# **User Manual**



Please read the manual before installation and operation.

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# K1100F Automatic Kjeldahl Analyzer

**Product** Manual

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Warning: The instrument can not provide the designed protection for operators who do not follow the right procedures and requirements given by the manufacturer.

Warning: All solutions must be handled with care according to the lab's safety regulation. Please make a reference to the related material safety data sheet. Wear the lab-gown, goggle and rubber gloves all the time. Be care of hot reagents.



Warning: The waste liquid can be directly discharged into the sewer if it does not conflict with the local waste treatment regulations. Ensure that waste discharge pipe do not bend or flow up, and it should be short as much as possible. The export should not lower than the surface of the sewer (there will be a noise when the hot water discharged into cold water) Meanwhile the pipe should be fixed, because the discharged waste liquid has a certain pressure.

# I. Summary

#### **1.1 Application**

K1100F 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 K1100F Automatic Kjeldahl Analyzer.

K1100F 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.

#### **1.2 Principles**

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



K1100F 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:

$$[1]. (NH)_2 SO_4 + 2NaOH \xrightarrow{\text{high-temperature steam}} Na_2 SO_4 + 2H_2 O + 2NH_3 \uparrow$$

[2]. 
$$2NH_3 + 4H_3BO_3 = (NH_4)_2B_4O_7 + 5H_2O_7$$

[3]. 
$$(NH_4)_2 B_4 O_7 + 5H_2 O + 2HCl = 4H_3 BO_3 + 2NH_4 Cl$$

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:

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

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

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

W=sample weight (g);

 $V_0$ = 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.

### **1.3 Operating Flow Chart**



# II. Main performance

#### 2.1 Technical parameters

- a. Sample capacity: solid≤5g, liquid≤20mL;
- b. Measuring range: 0.1mg~200mg nitrogen;
- c. Analysis time: 5~10 min/sample;
- d. Recovery: ≥99.5%;
- e. Titration accuracy: 1.0µL/step;
- f. Repeated errors: ±0.5%;
- g. Data storage capacity: 1,800 pieces;
- h. Consumption of condensate water: 1.5L/min;
- i. External interface: USB, RS485;
- j. Dimension size (length × width × height): 455mm × 391mm × 730mm;
- k. Net weight: 38kg.

#### 2.2 Use conditions

- a. Power supply: 220 VAC ±10% 50Hz;
- b. Rated power: 2KW;
- c. Condensate water pressure: 0.02Mpa-1 Mpa; flow: 4.5-6L/min;
- d. Condensate water temperature: below 20°C;
- e. Ambient temperature: 10°C~28°C.

#### III. 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:





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
 "Digestion tube in-place" detector 9. Security door 10. Waste receiving tank 11. Touch screen 12. Panel 13. Printer 14. Receiving cup 15. Titration acid tank



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



Fig 3.3

19. Power switch 20. Power plug 21. RS485 interface 22. USB interface 23. Distilled water level detection interface 24. Boric acid level detection interface 25. Alkali solution level detection interface 26. Alkali inlet

27. Boric acid inlet 28. Distilled water inlet



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

# **IV. 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  $10^{\circ}$ C and  $28^{\circ}$ 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.

# **V. Function introduction**

#### 1. Function of operating panel

a. The display: 24-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.

Protection upon test tube is not in place: when the system detects that test tube is not in place during operation, the device will stop and make alarm to prevent overflow of alkali solution.

Level protection of solution tank: the solution tank is provided with level sensors;

when the level of any of the acid tank, alkali tank or water tank is so low that refilling is needed, the system will make alarm.

#### 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.

Titrating: 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, including two test modes, i.e., automatic test and manual tests.

[Automatic test] After setting experimental parameters, the device will carry out automatic experimental test.

[Manual test] It's used to step by step carry out experimental test and debug and test with respect to each function of the device. See Fig. 5.5.2.



Fig. 5.5.2

Touch and choose automatic test mode to enter corresponding mode interface, wherein four functions can be attained as follows:

- 1. Choose type
- 2. Input parameters
- 3. Commence experiment
- 4. Save mode

#### 1) Automatic test:

The system initially displays the interface for optional types, and there're ten optional types in total. Choose the experiment type according to the samples. See Fig. 5.5.3.

Automati	c Kjeldah	l Analyzer	vpe select	ion		
Cor	nvention	Peanut	Soybean	Corn	Sunflow	ver
	Flour	Barley	Pure Milk	Rice	Produ User Mod	et et
*	0	E	P	%	¢°	*
Menu	Test	Clean	Search	Debug	Setup	Help



Choose the right type. For example, choose [Convention] to enter parameter input interface, wherein last experiment data will be displayed. A keyboard pop-up appears upon click on each data region, and corresponding data may be input as required. Press [OK], and enters into experiment start interface; press [Back], and returns to the last interface. See Fig. 5.5.4.





Upon choosing [User mode], enter the following interface. [User mode] allows users to save 100 kinds of measurement mode in the process of measurement for convenience of future measurement of similar experiments. It can provides previous information, such as concentration of titration acid, volume of diluting water, volume of boric acid, volume of alkali solution and distilling duration. It can also be corrected according to desired information by clicking the data region. The sample No. can be chosen via the left and right triangle icon, as well as keyboard input. The illustration shows the user has chosen mode 1, and input parameters such as sample weight, blank volume, protein coefficient, etc. See Fig. 5.5.5.



Fig. 5.5.5

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, digestion tube in place, presence of solution in solution tank, etc. If any abnormal condition is detected, the task will be suspended. For example, upon lack of condensate water or digestion tube not-in-place, the following interface will appear. See Fig. 5.5.6.



Fig. 5.5.6

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.5.5.7.



Fig.5.5.7

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.





When a parameter is set, after filling of diluting water is completed, the system will start filling of alkali. After alkali adding, it will add 10ml distilled water more, the interface (figure 5.5.9) as follow:





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. 5.5.10.





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

Automatic Kjeldahl Analyzer Test Distilling ... Pause NaOH HaBO: O × Menu Clean Search Debug Setup Help Test



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. 5.5.13.

			est		
		<b>∮ µ</b>	Distilling		
door	tube d	condenser Generator			
H2O	NaOH	HsBOs Receiving oup		Paus	•
	2		0 %	20	22

Fig. 5.5.12

started; meanwhile the suspend or back can be attained. See Fig. 5.5.11.





Upon the system enters into the process of titration, See Fig. 5.5.14.

		1	Test		
ก	Ĩ		п	trating	
door E	tube cor	denser Genera	hor		
H <sub>2</sub> O	NaOH I	HaBOs Receivin	ng		
	~	-	0	30	 (1)
n	$\underline{\smile}$			10	\$

Fig. 5.5.14

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. 5.5.15.



Fig. 5.5.15

Cleaning will be automatically started upon completion of discharging. The digestion tube will first be cleaned. See Fig. 5.5.16.

		1	Test			
Ð			[1	leaning dige	stion tube .	
door	tube co	Andenser Gener	ator 1			
H2O	NaOH	HsBOs Receiv oup	ing			
~	2	6	0	30	D°	23

Fig. 5.5.16

The receiving cup will be cleaned following the completion of digestion tube cleaning. See Fig. 5.5.17.





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 and saving mode can be carried out. See Fig.5.5.18.



Fig.5.5.18

In case of blank experiment, the titration result interface will only display titrated volume. See Fig.5.5.19.





Choosing save mode can save the present experiment mode, with interface cuing user No. input and showing dialog box for input. See Fig. 5.5.20.



Fig. 5.5.20

After number input, press [OK] to save or press [Back] to return to the previous interface. The user No. will be automatically generated, and can be modified. See Fig. 5.5.21.



Fig. 5.5.21

After completion of saving mode, it will automatically return to the parameter input interface and enter into standby status. See Fig. 5.5.4.

2) Manual test

Upon touching and choosing the manual test mode in the interface in Fig. 5.5.2, it will enter into the parameter test interface. See Fig. 5.5.22.





After inputting corresponding parameters, press [OK] to enter the following interface, wherein the following functions can be manually attained: add diluting water, add boric acid, add alkali, distill, titrate, calculate and print, discharge, digestion tube clean, receiving cup clean, with each step manually manipulated. See Fig. 5.5.23.

Add boric acid	Add dilution water	Add alkali	Distillation (min)
025	010	010	05
Titrate	alculate nd Print Discharge	Digestion tube clean	Receiving cup clean
	ок	Back	
	ок	Back	



For example, choose Add diluting water (the triangle icon is directed to the present option), input corresponding volume, and touch to start adding of diluting water. After the completion of adding, it will automatically return to the present interface, ready for choice of other functions. All functions of this interface are optional. See Fig. 5.5.24.

	<u></u>	eyboar	d	
Add boric acid				Distillation (min)
025	1	2	3	05
Titrate Cak and	4	5	6	Receiving cup clean
(		8	9	
	•	.0	-	
~ 0	ОК		Cancel	
🗅 👱 j		~	10	

Fig. 5.5.24

During the experiment, the system will collect all aspects of status information with respect to the device on a real-time basis similar to automatic test.

# Note: each step of manual test will display the ongoing status and content of experiment result similar to automatic test.

#### Cleaning

Choose [Cleaning], with interface as shown in the figure, including Digestion tube cleaning, Receiving cup cleaning, Alkali pipeline cleaning, Boric acid pipeline cleaning, Acid washing, Steam bottle evacuation. Choose Cleaning to start cleaning. See Fig. 5.5.25.

	Digestion tube	Boric acid
	clean	pipeline cleaning
	cup clean	Pickling
	Alkali pipeline cleaning	discharge bottle
<u>a</u>		2 % 🕫 🛯

Fig. 5.5.25

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. 5.5.26.



Fig. 5.5.26

#### 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.: 1-1800). [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. 5.5.27.

Sample NO.     Sample (g) weight (g) weight (g)     Titration acid (mol/L) concentration     Titrated (mL) volume (mL)       Image: Optimized concentration     0,0000     0,0000     0,0000     0,0000       Blank (mL) volume (mL)     Protein coefficient     Nitrogen (%) content (%)     Protein (%)       Image: Optimized concentration     0,0000     0,0000     0,0000
Experiment date:

Fig. 5.5.27

#### 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,  $\sqrt{}$  indicates initiation, × indicates closure; the white icon represents detection of level sensors, etc.  $\sqrt{}$  and × indicate the present working status of sensors. For example,  $\sqrt{}$  appears upon detection of security door in place, otherwise × appears. After completion of choosing, the debug of the device commences. See Fig. 5.5.28. 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.

Flefill valve of Steam Generator	x	Discharge valve of Steam Generator	×	Steam valve	×	Distillation valve	×	Pinch valve	×	Discharge valve of digestion tube	>
Discharge valve of liquid bottle	×	Refill valve of receiving cup	×	Discharge valve of receiving cup	×	Three-way valve for titration	×	Titration lamp	×	Mixing motor	,
Titrator	×	Refill valve for condensate water	×	Dilution water valve	×	Alkali valve	×	Alkali pump	×	Boric acid pump	,
Lift pump	×	Discharge pump	×	Heating pipe	×	Printer	×	Alarm	×	Protective door in place	
Digestion tube in place		Tumbler		Barrel level		Alkali barrel level		Boric acid barrel level		Receiving cup lower level	
Condensate water flow Condensate water temp	(C)	000	SEC.E	team Generato amp leceiving quid temp	*(1)*	> 000 > 000	)			Receiving cup higher level	

Fig. 5.5.28

#### Setup

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

[Steam flow] can choose the steam flow rate.

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

[Distillation mode] provides two optional distillation modes: distillation before adding alkali and distillation after adding alkali.

[Titration mode] the system titration mode, including two optional titration modes: distillation before titration and distillation accompany with titration.

[Discharge from digestion tube] with respect to automatic test which determines whether to carry out discharge from digestion tube after titration.

[Digestion tube cleaning] with respect to automatic test which determines whether to carry out digestion tube cleaning after titration.

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

[Print mode] provides two print modes: simple print and ordinary print.

[Color calibration] is used for titration sensor calibration.

[Color trimming] is to adjust the color value.

[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.

[Data transmission] communicate with upper computer by USB or RS485.

Set up corresponding function in this interface before the commencement of experimental test. See Fig. 5.5.29.

	Steam flow	Water fine test	Distillation	Titration mode
	Otean now	water now test	mode	The action mode
	Discharge from digestion tube	Digestion tube cleaning	Receiving cup cleaning	Print mode
	Color calibration	Color trimming	Titrator calibration	Calibration coefficient
	System time	Historical data	Data transmission	
_		_		

Fig. 5.5.29

#### Steam flow

Choose [Steam flow] to enter into the interface for choosing parameters, wherein dialog box for data of steam flow rate appears. Choose right parameter, click on [OK] to continue; [Back] to return to initial Setup interface. See Fig. 5.5.30.



Fig. 5.5.30

#### 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. 5.5.31.



Fig. 5.5.31

Distillation mode

Choose [Distillation mode] to enter into choice interface, wherein there're two optional modes, distillation before adding alkali and distillation after adding alkali. For example, choose distillation after adding alkali, press [OK] to complete setup; choose [Back] to maintain original default mode return to the initial Setup interface. See Fig. 5.5.32.



Fig. 5.5.32

#### Titration mode

Choose [titration mode] to enter into choice interface, choose [Yes] to execute distillation accompany with titration, choose [No] to execute distillation before titration. See Fig. 5.5.33.



Fig. 5.5.33

Discharge from digestion tube

Choose [Discharge from digestion tube] to enter into choice interface; choose [Yes], [OK] to enable discharge from digestion tube after completion of test; choose [No], [OK] to disable discharge from digestion tube after completion of test; choose [Back] to maintain the original setting and return to the Setup interface. See Fig. 5.5.34.



Fig. 5.5.34

Digestion tube cleaning

Choose [Digestion tube cleaning] to enter into choice interface; choose [Yes], [OK] to enable digestion tube cleaning after completion of test; choose [No], [OK] to disable digestion tube cleaning after completion of test; choose [Back] to maintain the original setting and return to the Setup interface. See Fig. 5.5.35.

Setup
Digestion tube   Yes   No
ОК Васк
2 💼 🔎 🕺 💕 🛎

Fig. 5.5.35

#### 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. 5.5.36.



Fig. 5.5.36

#### Print mode

Choose [Print mode] to enter into choice interface, choose[simple print], the system will print related parameters one by one; choose [ordinary print], the system will just print test results. See Fig. 5.5.37.



Fig. 5.5.37

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. 5.5.38 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. 5.5.38

Color trimming

Choose [Color trimming] to enter into color trimming interface, Press[-] or[+] can adjust the date, after completed, press [OK] to save, otherwise, press [Back] to exit. See Fig. 5.5.39.



Fig. 5.5.39

Titrator calibration

Make the special receiver equipment ready before titrator calibration. Choose [Start] with respect to [Titrator calibration], the titrator to absorb fluid first, after alarming, the titrator 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 titrator calibration. In case of choosing [No], [OK] or [Back], it will maintain the original setting and enter into the initial Setup interface. See Fig. 5.5.40.

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.

	Setup
	Titrator calibration : Start Actually-measured () mL volume :
	OK Back
~ ~	

Fig. 5.5.40

#### 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. 5.5.41.

	Setup
	Input calibration 1.0000
	OK Back
*	े 📻 🔎 💥 💕 🛋

Fig. 5.5.41

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. 5.5.42.



Fig. 5.5.42

Historical data

Choose [Historical data] to enter into choice interface, choose [OK] to delete all historical date, choose [Back] to exit. See Fig. 5.5.43.

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



Fig. 5.5.43

Data transmission

Choose [Data transmission] to enter into choice interface, choose [USB], the system will communicate with upper computer by USB. Choose [RS485], the system will communicate with upper computer by RS485. See Fig. 5.5.44 and Fig. 5.5.45.



Fig. 5.5.44



Fig. 5.5.45

#### Help

Choosing [Help] in the original interface will display interface as shown in Fig. 5.5.46.



Fig. 5.5.46

# VI. 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(1/2H2SO4)=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) Mixed indicator: Put methyl red 0.1g and bromcresol green 0.5g soluble in ethanol without water, constant volume to 100mL.

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	Nitrogen content in sample to		
standard titration acid (mol/L)	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<sub>1</sub>: 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.

## VII. Search of test record

The device can store 1,800 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 (1,800 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 1,800 test records for search and print by the user.

### **VIII. 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.

Serial No.	Failure	Cause	Solution
1	The device can not be powered on	<ol> <li>Shortage of power supply ;</li> <li>power line not properly connected;</li> <li>Fuse is broken.</li> </ol>	<ol> <li>Cheak the power supply;</li> <li>Properly connect the power line;</li> <li>Change the fuse.</li> </ol>
2	There's no steam when distillation	<ol> <li>Steam generator water shortage;</li> <li>The heating controller fails;</li> <li>The conductor wire between the heating controller and the distillation bottle is not well connected;</li> <li>Heating tube fails;</li> <li>Pressure-head switch fails;</li> <li>Temperature protection switch fails;</li> <li>Steam valve fails.</li> </ol>	<ol> <li>Add water to bucket, then press "continue";</li> <li>Change the heating controller;</li> <li>Check whether the conductor wire is loosened;</li> <li>Change heating tube;</li> <li>Change pressure-head switch;</li> <li>Change temperature protection switch;</li> <li>Change steam valve.</li> </ol>

# **IX. Troubleshooting**

3	Alkali, boric acid and dilution water can not be added properly	<ol> <li>Insufficient solution in the solution tank, and the pipette is located above the level;</li> <li>The solution adding pipeline is not air-tight;</li> <li>The alkali adding pump can not work in order; can not be started; the pipeline is blocked;</li> <li>The pump can not work</li> <li>Solution tank is damaged.</li> </ol>	<ol> <li>Add solution in the solution tank;</li> <li>Check the connectors for the pipeline are sealed;</li> <li>Clean the solution pump;</li> <li>Change the pump;</li> <li>Change solenoid valve.</li> </ol>
4	Titration can not be carried out	<ol> <li>Insufficient titration acid;</li> <li>Failure in titration system;</li> <li>Clogging of titration electromagnetic valve.</li> </ol>	<ol> <li>Add titration acid;</li> <li>Change the titration system;</li> <li>Change electromagnetic valve.</li> </ol>
5	Unstable measurement data	<ol> <li>The receiving cup is not clean;</li> <li>The steam generator is not clean;</li> <li>The position of burette is not installed correctly.</li> <li>Too much liquid in the digestion tube;</li> <li>Insufficient alkali;</li> <li>Titration color error.</li> </ol>	<ol> <li>Clean receiving cup;</li> <li>Clean steam generator</li> <li>Install burette correctly;</li> <li>Reduce liquid in the digestion tube;</li> <li>Add sufficient alkali;</li> <li>Calibrate the color again.</li> </ol>
6	Wrong measurement data	<ol> <li>Wrong input of titration acid concentration;</li> <li>Inaccurate device calibration.</li> </ol>	<ol> <li>Input hydrogen ion concentration as titration acid concentration;</li> <li>Recalibrate the device.</li> </ol>
7	The test is paused	<ol> <li>The door is not closed well;</li> <li>Digestion tube is not put well;</li> <li>Insufficient condensate water;</li> <li>Insufficient liquid in steam generator;</li> <li>Overtemperature of steam generator;</li> <li>Overflow of receiving cup;</li> <li>Overtemperature of receiving liquid.</li> </ol>	<ol> <li>Close the door well;</li> <li>Fixed the digestion tube well;</li> <li>Cheak condensate water;</li> <li>Cheak steam generator;</li> <li>Cheak receiving cup;</li> <li>Cheak condensate water.</li> </ol>

# X. Supplements

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.

# **XI. 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 1800. 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.

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.



### Back elevation of K1100F Automatic Kjeldahl Analyzer

Fig.2

1. Level sensor of steam generator 2. Steam generator 3. Piston pump

#### 4. Steam bottle

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The enterprise continuously commits to product quality and performance improvement for product technology and design update, without prior notice.

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