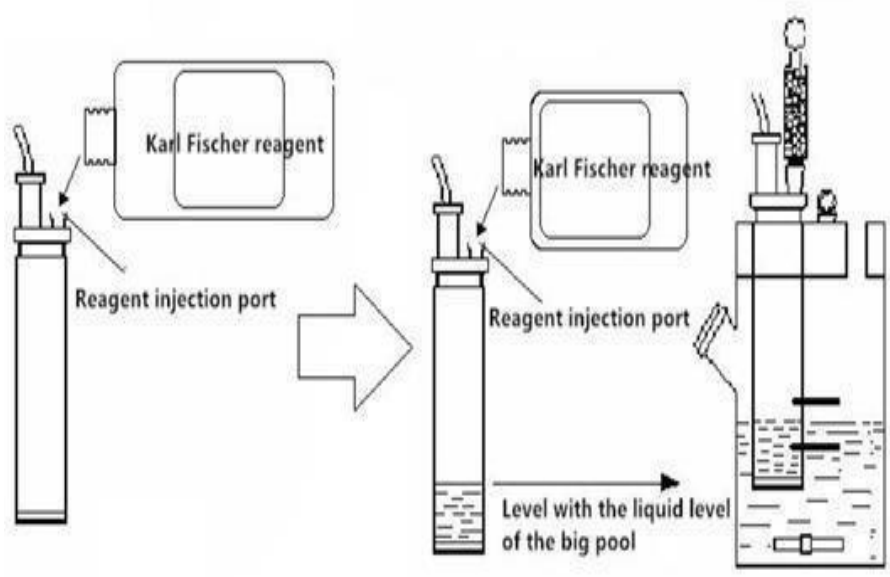

Figure.5

- After the reagent is injected into the electrolysis electrode make it level with the liquid surface of the large pool bottle.

- After completing the above process, carefully place the stirrer through the inlet, and then evenly apply a layer of vacuum grease to the measuring electrode, electrolytic electrode, drying tube, and inlet spigot grinding, on the part. Gently turn it to make it better sealed. And connected to the host.
- Place the cell into the instrument cell compartment. Insert the electrolytic electrode connecting wire with the aviation plug and the measuring electrode connecting wire into the electrolytic electrode socket and the measuring electrode socket respectively; the notch of the plug is inserted into the protruding head of the socket.
- Adjusting the stirring rod speed: There is a vortex in the electrolyte surface, but no excessive bubbles can be generated in the reagent.

3) Reagent failure phenomenon

- The reagent is used for more than one month; the color of the reagent isdarkened, and the display has a regular electrolysis count.
- It is difficult to reach the endpoint in the electrolysis process (the reagent isdark and repeatedly shakes the electrolytic cell repeatedly and repeatedly).

4) Cleaning and drying of electrolytic cell bottles

- The upper and lower platinum mesh inside the electrolytic electrode in the electrolytic cell should be cleaned in time if a black precipitate is found:
- The electrolytic cell bottle, electrolytic electrode, and the like are washed withabsolute ethanol. After washing, it is dried in an oven of less than 50 ° C forabout three hours, and then naturally cooled to add a reagent.

5) Injection seal replacement

- The long-term use of the silicone pad of the inlet will make the pinhole non-shrinking so that the moisture in the air will enter and affect the measurement and should be replaced in time.
- The milky white silicone pad was placed in a cock and screwed in with a fastening stud.

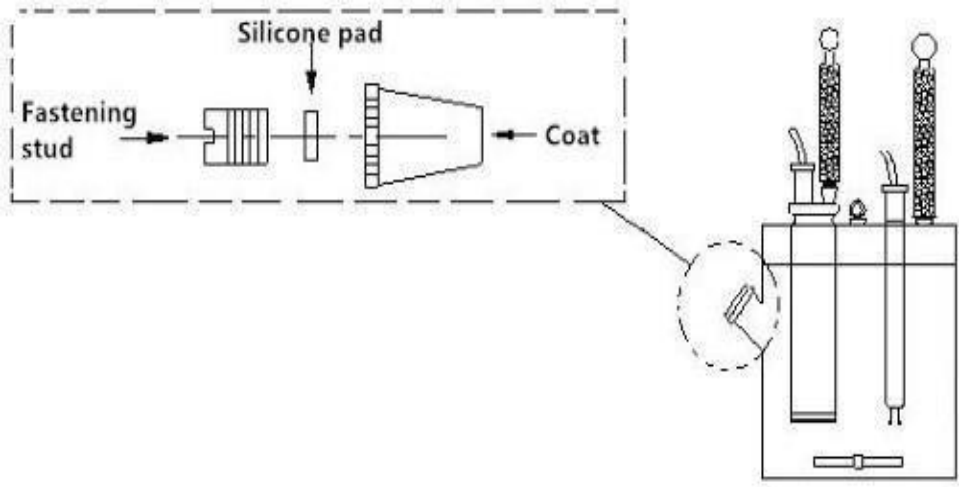

Figure.6

8.2 Reagent dubbing (important step)

The reagent is stored in a tan color, and the color in normal use is light yellow.Therefore, after the reagent is added to the bottle, it is necessary to inject anamount of distilled water, and the reagent is debugged to a pale-yellow colorto achieve a balanced state of the measurable sample. (just need to debug once after replacing the reagent).

1) Boot screen

Turn ON the power switch, and the screen displays "Welcome to the trace moisture meter" analyze the measurement, parameter setting, and history reminder menu.

2) Debugging steps

Connect the electrolytic cell bottle to the instrument socket and press [Analytical Determination] to enter the measurement interface.

- Using a 50 μl sampler, draw 20 μl of distilled water through the inlet and
- slowly inject it into the reagent.
- Repeat the injection of distilled water until it turns pale yellow; (As thedistilled water continues to inject, the color will gradually turn pale yellow.)
- Observe whether the display starts to count electrolysis **[** Reagent status**]** indicates whether **[**Water**]** is displayed. If there are two phenomena above, it.
- It means that too much-distilled water is injected, and water injection must be stopped immediately; wait for the instrument to automatically inject excess water. The buzzer alarms and the debugging are completed.

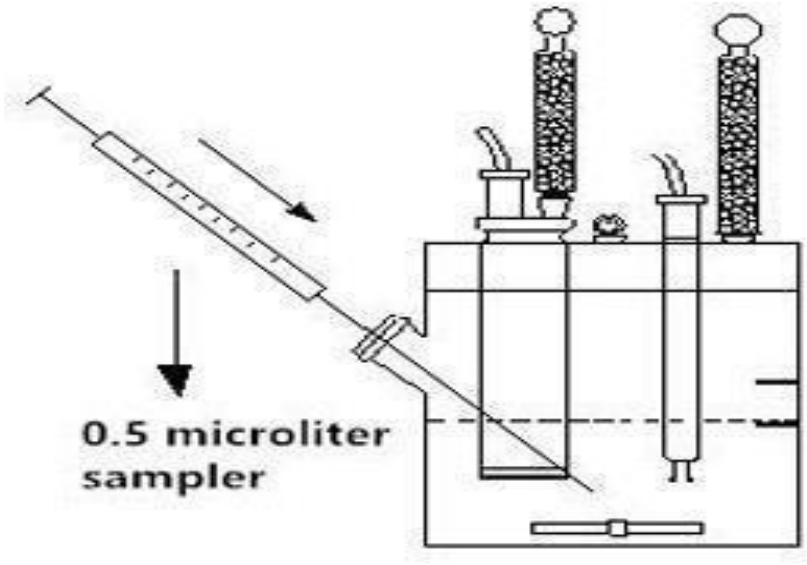

Figure.7

Note:

- When injecting distilled water, it must be injected slowly. When the reagent reaches a pale-yellow color, shake the bottle repeatedly or try to use a 0.5 μl injector to distill the distilled water.
- If the reagent in the electrolysis electrode is still dark brown, it will cause the instrument to jump back and forth between **[**over-water**]** and 【 over-iodine 】 It is normal.
- During the debugging process, observe the reagent color at any time, whether the instrument counts and does not inject too much pure water,causing the reagent to directly fail.
- The electrolysis electrode is not dried. After pouring the reagent, it turns paleyellow, which will cause the reagent to electrolyze for a long time, causingthe reagent to directly fail
- If the instrument is not used for a long time, the reagent in the electrolytic cell bottle absorbs too much water vapor, which will cause the reagent to directly fail.
- Electrolytic counting for a long time, the reagent in the electrolysis electrodechanges from light yellow to dark red, and the reagent fails.

8.3 Calibration of reagents

After the instrument is turned on, whether the reagent can be normallymeasured can be judged by calibration with a 0.5 μl micro-injector. When the instrument reaches equilibrium and is relatively stable, the calibration method is as follows

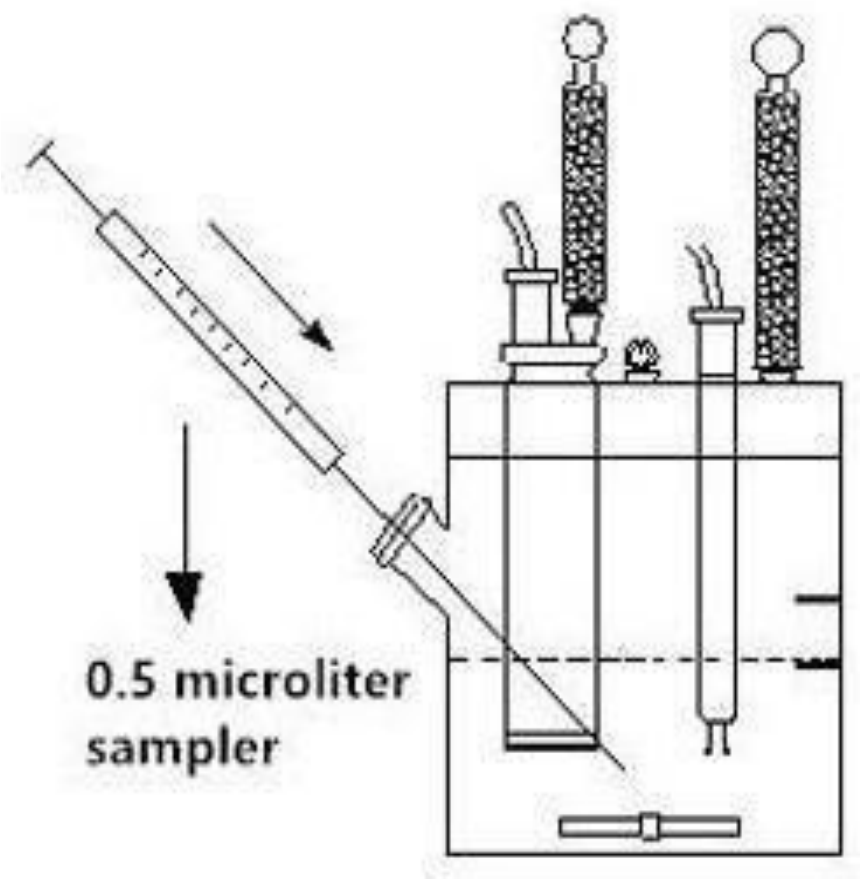

Figure.8

Press **[** Analyze**]** to enter When the reagent prompts “Balance”, the currentoperation displays “press the start button, inject sample” prompt, press **[**Start**]** anduse 0.1ul pure injector to extract 0.1ul pure water into the reagent. The display result should be “100±10ug”, generally calibrated 2-3 times, andthe result is within the error range, and the sample measurement can be performed.

8.4 Sample measurement

To obtain accurate measurement results, the appropriate injection volume should be selected according to the water content of the sample. The injection volume control is preferably between 30 μ g and 200 μ g on the display and the out-of-range can reduce the injection volume, and vice versa.

The sampler is cleaned 5 to 7 times with the test sample, and then the sample size isdetermined according to the sample's moisture content to prepare for the injection.

Sample moisture content (ppm)	Sample size (mL)
0 – 10	2 – 5
10 – 100	1 – 2
100 – 1000	0.1 – 1
> 1000	< 0.1

8.5 Parameter setting

After entering, press the item to be modified, select the corresponding number in the digital area, modify the data, and press **[** Back**]** to exit.

The density and volume of known samples can determine the equation

The sample volume refers to the amount of sample added, and the instrument automatically calculates the sample moisture content according to the set sample volume after the measurement is completed.

Formula 2 can be determined by the weighing method. Modify the total mass in the parameter settings. When the instrument is measured, input the tare weight according to the interface requirements, and the instrument automatically calculates the result.

Injection sample

Press the **[** Start**]** button to confirm that **[** Reagent Status**]** is “Balanced”, thenpress **[**Start**]** to quickly inject the sample into the reagent in the electrolyticcell, pull out the needle, and the instrument will automatically measure. When the instrument is measured, the screen will display the sample's water content, and the buzzer will alarm.

The water content calculation formula is as follows

$$\text{Water content(ppm)} = \frac{\text{Test result(microgramwater)}}{\text{Sample mass(g)}} = \frac{\text{Test result(microgramwater)}}{\text{Sample mass(g)sample density(g/ml) x sample volume(ml)}}$$

Water content (%) = ppm ÷ 10000

9. Maintenance

1) It is not advisable to apply vacuum grease at the grinding port of the electrolyticcell. It is recommended to rotate the joint of the grinding ring once a week. If it does not rotate for a long time, the grinding mouth will be firmly bonded together and it is not easy to disassemble.
2) When replacing the electrolytic electrode reagent, if the new reagent is poured into the light color immediately, it means that it is not dry. There is still wateron the filter plate. It can be dried again. After it is naturally cooled, add the reagent.
3) When the reagent turns dark, turn off the machine immediately and check if the electrode is open, if the stir bar has stopped rotating, or if the reagent hasfailed.
4) The two platinum legs of the measuring electrode are normally separated vertically. When they are connected, it will cause a short circuit, indicating that the iodine has passed.
5) Do not use the reagent color if it has turned pale after the reagent bottle is turnedON.
6) When the color-changing silica gel of the drying tube changes from blue to lightblue, the new color-changing silica gel should be replaced.
7) The measuring electrode, the plug, and the socket of the electrolytic electrodemay cause poor contact due to frequent activities. Make sure that the protrusion of the socket on the instrument is aligned with the notch of the electrode plug and then inserted and should not be rigidly rotated.
8) The platinum mesh in the electrolysis electrode cannot be lifted, which affectsthe test results.

1) Maintenance of electrolytic electrode

The following phenomena may occur when the electrolytic electrode iscontaminated:

- Reduce the electrolysis efficiency and extend the measurement time.
- The blank current is increased due to the adhesion of the contaminated portion to absorb moisture.
- The titration rate is unstable and cannot reach the endpoint.
- If the above situation occurs, the surface of the glassware and the dirt on the platinumnet can be cleaned with anhydrous ethanol (be careful not to damage the platinum wire and the platinum mesh).
- The electrolysis electrode can be dried by a hair dryer. The core part of thesand is difficult to dry, so it should be thoroughly dried.
- Pour a little reagent into the electrode and shake it gently. If the reagent turns pale, itmeans not drying.

2) Instrument maintenance

- During the use of the instrument, the stirring must be kept open.
- If it is closed, theelectrolyte can be forced to iodine.
- When iodine is over, a small amount of distilled water can be injected into the micro-injector, and the sample is adjusted after adjusting to the equilibrium point.
- The reagent must be fully balanced before the sample can be measured.
- Do not use the same injector for multiple samples, so as not to affect the measurement error.

10. Troubleshooting

1) The reagent contains too much iodine

1. Evaluate whether the reagent is true iodine. If it is true, use 0.2 to 0.4 microlitersof water to inject with a 0.5 microliter sampler. Do not inject with a 50 μl andlarger injector.
2. Check the measuring electrode and whether the platinum wire at the lowerend of the measuring electrode is connected, causing a short circuit.

2) Electrolysis does not end

1. Check whether the reagent has failed, or do not use it together for a long time. There is a lot of water in the electrolytic cell, which leads to counting.
2. When the instrument is not used, a lot of air will enter the pool bottle, and alarge amount of water vapor will be absorbed by the ceramic filter plate in the electrolytic electrode. After the start-up, the moisture in the ceramic filter plate will be slowly released into the large pool bottle.
3. It is difficult toreach the balance point for a long time. It is recommended to start the workevery day or put the bottle into the drying cylinder when not in use to avoidthe ingress of water vapor, causing long-term electrolysis and shortening the life of the reagent.
4. The electrolysis electrode was not dried before the reagent was debugged. When the reagent is being debugged, too much water is injected.

3) Bad repeatability

1. There is residual moisture in the inner wall of the bottle and the ceramic filter plate of the electrolysis electrode. You can press the start or start button but do not enter the sample, and the instrument does not count.
2. If the count indicates that there is water on the filter plate or the inner wall ofthe bottle, Shake the bottle repeatedly and consume this part of the water before measuring.
3. For ketone and aldehyde samples, please choose a special ketone aldehyde reagent.
4. The platinum electrode and the ceramic filter plate in the electrolysis electrode are caused by sediments. Kindly clean them in time before measuring.