Micro moisture tester for insulating oil

Operation Manual

Contents

Ⅰ Summarization

The instrument is a new developed micro moisture measurement and analysis apparatus with high-resolution color touch LCD, convenient and visual HMI and operability. The apparatus adopts high performance ARM processor, which has large data storage, fast and stable operation and outstanding anti-disturbance performance, has the advantages of high detection speed and high precision. The instrument has (i) self-diagnosis of trouble function, display and print the results when the test finished; (ii) measuring dynamic curve indicating function which makes the test more intuitive; (iii) large data storage function which could up to 1000 data records; (iiii) delay measuring function, which is very effective in the testing of lower moisture content of specimen. The instrument adopts the sliding touch stirring to control the speed; Moisture content calculation formula contains various algorithms that based on the volume and weight etc.; During the testing, people could modify the related parameters of the formula timely and will not affect the measurement value of moisture content which is convenient to the user.

The instrument adopts Karl-Fischer coulomb titration method to determine the moisture content of the liquid, gas and solid sample reliably. During the test, the instrument will make indirect measurement with corresponding sample injector of solid, gas and liquid to the solid of insoluble to the reagent and material that will pollute the electrode and affect reagent reacting, therefore, it is a kind of highly efficient, fully automatic analysis instrument which will be widely used in electric power, petroleum, chemical industry, medicine, railway, environmental protection, scientific research institutions and other industries.

Ⅱ Technical Specification

Titration method:	Coulometric titration
Testing scope:	0ug～200mg
Sensitivity:	0.1μg
Precision:	5μg～100μg±3μg
	3% above 100μg (not including sampling error)
Specimen form:	solid, liquid and gas
Display:	Color LCD monitor
Controlling:	Touch screen

Data storage:	1000 testing results can be stored
Status indicator：	dynamic curve, text display
Agitation speed control：	sliding touch control panel
Duration：	10 years with power down
Printer:	Thermal printer
Power supply:	AC220V±11V 50Hz±2.5Hz
Power:	≤50VA
Environment temperature:	5℃—35℃
Environment humidity:	≤85%RH
Dimension:	330mm X 240mm X 180mm
Weight:	about 10Kg

Ⅲ Working Theory

Equation of Karl Fischer reagent and water is as follows:

$$I_2+SO_2+3O_5H_5N+H_2O\text{-}2C_5H_5N \cdot HI+C_5H_5N \cdot SO_3 \cdots\cdots (1)$$

$$C_5H_5N \cdot SO_3+CH_3OH\text{-}C_5H_5N \cdot HSO_4CH_3 \cdots\cdots\cdots\cdots (2)$$

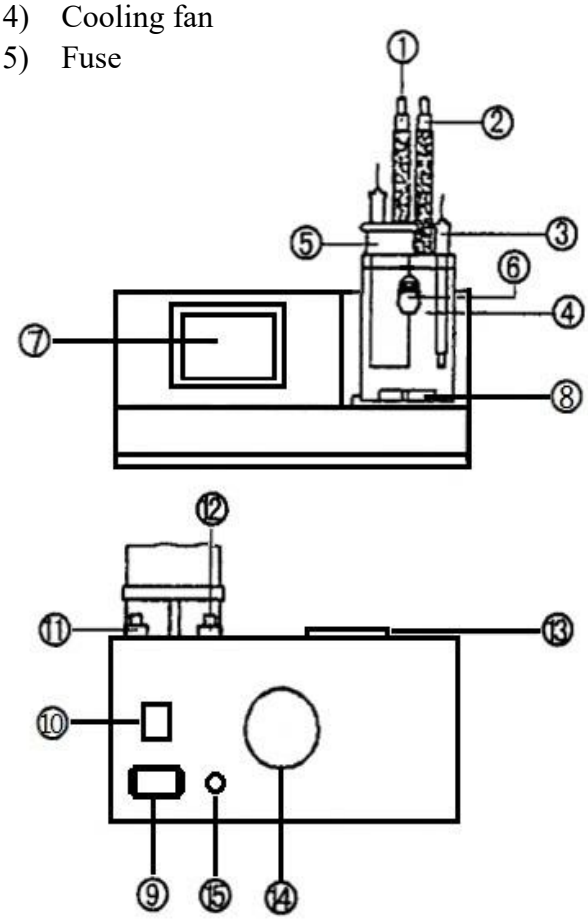
The reagent solution is composed by the majority iodine, pyridine with sulfur dioxide and carbinol etc. Iodine is formed through electrolyzing at anode. According to Faraday's Law, iodine is in the direct ratio with quality of electric charge. The equation is as follows:

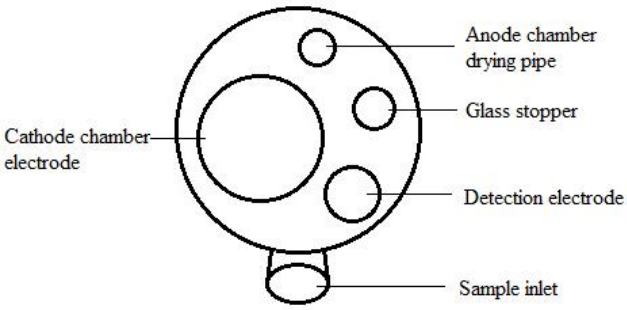
$$2I^{-}\text{-}2e\text{-}I_2 \cdots\cdots \quad (3)$$

From (1), molecules of iodine are equal to molecules of water. Put the sample in Karl Fischer reagent, water in the sample consumes iodine in the reagent. The instrument can display the consumption of iodine during the titration. The iodine consumption is in the ratio with the quantity of electric charge. The moisture content is decided by the quantity of electric charge. This instrument adopts automatic electrolysis current controlling system. The electrolysis current can be decided by the water content of the sample, the maximum is 400mA.

Ⅳ Structure Characteristics (chart 1)

(1) Cathode chamber drying pipe
(2) Anode chamber drying pipe
(3) Detection electrode
(4) Titration pond (anode chamber)
(5) Cathode chamber electrode
(6) Sample inlet
(7) Monitor
(8) Stirrer
(9) Power socket
(10) Power switch
(11) Testing socket
(12) Titration socket
(13) Printer
(14) Cooling fan
(15) Fuse





Whole machine structure　　Titration pond structure

Chart 1

Ⅴ Operation Instructions

1. Cleaning, drying & installation of titration pond

①Uncover all the glass meatus, titration pond, drying pipe and seal plug can be cleaned by water and dried in an oven at about 60℃, then cooled naturally. Cathode chamber and testing

electrode can not be cleaned by water, instead by acetone or carbinol, and should be dried by fan. Please be careful not to clean the connecting part of electrode (See chart 2), in case the testing error may be caused.

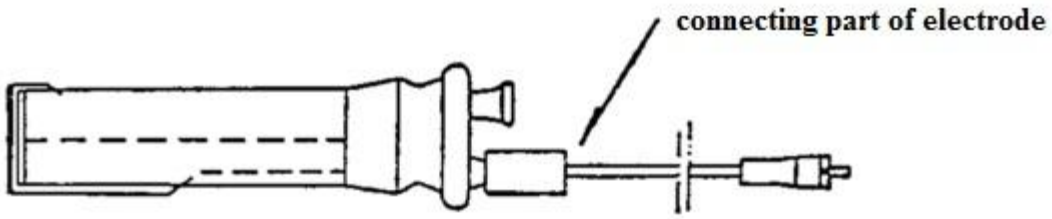


Chart 2

②Put silica gel, not the powder, into the drying pipe. Tuck the sample inlet with faucet (see chart 3)

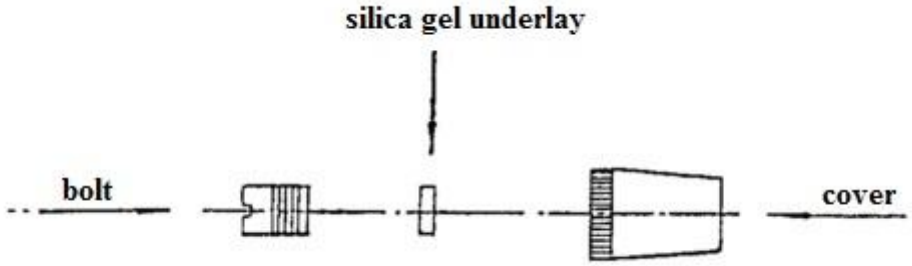


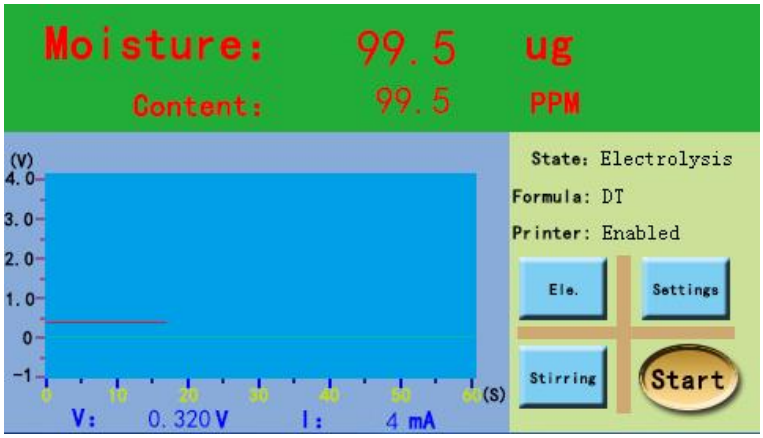
Chart 3

Put in the stirrer carefully through the sample inlet. Coat equably vacuum lubricator on the opening end of testing electrode, cathode chamber electrode, drying pipe, faucet and seal plug. Then install them, except the drying pipe and seal plug, rotate slightly to make a good seal.

Put the reagent through the seal plug by funnel into the anode chamber to the lowest reticle, then put the reagent through the drying pipe into the cathode chamber, making the same height of the reagent in both chambers. Install the drying pipe and the seal plug, rotate slightly to make a good seal (input of reagent should be taken in vent-cabinet). Plug the detection electrode and cathode chamber electrode into “DET”(Detection) and “TITR”(Titration) inlet separately.

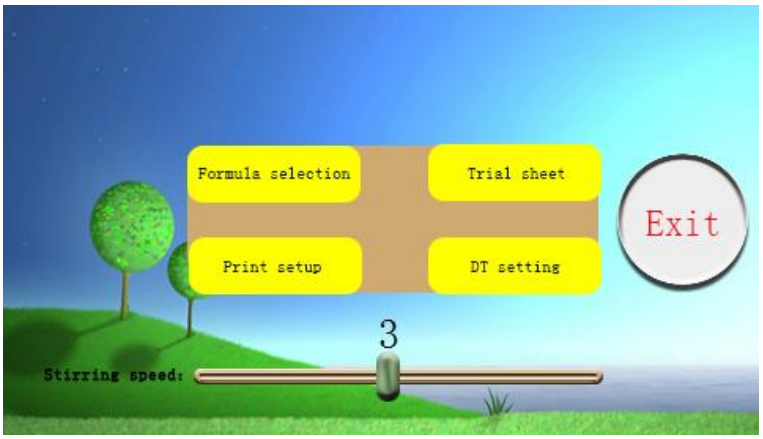
2. Operation Interface Introduction：

Starting up，the instrument automatically enter the test interface after few seconds display of the boot interface



Click up the button of “Ele.”(it means “Electrolysis”) and “Stirring” to control electrolytic current and stirring. Click on the “Start” button, inject the sample through sample injection cock when the instrument display “Be titrating”, then the titration autostart and moisture actual measured value are shown on the display and printed. If you need to change the formula, click the "Settings" button to enter the settings menu.(Remark: “Error 1” indicates”Measuring open circuit”;”Error 2” indicates “”Reagent excess iodine”;”Error 3” indicates “Electrolysis open circuit”;”Error 4” indicates “Non-electrolysis”.

The operator can set calculation formula, set printer, set the delay time, and adjust the stirring speed of the stirrer in the titration pond which could be turn up and turn down through slid the slider button rightwards and leftwards (Generally, set the stirring speed as the fifth grade).



(1) Formula selection

The operator could choose the required formula in this UI. Please click the “Formula parameter settings" button at the upper right to enter into the parameter settings UI which has given a corresponding note to the parameters:

Formula 1：F1=DT/(V*SG) ppm

Formula 2：F2=DT/(W-w) ppm

Formula 3：F3=DT/(V*SG)　%

Formula 4:　F4=DT/(W-w)　%

Formula 5：F5=DT　　ug

Thereinto, DT—Actual measured moisture value，unit：ug；

V—Sample injection volume，unit：ml；

SG—Sample density，unit：g/ml；

W—Sample total weight，unit：mg；

w—tare weight, unit：mg.

(2) Trial sheet

The UI is a test data recording interface, in which you can view the previous test results. The operator could check the record one by one through clicking the “Up” and “Down” button and delete all data records through clicking the “Yes” button on the popup of “Clear” button.

(3) Print setup

The operator could set the printer in this UI. When enabled, the instrument would autotype the result after testing, or will not.

(4) DT setting

The operator could set the time to switch on the current after clicking the “Start” button, the unit is seconds. That means, if the delay time is 10 seconds, then 10 seconds after clicking the "Start", the titration current is switched on. This method applies to measure the sample of smaller moisture content.

3. Process of equilibrium and stability of electrolyte:

(1) Please turn on the power switch, enter the test UI, and the instrument automatically turn on the mixing and electrolysis. Generally, the speed of stirrer in titration pool has been adjusted well, and do not need to adjust, if will, enter into the Menu setting UI to change, which aiming to make the stirrer rotate placidly, and no reagent will splash to the wall.

(2) “Electrolytic solution over iodine” indication on the test UI means that electrolytic iodine in the electrolyte is in excess, and please inject moderate distilled water through the sprue until the instrument work voltage curve close to zero and reach equilibrium levels.

4. Instrument calibration：

The instrument could be calibrated using pure water when it reaches the initial equilibrium point

and is relatively stable. Detail as follows:

(1) Extract 0.1ul pure water using 0.5ul sample injector, to make preparation for calibration.

(2)Press the "start" button, and then inject the pure water into anode chamber through sample injection cock. Notes: the injector tip should insert into the reagent, but avoid contact with the inner wall of the titration cell and electrode. The titration will autostart after pure water injection.

(3)Buzzer ring, shown “test finished” on the display, and the result is 100±3ug （without the sample error), generally, after 2 ~ 3 times calibration and the result is in the error range, you could began to test the sample.

5. Measurement operation：

During the process of using new reagents or testing the sample, reagent in the anode chamber will naturally produce a small amount of iodine which will damage the balance point of the instrument. In this case, extract little pure water using injector and inject to the anode chamber through sample injection cock, until the instrument is recovered to the balance point, then began to measure the sample.

Similar to the calibration of the instrument, when the instrument is equilibrium - voltage baseline is a line near zero level (equilibrium point), sample test could begin (example: liquid samples, content calculation formula is F1).

(1)Sampling:

Taking sample to wash the 1ml injector.

(2)Sample injection and determination

Click on the “start” button after sampling, inject the sample to the anode chamber through sample injection cock when the instrument display “be titrating”, then the titration autostart and moisture value are ever-increasing. Titration finish, buzzer ring, and “test finish” information shown on the display. The printer in the enabled state will print out the measurement results.

The operator could change the calculation formula or the relevant parameters in the formula before the end of the titration through clicking “setting”- “formula selection” (-“formula parameters setting”) button.

Ⅵ Points of Attention

1. Points of attention of the reagent

(1) In normal titration, every 100ml reagent reacts with 1g water at least. The sensitivity

declines if the titration period is too long. The reagent should be changed.

(2) During the titration, the reagent in anode chamber is found to release large quantity of air bubble or be polluted to light henna, the extra current augments, which decreases the precision of the titration. In this situation, change the reagent as soon as possible.

(3) If the titration has lasted more than half an hour and the titrator is still not stable, press "Stirring" to stop stirring. Observe if brown iodine is obviously formed at the anode under the ceramic filter. If there is no or little iodine, change the reagent.

(4) Pay attention not to inbreathe or touch the reagent. Wash the skin with water if it is contacted with the reagent.

2. Points of attention of testing

(1) When the sample is injected into the titration pond, the pinhead of the sample injector must be under the liquid level. The sample can not contact with the inner wall and the electrode of the titration pond.

The typical testing range of this titrator is 10 μg-10mg. In order to get a precise testing result, the quantity of the sample injection should be controlled according to the moisture content of the sample.

(2) To guarantee the precision of the measurement, customer must use the original electrolyte (Karl Fischer reagent) provided by the manufacturer.

Ⅶ Maintenance

1. Location of installation

(1) The instrument can not be installed in a room with caustic gas, in case it is eroded and the lifespan is shortened.

(2) Room temperature should be beyond 5℃ and under 40℃.

(3) The instrument can not be installed in a place shot straight by the sunlight.

(4) The instrument can not be installed in a high humid place.

2. Maintenance of reagent

(1) The reagent should be put a breezy place, where the temperature is at 5-25℃ and the relative humidity is less than 65%. If it is shot straightly by the sunlight or is placed at a high temperature,

the sulfur dioxide and iodine are released from pyridine.

(2) Be careful with toxicity, odor and flammability of the reagent, it may be loaded and changed on a breezy test-bed.

3. Changing of silica gel underlay

If the underlay at the inlet has been used for a long period, the pinholes may lose contractibility. The moisture in the air may enter the titration pond and cause error. In this situation, the silica gel underlay needs to be changed.

4. Changing of silica gel

(1) When the color the silica gel in the drying pipe is changing from blue to light blue, change the silica gel.

(2) Pay attention not to load the silica gel powder in the drying pipe. Otherwise, the following malfunctions are occurred:

① The reagent is released from the cathode chamber, there is no reagent in the cathode chamber and the electrolytic current stops.（See chart 4a）

② The reagent in the anode chamber immerges into the cathode chamber. The iodine ion assembles and settles on the ceramic filter, decreasing the efficiency of titration. （See chart 4b）

a

b

Chart 4

5. Maintenance of the connecting part the titration pond

Rotate the connecting part of the titration pond about one week. Recoat it thin with vacuum lubricator if it is not rotated easily. Otherwise, the vacuum lubricator stiffens and the connecting accessory may not be dismantled. Therefore, maintenance is very important, making easy for the

dismantlement and cleaning.

Remarks:

The vacuum lubricator can not be coated too much, in case it enters into the titration pond and causes the testing error.

6. Treatment of the agglutination of the connecting part of the titration pond.

If the connecting part of the titration pond sticks solidly and can not be easily dismantled, please refer to the following procedure:

(1) Release the reagent in the titration pond and clean the titration pond.

(2) Inject some acetone around the connecting part, rotate the accessory slightly at the connecting part, it can be dismantled.

(3) If it is not working, put the titration pond in a 2-liter beaker, load 5% potassium chloride solution slowly. See Chart 5 for the level of the liquid. Pay much attention that the down-lead end of the testing electrode and the cathode chamber electrode does not immerge into the liquid. It can be dismantled after more than ten hour or 24-hour soak. (This method can be repeated.)

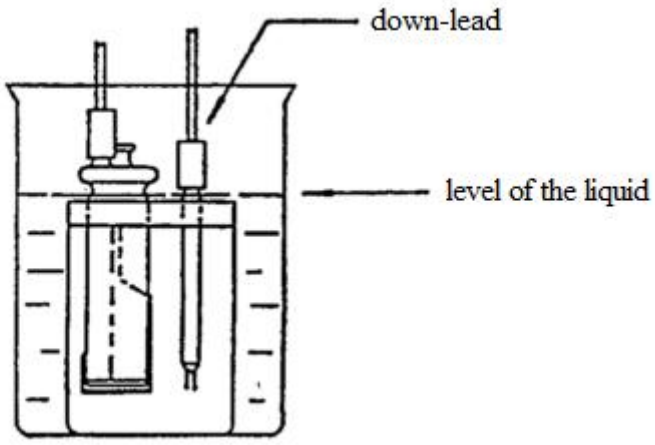


Chart 5

7. Maintenance of the testing electrode

(1) When the stirrer is rotating quickly, pay attention the stirrer may jump to destroy the electrode.

(2) During the putting-in and taking-out of the testing electrode, stop the stirring and pay attention the testing electrode not to touch the inner wall of the titration pond.

(3) When the testing electrode is crooked and not short circuit, it can be used and also can be

repaired. Clamp the root of the platonic electrode with forceps, restore the end of the platinic electrode slowly. (See chart 6).

(4) When the testing electrode is polluted, clean it with acetone. If it is not working, burn the end of the platinic thread with alcohol burner. (See chart 7)

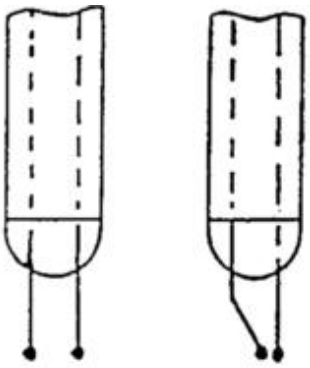


Chart 6

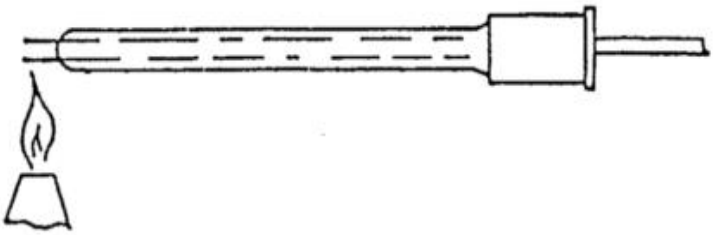


Chart 7

Remarks:

Move the flame toward the end of the platinic thread slowly, avoiding the burst of glass part of the electrode by the quick heating.

(5) If there is a leakage and some reagent exists obviously in the electrode, (See chart 8), measure the electrode with multimeter. If the resistance is big than 100kΩ, the electrode can still be used. Otherwise, change it.

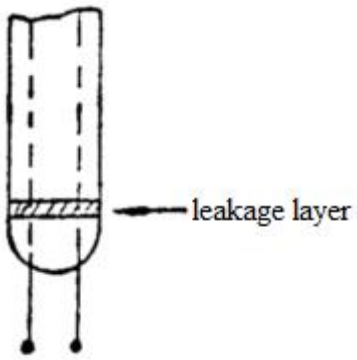


Chart 8

8. Maintenance of the cathode chamber

(1) During the dismantlement of the cathode chamber, for the platinic thread and platinic net are sticking out of the cross section of the connecting part of the cathode chamber, pay attention not to touch the top and the hole wall of the titration unit. (See chart 9)

(2) Cleaning of the cathode chamber

The following phenomena may occur if the cathode chamber is polluted:

① The efficiency is reduced and the testing time is extended.

② The extra current augments because the polluted part absorbs moisture.

③ The titration speed is unstable and the titration can not finish.

If the above phenomena occur, clean the outer of the glass part and dirt on the platinic net with acetone. (Pay attention not to damage the platinic thread and platinic net.) Inject the acetone in the cathode chamber, use rubber plug or something similar to seal the drying pipe, shake it thoroughly to get rid off the dirt inside. (This procedure can be taken repeatedly.) Then pour the acetone at the outer of the glass part for cleaning. Do not clean the down-lead.

If it is not working, immerge the cathode chamber into vitriol which is loaded in a beaker(See chart 10).

Remarks:

Do not damage the plaitnic thread and the platinic net.

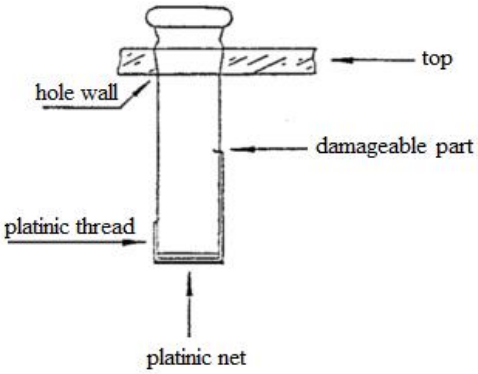


Chart 9

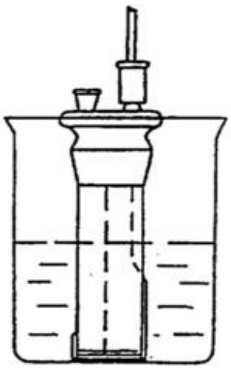


Chart 10

(3) Drying of the cathode chamber

Use fan to dry the cathode by hot wind. The difficult drying part should be dried thoroughly. (See chart 11) If the extra moisture may exist, put the cathode chamber into the vacuum drying desiccator for drying about 12 hours.

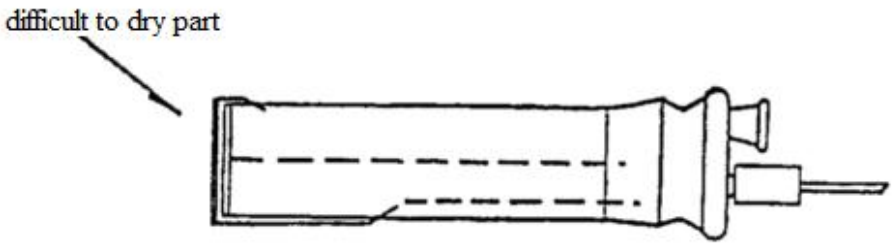


Chart 11