

DW-K9860 Kjeldahl Analyzer User Manual



Please read operating manual before installation and operation.

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I. Safety

1. Overview

This operating manual is prepared for the laboratory instrument operators. Please read this manual carefully before using the instrument, and conduct the operation according to the manual. Personnel who are not familiar with the instrument operation or safety information shall not operate the instrument.

This instrument adopts the current most advanced technology for design and manufacturing. But the improper use of instrument will cause the potential safety hazard.

The instrument manufacturer has made the assessment on the possible residual hazard:

Inexperienced personnel operate the instrument

Failure to operate the instrument according to the normal regulations

There is the warning information in this operating manual, and operators shall know these residual hazards.

2. Safety



Warning: This instrument is only used for the laboratory. If the instrument is damaged due to the failure of conducting the operation according to the manual during the use, the manufacturer refuses to bear all consequences.



Warning: If the failure of using the instrument properly or conducting the operation according to the operating manual causes the consequences, the company will not bear the safety problem of instrument use.



Warning: The failure of operating the instrument as required by this manual will weaken the safety performance of the instrument.



Warning: Please conduct the treatment of various solutions used in the test according to the laboratory safety regulations. The operators shall wear the rubber gloves, lab coats and goggles.



Warning: Oral take-in of boric acid will cause the acute poisoning, which is mainly embodied in gastrointestinal symptoms, nausea, emesis, abdominal pain, diarrhea, etc., and then dehydration, shock, coma or acute renal failure, or high fever, liver and kidney damage and convulsions, and even cause death if serious. It is easily absorbed by the damaged skin and causes the poisoning. Chronic poisoning Long-term absorption of small amount of boric acid in gastrointestinal tract or skin will lead to mild digestive tract symptoms, dermatitis, baldness and liver and kidney damage.



Warning: Sodium hydroxide has intense irritation and corrosivity. The powder or smog will irritate the eyes and respiratory tract, and corrode nasal septum. Direct contact of skin or eyes with sodium hydroxide may cause burns, and accidental take-in can cause gastrointestinal burns, mucosal erosion, bleeding and shock.



Warning: Sulfuric acid (96-98%) has intense irritation and corrosive effect upon the skin, mucosa and other organs. Vapor or mist may cause conjunctivitis, conjunctival edema, and corneal opacity, and consequently blindness; it may cause respiratory irritation, and may cause dyspnea and pulmonary edema in severe cases. High concentration may cause the laryngospasm or glottic edema, and finally death by suffocation. Oral take-in may cause digestive tract burns and thus elcosis. It may cause gastric perforation, peritonitis, renal damage, shock, etc. in severe cases.



Warning: The temperature of digestive tract will reach 100°C during the experiment. After completing the distillation, take the testing tube out with test tube holder for avoiding scald.



Warning: Please use the power line provided by Drawell.
Other powerlines will affect the safety performance of the instrument.



Warning: This instrument is equipped with the special grounding power

plug for preventing electric shock. Please use the grounding receptacle.



Warning: Electric shock hazard only professional and qualified persons are permitted to open the machine cover and the panel.



Warning: The instrument shall not be used under the environment with the potential explosion risk.



Warning: In the case of not conflicting with the local waste disposal regulations, waste liquor can be directly discharged into the sewer. Make sure that the exhaust line is not bent or doesn't keep the upward flow and is as short as possible, and the outlet end shall not be lower than the liquid level of the sewer. (exhausting hot water below cold water will produce the noise). The pipeline shall be fixed, because the waste liquid discharged from the system has certain pressure.



Warning: The power switch in the lower right side of the instrument can be reached easily at any time, making sure that the power can be turned off at any time.



Notes: Don't touch or open the protective door during the instrument operation.



Notes: Make sure that the liquid solution will not contact the power line and electric parts inside the instrument.



Notes: Stop using the instrument in the case of failure, and timely contact the nearby Drawell service center.



Notes: This instrument shall be repaired by the personnel authorized by Drawell . Drawell recommends using the original spare parts. If the spare parts from other sources are used, the quality guarantee will be invalid.



Notes: The instrument is designed and tested according to EU standard (CE). To guarantee the continuous conformance to the standard, the instrument can only be connected with the equipment meeting CE requirements.



Notes: The unboxing, assembly and installation of the instrument shall be completed by the personnel authorized by Drawell .

3. Guidance for waste disposal



It is not allowed to discard the electronic devices together with the non-classified general waste. Improper disposal will produce the damage to the environment and human health. Please refer to the local waste disposal regulations for collection and disposal of the equipment.

4. Quality guarantee policy

The quality guarantee is generally specified in the purchasing order or contract, and is only applicable to:

The user shall abide by all written explanations and documents of Drawell .

The equipment shall be installed, maintained, adjusted and calibrated according to the methods described and recommended in the documents.

The equipment is not used for other purposes beyond those specified by Drawell .

The equipment is not refitted or repaired with spare parts not from Drawell , and is not repaired by the personnel beyond those authorized by Drawell .

Only use the accessories and consumables provided by Drawell for from the source recommended by Drawell .

The equipment can't be operated according to the way which is inconsistent with the ordinary practice.

Only software authorized by Drawell can be installed in the PC of the equipment.

Use the external PC meeting the requirements recommended by Drawell .

Don't play game with PC, including the game installed together with operating system.

The equipment shall conduct the correct maintenance according to the requirements recommended by Drawell .

The equipment contains some quick-wear parts, and these spare parts can be inquired in the user's manual and user's guide.

The quality guarantee responsibility of quick-wear parts is only limited to the damage caused by the material defects or production problem.

II. Summary

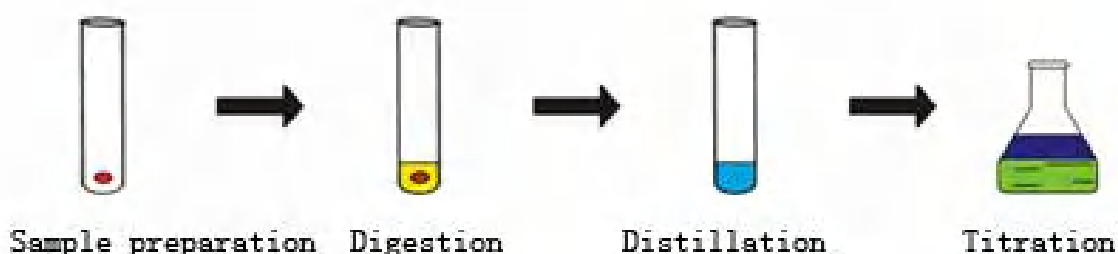
2.1 Application

Automatic Kjeldahl Analyzer is an automatic, smart device determining nitrogen content based on Kjeldahl method. It can be widely used in food processing, feed production, tobacco, livestock, soil fertility, environmental monitoring, medicine, agriculture, scientific research, teaching, quality control and other fields for the test of nitrogen or protein content with respect of macro and semimicro samples and can also be used for the test of ammonium, volatile fatty acid / alkali, and so on. Upon test of samples by using Kjeldahl method, the processes of digestion, distillation and titration are required, wherein distillation and titration are main determination processes with respect to Automatic Kjeldahl Analyzer.

Automatic Kjeldahl Analyzer is an automatic nitrogen determination system integrating distillation and titration based on classic Kjeldahl method; the instrument provides great convenience for lab staff in determining nitrogen-protein. It's safe, reliable, simple for use and time-saving. A friendly user interface in English allows easy operation and displays abundant information, enabling users to quickly master the use of the instrument.

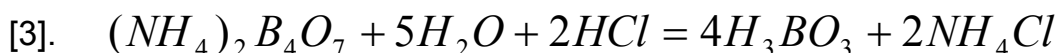
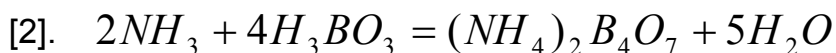
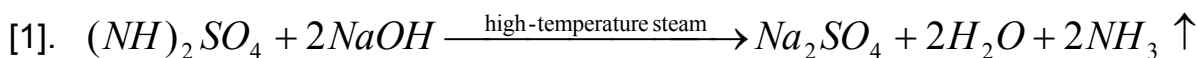
2.2 Principles

According to Kjeldahl principles, the determination requires three steps, which are digestion, distillation and titration.



Automatic Kjeldahl Analyzer can automatically complete the processes of distillation and titration. Upon the samples to be determined is fully digested, it's

subjected to the following chemical reactions on the instrument:



The ammonia gas emitted during the reaction, together with the steam, is collected in the receiving cup added with boric acid absorbing solution (containing mixed indicator) after condensed by a condensing tube. Then the automatic titrator carries on titration and record the volume of consumed standard titration acid. Based on the volume of consumed standard titration acid, the calculating system calculates the nitrogen content and crude protein content based on the following formulae.

Nitrogen content:

$$\text{Crude protein content: } N(\%) = \frac{1.401 \times M}{W} (V - V_0)$$

$$\text{Wherein: } P(\%) = N(\%) \times C$$

M=molar concentration of titration acid (mol/L);

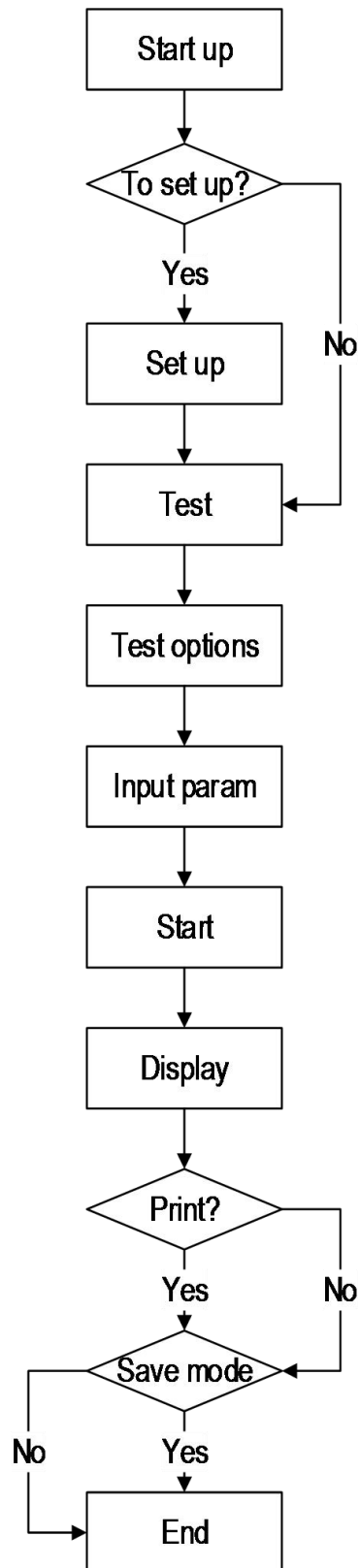
W=sample weight (g);

V₀= volume of consumed standard titration acid during blank sample titration (mL);

V= volume of consumed standard titration acid during sample titration (mL);

C=conversion coefficient for crude protein.

2.3 Operating Flow Chart



III. Main performance

3.1 Technical parameters

- a. Sample capacity: solid \leq 5g, liquid \leq 20mL;
- b. Measuring range: 0.1mg \sim 200mg nitrogen;
- c. Analysis time: 5 \sim 10 min/sample;
- d. Recovery: \geq 99.5%;
- e. Titration accuracy: 1.0 μ L/step;
- f. Repeated errors(RSD): \leq 0.5%;
- g. Data storage capacity: 1,000 pieces;
- h. Consumption of condensate water: 1.5L/min;
- i. External interface: USB, RS485;
- j. Dimension size (length \times width \times height): 455mm \times 391mm \times 730mm;
- k. Net weight: 38kg.

3.2 Use conditions

- a. Power supply: 220 VAC \pm 10% 50Hz;
- b. Rated power: 2KW;
- c. Condensate water pressure: $>$ 0.02Mpa; flow: $>$ 1.5L/min;
- d. Condensate water temperature: \leq 20 $^{\circ}$ C;
- e. Ambient temperature: 10 $^{\circ}$ C \sim 35 $^{\circ}$ C.

IV. Name of instrument components

The instrument is a system conducting automatic distillation, titration, calculation, printing, discharge and cleaning on the fully-digested sample and capable of displaying operating procedures, with a microcomputer calculating results and a printer outputting data. The system substantially consists of microcomputer controller, steam generator, distilling system, alkali adding system, boric acid adding system, titrating system, micro-printing, discharge system and cleaning system.

The structure of the instrument is composed as shown in the figure below:



Fig 3.1

1. Label 2. "Security door in-place" detector 3. Anti-splash bottle 4. Condensate water interface 5. Distilling head 6. Fixed holder for digestion tube 7. Digestion tube 8. Digestion tube fixing block 9. Security door 10. Waste receiving tank 11. Touch screen 12. Panel 13. Printer 14. Receiving cup 15. Titration acid tank

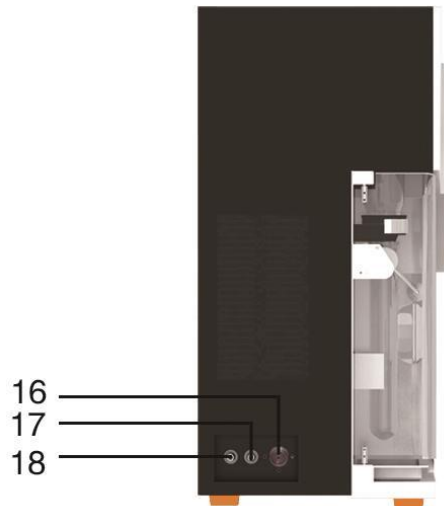


Fig 3.2

16. Condensate water inlet 17. Condensate water outlet 18. Discharge port

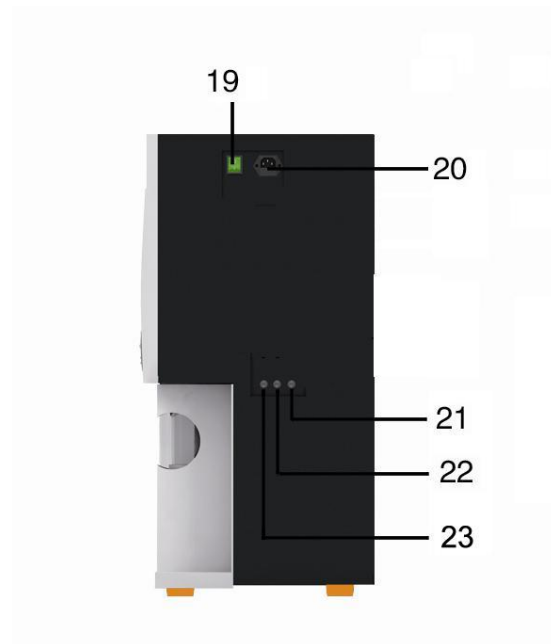


Fig 3.3

19. Power switch 20. Power plug 21. Alkali inlet 22 Boric acid inlet 23. Distilled water inlet

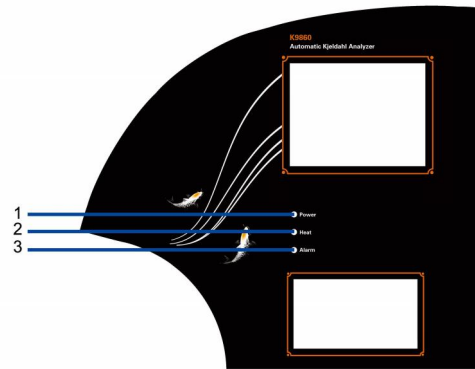


Fig 3.4

1. Power indicator light 2. Heating indicator light 3. Alarm indicator light

V. Device installation methods

1. Inspection before installation

After the package is removed, check the instrument and its fittings as specified in the appended packing list and inspect for damage. In case of damage, immediately contact the manufacturer. (Please retain the damaged components)

2. Installation conditions

- a. The device shall avoid direct sun radiation and places without extreme temperature or moisture. In general, the room temperature shall be kept between 10°C and 35°C.
- b. The device shall be installed at places adjacent to water source and drain tank and provided with power sockets; the distance between the location of water supply valve and power supply and the device shall be less than one meter for convenience of operation.
- c. The water supply shall comply with requirements of water pressure and water temperature.

- d. The drain tank shall at least be 50cm lower than discharge port of the device to ensure smooth natural discharge.
- e. The power arrangement shall comply with requirements of power supply. It shall be provided with ground lead, and separate power switches and safeguards to ensure users' safety.
- f. The device shall be installed far away from large electrical equipments, and the operating location shall be free of quake, corrosive liquid and strong electromagnetic field interference.

3. Installation

Lay the device flat on the test bench, leaving a distance of at least 20cm between the back side of the device and the wall. Arrange a power socket within a distance of one meter from the device, and meanwhile provide air-break switch, anti-creeping switch and reliable grounding.

Connect pipelines according to each pipe interfaces listed in the back elevation of the device. The condensate water inlet (16) is connected to the tap water valve; condensate water outlet (17)discharge port (18) are respectively connected to the drain tank through the pipe drains and discharge pipe to ensure smooth discharge.

VI. Function introduction

1. Function of operating panel

- a. Display: True-color touch display.
- b. Power indicator light (green) : it shows power supply status; the light is on upon the device is powered on. When the light is on, it means the device enters into standby status.
- c. Heating indicator light (red) : When the light is on, it means the water in the steam generator is being heated and the light turns off in non-operating status.
- d. Alarm indicator light (yellow) : When the light is on, it means the failure of system occurs or certain necessary conditions are not met upon distillation and the light will be off when the system is in efficient working order. Note: the display will simultaneously show alarming words and sound under alarming status.

2. The functions of the buttons of the printer are as follows

a. [SEL] button

Upon pressing this button, if the operating indicator light of the printer is on, it means the printer is in line; if the light is off, it means the printer is off line. If printing is desired, please maintain the print in line;

b. [LF] button

When paper needs to be reloaded, press [SEL] button to turn off the operating indicator light of the printer; press [LF] button to make the printer automatically loading paper; press this button again, the printer will stop loading paper; at last press [SEL] again to turn on the operating indicator light of the printer, and the paper loading process of the printer completes.

3. Safety performance

Protection upon condensate water supply is suspended: where the tap water supply is cut off or suspended as the device is running, the device will automatically stops operation to prevent spraying of heating steam scalding people.

Protection upon steam generator lacks water: the steam generator is provided with water refilling functions; when the tank supplying the steam generator is lacking water, the steam generator will automatically stops operation to prevent the damage from heating without water.

Protection of security door: a transparent anti-corrosive security door enables observation of the whole process of distillation; if the security door is not closed upon operation, the device will lock up the operating button to provide safety protection.

Protection upon steam generator is under overpressure: the steam generator has protective functions against overpressure, in case of overpressure, the pressure sensor will automatically cut off the power supply to prevent explosion due to overpressure.

4. Operation of device functions

The operations of functions attainable by the device in testing samples are as follows: diluting, adding reagent, distilling, titrating, discharging waste, result calculating and printing.

Diluting: add distilled water into the digested sample in the digestion tube to dilute.

Adding reagent: includes adding of alkali solution, boric acid absorption solution, titration acid, etc.

Distilling: add hot steam into the sample in the digestion tube to distill the ammonia of sample.

Titration: titrate absorption liquid when distilling or complete distilling.

Discharge waste: discharge wastes in the digestion tube, receiving cup.

Result calculating and printing: calculate the result and print it according to operation.

5. Operation of function interface

Main interface

After the startup animation, six functions will be displayed, including [Test], [Clean], [Search], [Debug], [Setup], [Help]. Press corresponding icon to enter respective interface. See Fig.5.5.1



Fig. 5.5.1

Press [Test], the menu system will automatically enter into operating mode interface, [Automatic test] After setting experimental parameters, the device will carry out automatic experimental test.

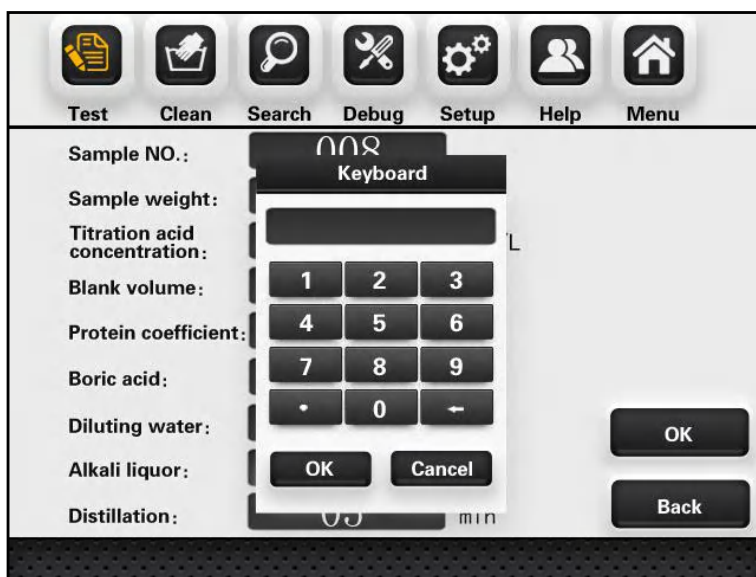


Fig. 5.5.2

After inputting corresponding experimental parameters, press [OK] to enter experiment interface and start the experiment. The steps of experiment are respectively: add diluting water, add boric acid, add alkali, distill, titrate, calculate and print, discharge, digestion tube cleaning and receiving cup cleaning, of there the functions of digestion tube cleaning and receiving cup cleaning are set up as required in [Setup] interface before the experiment starts.

To ensure the safety of the experiment, the system collects and displays all aspects of status information with respect to the device on a real-time basis during the test process, such as level of steam generator, security door in place, presence of condensate water, etc. If any abnormal condition is detected, the task will be suspended. For example, upon lack of condensate water, the following interface will appear. See Fig. 5.5.3.



Fig. 5.5.3

The system displays current operating items and real-time values on a real-time basis. When the set value is reached, the system will stop the current function and turn to the next experimental function. If the parameter is set with value “0”, then the system will not start corresponding function. Taking adding boric acid as an example, when a parameter is set, the system will start filling of boric acid. See Fig.5.5.4.



Fig.5.5.4

When a parameter is set, after filling of boric acid is completed, the system will start filling of diluting water and display that diluting water is being added.

Note: the dilution function of the device is to dilute the digested sample in the digestion tube by adding distilled water.



Fig.5.5.5

After parameter setting and dilution water adding completed, Alkali adding starting, the display (figure 5.5.6) as follow:



Fig. 5.5.6

During foregoing operation, the experiment can be suspended at any time by the operator as necessary. The experiment can be either continued or quitted after suspension. If the alkali is not sufficient due to certain causes, then the experiment can be suspended upon distillation. Press [Add alkali] to add fixed volume of alkali solution. A 5ml solution can be added for each pressing of [Add alkali]. Press [Continue] to continue the experiment. See Fig. 5.5.7.



Fig. 5.5.7

After completion of adding alkali, the system will start the distillation process. If the time parameter of distillation is set 0, then the system will not start the distillation process; if certain parameter is set, the distillation process will be automatically started; meanwhile the suspend or back can be attained. See Fig. 5.5.8.



Fig. 5.5.8

During the process of distillation, in case of abnormal conditions, it will be notified, such as overheating of ammonia water, See Fig.5.5.12, overheating of steam generator, See Fig. 5.5.9.

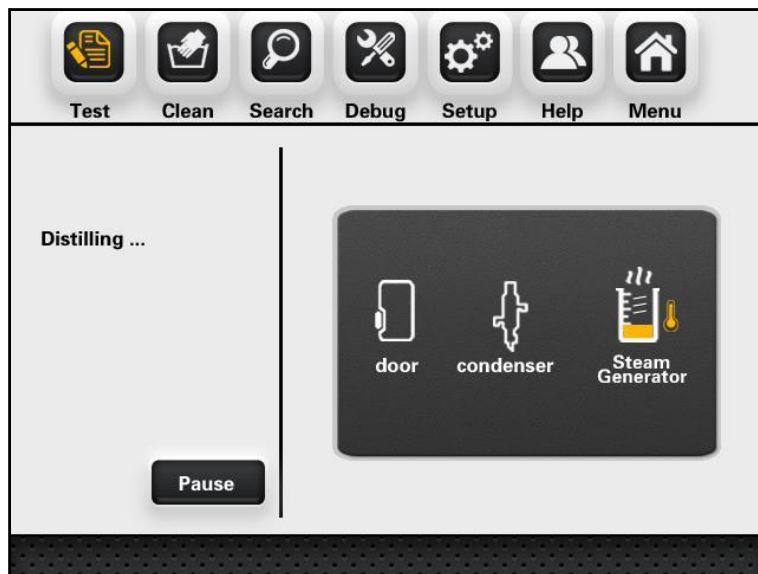


Fig. 5.5.9

Upon the system enters into the process of titration, See Fig. 5.5.10.

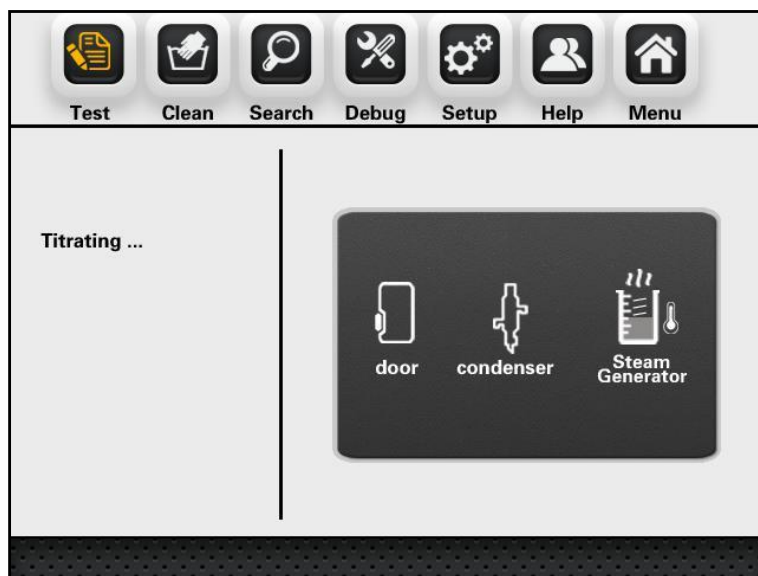


Fig. 5.5.10

In case discharging and cleaning are set in [Setup], then system will automatically discharge and clean after completion of titration. The cleaning of digestion tube and receiving cup will be respectively carried out following the discharging of the same. The system can not suspend or back upon commencement of cleaning or discharging till the completion of cleaning or discharging. See Fig. 5.5.11.



Fig. 5.5.11

After completion of cleaning, it will enter into the titration result interface, wherein the titrated volume, nitrogen content and protein content will be simultaneously displayed. Under such interface, printing can be carried out. See Fig.5.5.12.



Fig.5.5.12

Cleaning

Choose [Cleaning], with interface as shown in the figure, including , Receiving cup cleaning, Alkali pipeline cleaning, Boric acid pipeline cleaning, Acid washing, you will see this interface when you access cleaning page, See Fig. 5.5.13.

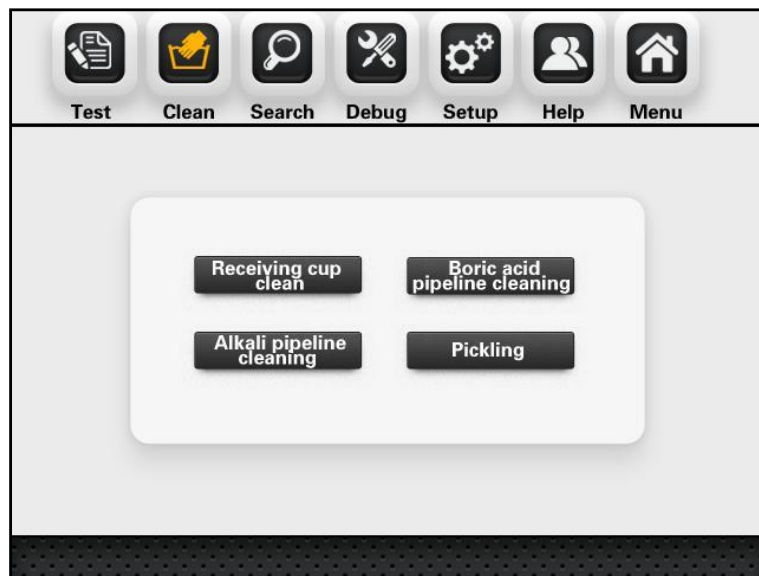


Fig. 5.5.13

For example, choose [Receiving cup cleaning], click on [Receiving cup cleaning] to start cleaning of receiving cup; the interface displays "cleaning receiving cup "; upon touching [Back], the cleaning stops and the interface returns to cleaning function interface. See Fig. 5.5.14.

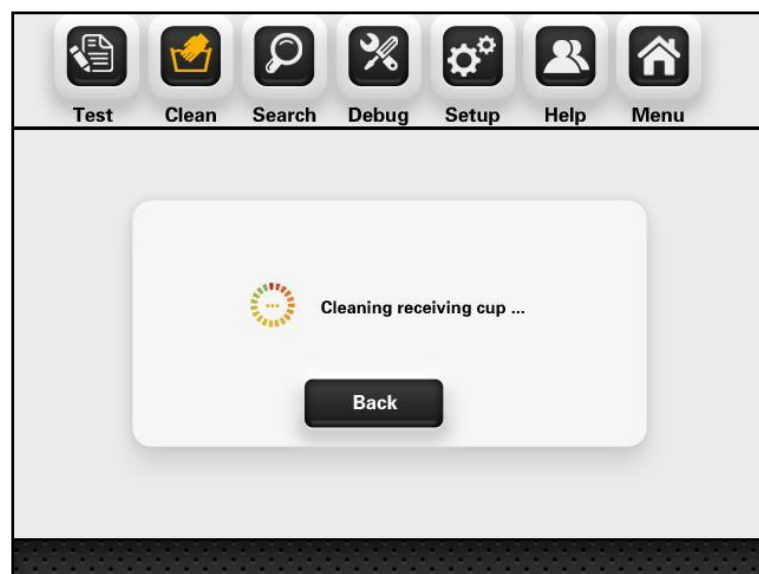


Fig. 5.5.14

Search

Choose [Search] to enter into search interface for historical record, input sample No. to display corresponding experiment results and storage time. In case no reference number is input, the system will display the record of latest experiment; the system will display corresponding record following manual input of reference number (Range of sample No.: 1-1000). [Print] operation is available; click on Print to print data of corresponding reference number. Clicking on menu icon can exit search interface and return to the main interface. Also, you can click on other icons in the tool bar as necessary to enter into other function interfaces. See Fig. 5.5.15.

Test	Clean	Search	Debug	Setup	Help	Menu
Sample NO. :	◀ 007 ▶					
Sample weight:	0.0000		g			
Titration acid concentration:	0.0000		mol/L			
Titrated volume:	0.0000		mL			
Blank volume:	0.0000		mL			
Protein coefficient:	6.25					
Nitrogen content:	0.0000		%			
Protein content:	0.0000		%			
Experiment date:	00/00/00 00:00:00				Print	

Fig. 5.5.15

Debug

Choose [Debug] to enter into the debug interface, which tests all electronic parts within the device. With respect to the back of corresponding button, √ indicates initiation, × indicates closure; the white icon represents detection of level sensors, etc. √ and × indicate the present working status of sensors. For example, √ appears upon detection of security door in place, otherwise × appears. After completion of choosing, the debug of the device commences. See Fig. 5.5.16. Choose [Back] to return to the previous interface.

Note: the consequences arising from unauthorized use of debug interface will be borne by the user; this interface is only for use by technicians with the manufacturer.



Fig. 5.5.16

Setup

Choose [Setup] to enter Setup interface, wherein setup of the following functions can be attained:

[Water flow test] determines whether to detect the presence of condensate water during the test.

[Receiving cup cleaning] with respect to automatic test which determines whether to carry out receiving cup cleaning after titration.

[Color calibration] is used for titration sensor calibration.

[Titrator calibration] is conducted by titrator.

[Calibration coefficient] upon measuring samples, there will be system errors due to connection to different systems. To ensure accurate measurement, a deviation calibration coefficient is established to enable calibration by the user.

[System time] is used to set up system time.

[Historical data] reset storage data.

Set up corresponding function in this interface before the commencement of experimental test. See Fig. 5.5.17.

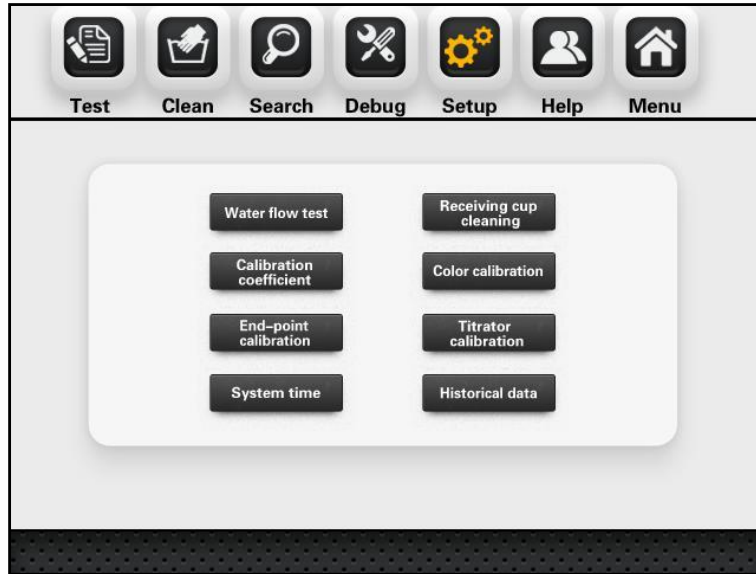


Fig. 5.5.17

Water flow test

Choose [Water flow test] to display corresponding dialog box. Choosing [Yes], [OK] means the system will suspend the experiment in case no water is present in the condensate water tube during the test till the presence of water. Choosing [No], [OK] means there will be no alarm regardless of presence of water. Pressing [Back] will maintain the original setting and enter into the original Setup interface. See Fig. 5.5.18.

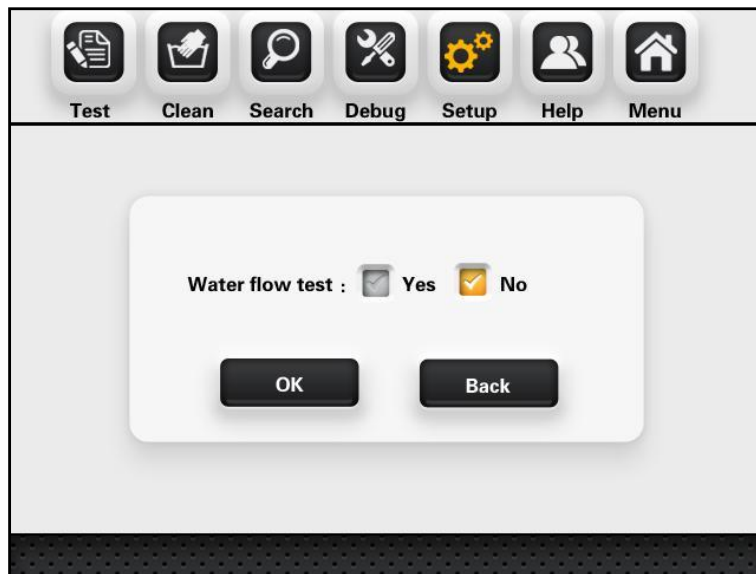


Fig. 5.5.18

Receiving cup cleaning

Choose [Receiving cup cleaning] to enter into choice interface; choose [Yes], [OK] to enable receiving cup cleaning after completion of test; choose [No], [OK] to disable receiving cup cleaning after completion of test; choose [Back] to maintain the original setting and return to the Setup interface. See Fig. 5.5.20.

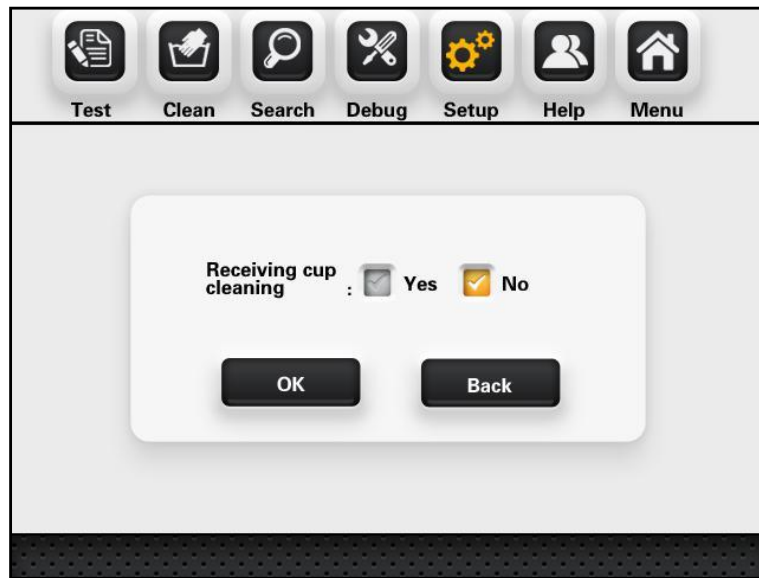


Fig. 5.5.20

Calibration coefficient

Choose [Calibration coefficient] to enter into the following interface, wherein corresponding calibration coefficient is to be input. Press [OK] to enable calculation in the process of test based on the input coefficient. See Fig. 5.5.21.

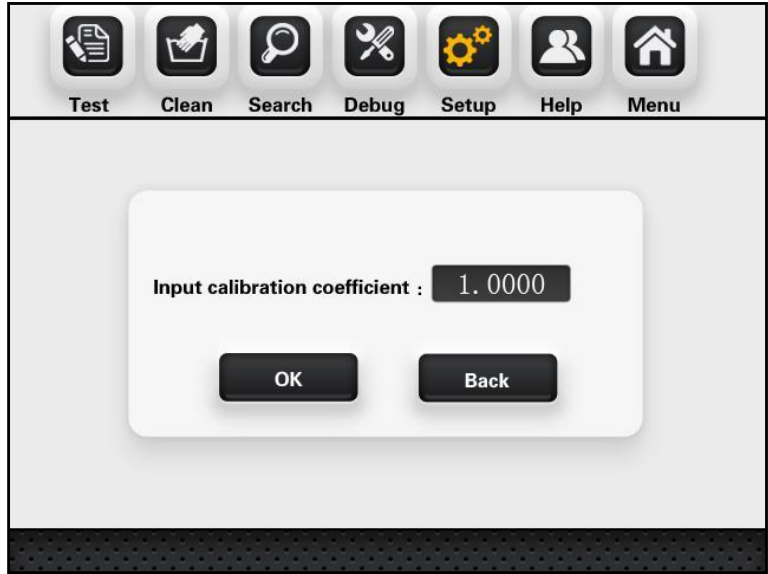


Fig. 5.5.21

Color calibration

Choose [Color calibration] to enter into color calibration interface, wherein the color calibration is carried out with respect to the recognition of titration end-point color change. See Fig. 5.5.22 for the interface.

Note: the consequences arising from unauthorized use of debug interface will be borne by the user; this interface is only for use by technicians with the manufacturer.

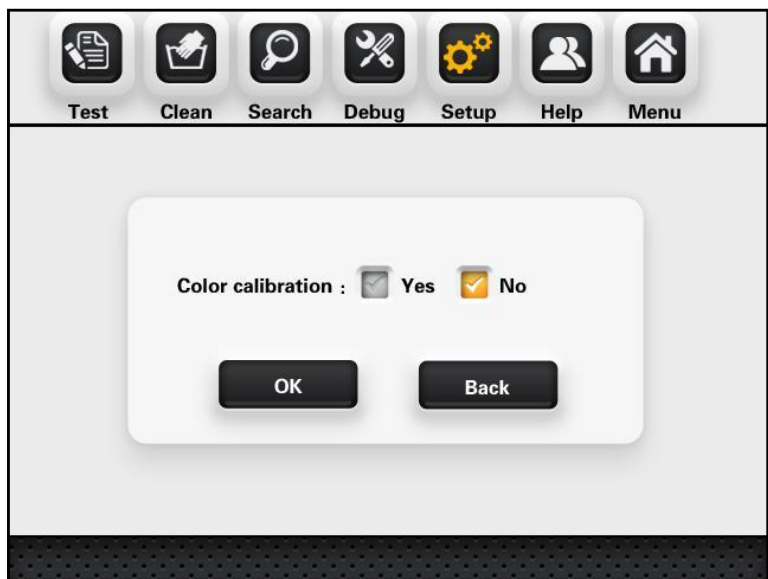


Fig. 5.5.22

Titration calibration

Make the special receiver equipment ready before titration calibration. Choose [Start] with respect to [Titration calibration], the titration to absorb fluid first, after alarming, the titration pushes out a fixed volume of solution to the receiver equipment; then the input data of actually pushed out volume is obtained through weighing. Press [OK] to complete the process of the titration calibration. In case of choosing [No], [OK] or [Back], it will maintain the original setting and enter into the initial Setup interface. See Fig. 5.5.23.

Note: the consequences arising from unauthorized use of debug interface will be borne by the user; this interface is only for use by technicians with the manufacturer.

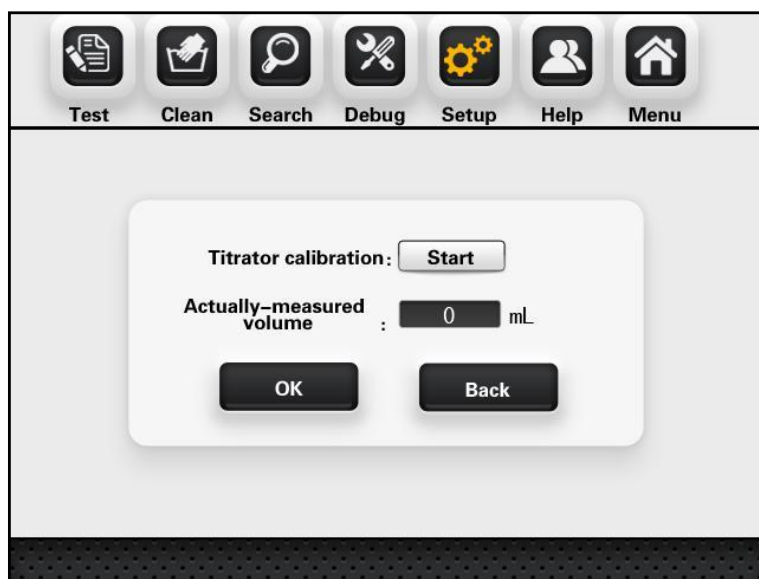


Fig. 5.5.23

System time

Choose [System time] to display dialog box for time setting, and input right time. Press [OK] to complete time setting; [Back] to maintain original setting and enter into the original Setup interface. See Fig. 5.5.24.

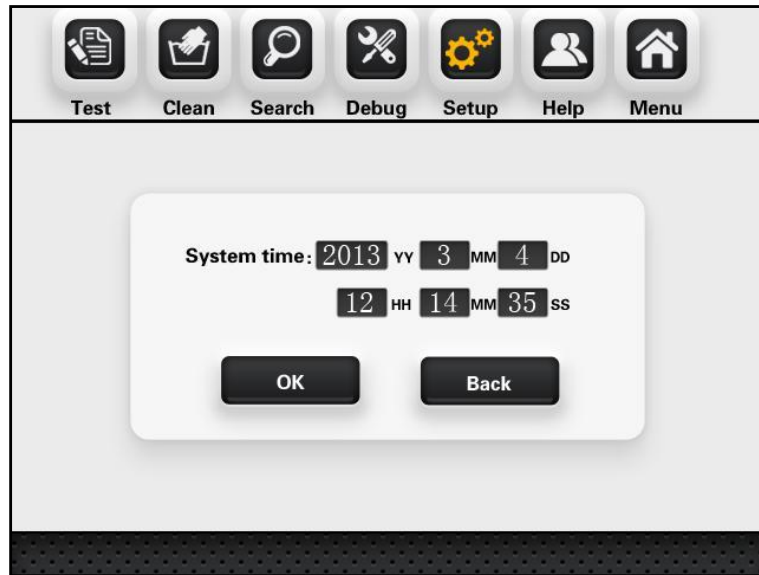


Fig. 5.5.24

Historical data

Choose [Historical data] to enter into choice interface, choose [OK] to delete all historical data, choose [Back] to exit. See Fig. 5.5.26.

Note: Once reset historical data it can't recover, please careful operate carefully.

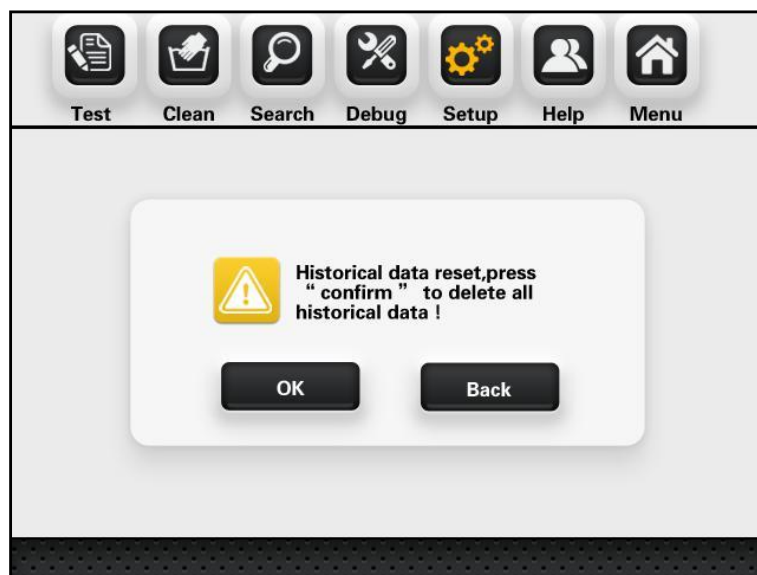


Fig. 5.5.26

Help

Choosing [Help] in the original interface will display interface as shown in Fig. 5.5.27.

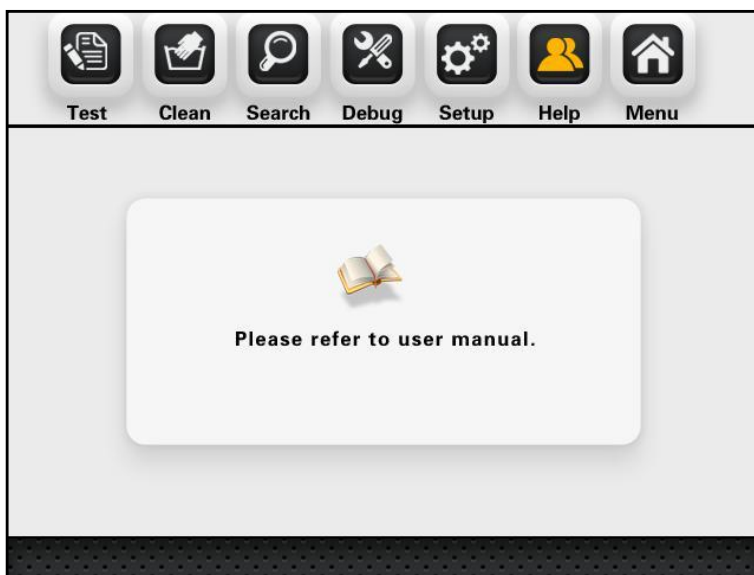


Fig. 5.5.27

VII. Sample test

1. Reagent preparation

(1) Boric acid solution (20g/L): weigh and dissolve 20.00g boric acid in distilled water, with volume defined in a 1,000mL measuring flask; shake it till evenly mixed. Add 100:1 mixture of methyl red and bromcresol green and make it uniformly mixed.

(2) Sodium hydroxide solution (400g/L): weigh and dissolve 400.00g sodium hydroxide in distilled water, with volume defined in a 1,000mL measuring flask; shake it till evenly mixed.

(3) Standard titration solution

a) Standard titration solution of sulfuric acid [$c(1/2H_2SO_4)=0.1000$ mol/L]: take and dilute 2.73mL concentrated sulfuric acid (density: 1.8419g/mL) with distilled water, with volume defined in a 1,000mL measuring flask; shake it till evenly mixed, and calibrate.

b) Standard titration solution of hydrochloric acid: take and dilute 8.30mL concentrated hydrochloric acid (concentration: 36%-38%) with distilled water, with

volume defined in a 1,000mL measuring flask; shake it till evenly mixed, and calibrate.

(4) Standard solution of ammonium sulfate: take and dissolve 6.6065g dried ammonium sulfate (G/R) with distilled water, with volume defined in a 1,000mL measuring flask; shake it till evenly mixed.

(5) Mixing 1 part of methyl red was dissolved in 0.1% ethanol solution of 95% ethanol (1g / L) and 5 parts was dissolved in 0.1% bromocresol green ethanol solution of 95% ethanol (1g / L), please make it just before use.

Note: sulfuric acid (H₂SO₄), copper sulfate (CuSO₄), potassium sulfate (K₂SO₄) shall be prepared for the digested sample.

Tips: Weigh 0.5-1g sample; add in 8mL-10mL concentrated sulfuric acid; and add in copper sulfate: 3.2g potassium sulfate of 1:15 mixture. (Refer to national standard and relevant industry standards for specific dosage)

When using Kjeldahl Analyzer, it is suggested as follows in order to reduce the measurement error of the device to the greatest extent:

- (1) The pH of 2% boric acid absorption solution shall be regulated to 4.5;
- (2) The reaction between strong acid and strong base is vigorous, thus it's suggested to dilute it by first adding 10-20 ml distilled water;
- (3) The volume of 40% or approximate concentration of sodium hydroxide to be added is advantageously 4 times of that of concentrated sulfuric acid;
- (4) Upon distillation, the total volume of liquid in the digestion tube is advantageously kept below 1/3 of the capacity of the digestion tube;

2. Reference value for test

The nitrogen content in the sample shall be considered in weighing sample. Where sample features high nitrogen content, fewer samples shall be taken provided the weighing error is reduced to the greatest extent. In case of low nitrogen content in sample, more samples shall be weighed. It can be generally advantageous that the weighed sample contains 15-50mg nitrogen.

Upon digestion of sample, add 10mL distilled water to dilute the digested sample. After it is fully cooled and leave it for measurement. Generally 0.1mol/L standard acid is taken as titration acid.

Table I Relationship between standard titration acid to be chosen and nitrogen

content in sample to be measured

3. Test parameter setup

Molar concentration of standard titration acid (mol/L)	Nitrogen content in sample to be measured (mg)
0.02	0.1~20
0.1	15~100
0.15	30~200

- (1) Install the device, and properly connect the pipeline.
- (2) Turn on the condensate water; place an empty digestion tube, start the device to vaporize water for 5-10 min in order to clean the pipeline and stabilize the steam flow.
- (3) Put the digestion tube containing digested sample in place and close the security door. Set up corresponding parameters and functions to start the test. The device simultaneously starts the real-time test function. Add boric acid absorption solution, diluting water and concentrated alkali solution in the device. Ammonia gas emitted from distillation of steam is absorbed via condensate boric acid; and then standard acid is used for titration.
- (4) After completion of the test, the result is displayed. Printing, automatic discharge and automatic cleaning are attainable. It returns to initial parameter input interface after completion of the test.

4. Device calibration

(1) Meaning of calibration coefficient K

Upon measuring sample by using the device, there will be system error due to connection to different systems. Thus, in order to make accurate measurement, a error calibration coefficient K shall be established. The users can carry out calibration by themselves.

The relationship between nitrogen content calculation and K value:

$$N\% = \frac{1.401 \times M \times (V - V_0) \times K}{W}$$

(2) Measurement of standard sample

Ammonium sulfate (purity level: at least A/R) may be selected as a standard sample for device calibration. Both liquid and solid samples can be used for measurement.

Solid sample: weigh and put 0.1-0.2g ammonium sulfate into the digestion tube for direct measurement on the device.

Liquid sample: weigh 6.6065g ammonium sulfate and define its volume to 1,000mL with distilled water, producing a standard solution with 1.4mg/mL nitrogen content.

Use a volumetric pipette to take a standard sample with nitrogen content close to that of the actually-measured sample. Upon blank measurement, choose 1 as K value.

(3) Calculation of K

It can be derived from above calculating formula that:

$$K = \frac{N}{N_1}$$

N: Nitrogen content in the standard sample (%);

N_1 : Nitrogen content in the standard sample as measured by the device (%).

For example: take three standard solution samples, each 10ml 1.4mg/mL of standard solution and respectively obtain nitrogen content in the standard sample: N_1 , N_2 , N_3 .

Calculate K_1 , K_2 , K_3 based on calculating formula for K, and obtain a final K which is an arithmetical mean of the three.

After the calibration coefficient K is calculated, input this coefficient in [Calibration coefficient] in the sample parameter input interface, and re-measure with a standard sample. In case the error remains, it shall be re-calibrated. (It's suggested to approximate the nitrogen content in the standard solution to that in single sample of substance to be measured in the experiment.)

Where corresponding crude protein content is required, a protein conversion coefficient shall be input in the test parameters, and the device will automatically calculate the crude protein content. When crude protein content is undesired, please set the protein conversion coefficient to 0. Thus the obtained crude protein content will be 0 and there will be no crude protein content the report to be printed.

VIII. Search of test record

The device can store 1,000 records for reference to previous test record by the user. After completion of sample test, the sample No. will be automatically incremented. After the record reaches the maximum storage capacity (1,000 pieces), it will restart to record from reference number one (the previous first record). The test record can be stored for a long period (typically greater than 10 years), with a circular storage of 1,000 test records for search and print by the user.

IX. Routine maintenance of device

1. The device socket shall be kept clean and dry and away from acid and alkali solution to ensure insulation high input impedance performance.
2. The alkali solution tank, boric acid solution tank, distilled water tank, titration acid tank shall be cleaned on a regular basis.
3. In case any liquid remains in the waste receiver tank at the bottom of security door of the device, please immediately clear it way.
4. There will be scale deposit in the distillation bottle after the device is used for a long period, which will affect heating efficiency (it's suggested to clean for every 6 months). The cleaning frequency may be increased in case of frequent use. The scale deposit is removed by introducing a detergent or a certain concentration of weak acid solution in through distilled water pipeline and discharged via the distilled water drain valve. Then the pipeline will be properly connected following several flushing using the distilled water.
5. When the titration acid concentration is modified, the titration acid in the pipeline and the titrator is fully discharged by manual titration, and new acid is used to flush for at least 6 times and discharged immediately.
6. In order to prolong the service life of the glass components, please clean it at least once after completion of work every day, i.e., 100mL water is added and evaporated for 5 min.

7. There may be bubbles in the pipeline of the piston pump, please remove it before experiment.
8. It is advantageous to calibrate the titrator at least once a year.
9. Fix the pipeline connected to the discharge ports of the steam generator and the digestion tube to prevent splash of high temperature corrosive liquid.
10. The receiving cup and alkali pump shall be cleaned after completion of experiment.
11. When the device is heated with full power and sufficient condensate water, the liquid discharged shall not be less than 150mL for discharge time of 5 min.
12. Wear protective glasses and gloves upon maintenance on the device.

X. Troubleshooting

Serial No.	Failure	Cause	Solution
1	The device can not be powered on	1. Shortage of power supply ; 2. power line not properly connected; 3. Fuse is broken.	1. Check the power supply; 2. Properly connect the power line; 3. Change the fuse.
2	There's no steam when distillation	1. Steam generator water shortage; 2. The heating controller fails; 3. The conductor wire between the heating controller and the distillation bottle is not well connected; 4. Heating tube fails; 5. Pressure-head switch fails; 6. Temperature protection switch fails; 7. Steam valve fails.	1. Add water to bucket, then press "continue"; 2. Change the heating controller; 3. Check whether the conductor wire is loosened; 4. Change heating tube; 5. Change pressure-head switch; 6. Change temperature protection switch; 7. Change steam valve.
3	Alkali, boric acid and dilution water can not be added properly	1. Insufficient solution in the solution tank, and the pipette is located above the level; 2. The solution adding pipeline is not air-tight; 3. The alkali adding pump can not work in order; can	1. Add solution in the solution tank; 2. Check the connectors for the pipeline are sealed; 3. Clean the solution pump ; 4. Change the pump; 5. Change solenoid

		not be started; the pipeline is blocked; 4. The pump can not work 5. Solution tank is damaged.	valve.
4	Titration can not be carried out	1. Insufficient titration acid; 2. Failure in titration system ; 3. Clogging of titration electromagnetic valve.	1. Add titration acid; 2. Change the titration system; 3.Change electromagnetic valve.
5	Unstable measurement data	1. The receiving cup is not clean; 2. The steam generator is not clean; 3. The position of burette is not installed correctly. 4. Too much liquid in the digestion tube; 5. Insufficient alkali; 6. Titration color error.	1. Clean receiving cup; 2.Clean steam generator 3. Install burette correctly; 4. Reduce liquid in the digestion tube; 5.Add sufficient alkali; 6.Calibrate the color again.
6	Wrong measurement data	1. Wrong input of titration acid concentration; 2. Inaccurate device calibration.	1. Input hydrogen ion concentration as titration acid concentration; 2. Recalibrate the device.
7	The test is paused	1. The door is not closed well; 2.Insufficient condensate water; 3.Insufficient liquid in steam generator; 4.Overtemperature of steam generator; 5. Overflow of receiving cup; 6.Overtemperature of receiving liquid.	1.Close the door well; 2.Fixed the digestion tube well; 3.Cheak condensate water; 4.Cheak steam generator; 5.Cheak receiving cup; 6.Cheak condensate water.

The device is warranted for a year from the date of purchase (subject to the date on the issued invoice) except for the following conditions:

1. The warranty period expires;
2. Damage caused by misuse;
3. Damage caused by disassembly unauthorized by the manufacturer;
4. Damage caused by improper transportation and custody.

XII. Cautions

1. Make sure there's sufficient water in the distilled water tank before using the device to ensure normal operation of the device. The manufacturer will not be responsible for device failure caused by insufficient distilled water.
2. The interior storage of the device stores data based on sample No.. Notice the setting of sample No.; the storage is reusable, with a sample No. ranging from 1 to 1000. The user shall not be free to modify the sample No..
3. The preparation of alkali solution and acid solution requires careful operation to avoid being burnt by chemical reagent.
4. The device contains glassware, which requires careful handling during conveyance.
5. Upon repairing internal components of the device, make sure to turn off the device and plug off the power line and wait for the cooling down of the distillation system.
6. The outlet of the discharge pipe shall be located below the installation location of the device to ensure smooth discharge.
7. Upon the device is left unused for a long period, the alkali solution in the alkali solution tank shall be replaced with distilled water; the digestion tube is put in place; the alkali is added manually to remove the alkali solution in the pipeline and the pipeline is cleaned to prevent crystallization clogging.
8. A blank test shall be carried out before start-up to clean the pipeline of the device in order to ensure accurate test results.
9. The device has been subject to test before delivery, thus it's normal for presence of residual solution.
10. It is suggested that the maximum solution volume in the digestion tube shall not exceed 2/3 of the capacity of the digestion tube.

Note: before using the device, please turn on the condensate water and examine whether solutions in each solution tank (alkali solution tank, boric acid solution tank, distilled water tank and titration acid tank) meet the requirements of the test, or otherwise, add immediately.

XIII. Annex

The assembly and disassembly of the digestion tube is illustrated as follows:

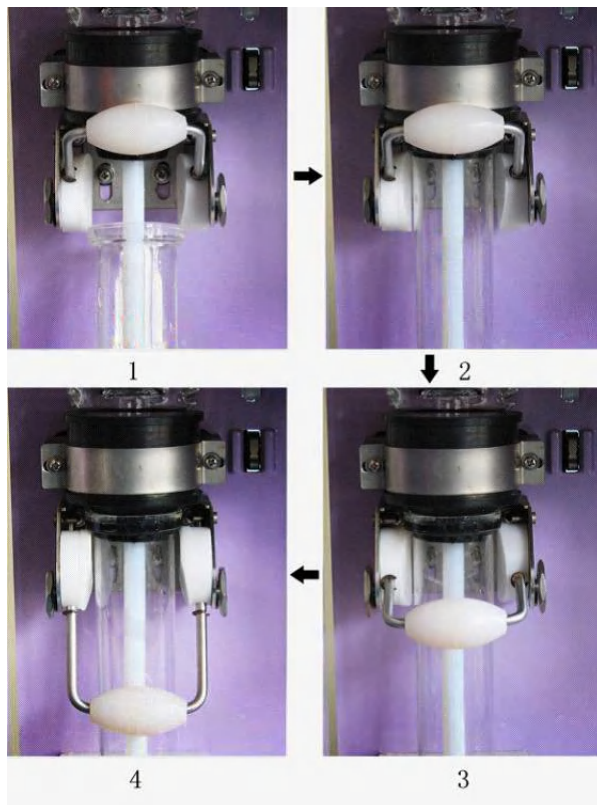


Fig.1

Steps of installing the digestion tube:

First lift up the fixed tube handle, and put the mouth of the digestion tube in place from below and pull down the fixed tube handle.

Steps of dismantling the digestion tube:

Lift up the fixed tube handle and remove the digestion tube in a vertically downward direction.

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